Food and Agriculture Organization of the United Nations

World Health Organization

EVALUATION 2021

PART I - RESIDUES

Pesticide residues in food

Extra Joint FAO/WHO Meeting on Pesticide Residues

Evaluation
Part I - Residues

Pesticide Residues in Food 2021

Extra Joint FAO/WHO Meeting on Pesticide Residues

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, 17-21 May and 7-11 June 2021

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Monographs containing summaries or residue data and toxicological data considered at the 2019 Extra JMPR, together with recommendations, are available upon request from FAO or WHO under the title:
Pesticide residues in food 2021
Evaluations
Part I: Residues

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Abbreviations

ADI	acceptable daily intake
AGF	aspirated grain fractions
AR	applied radioactivity
As	as received
ARfD	acute reference dose
BBCH	Biologische Bundesanstalt, Bundessortenamt und Chemische Industrie
bw	body weight
CAC	Codex Alimentarius Commission
CAS	Chemical Abstracts Service
CCPR	Codex Committee on Pesticide Residues
cGAP	critical GAP
DALA	days after last application
DAT	days after treatment
DM	dry matter
DT_{50}	time required for 50% dissipation of the initial concentration
Dw	dry weight
EFSA	European Food Safety Authority
eq	equivalent(s)
ESI	electrospray ionization
EU	European Union
FAO	Food and Agriculture Organization of the United Nations
GAP	good agricultural practice
GC-ECD	gas chromatography – electron capture detector
GC-FPD	gas chromatography – flame photometric detector
GC-MS	gas chromatography – mass spectrometry
GEMS	Global Environment Monitoring System – Food Contamination Monitoring and Assessment Programme
GI	gastrointestinal tract
GLP	good laboratory practice
HPLC	high performance liquid chromatography
HR	highest residue level in the edible portion of a commodity

HR-P	highest residue level in a processed commodity
IEDI	International Estimated Daily Intake
IESTI	International Estimate of Short-Term Dietary Intake
ILV	independent laboratory validation
ISO	International Organization for Standardization
IUPAC	International Union of Pure and Applied Chemistry
JECFA	Joint FAO/WHO Expert Committee on Food Additives
JMPR	Joint FAO/WHO Meeting on Pesticide Residues
LC-MS/MS	liquid chromatography-tandem mass spectrometry
LOD	limit of detection
LOQ	limit of quantification
MRL	maximum residue limit
NOAEL	no-observed-adverse-effect level
OECD	Organisation for Economic Co-operation and Development
PBI	plant-back interval
PF	processing factor
PHI	pre-harvest interval
Po	post-harvest
ppm	parts per million
QuEChERS	quick, easy, cheap, effective, rugged and safe
QSAR	quantitative structure–activity relationship
RAC	raw agricultural commodity
RSD	relative standard deviation
RTI	re-treatment interval
SC	suspension concentrate
SPE	solid phase extraction
STMR	supervised trials median residue
STMR-P	supervised trials median residue in a processed commodity
TRR	total radioactive residues
TTC	threshold of toxicological concern
UK	United Kingdom
USA	United States of America
WHO	World Health Organizatio

Use of JMPR Reports and Evaluations by registration authorities

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Introduction

An Extra 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 virtually over two sessions from 17 to 21 May and 7 to 11 June.

The meeting was opened by the JMPR Secretariats, Madame YongZhen Yang of FAO and Dr Kim Peterson of the WHO. Madame Yang welcomed and conveyed her appreciation to the panel members for their efforts in participating in preparatory virtual sessions prior the current JMPR Meeting. Dr Peterson also thanked the participants for providing their expertise and for devoting significant time and effort to the important work of the JMPR. He highlighted that holding the current Meeting virtually was an important initiative but that the practice would not be unfamiliar to participants and that despite the challenge of significantly different time zones, was confident the goals of the Meeting would be achieved.

In concluding Madame Yang referenced recent remarks of the FAO Director General Dr Qu Dongyu that the challenges of the pandemic, has meant that new modes of working and innovative systems have had to be created in adapting to this new normal and that working remotely is now part of our daily working life. The current virtual meeting will be an opportunity for the JMPR to identify possibilities of working remotely.

During the meeting, the FAO Panel of Experts on Pesticide Residues in Food 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 for estimating the maximum levels of residues that might occur as a result of use of the pesticides according to good agricultural practice. The methodologies are described in detail in the FAO Manual on the provision and evaluation of pesticide residue data for the estimation of maximum residue levels in food and feed (2016) hereafter referred to as the FAO Manual. The WHO Core Assessment Group on Pesticide Residues was responsible for reviewing toxicological and related data where necessary and possible.

The Meeting evaluated 29 pesticides for residues with regard to additional uses. The Meeting estimated maximum residue levels and recommended them for use by CCPR and estimated supervised trials median residue (STMR) and highest residue (HR) levels as a basis for estimating dietary exposures.

The Meeting also estimated the dietary exposures (both acute and long-term) of the pesticides reviewed and, on this basis, performed a dietary risk assessment in relation to the relevant ADI and where necessary the ARfD. Cases in which ADIs or ARfDs may be exceeded, if they occur, are clearly indicated in

ACETAMIPRID (246)

First draft prepared by C.M. Mahieu, Centre for Nutrition, Prevention and Health Services (VPZ), National Institute for Public Health and the Environment (RIVM), The Netherlands

EXPLANATION

Acetamiprid is a neonicotinoid insecticide with contact and stomach action against a range of *Hemiptera, Thysanoptera* and *Lepidoptera* plant pests, 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 North America, Europe and in a number of countries in Asia and the Pacific.

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. It was evaluated again in 2012, 2015 and 2017. At the 2017 JMPR data supporting the use on pistachio was provided, but without a registered use label. As such the 2017 Meeting could not estimate a maximum residue level for pistachio. Iran provided a new registered label on use in pistachio to the present Meeting, based on field trial data provided in 2017.

For both compliance with the MRL and dietary risk assessment, the residue is defined as *acetamiprid* for plant commodities and the *sum of acetamiprid and N-desmethyl-acetamiprid, expressed as acetamiprid* for animal commodities.

The residue is not fat-soluble.

USE PATTERN

The GAP information for pistachio in Iran is summarized in Table 1.

Table 1 Registered pre-harvest uses of acetamiprid on pistachio

Crop	Country	Form	Application				PHI, days
			Method	Rate kg ai/ha	Spray conc, kg ai/hL	Number (RTI in days)	
Pistachio	Iran	SP 20%	high volume spraying [a]	0.05	0.005	3 [a]	28-42

RTI = retreatment interval; PHI = Preharvest interval

[a] Information from the Iranian Government: First application at immature fruit & fruiting stage with third application when the kernels of pistachio are full (mature fruiting). The time between the first and the second application is not a fixed time, since it depends on the economic injury level (EIL) of the pest (psylla). In normal agricultural practice, the time between the second and third application is usually 30 days.

RESULTS OF SUPERVISED RESIDUE TRIALS ON CROPS

Four independent trials were conducted on pistachios in Iran in 2015 and were evaluated by the 2017 JMPR. For maximum residue level estimation, the results are included in the current evaluation in Table 2.

Table 2 Residues of a 20% SP formulation of acetamiprid in pistachio (nutmeat) after three foliar pre-harvest treatments using hydraulic handgun sprayers

Location, year, (variety) PISTACHIO NUTMEAT	No (RTI)	kg ai/ha	kg ai/hL	GS & last treatment day	DAT	residues, mg/kg [a]	reference
Zarand, Iran, 2015 (Fandoghi) [b]	3 (45, 31)	0.05	0.005	fruiting, 05 Aug	28	0.32/ 0.30/ 0.34 (0.32) [b]	Trial 001 summary - Zarand
Kerman, Iran, 2015 (Fandoghi) [b]	3 (45, 31)	0.05	0.005	fruiting, 05 Aug	28	0.50/ 0.47/ 0.51 (0.49) [b]	Trial 001 summary – Kerman
Qazvin, Iran, 2015 (Kale bozi)	3 (39, 29)	0.05	0.005	fruiting, 05 August	30	0.36/ 0.30/ 0.35 (0.34)	Trial 001 summary - Qazvin
Qhom, Iran, 2015 (Abbas Ali)	3 (60, 32)	0.05	0.005	fruiting, 07 August	30	0.22/ 0.17/ 0.22 (0.20)	Trial 001 summary - Qhom

DAT = Days After Last Treatment; GS = Growth Stage; RTI = Retreatment Interval

[a] Residues were measured in triplicate field samples. Mean is given in brackets.

[b] Zarand is a city in Kerman province. Most of the pistachio orchards are in this region. Though both experiments were performed on the same day and on the same variety, the locations were 120 km apart and can therefore be considered independent trials.

Note by the reviewer: The timing between the first two treatments (39–60 days) is pest driven and not expected to be of great impact on the final residue level, since the nuts are still immature and the nutmeat is completely protected by the epicarp, residues from early applications tend to decline, and acetamiprid is only moderately systemic (some penetration in lower parts, but no translocation (JMPR 2011)). In addition, the short environmental half-life of approximately 1 week as well as a significant decline of residues over one to two weeks observed in fruiting vegetables and leafy vegetables (JMPR 2011) further support the expectation that the first treatment will not drive the residues by >25%. The trials were considered suitable for maximum residue level estimation.

APPRAISAL

Acetamiprid is a neonicotinoid insecticide. The compound was evaluated for the first time by the JMPR in 2011 (T,R), where an ADI of 0–0.07 mg/kg bw and an ARfD of 0.1 mg/kg bw was established. Maximum residue levels were recommended by the JMPR in 2011, 2012 (response to concerns raised on several leafy vegetables) and 2015.

The residue definition for acetamiprid in plant commodities for compliance with the MRL and dietary risk assessment is acetamiprid. The residue definition for animal commodities for compliance with the MRL and dietary risk assessment is the sum of acetamiprid and N-desmethyl-acetamiprid, expressed as acetamiprid.

The residue is not fat-soluble.

The 2017 JMPR evaluated acetamiprid for an additional use on pistachio in Iran, but concluded that the provided data did not match the GAP. At the Fiftieth Session of the CCPR it was indicated an alternative GAP would be provided. The current Meeting received a new label to support a maximum residue level recommendation for use of acetamiprid on pistachio, based on field trial data provided in 2017.

Results of supervised residue trials on crops

Pistachio

The critical GAP for acetamiprid on pistachio in Iran is for 3 foliar applications at 0.05 kg ai/ha and a PHI of 28 days. The 2017 Meeting received four trials, conducted in Iran, using an application rate of 3 × 0.05 kg ai/ha, a re-treatment interval (RTI) of 39–60 days between the first two treatments and 29–32 days between the second and third treatments and a PHI 28–30 days. Information from the Iranian government confirms that these RTI ranges are common practice in the country.

The Meeting decided that the four trials were suitable for maximum residue level estimation. Residue levels (in nutmeat) were (n = 4): 0.20, 0.32, 0.34 and 0.49 mg/kg.

The Meeting estimated a maximum residue level of 1 mg/kg and an STMR and HR of 0.33 and 0.51 mg/kg (highest individual value), respectively for pistachio. The Meeting decided to withdraw the maximum residue level for the Group of Tree nuts of 0.06 mg/kg and to recommend a maximum residue level of 0.06 mg/kg for the Group of Tree nuts, except Pistachio.

Residues in animal commodities

Pistachio is not part of the diets listed in the most recent version of the OECD livestock dietary burden calculator. The Meeting therefore did not recalculate the livestock dietary burden and confirms its previous recommendations for animal commodities.

RECOMMENDATIONS

On the basis of the data obtained from supervised trials, the Meeting concluded that the residue levels listed below in Table 1 are suitable for establishing maximum residue limits and for IEDI and IESTI assessments.

Definition of the residue for compliance with the MRL and dietary risk assessment for plant commodities: *Acetamiprid*

Definition of the residue for compliance with the MRL and dietary risk assessment for animal commodities: *Sum of acetamiprid and N-desmethyl-acetamiprid, expressed as acetamiprid*.

The residue is not fat-soluble

Table 1 Recommendations for residues of acetamiprid from the 2021 Extra JMPR

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg eq	HR or HR-P mg/kg eq
		New	Previous		
TN 0085	Tree nuts, Group of	W	0.06	0.01	0.05
TN 0085	Tree nuts, Group of, except Pistachio nut	0.06	-	0.01	0.05
TN 0675	Pistachio nut	1	-	0.33	0.51

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for acetamiprid is 0–0.07 mg/kg bw. The International Estimated Daily Intakes (IEDIs) for acetamiprid were estimated for the 17 GEMS/Food Consumption Cluster Diets using the STMR or STMR-P values estimated by the JMPR. The results are shown in Annex 3 of the 2021 Extra JMPR Report.

The IEDIs ranged from 0–3% of the maximum ADI. The Meeting concluded that long-term dietary exposure to residues of acetamiprid from uses considered by previous and current JMPR is unlikely to present a public health concern.

Acute dietary exposure

The ARfD for acetamiprid is 0.1 mg/kg bw. The International Estimate of Short-Term Intakes (IESTIs) for acetamiprid were calculated for the food commodities and their processed commodities for which HRs/HR-Ps or STMRs/STMR-Ps were estimated by the present Meeting and for which consumption data were available. The results are shown in Annex 4 of the 2021 Extra JMPR Report.

The IESTI for pistachio varied from 2% of the ARfD for the general population to 3% of the ARfD for children. The Meeting concluded that acute dietary exposure to residues of acetamiprid from use considered by the present Meeting is unlikely to present a public health concern.

BIXAFEN (262)

First draft prepared by Mr C. Sieke, Federal Institute for Risk Assessment, Germany

EXPLANATION

Bixafen (ISO common name) is a pyrazole-carboxamide fungicide used to control diseases on multiple crops. Bixafen inhibits fungal respiration by binding to mitochondrial respiratory complex II. It was considered for the first time by the 2013 JMPR for toxicology and residues, when an ADI of 0–0.02 mg/kg bw and an ARfD of 0.2 mg/kg bw were established. Bixafen was last reviewed for residues by the 2016 JMPR, where residues in rotational crops were evaluated and recommendations were made for maximum residue levels in plant and animal commodities.

The 2013 JMPR recommended the following residue definition for bixafen:

Definition of the residue for compliance with the MRL for plant commodities: *bixafen*

Definition of the residue for compliance with the MRL for animal commodities and for dietary risk assessment for plant and animal commodities: *sum of bixafen and N-(3',4'-dichloro-5-fluorobiphenyl-2-yl)-3-(difluoromethyl)-1H-pyrazole-4-carboxamide (bixafen-desmethyl), expressed as bixafen*

The residue is fat-soluble.

Bixafen was scheduled at the Fifty-first Session of the CCPR for the evaluation of additional uses by the 2020 JMPR Meeting, which was postponed to the 2021 Extra JMPR. The current Meeting received new information on use patterns for bixafen in pulses, potatoes, cereal grains and oilseed crops, supported by additional plant metabolism studies, field rotational crop studies, analytical methods and recovery data, supervised field trials and studies simulating typical processing conditions.

METABOLISM AND ENVIRONMENTAL FATE

The current Meeting received metabolism studies conducted using [pyrazole-^{14}C]-bixafen and [dichlorophenyl-^{14}C]-bixafen. The positions of the labels are presented in the following figures:

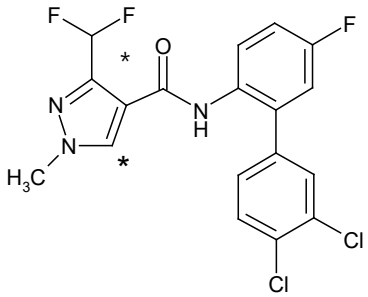


Figure 1 [pyrazole-^{14}C]-bixafen

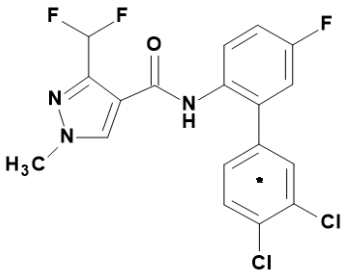
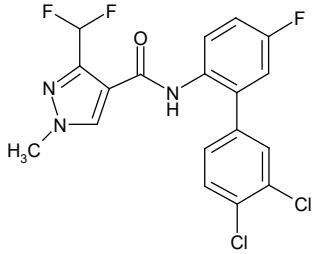
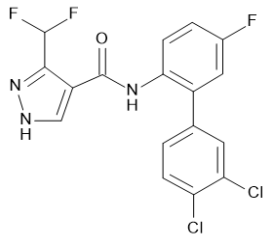
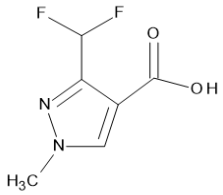
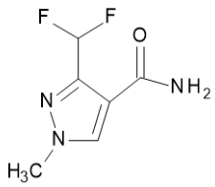
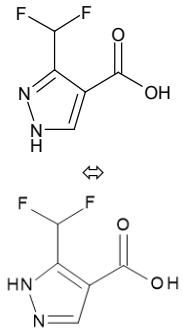
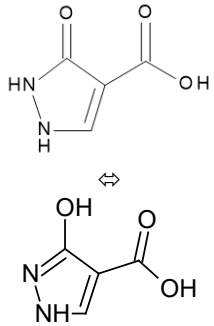


Figure 2 [dichlorophenyl-UL-^{14}C]-bixafen

Chemical names, structures and code names of metabolites and degradation products of bixafen discussed within this document are shown below. For a complete list of metabolites, please refer to the 2013 JMPR evaluation report.

Table 1 Metabolites of bixafen discussed within this document

Code Names	Chemical name	Structure	Where found
Bixafen BYF 00587 (AE1698406)	N-(3',4'-dichloro-5-fluorobiphenyl-2-yl)-3-(difluoromethyl)-1-methyl-1H-pyrazole-4-carboxamide (IUPAC) N-(3',4'-dichloro-5-fluoro[1,1'-biphenyl]-2-yl)-3-(difluoromethyl)-1-methyl-1*H*-pyrazole-4-carboxamide (CAS)		Plants Soil Livestock animals Rats
Bixafen-desmethyl (M21) (BCS-AA10008)	N-(3',4'-dichloro-5-fluorobiphenyl-2-yl)-3-(difluoromethyl)-1H-pyrazole-4-carboxamide		Plants Soil Livestock animals Rats
Bixafen-pyrazole-4-carboxylic acid (M42) (AE 1954999)	3-(difluoromethyl)-1-methyl-1H-pyrazole-4-carboxylic acid		Rotational crops Soil Livestock animals Rats
Bixafen-pyrazole-4-carboxamide (M43)	3-(difluoromethyl)-1-methyl-1H-pyrazole-4-carboxamide		Rotational crops Rats

Code Names	Chemical name	Structure	Where found
Bixafen-desmethyl-pyrazole-4-carboxylic acid (tautomer 1 & 2) (M44 & M45) (BCS-AA10651)	3-(difluoromethyl)-1H-pyrazole-4-carboxylic acid ⇔ 5-(difluoromethyl)-1*H*-pyrazole-4-carboxylic acid		Plants Rotational crops Rats
Bixafen-pyrazolone-4-carboxylic acid (M47)	3-oxo-2,3-dihydro-1H-pyrazole-4-carboxylic acid or 3-hydroxy-1H-pyrazole-4-carboxylic acid		Plants Rotational crops

Plant metabolism

The Meeting received new plant metabolism studies with bixafen on potatoes and tomatoes, each treated with [pyrazole-^{14}C]-bixafen or [dichlorophenyl-^{14}C]-bixafen.

Potato

The metabolic fate of [pyrazole-^{14}C]-bixafen (Moderegger D., 2015, BIXAFEN_079) and [dichlorophenyl-UL-^{14}C]-bixafen (Moderegger D., 2015, BIXAFEN_080) in potatoes (*Solanum tuberosum L., variety "Granola"*) was investigated in two parallel studies. The crops were cultivated in containers being subjected to natural sunlight but were covered during rainfall periods. Plants were separately treated for each radiolabel with three foliar applications of 0.24 kg ai/ha each at BBCH 61 (beginning of flowering), 14 days later at BBCH 70 (fruit development; first berries visible) and finally 42 days later at BBCH 97 (leaves and stem dead). Potato leaves were harvested at an intermediate growth stage (BBCH 92, beginning of senescence) 28 days after the second application. Mature tubers were sampled 7 days after the final treatment.

Potato leaves and tubers were conventionally extracted three times with a mixture of acetonitrile/water (4/1; v/v). The solids were separated from the extract by filtration. The radioactivity in the extracts was determined by LSC. The post extraction solids (PES) were radioassayed by combustion. The released $^{14}CO_2$ was trapped in an alkaline scintillation cocktail and the radioactivity was determined by LSC. For profiling of parent compound and metabolites the combined extracts were purified using an SPE cartridge. All fractions were radioassayed by LSC.

The extracts of potato leaves and potato tubers were analysed by HPLC. Detection was performed with a variable wavelength detector (set at 254 nm) connected in-line with a flow-through radioactivity detector equipped with a solid glass scintillator in the measuring cell. Parent compound and metabolites were quantified by integration of the radioactivity chromatograms and identified by spectroscopic methods, HPLC co-chromatography with non-radiolabelled reference compounds (bixafen, -desmethyl, -pyrazole-4-

carboxylic acid, -desmethyl-pyrazole-4-carboxylic acid and –amino-biphenyl) or by comparison of retention times with profiles of the confined rotational crops study. Unknown metabolites were characterised based on their extraction and chromatographic behaviour.

All extraction experiments and the HPLC analyses were performed within three months after sampling of the potato samples. The first metabolite profiling in the extract of tubers as well as the quantification of parent compound and metabolites were performed within one day after start of extraction, for potato leaves within nine days after start of extraction. Repeated analyses of potato leaf extracts showed no indication of degradation of parent compound and metabolites in the profiles.

TRR levels found were highest in potato leaves, while tubers contained only very low residues near the LOQ (Table 2).

Table 2 Total radioactivity in potato matrices following application of ^{14}C-bixafen (3 × 0.24 kg ai/ha)

Matrix	DAT	[pyrazole-^{14}C]-bixafen TRR in mg eq/kg	[dichlorophenyl-UL-^{14}C]-bixafen TRR in mg eq/kg
Potato leaves	28 (after 2nd treatment)	24.4	21.8
Potato tubers	7 (after 3rd treatment)	0.003	0.002

The major part of the radioactivity was extracted by conventional extraction with acetonitrile/water (4/1; v/v) from potato leaves (97.7–98% of the TRR, 21.3–23.8 mg eq/kg) and potato tubers (84.8% of the TRR, 0.003 mg eq/kg, pyrazole-label only). The radioactivity in the remaining solids (PES) amounted to 2.0–2.3% of the TRR (0.445–0.558 mg eq/kg) in potato leaves and 15.2% of the TRR (<0.001 mg eq/kg, pyrazole-label only) in potato tubers. The radioactivity in potato tubers from the dichlorophenyl-label and the solids from both radiolabels was not further investigated.

Table 3 Distribution of radioactivity in the extracts of potato leaves and tubers after foliar application of ^{14}C-bixafen (3 × 0.24 kg ai/ha)

Fraction	[pyrazole-^{14}C]-bixafen				[dichlorophenyl-UL-^{14}C]-bixafen	
	Potato leaves		Potato tubers		Potato leaves	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
TRR	100	24.4	100	0.003	100	21.8
Extraction with acetonitrile/water (4/1; v/v)	97.7	23.8	84.8	0.003	98.0	21.3
Concentrate used for quantitation of metabolites	97.3	23.7	84.8	0.003	97.4	21.2
Not analysed fraction (SPE)	0.4	0.088	--	--	0.6	0.124
PES (Unextractable residues)	2.3	0.558	15.2	<0.001	2.0	0.445
Total recovery	100	24.4	100	0.003	100	21.8

The metabolic profile following characterization and identification of the radioactivity in potato leaves and tubers is presented in the following table. In potato tubers, identification was only performed for the pyrazole label.

Table 4 Identification of radioactivity in potato matrices following application of ^{14}C-bixafen (3 × 0.24 kg ai/ha)

Fraction/Compound	[pyrazole-^{14}C]-bixafen				[dichlorophenyl-UL-^{14}C]-bixafen	
	Potato leaves (28 DAT after 2nd treatment)		Potato tubers (7 DALT)		Potato leaves (28 DAT after 2nd treatment)	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Extract with acetonitrile/water (4/1; v/v)	97.7	23.8	84.8	0.003	98.0	21.3
Bixafen (parent)	95.6	23.3	31.4	0.001	95.9	20.9
Bixafen-desmethyl (M21)	0.6	0.135	---	---	0.6	0.121
Bixafen-desmethyl-pyrazole-4-carboxylic acid (tautomer 1 & 2) (M44 & M45)	–	–	12.5	<0.001	--	--
Bixafen-pyrazole-4-carboxamide (M43)	0.1	0.025	9.0	<0.001	--	--
Bixafen-pyrazolone-4-carboxylic Acid (M42)	<0.1	0.006	25.9	0.001	--	--
Bixafen-pyrazole-4-carboxylic Acid (M47)	0.1	0.023	5.9	<0.001	--	--
Unknown 1	0.1	0.018	–	–	0.1	0.012
Unknown 2	0.2	0.041	–	–	0.2	0.034
Unknown 3	0.1	0.032	–	–	0.1	0.023
Unknown 4	0.4	0.086	–	–	0.3	0.075
Unknown 5	0.1	0.023	–	–	0.1	0.020
Unknown 6	0.1	0.019	–	–	0.1	0.019
Unknown 7	0.1	0.030	–	–	0.1	0.027
Not analysed fractions (SPE)	0.4	0.088	–	–	0.6	0.124
PES	2.3	0.558	15.2	<0.001	2.0	0.445
Total recovery	100.0	24.4	100.0	0.003	100	21.8

A proposed metabolic pathway for bixafen in potatoes (leaves and tubers) is presented in the following figure.

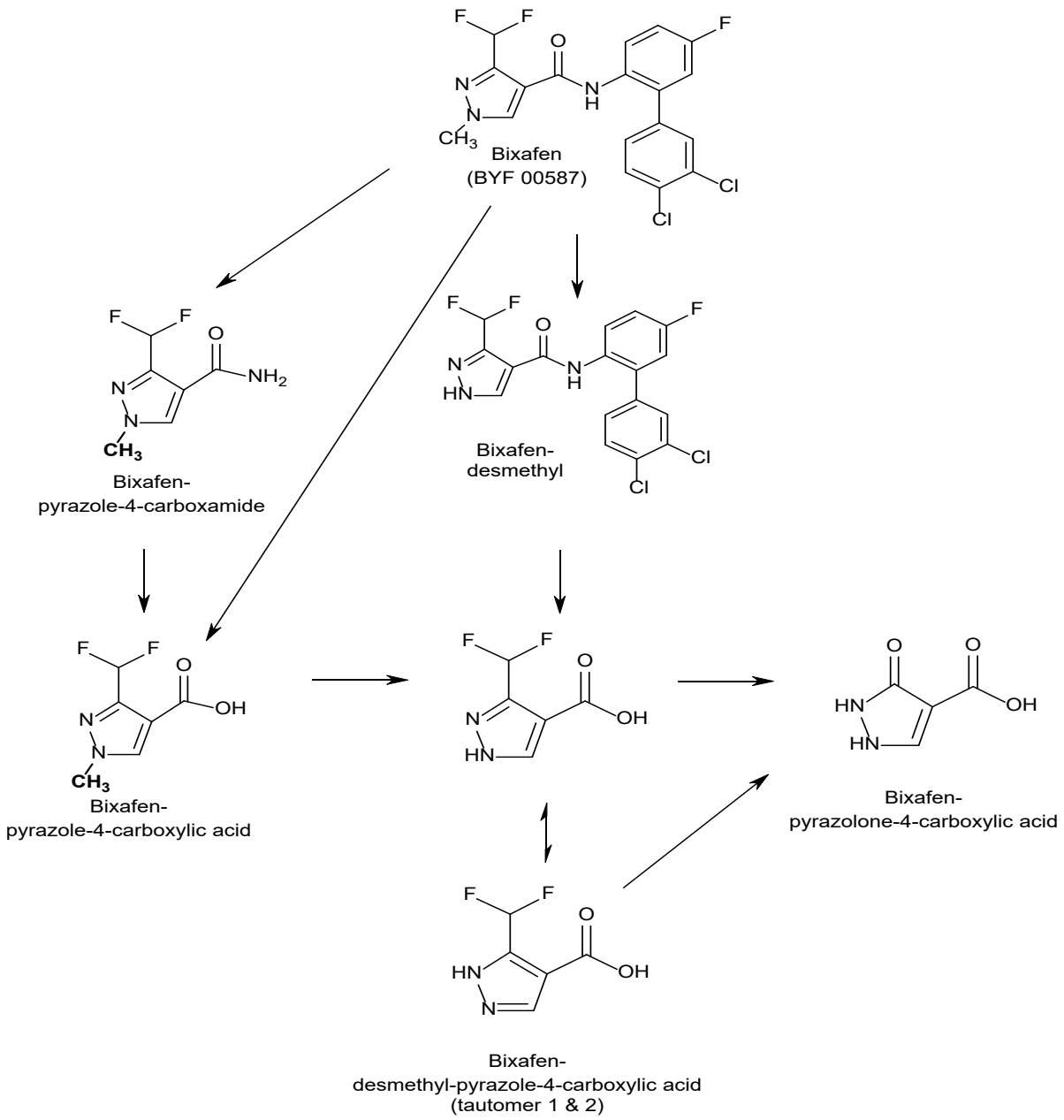


Figure 1: Proposed metabolic pathway for bixafen in potatoes

Tomato

For tomatoes (*Lycopersicon lycopersicum L. cv.* "Philona"), the metabolic fate of [pyrazole-^{14}C]-bixafen (Moderegger D., 2016, BIXAFEN_081) and [dichlorophenyl-UL-^{14}C]-bixafen (Moderegger D., 2016, BIXAFEN_082) was investigated in two parallel studies conducted under greenhouse conditions.

The tomato plants were treated with either EC 125 formulated [pyrazole-5-^{14}C]-bixafen or [dichlorophenyl-UL-^{14}C]-bixafen by foliar spray application. The application conditions simulated three foliar applications with single application rates of about 0.21–0.23 kg ai/ha for the applications at BBCH 78–80 (development of fruit: fruits have reached typical form and size), 21 days later at BBCH 84–85 (ripening of fruit : 40–50% of fruits show typical fully ripe colour) and 14 days later at BBCH 87–89 (ripening of fruit : 70–100% of fruits show typical fully ripe colour). Samples of fruits and leaves were collected 3 days after the last treatment (DALT).

A subset of fruits were surface-washed by dipping the fruits into a dichloromethane bath and subsequent rinsing with dichloromethane. Afterwards, an aliquot of the surface-washed tomato fruits was subsequently extracted three times with acetonitrile/water (4/1; v/v) using a high-speed blender. Solids were separated from the extracts by centrifugation. Aliquots of extracts and the remaining post extraction solids (PES) were radioassayed by LSC. The first two of three acetonitrile/water extracts were combined and purified using an SPE cartridge.

The surface wash and conventional extract of tomato fruits were analysed by HPLC. Detection was performed with a variable wavelength detector (set at 254 nm) connected in-line with a flow-through radioactivity detector. Parent compound and metabolites were quantified by integration of the radioactivity chromatograms and identified by spectroscopic methods and by HPLC and TLC co-chromatography with non-radiolabelled reference compounds. Unknown metabolites were characterised based on their extraction and chromatographic behaviour.

All extraction experiments and the HPLC analyses were performed within three weeks after sampling of the tomato samples. The first metabolite profiling in the surface wash and the extract of tomato fruits as well as the quantitation of parent compound and metabolites were performed within six days after surface wash and four days after the start of extraction, respectively. Repeated analyses of surface wash and extract of tomato fruits showed no indication of degradation of parent compound and metabolites in the profiles.

TRR levels found in fruits from the different experiments is shown below (Table 5).

Table 5 Total radioactivity in tomato fruits following application of ^{14}C-bixafen (3 × 0.21–0.23 kg ai/ha)

Matrix	DALT	[pyrazole-^{14}C]-bixafen TRR in mg eq/kg	[dichlorophenyl-UL-^{14}C]-bixafen TRR in mg eq/kg
Tomato fruits	3	1.75	3.19

From the treated fruits, the major part of the TRR was recovered in the surface wash (93.1–94.4% TRR). Nearly the complete remaining radioactivity was recovered in the extracts, leaving only 0.1% TRR unextracted. A summary of the extraction is presented in the following table:

Table 6 Distribution of radioactivity in the extracts of tomato fruits after foliar application ^{14}C-bixafen (3 × 0.21–0.23 kg ai/ha, 3 DALT)

Fraction	[pyrazole-^{14}C]-bixafen		[dichlorophenyl-UL-^{14}C]-bixafen	
	% TRR	mg eq/kg	% TRR	mg eq/kg
TRR	100	1.75	100	3.19
Surface wash with dichloromethane	94.4	1.65	93.1	2.97
Conventional extraction with acetonitrile/water (4/1; v/v)	5.6	0.098	6.9	0.219
Not analysed fraction	0.1	0.001	0.1	0.002
Total extracted	99.9	1.75	99.9	3.19
PES (Unextracted residues)	0.1	0.001	0.1	0.002
Total recovery	100	1.75	100	3.19

The identification of the radioactivity in tomato fruit revealed mostly unchanged parent bixafen for both labels (>99% TRR). The only metabolite identified was bixafen-desmethyl, present up to 0.1% of the TRR. A summary of the identified radioactivity is presented in the following table.

Table 7 Identification of radioactivity in tomato fruits following application of ^{14}C-bixafen (3 × 0.21–0.23 kg ai/ha, 3 DALT)

Fraction/Compound	[pyrazole-^{14}C]-bixafen		[dichlorophenyl-UL-^{14}C]-bixafen	
	% TRR	mg eq/kg	% TRR	mg eq/kg
Surface wash with dichloromethane	94.4	1.65	93.1	2.97
Bixafen (parent)	94.2	1.65	92.9	2.96
Unknown 2	0.2	0.003	0.2	0.005
Extraction with acetonitrile/water (4/1; v/v)	5.6	0.098	6.9	0.219
Bixafen (parent)	5.4	0.094	6.6	0.212
Bixafen-desmethyl	0.1	0.002	0.1	0.004
Unknown 1	<0.1	0.001	--	--
Not analysed fraction	0.1	0.001	0.1	0.002
PES (unextracted residues)	0.1	0.001	0.1	0.002
Total recovery	100	1.75	100	3.19

A proposed metabolic pathway for bixafen in tomatoes is presented in the following figure.

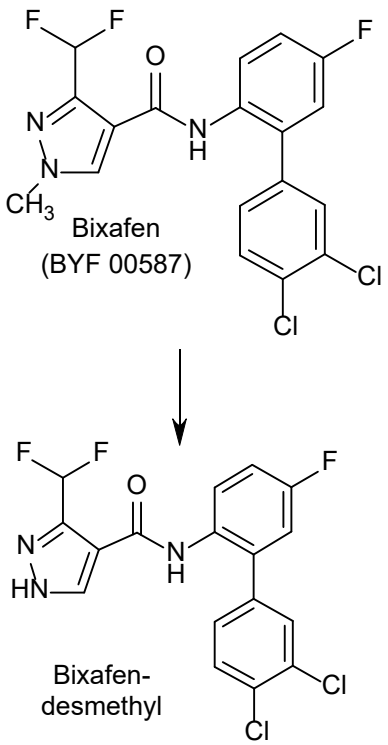


Figure 4 Proposed metabolic pathway for bixafen in tomatoes

Rotational crops

The Meeting received an additional field rotational crop study with bixafen on multiple crops.

Field rotational crop studies

The Meeting received a set of field rotational crop studies for bixafen conducted according to a similar layout at various experimental sites in Europe. In all studies, a single treatment of 1.13 kg ai/ha was applied

to bare soil and incorporated (8 cm depth). After nominal plantback intervals (PBIs) of 30, 120 and 365 days, rotational crops were planted/seeded.

In all trials soil samples were analysed according to method 00952/M002 (analytes: bixafen, bixafen-desmethyl, bixafen-desmethyl-pyrazole-4-carboxylic acid). The LOQ was 0.005 mg/kg in soil for each analyte (expressed as analyte equivalent).

Plant samples were analysed with two methods. Method 01013 was used to analyse parent compound and bixafen-desmethyl while the metabolites bixafen-desmethyl-pyrazole-4-carboxylic acid (M44/45) and bixafen-pyrazole-4-carboxamide (M43) were measured using method 01366/M001. The LOQ for both methods was 0.01 mg/kg per analyte (expressed as analyte equivalent).

Procedural recoveries for all methods and analytes were demonstrated being within acceptable ranges (70–120%, RSD <20%).

Table 8 Summary of residues in rotational crops after bare soil application of bixafen at 1.13 kg/ha in various European countries

Trial location / No.	PBI (DAT)	Sampling		Matrix	Residues (mg eq/kg)				
		Growth stage (BBCH)	DAT		Bixafen	Bixafen-desmethyl	Total	M44/45	M43
Strawberries (Freitag T., 2017, BIXAFEN_090; Maximum storage intervals: soil 458 d, fruits: 443 d)									
Belgium, Saint-Amand 15-2517-01	28	--	28	Soil	0.18	<0.005	--	<0.005	--
		85	84	Fruits	<0.01	<0.01	<0.02	<0.01	<0.01
		87	91	Fruits	<0.01	<0.01	<0.02	<0.01	<0.01
	155	--	155	Soil	0.16	<0.005	--	<0.005	--
		85	217	Fruits	<0.01	<0.01	<0.02	<0.01	<0.01
		87	223	Fruits	<0.01	<0.01	<0.02	<0.01	<0.01
	336	--	336	Soil	0.18	<0.005	--	<0.005	--
		85	398	Fruits	<0.01	<0.01	<0.02	<0.01	<0.01
		87	404	Fruits	<0.01	<0.01	<0.02	<0.01	<0.01
Netherlands, Zwaagdijk 15-2517-02	28	--	28	Soil	0.27	<0.005	--	<0.005	--
		85	83	Fruits	<0.01	<0.01	<0.02	<0.01	<0.01
		87	90	Fruits	<0.01	<0.01	<0.02	<0.01	<0.01
	176	--	176	Soil	0.16	<0.005	--	<0.005	--
		85	224	Fruits	<0.01	<0.01	<0.02	<0.01	<0.01
		87	230	Fruits	<0.01	<0.01	<0.02	<0.01	<0.01
	351	--	351	Soil	0.19	<0.005	--	<0.005	--
		85	399	Fruits	<0.01	<0.01	<0.02	<0.01	<0.01
		87	405	Fruits	<0.01	<0.01	<0.02	<0.01	<0.01
Spain, Gava 15-2517-03	28	--	27	Soil	0.32	<0.005	--	<0.005	--
		85	101	Fruits	<0.01	<0.01	<0.02	<0.01	<0.01
		87	150	Fruits	<0.01	<0.01	<0.02	<0.01	<0.01
	128	--	127	Soil	0.33	<0.005	--	<0.005	--
		85	201	Fruits	<0.01	<0.01	<0.02	<0.01	<0.01
		87	250	Fruits	<0.01	<0.01	<0.02	<0.01	<0.01
	311	--	310	Soil	0.24	<0.005	--	<0.005	--
		85	384	Fruits	<0.01	<0.01	<0.02	<0.01	<0.01
		87	433	Fruits	<0.01	<0.01	<0.02	<0.01	<0.01
Italy, Policoro 15-2517-04	26	--	26	Soil	0.13	<0.005	--	<0.005	--
		85	222	Fruits	<0.01	<0.01	<0.02	<0.01	<0.01
		87	244	Fruits	<0.01	<0.01	<0.02	<0.01	<0.01
	124	--	124	Soil	0.19	<0.005	--	<0.005	--
		85	320	Fruits	<0.01	<0.01	<0.02	<0.01	<0.01

Trial location / No.	PBI (DAT)	Sampling		Matrix	Residues (mg eq/kg)				
		Growth stage (BBCH)	DAT		Bixafen	Bixafen-desmethyl	Total	M44/45	M43
		87	342	Fruits	<0.01	<0.01	<0.02	<0.01	<0.01
	351	--	351	Soil	0.13	<0.005	--	<0.005	--
		85	534	Fruits	<0.01	<0.01	<0.02	<0.01	<0.01
		87	553	Fruits	<0.01	<0.01	<0.02	<0.01	<0.01
Leek (Freitag T., 2017, BIXAFEN_084; Maximum storage intervals: soil 349 d, stalks: 281 d)									
Belgium, Saint-Amand 15-2511-01	22	--	22	Soil	0.19	<0.005	--	<0.005	--
		49	161	Stalks	<0.01	<0.01	<0.02	<0.01	<0.01
	155	--	155	Soil	0.15	<0.005	--	<0.005	--
		49	273	Stalks	<0.01	<0.01	<0.02	<0.01	<0.01
	349	--	349	Soil	0.17	<0.005	--	<0.005	--
		49	467	Stalks	<0.01	<0.01	<0.02	<0.01	<0.01
Netherlands, Zwaagdijk 15-2511-02	29	--	29	Soil	0.23	<0.005	--	<0.005	--
		49	127	Stalks	<0.01	<0.01	<0.02	0.016	<0.01
	175	--	175	Soil	0.16	<0.005	--	<0.005	--
		49	264	Stalks	<0.01	<0.01	<0.02	<0.01	<0.01
	350	--	350	Soil	0.22	<0.005	--	<0.005	--
		49	439	Stalks	<0.01	<0.01	<0.02	0.011	<0.01
Spain, Alginet 15-2511-03	21	--	22	Soil	0.23	<0.005	--	<0.005	--
		49	117	Stalks	<0.01	<0.01	<0.02	<0.01	<0.01
	179	--	180	Soil	0.15	<0.005	--	<0.005	--
		49	294	Stalks	<0.01	<0.01	<0.02	<0.01	<0.01
	313	--	314	Soil	0.15	<0.005	--	<0.005	--
		49	428	Stalks	<0.01	<0.01	<0.02	<0.01	<0.01
Italy, Manfredonia 15-2511-04	30	--	30	Soil	0.13	<0.005	--	<0.005	--
		49	189	Stalks	<0.01	<0.01	<0.02	<0.01	<0.01
	136	--	137	Soil	0.13	<0.005	--	<0.005	--
		49	244	Stalks	<0.01	<0.01	<0.02	0.034	<0.01
	365	--	365	Soil	0.074	<0.005	--	<0.005	--
		49	473	Stalks	<0.01	<0.01	<0.02	0.014	<0.01
Cabbage (Freitag T., 2017, BIXAFEN_087; Maximum storage intervals: soil 344 d, heads: 334 d)									
Belgium, Saint-Amand 15-2514-01	20	--	20	Soil	0.22	<0.005	--	<0.005	--
		48	133	Head	<0.01	<0.01	<0.02	<0.01	<0.01
		49	147	Head	<0.01	<0.01	<0.02	<0.01	<0.01
	155	--	155	Soil	0.15	<0.005	--	<0.005	--
		48	253	Head	<0.01	<0.01	<0.02	<0.01	<0.01
		49	267	Head	<0.01	<0.01	<0.02	<0.01	<0.01
	349	--	349	Soil	0.14	<0.005	--	<0.005	--
		48	447	Head	<0.01	<0.01	<0.02	<0.01	<0.01
		49	461	Head	<0.01	<0.01	<0.02	<0.01	<0.01
Netherlands, Zwaagdijk 15-2514-02	28	--	29	Soil	0.20	<0.005	--	<0.005	--
		47	113	Head	<0.01	<0.01	<0.02	0.01	<0.01
		49	127	Head	<0.01	<0.01	<0.02	<0.01	<0.01
	168	--	168	Soil	0.16	<0.005	--	<0.005	--
		48	258	Head	<0.01	<0.01	<0.02	<0.01	<0.01
		49	272	Head	<0.01	<0.01	<0.02	<0.01	<0.01
	343	--	343	Soil	0.16	<0.005	--	<0.005	--
		48	433	Head	<0.01	<0.01	<0.02	<0.01	<0.01
		49	447	Head	<0.01	<0.01	<0.02	<0.01	<0.01
Spain, Alginet 15-2514-04	24	--	25	Soil	0.32	<0.005	--	<0.005	--
		48	83	Head	<0.01	<0.01	<0.02	<0.01	<0.01
		49	97	Head	<0.01	<0.01	<0.02	<0.01	<0.01

Trial location / No.	PBI (DAT)	Sampling		Matrix	Residues (mg eq/kg)				
		Growth stage (BBCH)	DAT		Bixafen	Bixafen-desmethyl	Total	M44/45	M43
	179	--	180	Soil	0.18	<0.005	--	<0.005	--
		47	267	Head	<0.01	<0.01	<0.02	<0.01	<0.01
		49	281	Head	<0.01	<0.01	<0.02	<0.01	<0.01
	325	--	326	Soil	0.14	<0.005	--	<0.005	--
		47	413	Head	<0.01	<0.01	<0.02	<0.01	<0.01
		49	427	Head	<0.01	<0.01	<0.02	<0.01	<0.01
Italy, Bitonto 15-2514-04	22	--	22	Soil	0.37	<0.005	--	<0.005	--
		45	92	Head	<0.01	<0.01	<0.02	<0.01	<0.01
		49	105	Head	<0.01	<0.01	<0.02	<0.01	<0.01
	164	--	164	Soil	0.26	<0.005	--	<0.005	--
		45	241	Head	<0.01	<0.01	<0.02	<0.01	<0.01
		49	255	Head	<0.01	<0.01	<0.02	<0.01	<0.01
	353	--	353	Soil	0.19	<0.005	--	<0.005	--
		45	430	Head	<0.01	<0.01	<0.02	<0.01	<0.01
		49	444	Head	<0.01	<0.01	<0.02	<0.01	<0.01
Courgettes (Freitag T., 2017, BIXAFEN_083; Maximum storage intervals: soil 372 d, fruits: 349 d)									
Belgium, Saint-Amand 15-2510-01	26	--	26	Soil	0.17	<0.005	--	<0.005	--
		72	74	Fruit	<0.01	<0.01	<0.02	<0.01	<0.01
		79	88	Fruit	<0.01	<0.01	<0.02	<0.01	<0.01
	157	--	157	Soil	0.21	<0.005	--	<0.005	--
		71	203	Fruit	<0.01	<0.01	<0.02	<0.01	<0.01
		75	217	Fruit	<0.01	<0.01	<0.02	<0.01	<0.01
	335	--	335	Soil	0.18	<0.005	--	<0.005	--
		71	381	Fruit	<0.01	<0.01	<0.02	<0.01	<0.01
		75	395	Fruit	<0.01	<0.01	<0.02	<0.01	<0.01
Netherlands, Zwaagdijk 15-2510-02	28	--	28	Soil	0.18	<0.005	--	<0.005	--
		81	71	Fruit	<0.01	<0.01	<0.02	0.011	<0.01
		81	85	Fruit	<0.01	<0.01	<0.02	<0.01	<0.01
	169	--	169	Soil	0.12	<0.005	--	<0.005	--
		89	202	Fruit	<0.01	<0.01	<0.02	0.015	<0.01
		89	216	Fruit	<0.01	<0.01	<0.02	<0.01	<0.01
	344	--	344	Soil	0.14	<0.005	--	<0.005	--
		89	377	Fruit	<0.01	<0.01	<0.02	<0.01	<0.01
		89	391	Fruit	<0.01	<0.01	<0.02	<0.01	<0.01
Spain, Alginet 15-2510-03	28	--	29	Soil	0.20	<0.005	--	<0.005	--
		71	63	Fruit	<0.01	<0.01	<0.02	<0.01	<0.01
		74	78	Fruit	<0.01	<0.01	<0.02	<0.01	<0.01
	174	--	175	Soil	0.16	<0.005	--	<0.005	--
		71	239	Fruit	<0.01	<0.01	<0.02	<0.01	<0.01
		73	253	Fruit	<0.01	<0.01	<0.02	<0.01	<0.01
	344	--	345	Soil	0.13	<0.005	--	<0.005	--
		71	409	Fruit	<0.01	<0.01	<0.02	<0.01	<0.01
		73	423	Fruit	<0.01	<0.01	<0.02	<0.01	<0.01
Italy, Bitonto 15-2510-04	25	--	25	Soil	0.24	<0.005	--	<0.005	--
		71	54	Fruit	<0.01	<0.01	<0.02	<0.01	<0.01
		73	69	Fruit	<0.01	<0.01	<0.02	<0.01	<0.01
	162	--	162	Soil	0.25	<0.005	--	<0.005	--
		71	201	Fruit	<0.01	<0.01	<0.02	0.023	<0.01
		73	215	Fruit	<0.01	<0.01	<0.02	0.019	<0.01
	346	--	346	Soil	0.18	<0.005	--	<0.005	--
		71	385	Fruit	<0.01	<0.01	<0.02	0.018	<0.01

Trial location / No.	PBI (DAT)	Sampling		Matrix	Residues (mg eq/kg)				
		Growth stage (BBCH)	DAT		Bixafen	Bixafen-desmethyl	Total	M44/45	M43
		73	399	Fruit	<0.01	<0.01	<0.02	0.014	<0.01
Peas (Freitag T., 2017, BIXAFEN_086; Maximum storage intervals: soil 707 d, vines and seeds: 355 d)									
Belgium, Saint-Amand 15-2513-01	20	--	20	Soil	0.19	<0.005	--	<0.005	--
		73	95	Vines	<0.01	<0.01	<0.02	<0.01	<0.01
		89	125	Dry seed	<0.01	<0.01	<0.02	<0.01	<0.01
	123	--	123	Soil	0.14	<0.005	--	<0.005	--
		73	204	Vines	<0.01	<0.01	<0.02	<0.01	<0.01
		89	246	Dry seed	<0.01	<0.01	<0.02	<0.01	<0.01
	301	--	301	Soil	0.16	<0.005	--	<0.005	--
		73	382	Vines	<0.01	<0.01	<0.02	<0.01	<0.01
		89	424	Dry seed	<0.01	<0.01	<0.02	<0.01	<0.01
Netherlands, Zwaagdijk 15-2513-02	24	--	24	Soil	0.17	<0.005	--	<0.005	--
		73	92	Vines	<0.01	<0.01	<0.02	0.030	<0.01
		89	125	Dry seed	<0.01	<0.01	<0.02	0.037	<0.01
	141	--	141	Soil	0.25	<0.005	--	<0.005	--
		73	214	Vines	<0.01	<0.01	<0.02	0.011	<0.01
		89	239	Dry seed	<0.01	<0.01	<0.02	0.019	<0.01
	316	--	316	Soil	0.23	<0.005	--	<0.005	--
		73	389	Vines	<0.01	<0.01	<0.02	<0.01	<0.01
		89	414	Dry seed	<0.01	<0.01	<0.02	0.011	<0.01
Spain, Brenes 15-2513-03	23	--	24	Soil	0.098	<0.005	--	<0.005	--
		73	81	Vines	<0.01	<0.01	<0.02	<0.01	<0.01
		89	109	Dry seed	<0.01	<0.01	<0.02	<0.01	<0.01
	221	--	222	Soil	0.14	<0.005	--	<0.005	--
		73	284	Vines	<0.01	<0.01	<0.02	0.082	<0.01
		89	307	Dry seed	<0.01	<0.01	<0.02	0.11	<0.01
	364	--	365	Soil	0.12	<0.005	--	<0.005	--
		73	427	Vines	<0.01	<0.01	<0.02	0.047	<0.01
		89	451	Dry seed	<0.01	<0.01	<0.02	0.061	<0.01
Greece, Aronas 15-2513-04	27	--	28	Soil	0.15	<0.005	--	<0.005	--
		73	91	Vines	<0.01	<0.01	<0.02	0.055	<0.01
		89	124	Dry seed	0.033	<0.01	0.043	<0.01	<0.01
	195	--	196	Soil	0.13	<0.005	--	<0.005	--
		73	400	Vines	<0.01	<0.01	<0.02	<0.01	<0.01
		89	466	Dry seed	<0.01	<0.01	<0.02	<0.01	<0.01
	362	--	363	Soil	0.068	<0.005	--	<0.005	--
		73	428	Vines	<0.01	<0.01	<0.02	<0.01	<0.01
		89	474	Dry seed	<0.01	<0.01	<0.02	<0.01	<0.01
Potato (Freitag T., 2017, BIXAFEN_085; Maximum storage intervals: soil 692 d, tubers: 695 d)									
Belgium, Saint-Amand 15-2512-01	22	--	22	Soil	0.15	<0.005	--	<0.005	--
		45	105	Tuber	<0.01	<0.01	<0.02	0.012	<0.01
		49	179	Tuber	<0.01	<0.01	<0.02	<0.01	<0.01
	130	--	130	Soil	0.20	<0.005	--	<0.005	--
		45	229	Tuber	<0.01	<0.01	<0.02	0.016	<0.01
		49	288	Tuber	<0.01	<0.01	<0.02	0.010	<0.01
	311	--	311	Soil	0.18	<0.005	--	<0.005	--
		45	410	Tuber	<0.01	<0.01	<0.02	0.012	<0.01
		49	469	Tuber	<0.01	<0.01	<0.02	<0.01	<0.01
Netherlands, Zwaagdijk 15-2512-02	28	--	28	Soil	0.19	<0.005	--	<0.005	--
		45	101	Tuber	0.018	<0.01	0.028	0.064	<0.01
		49	115	Tuber	0.012	<0.01	0.022	0.040	<0.01

Trial location / No.	PBI (DAT)	Sampling		Matrix	Residues (mg eq/kg)				
		Growth stage (BBCH)	DAT		Bixafen	Bixafen-desmethyl	Total	M44/45	M43
	144	--	144	Soil	0.20	<0.005	--	<0.005	--
		45	209	Tuber	<0.01	<0.01	<0.02	0.050	<0.01
		49	228	Tuber	<0.01	<0.01	<0.02	0.032	<0.01
	319	--	319	Soil	0.23	<0.005	--	<0.005	--
		45	384	Tuber	<0.01	<0.01	<0.02	0.025	<0.01
		49	403	Tuber	<0.01	<0.01	<0.02	0.022	<0.01
France, Toulouse 15-2512-03	21	--	21	Soil	0.029	<0.005	--	<0.005	--
		45	90	Tuber	0.058	<0.01	0.068	0.061	<0.01
		49	125	Tuber	0.054	<0.01	0.064	0.012	<0.01
	152	--	152	Soil	0.10	<0.005	--	<0.005	--
		45	236	Tuber	0.019	<0.01	0.029	0.060	<0.01
		49	259	Tuber	0.015	<0.01	0.025	0.051	<0.01
	361	--	361	Soil	0.086	<0.005	--	<0.005	--
		45	445	Tuber	<0.01	<0.01	<0.02	0.038	<0.01
		49	468	Tuber	<0.01	<0.01	<0.02	0.035	<0.01
Spain, Alginet 15-2512-04	28	--	28	Soil	0.22	<0.005	--	<0.005	--
		45	107	Tuber	<0.01	<0.01	<0.02	<0.01	<0.01
		49	128	Tuber	<0.01	<0.01	<0.02	<0.01	<0.01
	173	--	172	Soil	0.12	<0.005	--	<0.005	--
		45	256	Tuber	<0.01	<0.01	<0.02	0.016	<0.01
		49	273	Tuber	<0.01	<0.01	<0.02	<0.01	<0.01
	343	--	342	Soil	0.11	<0.005	--	<0.005	--
		45	426	Tuber	<0.01	<0.01	<0.02	0.015	<0.01
		49	443	Tuber	<0.01	<0.01	<0.02	0.013	<0.01
Maize (Freitag T., 2017, BIXAFEN_089; Maximum storage intervals: soil 672 d, plants and seeds: 623 d)									
Belgium, Saint-Amand 15-2516-01	20	--	20	Soil	0.18	<0.005	--	<0.005	--
		85	147	Forage	<0.01	<0.01	<0.02	<0.01	<0.01
		89	196	Kernel	<0.01	<0.01	<0.02	<0.01	<0.01
		89	196	Stover	<0.01	<0.01	<0.02	<0.01	<0.01
	153	--	153	Soil	0.18	<0.005	--	<0.005	--
		85	286	Forage	<0.01	<0.01	<0.02	<0.01	<0.01
		89	330	Kernel	<0.01	<0.01	<0.02	<0.01	<0.01
		89	330	Stover	<0.01	<0.01	<0.02	<0.01	<0.01
	334	--	334	Soil	0.16	<0.005	--	<0.005	--
		85	467	Forage	<0.01	<0.01	<0.02	<0.01	<0.01
		89	511	Kernel	<0.01	<0.01	<0.02	<0.01	<0.01
		89	511	Stover	<0.01	<0.01	<0.02	<0.01	<0.01
Netherlands, Zwaagdijk 15-2516-02	27	--	28	Soil	0.19	<0.005	--	<0.005	--
		85	157	Forage	<0.01	<0.01	<0.02	0.030	<0.01
		89	170	Kernel	<0.01	<0.01	<0.02	0.063	<0.01
		89	170	Stover	<0.01	<0.01	<0.02	<0.01	<0.01
	141	--	141	Soil	0.16	<0.005	--	<0.005	--
		85	279	Forage	<0.01	<0.01	<0.02	<0.01	<0.01
		89	287	Kernel	<0.01	<0.01	<0.02	<0.01	<0.01
		89	287	Stover	<0.01	<0.01	<0.02	<0.01	<0.01
	316	--	316	Soil	0.18	<0.005	--	<0.005	--
		85	454	Forage	<0.01	<0.01	<0.02	<0.01	<0.01
		89	462	Kernel	<0.01	<0.01	<0.02	<0.01	<0.01
		89	462	Stover	<0.01	<0.01	<0.02	<0.01	<0.01
France, Castelnau	24	--	25	Soil	0.16	<0.005	--	<0.005	--
		85	140	Forage	<0.01	<0.01	<0.02	<0.01	<0.01

Trial location / No.	PBI (DAT)	Sampling		Matrix	Residues (mg eq/kg)				
		Growth stage (BBCH)	DAT		Bixafen	Bixafen-desmethyl	Total	M44/45	M43
d'estretefonds 15-2516-03		89	181	Kernel	<0.01	<0.01	<0.02	<0.01	<0.01
		89	181	Stover	<0.01	<0.01	<0.02	<0.01	<0.01
	169	--	170	Soil	0.12	<0.005	--	<0.005	--
		85	301	Forage	<0.01	<0.01	<0.02	<0.01	<0.01
		89	338	Kernel	<0.01	<0.01	<0.02	<0.01	<0.01
		89	338	Stover	<0.01	<0.01	<0.02	0.025	<0.01
	363	--	364	Soil	0.10	<0.005	--	<0.005	--
		85	495	Forage	<0.01	<0.01	<0.02	<0.01	<0.01
		89	532	Kernel	<0.01	<0.01	<0.02	<0.01	<0.01
		89	532	Stover	<0.01	<0.01	<0.02	0.018	<0.01
Italy, Bologna 15-2516-04	20	--	20	Soil	0.14	<0.005	--	<0.005	--
		85	116	Forage	<0.01	<0.01	<0.02	<0.01	<0.01
		89	152	Kernel	<0.01	<0.01	<0.02	<0.01	<0.01
		89	152	Stover	<0.01	<0.01	<0.02	0.017	<0.01
	177	--	176	Soil	0.10	<0.005	--	<0.005	--
		85	302	Forage	<0.01	<0.01	<0.02	<0.01	<0.01
		89	334	Kernel	<0.01	<0.01	<0.02	<0.01	<0.01
		89	334	Stover	<0.01	<0.01	<0.02	<0.01	<0.01
	365	--	364	Soil	0.094	<0.005	--	<0.005	--
		85	490	Forage	<0.01	<0.01	<0.02	<0.01	<0.01
		89	522	Kernel	<0.01	<0.01	<0.02	<0.01	<0.01
		89	522	Stover	<0.01	<0.01	<0.02	0.023	<0.01
Oilseed rape (Freitag T., 2018, BIXAFEN_088; Maximum storage intervals: soil 558 d, plants and seeds: 422 d)									
Belgium, Saint-Amand 15-2515-01	21	--	21	Soil	0.21	<0.005	--	<0.005	--
		77	299	Forage	<0.01	<0.01	<0.02	<0.01	<0.01
		89	348	Seeds	<0.01	<0.01	<0.02	<0.01	<0.01
	156	--	156	Soil	0.15	<0.005	--	<0.005	--
		77	434	Forage	<0.01	<0.01	<0.02	<0.01	<0.01
		89	483	Seeds	<0.01	<0.01	<0.02	<0.01	<0.01
	323	--	323	Soil	0.12	<0.005	--	<0.005	--
		59	530	Forage	<0.01	<0.01	<0.02	<0.01	<0.01
		89	635	Seeds	<0.01	<0.01	<0.02	<0.01	<0.01
Germany, Burscheid 15-2515-02	18	--	18	Soil	0.073	<0.005	--	<0.005	--
		59	249	Forage	<0.01	<0.01	<0.02	<0.01	<0.01
		89	343	Seeds	<0.01	<0.01	<0.02	<0.01	<0.01
	129	--	129	Soil	0.15	<0.005	--	<0.005	--
		59	360	Forage	<0.01	<0.01	<0.02	<0.01	<0.01
		89	454	Seeds	<0.01	<0.01	<0.02	<0.01	<0.01
	340	--	340	Soil	0.092	<0.005	--	<0.005	--
		59	563	Forage	<0.01	<0.01	<0.02	<0.01	<0.01
		89	667	Seeds	<0.01	<0.01	<0.02	<0.01	<0.01
France, Castelnau	20	--	21	Soil	0.14	<0.005	--	<0.005	--
		59	215	Forage	<0.01	<0.01	<0.02	<0.01	<0.01
d'estretefonds 15-2515-03		89	300	Seeds	<0.01	<0.01	<0.02	<0.01	<0.01
	168	--	169	Soil	0.10	<0.005	--	<0.005	--
		59	363	Forage	<0.01	<0.01	<0.02	<0.01	<0.01
		89	448	Seeds	<0.01	<0.01	<0.02	<0.01	<0.01
	368	--	369	Soil	0.12	<0.005	--	<0.005	--
		59	571	Forage	<0.01	<0.01	<0.02	0.014	<0.01
		89	668	Seeds	<0.01	<0.01	<0.02	<0.01	<0.01
Italy,	27	--	28	Soil	0.057	<0.005	--	<0.005	--

Trial location / No.	PBI (DAT)	Sampling		Matrix	Residues (mg eq/kg)				
		Growth stage (BBCH)	DAT		Bixafen	Bixafen-desmethyl	Total	M44/45	M43
Palidoro Fiumicino 15-2515-04		59	182	Forage	<0.01	<0.01	<0.02	0.015	<0.01
		89	257	Seeds	<0.01	<0.01	<0.02	<0.01	<0.01
	139	--	140	Soil	0.086	<0.005	--	<0.005	--
		59	294	Forage	<0.01	<0.01	<0.02	0.01	<0.01
		89	369	Seeds	<0.01	<0.01	<0.02	<0.01	<0.01
	323	--	323	Soil	0.11	<0.005	--	<0.005	--
		59	496	Forage	<0.01	<0.01	<0.02	0.042	<0.01
		89	573	Seeds	<0.01	<0.01	<0.02	<0.01	<0.01

RESIDUE ANALYSIS

Analytical methods

For the analysis of bixafen and its metabolites new analytical methods for plant matrices and soil were provided. Additionally, new validation data on methods evaluated by previous JMPR Meetings were provided. In the following table an overview of the new methods and of the additional validation data is presented.

Table 9 Overview of analytical methods for bixafen residues

Method	Matrix	Extraction	Clean-Up	Detection, LOQ
New methods				
01367 (QuEChERS)	High water High oil High starch High acid High protein	Acetonitrile + salts for phase separation	SPE with primary secondary amine	HPLC-MS/MS Bixafen m/z: 414→394 (quant.) m/z: 414→266 (conf.) LOQ: 0.01 mg/kg
Additional validation data				
01012 (evaluated by 2013 JMPR)	New data for: High water High oil High starch High acid High sugar	acetonitrile/water (4/1, v/v) containing cysteine hydrochloride	filtration	HPLC-MS/MS with isotopically-labelled ISTD Bixafen: m/z 414 → 394 and m/z 414→ 266 LOQ: 0.01 mg/kg M21: m/z 400 → 380 and m/z 400→ 266 / 360 LOQ: 0.01 mg/kg
01013 (evaluated by 2013 JMPR)	New data for: High water High oil High starch High acid	acetonitrile/water (4/1, v/v) containing cysteine hydrochloride	filtration	HPLC-MS/MS Bixafen: m/z 414 → 394 LOQ: 0.01 mg/kg M21: m/z 398 → 378 LOQ: 0.01 mg/kg
01633/M001 (evaluated by 2013 JMPR)	New data for: High water High oil High starch High acid	acetonitrile/water (4/1, v/v) containing cysteine hydrochloride	filtration	HPLC-MS/MS M43: m/z 176 → 136 LOQ: 0.01 mg/kg M44/45: m/z 163 → 68

Method	Matrix	Extraction	Clean-Up	Detection, LOQ
				LOQ: 0.01 mg/kg
00952 (evaluated by 2013 JMPR)	Soil	acetonitrile/water (4/1, v/v)	filtration	HPLC-MS/MS Bixafen: m/z: 414→394 (quant.) m/z: 414→266 (conf.) LOQ: 0.01 mg/kg M21: m/z 398 → 378 and m/z 398→ 358 LOQ: 0.01 mg/kg M44/45: m/z 163 → 123 m/z 163 → 68 LOQ: 0.005 mg/kg

Plant materials

Method 01367 (Schoening R., 2013, BIXAFEN_100, Konrad S., 2014, BIXAFEN_101)

Analyte: Bixafen

Principle: LC-MS/MS (QuEChERS)

LOQs: 0.01 mg/kg in orange fruits, tomato fruits, potato tubers, dry bean seeds and dry soya bean seeds

Description: The procedure is based on the published QuEChERS approach (EN 15662: 2008 "Foods of plant origin – Determination of pesticide residues using GC-MS and/or LC-MS/MS following acetonitrile extraction/partitioning and clean-up by dispersive SPE-QuEChERS method", German version 2009) with minor modifications. The residues were extracted with acetonitrile/water (1/1, v/v) and the salt mixture (Mg_2SO_4/NaCl) (4/1, w/w) was added. After shaking and centrifugation, an aliquot of the upper acetonitrile extract was taken and diluted with a mixture of acetonitrile/water (2/8, v/v) + 0.1 mL/L acetic acid. The solution was injected into a HPLC-MS/MS system operated in positive ESI mode and the residues were quantified by external standardisation in matrix matched standards. Two MRM transitions were monitored: m/z = 414 → 394 for quantification and m/z = 414 → 266 for confirmation of bixafen.

Table 10 Recovery data for method 01367 measuring bixafen in plant matrices

Matrix	Fortification level (mg/kg)	n	Bixafen quantification (m/z 414 → 394)		Bixafen confirmation (m/z 414 → 266)	
			% Recovery (mean)	% RSD	% Recovery (mean)	% RSD
Schoening R., 2013, BIXAFEN_100						
Orange, fruit	0.01	5	100-102 (101)	0.9	98-103 (101)	2.1
	0.1	5	101-103 (102)	0.9	99-102 (100)	1.1
Tomato, fruit	0.01	5	105-108 (106)	1.1	99-105 (102)	2.4
	0.1	5	100-104 (101)	1.7	97-101 (99)	1.8
Potato, tuber	0.01	5	96-103 (99)	3.0	96-100 (97)	1.7
	0.1	5	100-103 (101)	1.1	97-102 (100)	1.9
Bean, dry seed	0.01	5	98-107 (101)	3.6	95-100 (96)	2.2
	0.1	5	99-102 (101)	1.6	98-100 (99)	1.3
Soya bean,	0.01	5	94-97 (96)	1.4	92-96 (95)	1.8

Matrix	Fortification level (mg/kg)	n	Bixafen quantification (m/z 414 → 394)		Bixafen confirmation (m/z 414 → 266)	
dry seed	0.1	5	99-102 (100)	1.1	98-101 (99)	1.1
Konrad S., 2014, BIXAFEN_101 (Independent laboratory validation)						
Orange, fruit	0.01	5	90-103 (98)	4.9	93-103 (97)	3.2
	0.1	5	97-99 (98)	1.0	96-102 (99)	2.4
Tomato, fruit	0.01	5	88-95 (90)	4.0	86-95 (91)	3.8
	0.1	5	97-101 (98)	1.8	93-99 (96)	2.2
Potato, tuber	0.01	5	94-101 (96)	2.7	94-99 (96)	1.9
	0.1	5	100-106 (104)	2.2	100-107 (103)	3.0
Bean, dry seed	0.01	5	100-104 (101)	1.6	97-104 (101)	2.6
	0.1	5	99-101 (99)	0.8	99-103 (100)	1.3
Soya bean, dry seed	0.01	5	71-97 (80)	12	73-95 (80)	11
	0.1	5	83-94 (87)	5.5	80-93 (86)	6.6

Stability of pesticide residues in stored analytical samples

Plant matrices

The Meeting received additional data on the storage stability of bixafen and bixafen-desmethyl under frozen conditions (Schoening, R., 2015, BIXAFEN_097).

The stability of bixafen and its metabolite bixafen-desmethyl was investigated in fortified control samples of material of plant origin (dry bean seed and orange fruit) during freezer storage (-18 °C or below) for 0 to 24 months. Nominal storage intervals were 0 days, 30, 60, 90, 180, 360, 540 and 720 days.

Control samples of dry bean seed and orange fruit were used for fortification. The control samples were homogenized with dry ice and an aliquot of 5 g was weighed into glass bottles. Individual amber glass bottles were used as storage containers for each sample in this study. This procedure allows extracting the whole fortified sample in the bottle. These samples were fortified individually, resulting in a fortification level of 0.10 mg/kg of bixafen and of 0.15 mg/kg of its metabolite bixafen-desmethyl as parent equivalents. After fortification, the solvent was allowed to evaporate.

Samples were analysed using method 01012 for bixafen and bixafen-desmethyl. Procedural recoveries for all analytes were demonstrated being within acceptable ranges (70–120%, RSD <20%).

Table 11 Storage stability data for bixafen and bixafen-desmethyl in plant matrices (Schoening, R., 2015, BIXAFEN_097)

Matrix	Storage period (days)	Bixafen% remaining (mean), 0.1 mg/kg	Bixafen concurrent recovery, 0.1 mg/kg[1]	Bixafen-desmethyl % remaining (mean), 0.15 mg/kg	Bixafen-desmethyl concurrent recovery, 0.1 mg/kg [a]
Orange, fruit	0	96, 109, 106, 110, 107 (106)	96, 109, 106, 110, 107 (106)	110, 108 (109)	97, 99, 103, 100, 99 (100)
	31	101, 105, 106 (104)	101, 105, 106 (104)	101, 107 (104)	87, 90, 80 (86)
	60	101, 75, 93 (90)	101, 75, 93 (90)	101, 92 (97)	93, 97, 79 (90)
	90	99, 105, 103 (102)	99, 105, 103 (102)	101, 103 (102)	91, 91, 93 (92)
	189	110, 107, 110 (109)	110, 107, 110 (109)	100, 106 (103)	91, 89, 95 (91)

Matrix	Storage period (days)	Bixafen% remaining (mean), 0.1 mg/kg	Bixafen concurrent recovery, 0.1 mg/kg[1]	Bixafen-desmethyl % remaining (mean), 0.15 mg/kg	Bixafen-desmethyl concurrent recovery, 0.1 mg/kg [a]
	360	101, 100, 103 (101)	101, 100, 103 (101)	97, 96 (97)	89, 94, 90 (91)
	551	113, 110, 108 (110)	113, 110, 108 (110)	99, 104 (102)	95, 92, 88 (92)
	719	90, 96, 97 (94)	90, 96, 97 (94)	96, 95 (96)	90, 92, 89 (90)
Beans, dry seed	0	105, 109, 109, 103, 106 106)	105, 109, 109, 103, 106 (106)	104, 104 (104)	92, 95, 95, 94, 99 (95)
	31	97, 101, 102 (100)	97, 101, 102 (100)	97, 106 (102)	94, 99, 97 (97)
	60	110, 110, 110 (110)	110, 110, 110 (110)	103, 104 (104)	95, 92, 97 (94)
	90	98, 107, 100 (102)	98, 107, 100 (102)	101, 100 (101)	94, 103, 101 (99)
	189	100, 101, 98 (100)	100, 101, 98 (100)	105, 106 (106)	95, 90, 98 (94)
	360	96, 95, 100 (97)	96, 95, 100 (97)	103, 101 (102)	90, 89, 87 (89)
	551	94, 101, 100 (98)	94, 101, 100 (98)	93, 99 (96)	93, 92, 94 (93)
	719	96, 96, 97 (96)	96, 96, 97 (96)	98, 101 (100)	87, 91, 87 (88)

[a]: Day zero concurrent recovery samples were fortified with 0.01 mg/kg instead of 0.1 mg/kg.

Soil

The storage stability of bixafen and bixafen-desmethyl in soil was already evaluated by the 2013 JMPR. The current Meeting received additional storage stability data for bixafen-desmethyl-pyrazole-4-carboxylic acid (M44/45) in two soils (Koch V., 2018, BIXAFEN_103).

The study was performed using the German soil "Höfchen" (silt loam, pH in water: 7.4, organic matter 1.58%) and soil "Dollendorf" (clay loam, pH in water 7.4, organic matter 8.6%). Actual storage intervals were 95, 179, 270, 363, 545, and 726 days. Untreated soil samples of 5 g each were weighed into 100 mL PP multi-purpose vessel with screw caps. The reference item was dissolved in acetonitrile to obtain fortification standard solutions with concentrations of about 1 mg/L. For the preparation of the storage stability samples 0.25 mL of the 1 mg/L fortification standard solution was diluted with acetonitrile/water (4+1, v/v) and was added to each of the corresponding sets of soil samples. The resulting concentration in all soil samples was approximately 0.05 mg/kg. The fortified samples were stored at -18 °C or below until analysis.

The residues of bixafen-desmethyl-pyrazole-4-carboxylic acid (M44/45) were determined according to the method 00952/M002 with a LOQ of 0.005 mg/kg for bixafen-desmethyl-pyrazole-4-carboxylic acid in soil (see section on analytical methods).

Table 12 Storage stability data for bixafen-desmethyl-pyrazole-4-carboxylic acid (M44/45) in soil (Koch V., 2016, BIXAFEN_103)

Soil	Storage period (days)	M44/45 % remaining (mean) 0.05 mg/kg fortification	M44/45 concurrent recovery 0.05 mg/kg fortification
Höfchen	0	96, 100, 97, 94 (97)	91, 92, 95, 90 (92)
	95	83, 81, 83, 86 (83)	92, 93, 89, 90 (91)
	179	79, 86, 90, 83 (85)	90, 92, 92, 99 (93)
	270	76, 71, 76, 80 (76)	88, 87, 85, 88 (87)
	363	70, 71, 71, 70 (71)	97, 99, 99, 100 (99)
	545	76, 73, 76, 77 (76)	96, 102, 105, 108 (103)
	726	63, 64, 65, 74 (67)	97, 101, 96, 96 (98)
Dollendorf	0	89, 105, 88, 104 (97)	93, 95, 91, 95 (94)
	95	86, 86, 91, 89 (88)	90, 96, 93, 96 (94)
	179	98, 99, 91, 107 (99)	109, 108, 108, 103 (107)
	270	79, 74, 74, 84 (78)	88, 86, 95, 85 (89)
	363	82, 77, 77, 80 (79)	92, 96, 92, 98 (95)

Soil	Storage period (days)	M44/45 % remaining (mean) 0.05 mg/kg fortification	M44/45 concurrent recovery 0.05 mg/kg fortification
	545	78, 81, 75, 88 (81)	104, 107, 104, 105 (105)
	726	89, 81, 89, 97 (89)	115, 114, 114, 108 (113)

USE PATTERN

Bixafen is registered in many countries for the control of fungal diseases. In the following table additional GAP information on all crops/crop groups supported with residue data are summarized.

Table 13 List of uses of bixafen

Crop or crop group	Country	Rate	Number of treatments (minimum interval)	Pre-harvest interval (PHI)
Pulses				
Soya bean	Brazil	0.063 kg ai/ha	2 (14 days)	30 days
Soya bean	USA	0.077 kg ai/ha	2 (10 days)	21 days (no feed of hay or threshings or grazing allowed)
Root and tuber crops				
Potato	USA	0.08 kg ai/ha	3 (10 days)	14 days
Cereal grains				
Barley	Brazil	0.063 kg ai/ha	4 (15 days)	30 days
Barley	USA	0.075 kg ai/ha	2 (14 days)	Forage: 10 days Hay: 20 days Stover and grain: 30 days
Maize (including sweet corn)	Brazil	0.063 kg ai/ha	2 (15 days)	15 days
Maize (incl. popcorn)	USA	0.077 kg ai/ha	2 (7 days), not after R4 stage (early dough stage = BBCH 83-85)	Forage: 10 days Stover and grain: 30 days
Oats	USA	0.075 kg ai/ha	2 (14 days)	Forage: 10 days Hay: 20 days Stover and grain: 30 days
Sorghum	USA	0.074 kg ai/ha	2 (growth stage dependent)	30 days
Wheat	Brazil	0.063 kg ai/ha	4 (15 days)	30 days
Wheat and triticale	USA	0.075 kg ai/ha	2 (14 days)	Forage: 10 days Hay: 20 days Stover and grain: 30 days
Oilseeds				
Cotton	Brazil	0.063 kg ai/ha	4 (15 days)	30 days
Peanuts	USA	0.10 kg ai/ha	2 (14 days)	14 days (no feed of hay or grazing allowed)
Sunflower	Brazil	0.063 kg ai/ha	2 (15 days)	30 days

Note: For all labels from the USA, rotational crop restrictions of 0 days for labelled crops, 30 days for leafy crops and 365 days for unlabelled crops were established.

RESULTS OF SUPERVISED RESIDUE TRIALS ON CROPS

Residue levels were reported as measured. Application rates were always reported as bixafen 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. Growth stages are reported according to the BBCH scale (https://www.julius-kuehn.de/media/Veroeffentlichungen/bbch%20epaper%20en/bbch%20skala%20englisch.html).

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.

Bixafen - supervised residue trials

Commodity	Indoor/Outdoor	Treatment	Countries	Table
Soya beans	Outdoor	Foliar	USA	Table 14
Potatoes	Outdoor	Foliar	USA	Table 15
Barley grain	Outdoor	Foliar	Brazil, USA	Table 16
Maize grain	Outdoor	Foliar	Brazil, USA	Table 17
Sweet corn	Outdoor	Foliar	Brazil	Table 18
Sorghum grain	Outdoor	Foliar	USA	Table 19
Wheat grain	Outdoor	Foliar	Brazil, USA	Table 20
Cotton seeds	Outdoor	Foliar	Brazil	Table 21
Peanut nutmeat	Outdoor	Foliar	USA	Table 22
Sunflower seeds	Outdoor	Foliar	Brazil	Table 23
Maize forage	Outdoor	Foliar	USA	Table 24
Maize fodder	Outdoor	Foliar	USA	Table 25
Sorghum forage	Outdoor	Foliar	USA	Table 26
Sorghum fodder	Outdoor	Foliar	USA	Table 27
Wheat forage	Outdoor	Foliar	USA	Table 28
Wheat hay and straw	Outdoor	Foliar	USA	Table 29
Peanut hay	Outdoor	Foliar	USA	Table 30

Soya beans

Residues of bixafen in soya beans cultivated in Northern America were investigated by Lenz C. (2016, BIXAFEN_091). Two foliar applications of a 125 g/L EC formulation were made at 108–122 g ai/ha at nominally 20 days before harvest. Two trials were conducted as decline trials with additional samples taken at nominally 10, 15, 25 and 30 days after the last application. In one trial an additional plot was established with two applications performed at an exaggerated rate of 574 and 556 g ai/ha to obtain samples used for processing of soya bean seeds into aspirated grain fraction (AGF), hulls, meal and refined oil. Applications were made to plots using spray volumes of 96–195 L/ha with ground equipment.

Soya bean (dry seeds) were analysed for bixafen and bixafen-desmethyl by residue analytical method 01012 with a LOQ of 0.01 mg/kg. Samples were also analysed for bixafen-desmethyl-pyrazole-4-carboxylic acid (M44/45) using residue analytical method 01366. For both methods, procedural recoveries for all analytes were demonstrated being within acceptable ranges (70–120%, RSD <20%).

Samples of soya bean seeds were stored deep-frozen for a maximum of 217 days (7 months) for bixafen and M21 and 387 days (12 months) for M44/45 prior to analysis. Processed commodities were stored deep-frozen for a maximum of 295 days (9 months).

Table 14 Residues of bixafen in soya beans.

Location, year (variety)	Application			Residues (mean), mg/kg					
Reference, Trial-ID	kg ai/ha	Interval in days	Max. growth stage	Sample	DALA	Bixafen	M21	Total	M44/45
USA (AL), Tallassee 2014 (Pioneer 95Y70) BIXAFEN_091, Trial A	0.11 0.11	-- 12	79 81	Seed, dry	20	0.01, 0.018 (0.014)	<0.01, <0.01 (<0.01)	0.02, 0.028 (0.024)	<0.01, <0.01 (<0.01)
USA (GA), Chula 2014 (Pioneer 95470) BIXAFEN_091, Trial B	0.11 0.11	-- 12	83 88	Seed, dry	9	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)	<0.01, <0.01 (<0.01)
					14	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)	<0.01, <0.01 (<0.01)
					20	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)	<0.01, <0.01 (<0.01)
					26	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)	<0.01, <0.01 (<0.01)
					28	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)	<0.01, <0.01 (<0.01)
USA (MO), Fisk 2014 (Pioneer P50T64R) BIXAFEN_091, Trial C	0.11 0.11	-- 12	81 87	Seed, dry	20	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)	<0.01, <0.01 (<0.01)
USA (AR), Stuttgart 2014 (MORSOY R248X02) BIXAFEN_091, Trial D	0.11 0.11	-- 12	81 86	Seed, dry	20	0.012, <0.01 (0.011)	<0.01, <0.01 (<0.01)	0.022, <0.02 (0.021)	<0.01, <0.01 (<0.01)
USA (AR), Winchester 2014 (Pioneer 45T11) BIXAFEN_091, Trial E	0.11 0.11	-- 12	79 85	Seed, dry	21	0.014, <0.01 (0.012)	<0.01, <0.01 (<0.01)	0.024, <0.02 (0.022)	<0.01, <0.01 (<0.01)
USA (IA), Kimballton 2014 (Asgrow AG2834) BIXAFEN_091, Trial F	0.11 0.11	-- 12	77 78	Seed, dry	20	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)	<0.01, <0.01 (<0.01)
USA (IA), Elk Horn 2014 (Asgrow AG2834) BIXAFEN_091, Trial G	0.11 0.11	-- 12	77 79	Seed, dry	9	0.036, 0.011 (0.024)	<0.01, <0.01 (<0.01)	0.046, 0.021 (0.034)	<0.01, <0.01 (<0.01)
					15	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)	<0.01, <0.01 (<0.01)

Location, year (variety) Reference, Trial-ID	Application kg ai/ha	Interval in days	Max. growth stage	Residues (mean), mg/kg Sample	DALA	Bixafen	M21	Total	M44/45
					20	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)	<0.01, <0.01 (<0.01)
					25	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)	<0.01, <0.01 (<0.01)
					30	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)	<0.01, <0.01 (<0.01)
USA (IA), Richland 2014 (92Y75) BIXAFEN_091, Trial H	0.11 0.11	-- 12	81 84	Seed, dry	18	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)	<0.01, <0.01 (<0.01)
USA (IL), Findlay 2014 (LG3989) BIXAFEN_091, Trial I	0.11 0.12	-- 12	79 79	Seed, dry	19	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)	<0.01, <0.01 (<0.01)
USA (IL), Stewardson 2014 (Pioneer 93Y84) BIXAFEN_091, Trial J	0.11 0.11	-- 12	79 81	Seed, dry	22	0.017, 0.012 (0.014)	<0.01, <0.01 (<0.01)	0.027, 0.022 (0.024)	<0.01, <0.01 (<0.01)
USA (KS), Stilwell 2014 (93Y72) BIXAFEN_091, Trial K	0.11 0.11	-- 12	79 82	Seed, dry	20	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)	<0.01, <0.01 (<0.01)
USA (KS), Lenexa 2014 (Willcross W1408) BIXAFEN_091, Trial L	0.12 0.11	-- 12	79 80	Seed, dry	22	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)	<0.01, <0.01 (<0.01)
USA (WI), Belleville 2014 (NK518-CS) BIXAFEN_091, Trial M	0.11 0.12	-- 11	79 79	Seed, dry	21	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)	<0.01, <0.01 (<0.01)
USA (NE), York 2014 (AG3034) BIXAFEN_091, Trial N	0.11 0.11	-- 12	78 79	Seed, dry	20	0.015, 0.043 (0.029)	<0.01, <0.01 (<0.01)	0.025, 0.053 (0.039)	<0.01, <0.01 (<0.01)
USA (ND), Northwood 2014 (NT0090RR) BIXAFEN_091, Trial O	0.11 0.11	-- 12	79 85	Seed, dry	21	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)	<0.01, <0.01 (<0.01)
USA (ND), Gardener 2014 (13R03 RR2Y) BIXAFEN_091, Trial P	0.11 0.12	-- 12	80 85	Seed, dry	19	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)	<0.01, <0.01 (<0.01)
USA (MN), St. Cloud 2014 (PB-0879NRR2) BIXAFEN_091, Trial Q	0.11 0.12	-- 12	79 91	Seed, dry	27	<0.01, <0.01, <0.01, <0.01 (<0.01)	<0.01, <0.01, <0.01, <0.01 (<0.01)	<0.02, <0.02, <0.02, <0.02 (<0.02)	<0.01, <0.01 (<0.01)
Canada (QC), St. Marc-sur-Richelieu 2014 (HS03RY33) BIXAFEN_091, Trial R	0.11 0.11	-- 11	79 84	Seed, dry	21	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)	<0.01, <0.01 (<0.01)

Location, year (variety) Reference, Trial-ID	Application			Residues (mean), mg/kg					
	kg ai/ha	Interval in days	Max. growth stage	Sample	DALA	Bixafen	M21	Total	M44/45
Canada (OR), Cambridge 2014 (Absolute RR Secan) BIXAFEN_091, Trial S	0.11 0.11	-- 11	79 84	Seed, dry	21	0.016, 0.032 (0.024)	<0.01, <0.01 (<0.01)	0.026, 0.042 (0.034)	<0.01, <0.01 (<0.01)
Canada (MB), Elm Creek 2014 (Libau) BIXAFEN_091, Trial T	0.11 0.11	-- 14	79 79	Seed, dry	20	0.020, <0.01 (0.015)	<0.01, <0.01 (<0.01)	0.030, <0.02 (0.025)	<0.01, <0.01 (<0.01)
Canada (MB), Morden 2014 (Libau) BIXAFEN_091, Trial U	0.11 0.11	-- 14	79 79	Seed, dry	21	0.011, <0.01 (0.01)	<0.01, <0.01 (<0.01)	0.021, <0.02 (0.02)	<0.01, <0.01 (<0.01)

DALA: days after last application

M21: bixafen-desmethyl, expressed as bixafen equivalents

Total: sum of bixafen and bixafen-desmethyl, expressed as bixafen equivalents

M44/45: bixafen-desmethyl-pyrazole-4-carboxylic acid

Potato

Residues of bixafen in potatoes cultivated in Northern America were investigated by Lenz C. (2017, BIXAFEN_092). Four foliar applications of a 125 g/L EC formulation were made at 53.7–62.2 g ai/ha at nominally 7 days before harvest. Two trials were conducted as decline trials with additional samples taken immediately before and after the last application and at nominally 3, 7, 10 and 14 days after the last application. In one trial an additional plot was established with four applications performed at an exaggerated rate in the range of 272–285 g ai/ha to obtain samples used for processing of potato into wet peel, flakes and chips. Applications were made to plots using spray volumes in the range of 94–197 L/ha with ground equipment.

Potato tubers were analysed for bixafen and bixafen-desmethyl by residue analytical method 01012 with a LOQ of 0.01 mg/kg. Procedural recoveries for all analytes were demonstrated being within acceptable ranges (70–120%, RSD <20%).

Samples of potato tubers were stored deep-frozen for a maximum of 253 days (8 months) prior to analysis.

Table 15 Residues of bixafen in potatoes

Location, year (variety) Reference, Trial-ID	Application			Residues (mean), mg eq/kg				
	kg ai/ha	Interval in days	Max. growth stage	Sample	DALA	Bixafen	M21	Total
USA (PA), Germansville 2014 (Dark Red Norland) BIXAFEN_092, Trial A	0.056 0.056 0.058 0.058	-- 7 8 7	61 69/41 44 47	Tuber	7	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)
USA (NJ), Baptistown 2014 (Dark Red Norland) BIXAFEN_092, Trial B	0.059 0.057 0.058 0.058	-- 7 8 7	40 42 45 46	Tuber	6	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)

Location, year (variety) Reference, Trial-ID	Application			Residues (mean), mg eq/kg				
	kg ai/ha	Interval in days	Max. growth stage	Sample	DALA	Bixafen	M21	Total
USA (NY), North Rose 2014 (Modoc) BIXAFEN_092, Trial C	0 056 0 056 0 056 0 056	-- 7 7 7	43 45 47 48	Tuber	7	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)
Canada (NB), Buctouche 2014 (Goldrush) BIXAFEN_092, Trial D	0.057 0.057 0.055 0.056	-- 7 7 7	407 408 408 409	Tuber	7	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)
Canada (NS), Woodville 2014 (Superior) BIXAFEN_092, Trial E	0.055 0.054 0.058 0.055	-- 6 7 7	406 406 408 408	Tuber	7	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)
USA (GA), Chula 2014 (Red Pontiac) BIXAFEN_092, Trial F	0.056 0.056 0.057 0.057	-- 7 7 7	44 45 47 48	Tuber	7	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)
USA (FL), Center Hill 2015 (Red Lasoda) BIXAFEN_092, Trial G	0.057 0.056 0.057 0.056	-- 7 7 7	43 45 47 47	Tuber	7	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)
USA (WI), Belleville 2014 (Superior) BIXAFEN_092, Trial H	0.057 0.056 0.062 0.055	-- 7 6 7	44 47 47 49	Tuber	-0	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)
					0	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)
					4	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)
					7	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)
					11	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)
					14	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)
USA (ND), Gardner 2014 (Red Pontiac) BIXAFEN_092, Trial I	0.057 0.060 0.058 0.058	-- 8 7 7	70 73 78 83	Tuber	7	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)
USA (IL), Stewardson 2014 (Kennebec) BIXAFEN_092, Trial J	0.057 0.056 0.056	-- 7 7	41 42 43	Tuber	6	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)

Location, year (variety) Reference, Trial-ID	Application			Residues (mean), mg eq/kg				
	kg ai/ha	Interval in days	Max. growth stage	Sample	DALA	Bixafen	M21	Total
	0.056	7	45					
Canada (MB), High Bluff 2014 (Russet Burbank) BIXAFEN_092, Trial K	0.058 0.059 0.057 0.058	-- 5 8 7	43 43 625 635	Tuber	8	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)
USA (UT), Lewiston 2014 (Ciklamen) BIXAFEN_092, Trial L	0.058 0.058 0.057 0.057	-- 6 6 7	48 48 48 48	Tuber	7	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)
USA (CA), Sanger 2014 (Yukon Gold) BIXAFEN_092, Trial M	0.056 0.056 0.056 0.056	-- 7 7 7	81 81 83 95	Tuber	7	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)
USA (ID), American Falls 2014 (Russet Burbank) BIXAFEN_092, Trial N	0.057 0.055 0.056 0.056	-- 7 7 7	46 47 48 48	Tuber	7	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)
USA (WA), Ephrata 2014 (Umatilla) BIXAFEN_092, Trial O Treatments: 22/07/14-12/08/14	0.057 0.056 0.057 0.056	-- 7 7 7	45 46 47 48	Tuber	-0	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)
					0	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)
					2	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)
					7	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)
					10	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)
					14	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)
USA (WA), Ephrata 2014 (Russet Burbank) BIXAFEN_092, Trial P Treatments: 07/08/14-28/08/14	0.057 0.057 0.057 0.056	-- 7 7 7	45 46 47 48	Tuber	7	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)
USA (WA), Yakima 2014 (Yukon Gold) BIXAFEN_092, Trial Q	0.056 0.056 0.056 0.055	-- 7 7 7	Small tubers	Tuber	7	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)

Location, year (variety) Reference, Trial-ID	Application			Residues (mean), mg eq/kg				
	kg ai/ha	Interval in days	Max. growth stage	Sample	DALA	Bixafen	M21	Total
Canada (BC), Okanagan Falls 2014 (Russet Burbank) BIXAFEN_092, Trial R	0.056 0.055 0.059 0.056	-- 9 7 7	81 43 43 45	Tuber	7	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)
Canada (BC), Oliver 2014 (Russet Burbank) BIXAFEN_092, Trial S	0.057 0.054 0.055 0.055	-- 7 8 7	89 91 93 48	Tuber	6	<0.01, 0.011 (0.01)	<0.01, <0.01 (<0.01)	<0.02, 0.021 (0.02)
Canada (AB), Josephburg 2014 (Russet Burbank) BIXAFEN_092, Trial T	0.056 0.056 0.057 0.056	-- 8 7 7	43 46 46 46	Tuber	7	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)

DALA: days after last application

M21: bixafen-desmethyl, expressed as bixafen equivalents

Total: sum of bixafen and bixafen-desmethyl, expressed as bixafen equivalents

Barley

Residues of bixafen in barley cultivated in Brazil were investigated in a set of studies (Resende G., 2016, BIXAFEN_104 & BIXAFEN_106 and Anonymous, 2013, BIXAFEN_105).

In total ten supervised residue trials were carried out on barley in Brazil during the 2012 and 2013 growing seasons. Four foliar applications of a 450 g/L EC formulation containing bixafen, prothioconazole and trifloxystrobin at 125 g/L, 175 g/L and 150 g/L, respectively, were made at 60–68 g bixafen/ha at nominally 30 days before harvest. All trials except two in 2012 were conducted as decline trials with additional samples taken at nominally 15, 20, 25 and 35 days after the last application. Applications were made to plots using a spray volume of 150 L/ha with ground equipment.

Barley grain was analysed for bixafen and bixafen-desmethyl by residue analytical method 01013 with a LOQ of 0.01 mg/kg. Procedural recoveries for all analytes were demonstrated, being within acceptable ranges (70–120%, RSD <20%).

Samples of barley seeds were stored deep-frozen for a maximum of 138 days (4.5 months) prior to analysis.

Table 16 Residues of bixafen in barley grain

Location, year (variety) Reference, Trial-ID	Application			Residues (mean), mg eq/kg				
	kg ai/ha	Interval in days	Max. growth stage	Sample	DALA	Bixafen	M21	Total
Brazil (Paraná), Ponta Grossa	0.066	--	60	Grain	25	0.66	<0.01	0.67
2012 (BRS Cave)	0.068	14	62		30	0.57	<0.01	0.58
BIXAFEN_104, BIXAFEN_105, F12-029-01	0.065	14	65		35	0.48	<0.01	0.49

Location, year (variety)	Application			Residues (mean), mg eq/kg				
Reference, Trial-ID	kg ai/ha	Interval in days	Max. growth stage	Sample	DALA	Bixafen	M21	Total
	0.067	14	65					
Brazil (Paraná), Londrina 2012 (BRS Cave) BIXAFEN_104, BIXAFEN_105, F12-029-02	0.065 0.065 0.068 0.065	-- 14 14 14	60 65 67 70	Grain	25 30 35	0.54 0.53 0.33	<0.01 <0.01 <0.01	0.55 0.54 0.34
Brazil (São Paulo), Paulínia 2012 (MN 743) BIXAFEN_104, BIXAFEN_105, F12-029-03	0.065 0.065 0.065 0.065	-- 14 14 14	51 57 65 71	Grain	25 30 35	0.56 0.77 0.72	0.01 0.01 0.01	0.57 0.78 0.73
Brazil (Paraná), Castro 2012 (BRS Cave) BIXAFEN_104, BIXAFEN_105, F12-029-04	0.064 0.068 0.060 0.064	-- 14 14 14	55 58 60 65	Grain	30	0.11	<0.01	0.12
Brazil (São Paulo), Capão Bonito 2012 (MN 743) BIXAFEN_104, BIXAFEN_105, F12-029-05	0.063 0.061 0.064 0.064	-- 14 14 13	51 59 65 73	Grain	29	0.30	0.02	0.32
Brazil (São Paulo), Paulínia 2013 (BRS Caue) BIXAFEN_106, F13-005-01	0.063 0.063 0.063 0.063	-- 14 14 14	51 57 71 73	Grain	15 20 25 30 35	0.38 0.73 0.86 0.53 0.58	<0.01 <0.01 <0.01 <0.01 <0.01	0.39 0.74 0.87 0.54 0.59
Brazil (Paraná), Londrina 2013 (BRS Caue) BIXAFEN_106, F13-005-02	0.066 0.065 0.065 0.060	-- 14 14 14	51 55 60 70	Grain	15 20 25 30 35	0.17 0.21 0.12 0.15 0.10	<0.01 <0.01 <0.01 <0.01 <0.01	0.18 0.22 0.13 0.16 0.11
Brazil (Paraná), Ponta Grossa 2013 (BRS Caue) BIXAFEN_106, F13-005-03	0.065 0.067 0.067 0.064	-- 14 14 14	51 59 65 75	Grain	15 20 25 30 35	0.22 0.14 0.14 0.17 0.16	<0.01 <0.01 <0.01 <0.01 <0.01	0.23 0.15 0.15 0.18 0.17

Location, year (variety)	Application			Residues (mean), mg eq/kg				
Reference, Trial-ID	kg ai/ha	Interval in days	Max. growth stage	Sample	DALA	Bixafen	M21	Total
Brazil (Paraná), Castro	0.067	--	51	Grain	15	0.21	<0.01	0.22
2013 (BRS Caue)	0.065	14	60		20	0.16	<0.01	0.17
BIXAFEN_106, F13-005-04	0.063	14	70		25	0.14	<0.01	0.15
	0.068	14	75		30	0.14	<0.01	0.15
					35	0.21	0.01	0.22
Brazil (São Paulo), Capão Bonito	0.063	--	51	Grain	15	0.35	0.02	0.37
2013 (BRS Caue)	0.063	14	57		18	0.51	0.03	0.54
BIXAFEN_106, F13-005-05	0.063	14	71		25	0.31	0.02	0.33
	0.063	14	73		30	0.30	0.02	0.32
					35	0.32	0.02	0.34

DALA: days after last application

M21: bixafen-desmethyl, expressed as bixafen equivalents

Total: sum of bixafen and bixafen-desmethyl, expressed as bixafen equivalents

Maize

Residues of bixafen in maize cultivated in Northern America were investigated by Lenz C. (2016, BIXAFEN_092).

In total sixteen supervised residue trials were carried out on maize in the USA during the 2014 growing season. Two foliar applications of a 125 g/L EC formulation were made at 107–118 g ai/ha at nominally 30 days before harvest of maize grain and the feed commodity stover. Two trials were conducted as decline trials with additional samples were taken at nominally 20, 25, 35 and 40 days after the last application. In one trial an additional plot was established with two applications performed at an exaggerated rate of 558 and 562 g ai/ha to obtain samples used for processing of maize grain into starch, meal/flour, grits and refined oil and the byproducts aspirated grain fraction (AGF) and germ. Applications were made to plots using spray volumes of 62–114 L/ha with ground equipment.

Samples were analysed for bixafen and bixafen-desmethyl by residue analytical method 01012 with a LOQ of 0.01 mg/kg. Procedural recoveries for all analytes were demonstrated being within acceptable ranges (70–120%, RSD <20%).

Samples were stored deep-frozen for a maximum of 136 days (4.5 months) prior to analysis.

Table 17 Residues of bixafen in maize grain

Location, year (variety)	Application			Residues (mean), mg eq/kg				
Reference, Trial-ID	kg ai/ha	Interval in days	Max. growth stage	Sample	DALA	Bixafen	M21	Total
USA (PA), Germansville 2014 (TA264-13VPR1B) BIXAFEN_092, 2014_RES-BAN1259-A	0.12 0.11	-- 21	77 87	Grain	29	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)
USA (AL), Tallassee 2014 (1690YHR) BIXAFEN_092, 2014_RES-BAN1259-B	0.11 0.11	-- 20	79 85	Grain	30	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)
USA (IA), Elk Horn 2014 (Mycogen 2V709a) BIXAFEN_092, 2014_RES-BAN1259-C	0.11 0.12	-- 20	85 87	Grain	20	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)
					25	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)
					30	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)
					35	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)
					40	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)
USA (IL), Stewardson 2014 (Burrus 6F74AMX) BIXAFEN_092, 2014_RES-BAN1259-D	0.11 0.11	-- 20	85 87	Grain	27	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)
	0.56 0.56	-- 20	85 87	Grain	27	0.0122 <0.01 <0.01 (0.0104)	<0.01, <0.01, <0.01 (<0.01)	0.0222, <0.02, <0.02 (0.0204)

Location, year (variety)	Application			Residues (mean), mg eq/kg				
Reference, Trial-ID	kg ai/ha	Interval in days	Max. growth stage	Sample	DALA	Bixafen	M21	Total
USA (KS), Stilwell 2014 (DKC62-08RIB) BIXAFEN_092, 2014_RES-BAN1259-E	0.11 0.11	-- 20	85 85	Grain	30	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)
USA (KS), Gardener 2014 (Producers Hybrid 7224VT3PRIB) BIXAFEN_092, 2014_RES-BAN1259-F	0.11 0.11	-- 19	85 87	Grain	29	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)
USA (IA), Richland 2014 (P1498 AM) BIXAFEN_092, 2014_RES-BAN1259-G	0.11 0.11	-- 20	85 86	Grain	19	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)
					24	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)
					31	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)
					33	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)
					40	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)
USA (WI), Oregon 2014 (DKC 49-29-RIB) BIXAFEN_092, 2014_RES-BAN1259-H	0.11 0.11	-- 20	83 86	Grain	29	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)
USA (MN), York 2014 (P0876CHR) BIXAFEN_092, 2014_RES-BAN1259-I	0.11 0.11	-- 20	73 85	Grain	30	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)
USA (MN), St. Cloud	0.11	--	72	Grain	29	<0.01, <0.01	<0.01, <0.01	<0.02, <0.02

Location, year (variety)	Application			Residues (mean), mg eq/kg				
Reference, Trial-ID	kg ai/ha	Interval in days	Max. growth stage	Sample	DALA	Bixafen	M21	Total
2014 (DKC 38-03-RIB) BIXAFEN_092, 2014_RES-BAN1259-J	0.11	21	83			(<0.01)	(<0.01)	(<0.02)
USA (KS),Stafford 2014 (P1105 AM) BIXAFEN_092, 2014_RES-BAN1259-K	0.11 0.11	-- 20	75 85	Grain	29	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)
Canada (QC), St. Marc-sur-Richelieu 2014 (829RA) BIXAFEN_092, 2014_RES-BAN1259-L	0.11 0.11	-- 19	79 85	Grain	30	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)
Canada (ON), Cambridge 2014 (3515 Maizex DBR) BIXAFEN_092, 2014_RES-BAN1259-M	0.11 0.11	-- 20	85 85	Grain	30	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)
Canada (MB), High Bluff 2014 (Dekalb RR) BIXAFEN_092, 2014_RES-BAN1259-N	0.11 0.12	-- 36	35 85	Grain	32	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)
USA (TX),Uvalde 2014 (DKC 69-43) BIXAFEN_092, 2014_RES-BAN1259-O	0.11 0.12	-- 21	71 87	Grain	28	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)
Canada (BC), Okanagan Falls 2014 (HLR219) BIXAFEN_092, 2014_RES-BAN1259-P Note: growth stage suggest sweet corn stage at sampling, plot not considered due to unclear matrix	0.11 0.11	-- 26	36 65	Grain	29	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)

DALA: days after last application

M21: bixafen-desmethyl, expressed as bixafen equivalents

Total: sum of bixafen and bixafen-desmethyl, expressed as bixafen equivalents

Sweet corn

Residues of bixafen in sweet corn cultivated in Brazil were investigated in a set of studies (Resende G., 2016, BIXAFEN_107 & Anonymous, 2016, BIXAFEN_108 and Resende G., 2016, BIXAFEN_109 & Anonymous, 2016, BIXAFEN_110).

In total ten supervised residue trials were carried out on maize in Brazil during the 2012 and 2013 growing seasons. Two foliar applications of a 450 g/L EC formulation containing bixafen, prothioconazole and trifloxystrobin at 125 g/L, 175 g/L and 150 g/L, respectively, were made at 0.059–0.068 kg bixafen/ha at nominally 15 days before harvest. All trials except two in 2012 were conducted as decline trials with additional samples taken at 10 and 20 days after the last application for 2012 trials and immediately after application and at 3, 7 and 21 days after the last application for 2013 trials. Applications were made to plots using a spray volume of 200 L/ha with ground equipment.

Samples were analysed for bixafen and bixafen-desmethyl by residue analytical method 01013 with a LOQ of 0.01 mg/kg. Procedural recoveries for all analytes were demonstrated, being within acceptable ranges (70–120%, RSD <20%).

Samples were stored deep-frozen for a maximum of 136 days (4.5 months) prior to analysis.

Table 18 Residues of bixafen in sweet corn

Location, year (variety)	Application			Residues (mean), mg eq/kg				
Reference, Trial-ID	kg ai/ha	Interval in days	Max. growth stage	Sample	DALA	Bixafen	M21	Total
Brazil								
Brazil (Paraná), Castro 2012 (DKB 350) BIXAFEN_107, BIXAFEN_108, F12-011-01	0.059 0.067	-- 14	65 71	Cob w/o husk	10 14 19	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.02 <0.02 <0.02
Brazil (São Paulo), Riberao Preto 2012 (Pioneer 30F53) BIXAFEN_107, BIXAFEN_108, F12-011-02	0.066 0.065	-- 14	61 71	Cob w/o husk	10 14 20	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.02 <0.02 <0.02
Brazil (Minas Gerais), Uberlandia 2012 (Maximus TL) BIXAFEN_107, BIXAFEN_108, F12-011-03	0.065 0.068	-- 14	61 69	Cob w/o husk	10 15 20	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.02 <0.02 <0.02
Brazil (São Paulo), Paulínia 2012 (P30F35)	0.063 0.067	-- 14	55 67	Cob w/o husk	14	<0.01	<0.01	<0.02

Location, year (variety)	Application			Residues (mean), mg eq/kg				
Reference, Trial-ID	kg ai/ha	Interval in days	Max. growth stage	Sample	DALA	Bixafen	M21	Total
BIXAFEN_107, BIXAFEN_108, F12-011-04								
Brazil (Paraná), Ponta Grossa 2012 (DKB 350) BIXAFEN_107, BIXAFEN_108, F12-011-05	0.068 0.062	-- 14	60 65	Cob w/o husk	14	<0.01	<0.01	<0.02
Brazil (São Paulo), Paulínia 2013 (P30F35HR) BIXAFEN_109, BIXAFEN_110, F13-007-01	0.064 0.063	-- 14	63 71	Cob w/o husk	0 3 7 15 21	<0.01 <0.01 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01 <0.01	<0.02 <0.02 <0.02 <0.02 <0.02
Brazil (Paraná), Castro 2013 (DKB 350) BIXAFEN_109, BIXAFEN_110, F13-007-02	0.065 0.060	-- 14	65 71	Cob w/o husk	0 3 7 15 21	<0.01 <0.01 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01 <0.01	<0.02 <0.02 <0.02 <0.02 <0.02
Brazil (Minas Gerais), Uberlandia 2013 (AG7088 PRO) BIXAFEN_109, BIXAFEN_110, F13-007-03	0.061 0.063	-- 14	67 71	Cob w/o husk	0 3 7 15 21	<0.01 <0.01 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01 <0.01	<0.02 <0.02 <0.02 <0.02 <0.02
Brazil (São Paulo), Ituverava 2013 (AG7088 PRO) BIXAFEN_109, BIXAFEN_110, F13-007-04	0.061 0.063	-- 14	67 71	Cob w/o husk	0 3 7 15 21	<0.01 <0.01 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01 <0.01	<0.02 <0.02 <0.02 <0.02 <0.02
Brazil (São Paulo), Riberao Preto 2013 (Bandeirante) BIXAFEN_109, BIXAFEN_110, F13-007-05	0.064 0.064	-- 14	55 71	Cob w/o husk	0 3 7 15 21	<0.01 <0.01 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01 <0.01	<0.02 <0.02 <0.02 <0.02 <0.02

DALA: days after last application

M21: bixafen-desmethyl, expressed as bixafen equivalents

Total: sum of bixafen and bixafen-desmethyl, expressed as bixafen equivalents

Sorghum

Residues of bixafen in sorghum cultivated in the USA were investigated by Lenz C. (2016, BIXAFEN_094).

In total nine supervised residue trials were carried out on sorghum in the USA during the 2014 growing season. Two foliar applications of a 125 g/L EC formulation were made at 112–116 g ai/ha at nominally 30 days before harvest of sorghum grain and the feed commodity stover. One trial was conducted as a decline trial with additional samples taken at 20, 25, 35 and 40 days after the last application. Two additional plots were established with two applications performed at an exaggerated rate in the range of 561 and 563 g ai/ha to obtain samples used for processing of syrup from stalks or the by-products aspirated grain fraction (AGF) from grain. Applications were made to plots using spray volumes of 60–86 L/ha with ground equipment.

Samples were analysed for bixafen and bixafen-desmethyl by residue analytical method 01012 with a LOQ of 0.01 mg/kg. Procedural recoveries for all analytes were demonstrated, being within acceptable ranges (70–120%, RSD <20%).

Samples were stored deep-frozen prior to analysis for a maximum of 347 days (11 months) for grain.

Table 19 Residues of bixafen in sorghum grain

Location, year (variety)	Application			Residues (mean), mg eq/kg				
Reference, Trial-ID	kg ai/ha	Interval in days	Max. growth stage	Sample	DALA	Bixafen	M21	Total
USA (MO), Fisk 2014 (Mycogen M3838 C) BIXAFEN_94, 2014-RES-BAN1260-A	0.11 0.11	-- 20	61 85	Grain	30	0.12, 0.14 (0.13)	0.044, 0.047 (0.046)	0.16, 0.19 (0.17)
USA (IA), Atlantic 2014 (Long-Tail Delight) BIXAFEN_94, 2014-RES-BAN1260-B	0.12 0.11	-- 21	23 55	Grain	20	0.044, 0.042 (0.043)	0.097, 0.092 (0.094)	0.14, 0.13 (0.14)
					25	0.032, 0.031 (0.032)	0.097, 0.089 (0.093)	0.13, 0.12 (0.12)
					30	0.012, 0.018 (0.015)	0.041, 0.044 (0.042)	0.053, 0.062 (0.058)
					35	0.020, 0.017 (0.018)	0.058, 0.059 (0.058)	0.078, 0.076 (0.077)

Location, year (variety) Reference, Trial-ID	Application kg ai/ha	Interval in days	Max. growth stage	Residues (mean), mg eq/kg Sample	DALA	Bixafen	M21	Total
					40	0.012, 0.017 (0.014)	0.033, 0.045 (0.039)	0.045, 0.062 (0.054)
USA (KS), Lenexa 2014 (K73-J6) BIXAFEN_94, 2014-RES-BAN1260-C	0.11 0.12	-- 19	83 87	Grain	29	0.82, 0.76 (0.79)	0.12, 0.11 (0.11)	0.94, 0.87 (0.90)
USA (NE), York 2014 (VARA 1005964) BIXAFEN_94, 2014-RES-BAN1260-D	0.11 0.11	-- 21	75 83	Grain	32	0.12, 0.11 (0.11)	0.042, 0.032 (0.037)	0.16, 0.14 (0.15)
USA (TX), East Bernard 2014 (DKS53-67) BIXAFEN_94, 2014-RES-BAN1260-E	0.11 0.11	-- 15	57 83	Grain	28	2.0, 1.6 (1.8)	0.11, 0.084 (0.097)	2.1, 1.6 (1.9)
USA (TX), Uvalde 2014 (Pioneer 83G19) BIXAFEN_94, 2014-RES-BAN1260-F	0.12 0.11	-- 22	51 87	Grain	28	0.16, 0.12 (0.14)	0.070, 0.059 (0.064)	0.23, 0.18 (0.20)
USA (ND), Carrington 2014 (LM 5009) BIXAFEN_94, 2014-RES-BAN1260-G	0.11 0.11	-- 21	69 71	Grain	27	0.26, 0.24 (0.25)	0.099, 0.10 (0.1)	0.36, 0.34 (0.35)
USA (TX), Groom 2014 (Y373) BIXAFEN_94, 2014-RES-BAN1260-H	0.11 0.11	-- 20	71 87	Grain	30	0.43, 0.52 (0.48)	<0.01, <0.01 (<0.01)	0.44, 0.53 (0.49)
USA (TX), Wall 2014 (DKS49-45) BIXAFEN_94, 2014-RES-BAN1260-I	0.11 0.11	-- 20	69 87	Grain	29	0.25, 0.25 (0.25)	0.035, 0.034 (0.034)	0.28, 0.28 (0.28)

DALA: days after last application

M21: bixafen-desmethyl, expressed as bixafen equivalents

Total: sum of bixafen and bixafen-desmethyl, expressed as bixafen equivalents

Wheat

Residues of bixafen in wheat cultivated in Brazil were investigated in a set of studies (Resende G., 2016, BIXAFEN_111, Anonymous, 2013, BIXAFEN_112 and Resende G., 2016, BIXAFEN_113).

In total ten supervised residue trials were carried out on wheat in Brazil during the 2012 and 2013 growing seasons. Four foliar applications of a 450 g/L EC formulation containing bixafen, prothioconazole and trifloxystrobin at 125 g/L, 175 g/L and 150 g/L, respectively, were made at intervals of 14 days and rates in the range of 60–69 g bixafen/ha at nominally 30 days before harvest. All trials except two in 2012 were conducted as decline trials with additional samples taken at 25 and 35 days after the last application for 2012 trials and at 15, 20, 25 and 35 days after the last application for 2013 trials. Applications were made to plots using a spray volume of 200 L/ha with ground equipment.

Samples were analysed for bixafen and bixafen-desmethyl by residue analytical method 01013 with a LOQ of 0.01 mg/kg. Procedural recoveries for all analytes were demonstrated, being within acceptable ranges (70–120%, RSD <20%).

Samples were stored deep-frozen for a maximum of 85 days (3 months) prior to analysis.

Residues of bixafen in wheat cultivated in Northern America were investigated by Lenz C. (2016, BIXAFEN_095).

In total twenty-six supervised residue trials were carried out on wheat in North America (10 in Canada and 16 in the USA) during the 2014/15 growing season. Two foliar applications of a 125 g/L EC formulation were made at 109–119 g ai/ha at nominally 30 days before harvest. Two trials were conducted as decline trials with additional samples were taken at nominally 25, 35, 40 and 45 days after the last application. In one trial an additional plot was established with two applications performed at an exaggerated rate of 553 and 574 g ai/ha to obtain samples used for processing of wheat grain into flour and the by-products aspirated grain fraction (AGF), bran, middlings, shorts and germ. Applications were made to plots using spray volumes of 60–114 L/ha with ground equipment.

Samples were analysed for bixafen and bixafen-desmethyl by residue analytical method 01012 with a LOQ of 0.01 mg/kg. Procedural recoveries for all analytes were demonstrated, being within acceptable ranges (70–120%, RSD <20%).

Samples were stored deep-frozen prior to analysis for a maximum of 151 days (5 months) for grain.

Table 20 Residues of bixafen in wheat grain

Location, year (variety)	Application			Residues (mean), mg eq/kg				
Reference, Trial-ID	kg ai/ha	Interval in days	Max. growth stage	Sample	DALA	Bixafen	M21	Total
Brazil								
Brazil (Paraná), Ponta Grossa 2012 (Supera) BIXAFEN_111, BIXAFEN_112, F12-028-01	0.063 0.069 0.061 0.064	-- 14 14 14	60 62 65 68	Grain	25 30 35	0.12 0.09 0.10	<0.01 <0.01 <0.01	0.13 0.10 0.11
Brazil (Paraná), Castro 2012 (Supera) BIXAFEN_111, BIXAFEN_112, F12-028-02	0.063 0.063 0.060 0.063	-- 14 14 14	51 55 60 65	Grain	25 30 35	0.02 0.01 0.02	<0.01 <0.01 <0.01	0.03 0.02 0.03
Brazil (São Paulo), Paulínia 2012 (CD 104)	0.064 0.063	-- 14	51 57	Grain	25 30	0.04 0.12	<0.01 <0.01	0.05 0.13

Location, year (variety) Reference, Trial-ID	Application kg ai/ha	Interval in days	Max. growth stage	Residues (mean), mg eq/kg Sample	DALA	Bixafen	M21	Total
BIXAFEN_111, BIXAFEN_112, F12-028-03	0.065 0.063	14 14	65 71		35	0.05	<0.01	0.06
Brazil (Paraná), Londrina 2012 (CD 104) BIXAFEN_111, BIXAFEN_112, F12-028-04	0.063 0.067 0.062 0.065	-- 14 14 14	60 65 67 70	Grain	30	0.05	<0.01	0.06
Brazil (São Paulo), Capão Bonito 2012 (IAC 381) BIXAFEN_111, BIXAFEN_112, F12-028-05	0.060 0.068 0.067 0.065	-- 14 14 14	51 59 69 73	Grain	29	0.10	0.04	0.14
Brazil (São Paulo), Paulínia 2013 (IAC 375) BIXAFEN_113, F13-009-01	0.063 0.063 0.063 0.063	-- 14 14 14	54 61 71 73	Grain	15 20 25 30 35	0.08 0.09 0.10 0.12 0.17	<0.01 <0.01 <0.01 <0.01 <0.01	0.09 0.10 0.11 0.13 0.18
Brazil (Paraná), Londrina 2013 (Supera) BIXAFEN_113, F13-009-02	0.067 0.068 0.065 0.064	-- 14 14 14	51 55 60 70	Grain	15 20 25 30 35	0.11 0.15 0.08 0.07 0.06	<0.01 <0.01 <0.01 <0.01 <0.01	0.12 0.16 0.09 0.08 0.07
Brazil (Paraná), Ponta Grossa 2013 (Supera) BIXAFEN_113, F13-009-03	0.064 0.065 0.063 0.065	-- 14 14 14	51 59 65 75	Grain	15 20 25 30 35	0.07 0.09 0.05 0.06 0.03	<0.01 <0.01 <0.01 <0.01 <0.01	0.08 0.10 0.06 0.07 0.04
Brazil (Paraná), Castro 2013 (Supera) BIXAFEN_113, F13-009-04	0.065 0.067 0.060 0.061	-- 14 14 14	51 60 70 75	Grain	15 20 25 30 35	0.13 0.09 0.06 0.06 0.06	<0.01 <0.01 <0.01 <0.01 <0.01	0.14 0.10 0.07 0.07 0.07
Brazil (São Paulo), Capão Bonito 2013 (IAC 375) BIXAFEN_113, F13-009-05	0.063 0.063 0.063 0.063	-- 14 14 14	52 59 71 73	Grain	15 18 25 30 35	0.03 0.05 0.11 0.10 0.06	<0.01 <0.01 0.02 0.01 <0.01	0.04 0.06 0.13 0.11 0.07
Canada and USA								
USA (AL), Tallassee 2015 (Pioneer 26R41) BIXAFEN_095, 2014RES-BAN1261-A	0.11 0.11	-- 34	25 69	Grain	32	0.034, 0.050 (0.042)	<0.01, 0.012 (0.011)	0.044, 0.062 (0.053)
USA (MI), Fisk 2015 (Roane) BIXAFEN_095, 2014RES-BAN1261-B	0.11 0.11	-- 34	29 71	Grain	30	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)
USA (IL), Stewardson 2014 (Becks 129)	0.11 0.11	-- 34	33 77	Grain	30	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)

Location, year (variety) Reference, Trial-ID	Application			Residues (mean), mg eq/kg				
	kg ai/ha	Interval in days	Max. growth stage	Sample	DALA	Bixafen	M21	Total
BIXAFEN_095, 2014RES-BAN1261-C								
USA (KS), Gardener 2014 (Agromax 413) BIXAFEN_095, 2014RES-BAN1261-D	0.11 0.11	-- 32	29 75	Grain	30	0.013, 0.016 (0.014)	<0.01, <0.01 (<0.01)	0.023, 0.026 (0.024)
USA (ND), Northwood 2014 (Faller) BIXAFEN_095, 2014RES-BAN1261-E	0.11 0.11	-- 42	25 71	Grain	27	0.015, 0.015 (0.015)	<0.01, <0.01 (<0.01)	0.025, 0.025 (0.025)
					30	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)
					35	0.011, 0.013 (0.012)	<0.01, <0.01 (<0.01)	0.021, 0.023 (0.022)
					41	0.010, 0.011 (0.011)	<0.01, <0.01 (<0.01)	0.02, 0.021 (0.021)
					44	0.011, 0.013 (0.012)	<0.01, <0.01 (<0.01)	0.021, 0.023 (0.022)
USA (TX), Uvalde 2015 (Greer) BIXAFEN_095, 2014RES-BAN1261-F	0.11 0.11	-- 73	26 77	Grain	35	0.054, 0.044 (0.049)	0.017, 0.014 (0.015)	0.070, 0.058 (0.064)
USA (ND), Carrington 2015 (Propper) BIXAFEN_095, 2014RES-BAN1261-G	0.11 0.12	-- 28	25 73	Grain	33	0.020, 0.020 (0.020)	<0.01, <0.01 (<0.01)	0.030, 0.030 (0.030)
USA (ND), Jamestown 2014 (Prosper) BIXAFEN_095, 2014RES-BAN1261-H	0.11 0.12	-- 18	25 65	Grain	33	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)
USA (NB), Grand Island 2014 (Proper HRS) BIXAFEN_095, 2014RES-BAN1261-I	0.11 0.11	-- 31	25 61	Grain	32	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)
USA (NB), York 2015 (Husker Genetics Overland) BIXAFEN_095, 2014RES-BAN1261-I2	0.11 0.11	-- 56	25 75	Grain	33	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)
Canada (SK), Hanley 2014 (Cardale)	0.11 0.11	-- 50	31 83	Grain	31	0.081, 0.083 (0.082)	<0.01, <0.01 (<0.01)	0.092, 0.093 (0.092)

Location, year (variety) Reference, Trial-ID	Application			Residues (mean), mg eq/kg				
	kg ai/ha	Interval in days	Max. growth stage	Sample	DALA	Bixafen	M21	Total
BIXAFEN_095, 2014RES-BAN1261-J								
Canada (SK), Pike Lake 2014 (CDC Utmost) BIXAFEN_095, 2014RES-BAN1261-K	0.11 0.11	-- 46	25 75	Grain	34	0.072, 0.064 (0.068)	0.015, 0.014 (0.014)	0.087, 0.078 (0.082)
Canada (AB), Taber 2014 (Carberry) BIXAFEN_095, 2014RES-BAN1261-L	0.11 0.12	-- 35	30 75	Grain	35	0.067, 0.037 (0.052)	0.030, 0.016 (0.023)	0.097, 0.053 (0.075)
USA (KS), Lamed 2015 (LCS Wizard) BIXAFEN_095, 2014RES-BAN1261-M	0.11 0.11	-- 65	25 83	Grain	25	0.028, 0.027 (0.027)	<0.01, <0.01 (<0.01)	0.038, 0.037 (0.037)
					30	0.018, 0.026 (0.022)	<0.01, <0.01 (<0.01)	0.028, 0.036 (0.032)
					35	0.023, 0.029 (0.026)	<0.01, <0.01 (<0.01)	0.033, 0.039 (0.036)
					41	0.025, 0.028 (0.026)	<0.01, <0.01 (<0.01)	0.035, 0.038 (0.037)
					46	0.032, 0.026 (0.029)	<0.01, <0.01 (<0.01)	0.042, 0.036 (0.039)
USA (OK), Hinton 2014 (Duster) BIXAFEN_095, 2014RES-BAN1261-N	0.11 0.11	-- 32	25 77	Grain	34	0.087, 0.091 (0.089)	0.021, 0.022 (0.021)	0.11, 0.11 (0.11)
USA (TX), Claude 2015 (not reported) BIXAFEN_095, 2014RES-BAN1261-O	0.11 0.11	-- 48	27 69	Grain	30	0.011, 0.016 (0.014)	<0.01, <0.01 (<0.01)	0.021, 0.026 (0.023)
USA (TX), Wall 2015 (Coronado) BIXAFEN_095, 2014RES-BAN1261-P	0.11 0.11	-- 46	32 77	Grain	35	0.030, 0.016 (0.023)	<0.01, <0.01 (<0.01)	0.040, 0.026 (0.033)
USA (WS), Ephrata 2014 (Expresso) BIXAFEN_095, 2014RES-BAN1261-Q	0.11 0.11	-- 45	25 77	Grain	35	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)
Canada (MB), Brandon 2015 (not reported) BIXAFEN_095, 2014RES-BAN1261-R2	0.11 0.11	-- 78	24 87	Grain	30	0.10, 0.11 (0.11)	<0.01, <0.01 (<0.01)	0.11, 0.12 (0.12)

Location, year (variety) Reference, Trial-ID	Application			Residues (mean), mg eq/kg				
	kg ai/ha	Interval in days	Max. growth stage	Sample	DALA	Bixafen	M21	Total
Treatment: 04.05.15 & 21.07.15								
Canada (BC), Oliver 2014 (Carberry) BIXAFEN_095, 2014RES-BAN1261-S	0.11 0.11	-- 26	25 85	Grain	35	0.036, 0.031 (0.034)	<0.01, <0.01 (<0.01)	0.046, 0.041 (0.044)
Canada (BC), Crawston 2014 (Common #1) BIXAFEN_095, 2014RES-BAN1261-T	0.11 0.12	-- 31	29 85	Grain	30	0.062, 0.037 (0.045)	0.012, <0.01 (0.011)	0.074, 0.047 (0.061)
Canada (SK), Wakaw 2014 (Cardale) BIXAFEN_095, 2014RES-BAN1261-U	0.11 0.11	-- 41	31 83	Grain	34	0.026, 0.035 (0.030)	<0.01, <0.01 (<0.01)	0.036, 0.045 (0.040)
Canada (SK), Rosthem 2014 (Cardale) BIXAFEN_095, 2014RES-BAN1261-V	0.11 0.11	-- 39	31 77	Grain	34	0.035, 0.041 (0.038)	<0.01, <0.01 (<0.01)	0.045, 0.051 (0.048)
Canada (AB), Josephburg 2014 (Harvest) BIXAFEN_095, 2014RES-BAN1261-W	0.11 0.11	-- 40	31 75	Grain	35	<0.01, 0.045 (0.028)	<0.01, <0.01 (<0.01)	<0.02, 0.055 (0.038)
Canada (AB), Gibbons 2014 (Utmost) BIXAFEN_095, 2014RES-BAN1261-X	0.11 0.12	-- 41	30 75	Grain	32	0.034, 0.038 (0.036)	<0.01, <0.01 (<0.01)	0.044. 0.048 (0.046)
Canada (MB), Brandon 2014 (Glen) BIXAFEN_095, 2014RES-BAN1261-Y Treatment: 02.07.14 & 08.08.14	0.12 0.12	-- 37	22 75	Grain	25	0.034, 0.029 (0.031)	<0.01, <0.01 (<0.01)	0.044, 0.039 (0.041)

DALA: days after last application

M21: bixafen-desmethyl, expressed as bixafen equivalents

Total: sum of bixafen and bixafen-desmethyl, expressed as bixafen equivalents

Cotton

Residues of bixafen in cotton cultivated in Brazil were investigated in a set of studies (Resende G., 2016, BIXAFEN_114, Anonymous, 2013, BIXAFEN_115 and Resende G., 2016, BIXAFEN_116).

In total ten supervised residue trials were carried out on cotton in Brazil during the 2012 and 2013 growing seasons. Four foliar applications of a 450 g/L SC formulation containing bixafen, prothioconazole and trifloxystrobin at 125 g/L, 175 g/L and 150 g/L, respectively, were made at 60–66 g bixafen/ha at nominally 30 days before harvest. All trials except two in 2012 were conducted as decline trials with additional samples taken at nominally 25 and 35 days after the last application for the 2012 trials and at

nominally 15, 20, 25 and 35 days after the last application for the 2013 trials. Applications were made to plots using a spray volume of 200 L/ha with ground equipment.

Cotton seeds were analysed for bixafen and bixafen-desmethyl by residue analytical method 01013 with a LOQ of 0.01 mg/kg. Procedural recoveries for all analytes were demonstrated, being within acceptable ranges (70–120%, RSD <20%).

Samples of cotton seeds were stored deep-frozen for a maximum of 192 days (6 months) prior to analysis.

Table 21 Residues of bixafen in cotton seed

Location, year (variety)	Application			Residues (mean), mg eq/kg				
Reference, Trial-ID	kg ai/ha	Interval in days	Max. growth stage	Sample	DALA	Bixafen	M21	Total
Brazil (São Paulo), Paulínia 2012 (FM 966 LL) BIXAFEN_114, BIXAFEN_115, F12-010-01	0.060 0.063 0.063 0.066	-- 14 14 13	81 81 83 85	Seeds	25 30 35	0.05 0.06 0.06	<0.01 <0.01 <0.01	0.06 0.07 0.07
Brazil (São Paulo), Ribeirão Preto 2012 (FM 966 LL) BIXAFEN_114, BIXAFEN_115, F12-010-02	0.063 0.064 0.063 0.065	-- 14 14 14	77 79 81 81	Seeds	25 30 34	0.01 0.01 <0.01	<0.01 <0.01 <0.01	0.02 0.02 <0.02
Brazil (Minas Gerais), Uberlândia 2012 (FM 966 LL) BIXAFEN_114, BIXAFEN_115, F12-010-03	0.064 0.063 0.063 0.063	-- 14 14 14	77 79 81 83	Seeds	25 30 35	0.06 0.02 0.03	<0.01 <0.01 <0.01	0.07 0.03 0.04
Brazil (São Paulo), Jaboticabal 2012 (FM 966 LL) BIXAFEN_114, BIXAFEN_115, F12-010-04	0.063 0.063 0.065 0.063	-- 14 14 14	77 77 77 81	Seeds	30	<0.01	<0.01	<0.02
Brazil (São Paulo), Ituverava 2012 (NU OPAL RR) BIXAFEN_114, BIXAFEN_115, F12-010-05	0.064 0.063 0.063 0.062	-- 14 14 14	77 78 79 81	Seeds	30	0.06	<0.01	0.07
Brazil (São Paulo), Paulínia 2013 (FM 966 LL) BIXAFEN_116, F13-004-1	0.063 0.063 0.063 0.063	-- 14 14 14	75 75 79 83	Seeds	15 20 25 30 35	0.02 0.03 0.03 0.02 <0.01	<0.01 <0.01 <0.01 <0.01 <0.01	0.03 0.04 0.04 0.03 <0.02
Brazil (São Paulo), Ribeirão Preto 2013 (FB 966 LL) BIXAFEN_116, F13-004-02	0.064 0.066 0.063 0.065	-- 14 14 14	77 79 81 83	Seeds	15 19 25 30 35	0.06 <0.01 <0.01 0.01 <0.01	<0.01 <0.01 <0.01 <0.01 <0.01	0.07 <0.02 <0.02 0.02 <0.02
Brazil (Minas Gerais), Uberlândia 2013 (FM 966 LL) BIXAFEN_116, F13-004-03	0.062 0.062 0.063 0.064	-- 14 14 14	79 80 81 82	Seeds	15 19 25 30 35	0.10 0.09 0.08 0.11 0.16	<0.01 <0.01 <0.01 <0.01 <0.01	0.11 0.10 0.09 0.12 0.17

Location, year (variety) Reference, Trial-ID	Application			Residues (mean), mg eq/kg				
	kg ai/ha	Interval in days	Max. growth stage	Sample	DALA	Bixafen	M21	Total
Brazil (São Paulo), Cravinhos 2013 (FB 966 LL) BIXAFEN_116, F13-004-04	0.064 0.062 0.063 0.063	-- 14 14 14	77 77 79 79	Seeds	15 20 24 30 35	<0.01 <0.01 [a] <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01 <0.01	<0.02 <0.02 <0.02 <0.02 <0.02
Brazil (Goias), Trindade 2013 (FM 975 WS) BIXAFEN_116, F13-004-05	0.066 0.065 0.060 0.065	-- 14 14 13	77 78 80 81	Seeds	15 18 25 30 35	<0.01 <0.01 <0.01 0.02 0.01	<0.01 <0.01 <0.01 <0.01 <0.01	<0.02 <0.02 <0.02 0.03 0.02

DALA: days after last application

M21: bixafen-desmethyl, expressed as bixafen equivalents

Total: sum of bixafen and bixafen-desmethyl, expressed as bixafen equivalents

[a] Residues in control 0.01 mg/kg

Peanuts

Residues of bixafen in peanuts cultivated in the USA were investigated by Lenz C. (2016, BIXAFEN_096).

In total fifteen supervised residue trials were carried out on peanut in the USA during the 2014 growing season. Two of them, trials 2014RES-BAN1251-A & B were performed at the same location and at the same time and are therefore not considered independent. Four foliar applications of a 125 g/L EC formulation were made at 54–58 g ai/ha at nominally 14 days before normal commercial harvest of peanut nuts and the feed commodity hay. One trial was conducted as a decline trial with additional samples of nuts and hay taken immediately before and after the last application and at 6, 21 and 28 days after the last application. In one trial an additional plot was established with four applications performed at an exaggerated rate in the range of 285–286 g ai/ha to obtain samples used for processing of peanut refined oil and the by-product meal. Applications were made to plots using spray volumes of 117–188 L/ha with ground equipment.

Samples were analysed for bixafen and bixafen-desmethyl by residue analytical method 01012 with a LOQ of 0.01 mg/kg. Samples were stored deep-frozen prior to analysis for a maximum of 132 days (4 months) for peanut nutmeat. Nutmeat samples were also analysed for bixafen-desmethyl-pyrazole-4-carboxylic acid (M44/45) using residue analytical method 01366. Residues in all samples were below the LOQ of 0.01 mg/kg and not reported in the following table. For both methods, procedural recoveries for all analytes were demonstrated, being within acceptable ranges (70–120%, RSD <20%).

Table 22 Residues of bixafen in peanut nutmeat

Location, year (variety) Reference, Trial-ID	Application			Residues (mean), mg eq/kg				
	kg ai/ha	Interval in days	Max. growth stage	Sample	DALA	Bixafen	M21	Total
USA (AL), Tallassee 2014 (GA06)	0.057 0.057	-- 14	73 79	Nutmeat	14	<0.01, <0.01	<0.01, <0.01	<0.02, <0.02

Location, year (variety) Reference, Trial-ID	Application kg ai/ha	Interval in days	Max. growth stage	Residues (mean), mg eq/kg Sample	DALA	Bixafen	M21	Total
BIXAFEN_96, 2014RES-BAN1251-A	0.057 0.057	13 15	85 87			(<0.01)	(<0.01)	(<0.02)
USA (AL), Tallassee 2014 (GA06) BIXAFEN_96, 2014RES-BAN1251-B	0.056 0.057 0.057 0.057	-- 13 15 14	71 77 83 87	Nutmeat	14	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)
USA (AL), Auburn 2014 (GA06) BIXAFEN_96, 2014RES-BAN1251-C	0.057 0.057 0.057 0.057	-- 14 14 14	75 75 85 87	Nutmeat	15	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)
USA (GA), Chula 2014 (GA09B) BIXAFEN_96, 2014RES-BAN1251-D	0.056 0.057 0.056 0.058	-- 14 14 14	75 75 79 85	Nutmeat	-0	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)
					0	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)
					6	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)
					14	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)
					21	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)
					28	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)
USA (GA), Abbeville 2014 (GA06G) BIXAFEN_96, 2014RES-BAN1251-E	0.056 0.056 0.056 0.056	-- 14 14 14	73 77 79 83	Nutmeat	14	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)
USA (GA), Weston 2014 (GA06G)	0.055 0.055	-- 14	72 74	Nutmeat	14	<0.01, <0.01	<0.01, <0.01	<0.02, <0.02

Location, year (variety) Reference, Trial-ID	Application kg ai/ha	Interval in days	Max. growth stage	Residues (mean), mg eq/kg Sample	DALA	Bixafen	M21	Total
BIXAFEN_96, 2014RES-BAN1251-F	0.055 0.055	14 14	76 80			(<0.01)	(<0.01)	(<0.02)
USA (NC), Seven Springs 2014 (Bailey) BIXAFEN_96, 2014RES-BAN1251-G	0.058 0.055 0.056 0.057	-- 14 14 14	81 82 84 87	Nutmeat	14	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)
USA (NC), Albertson 2014 (Bailey) BIXAFEN_96, 2014RES-BAN1251-H	0.057 0.055 0.056 0.057	-- 14 14 14	72 80 83 86	Nutmeat	14	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)
USA (FL), Center Hill 2014 (GA06G) BIXAFEN_96, 2014RES-BAN1251-I	0.054 0.057 0.058 0.057	-- 14 15 13	35 39 81 85	Nutmeat	14	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)
USA (OK), Ringwood 2014 (Jupiter) BIXAFEN_96, 2014RES-BAN1251-J	0.057 0.056 0.056 0.056	-- 14 14 14	79 79 79 79	Nutmeat	13	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)
USA (AR), Pocahontas 2014 (Georgia 09B) BIXAFEN_96, 2014RES-BAN1251-K	0.055 0.056 0.056 0.056	-- 14 13 14	71 75 84 87	Nutmeat	14	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)
USA (IA), Richland 2014 (Bailey) BIXAFEN_96, 2014RES-BAN1251-L	0.056 0.056 0.056 0.056	-- 14 14 14	79 82 83 84	Nutmeat	12	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)
USA (TX), East Bernard 2014 (Georgia 09B) BIXAFEN_96, 2014RES-BAN1251-M	0.056 0.056 0.056 0.056	-- 14 14 14	69 73 77 79	Nutmeat	14	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)
USA (OK), Hinton 2014 (TamNut 0L06) BIXAFEN_96, 2014RES-BAN1251-N	0.056 0.056 0.056 0.056	-- 15 13 14	73 79 79 85	Nutmeat	16	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)

Location, year (variety) Reference, Trial-ID	Application			Residues (mean), mg eq/kg				
	kg ai/ha	Interval in days	Max. growth stage	Sample	DALA	Bixafen	M21	Total
USA (TX), Wellington 2014 (TAMRUN) BIXAFEN_96, 2014RES-BAN1251-O	0.057 0.058 0.056 0.057	-- 14 14 14	79 81 84 86	Nutmeat	14	<0.01, <0.01 (<0.01)	<0.01, <0.01 (<0.01)	<0.02, <0.02 (<0.02)

DALA: days after last application

M21: bixafen-desmethyl, expressed as bixafen equivalents

Total: sum of bixafen and bixafen-desmethyl, expressed as bixafen equivalents

Sunflower

Residues of bixafen in sunflowers cultivated in Brazil were investigated in a set of studies (Resende G., 2016, BIXAFEN_117, Anonymous, 2013, BIXAFEN_118 and Resende G., 2016, BIXAFEN_119).

In total ten supervised residue trials were carried out on sunflower in Brazil during the 2012 and 2013 growing seasons. Two foliar applications of a 450 g/L SC formulation containing bixafen, prothioconazole and trifloxystrobin at 125 g/L, 175 g/L and 150 g/L, respectively, were made at 60–68 g bixafen/ha at nominally 30 days before harvest. All trials except two in 2012 were conducted as decline trials with additional samples taken at nominally 25 and 35 days after the last application for the 2012 trials and at nominally 15, 20, 25 and 35 days after the last application for the 2013 trials. Applications were made to plots using a spray volume of 150 L/ha with ground equipment.

Samples were analysed for bixafen and bixafen-desmethyl by residue analytical method 01013 with a LOQ of 0.01 mg/kg. Procedural recoveries for all analytes were demonstrated, being within acceptable ranges (70–120%, RSD <20%).

Samples of sunflower seeds were stored deep-frozen for a maximum of 273 days (9 months) prior to analysis.

Table 23 Residues of bixafen in sunflower seed

Location, year (variety) Reference, Trial-ID	Application			Residues (mean), mg eq/kg				
	kg ai/ha	Interval in days	Max. growth stage	Sample	DALA	Bixafen	M21	Total
Brazil (São Paulo), Paulínia 2012 (Catissol 01) BIXAFEN_117, BIXAFEN_118, F12-030-01	0.067 0.068	-- 14	67 75	Seeds	25 30 35	0.01 0.01 <0.01	<0.01 <0.01 <0.01	0.02 0.02 <0.02
Brazil (Minas Gerais), Uberlândia 2012 (Helio 152)	0.060 0.061	-- 14	65 75	Seeds	25 30 35	<0.01 0.08 0.05	<0.01 <0.01 <0.01	<0.02 0.09 0.06

Location, year (variety) Reference, Trial-ID	Application			Residues (mean), mg eq/kg				
	kg ai/ha	Interval in days	Max. growth stage	Sample	DALA	Bixafen	M21	Total
BIXAFEN_117, BIXAFEN_118, F12-030-02								
Brazil (São Paulo), Ribeirão Preto 2012 (BRS 321) BIXAFEN_117, BIXAFEN_118, F12-030-03	0.067 0.063	-- 14	65 83	Seeds	25 30 35	0.89 0.15 0.56	<0.01 <0.01 <0.01	0.90 0.16 0.57
Brazil (São Paulo), Piracicaba 2012 (Catissol) BIXAFEN_117, BIXAFEN_118, F12-030-04	0.065 0.063	-- 14	79 81	Seeds	30	<0.01	<0.01	<0.02
Brazil (São Paulo), Jaboticabal 2012 (BRS 321) BIXAFEN_117, BIXAFEN_118, F12-030-05	0.065 0.064	-- 14	65 81	Seeds	30	1.7 (mean of three analysis)	<0.01 (mean of three analysis)	1.7
Brazil (São Paulo), Paulínia 2013 (Catissol) BIXAFEN_119, F13-006-1	0.063 0.063	-- 14	65 75	Seeds	15 20 25 30 35	<0.01 0.04 0.06 0.03 0.05	<0.01 <0.01 <0.01 <0.01 <0.01	<0.02 0.05 0.07 0.04 0.06
Brazil (Minas Gerais), Uberlândia 2013 (SYN 042) BIXAFEN_119, F13-006-02	0.063 0.061	-- 14	65 80	Seeds	15 20 25 30 35	0.03 0.18 0.02 0.02 <0.01	<0.01 <0.01 <0.01 <0.01 <0.01	0.04 0.19 0.03 0.03 <0.02
Brazil (São Paulo), Ribeirão Preto 2013 (M-734) BIXAFEN_119, F13-006-03	0.064 0.063	-- 14	65 69	Seeds	15 20 25 30 35	0.11 0.78 0.21 0.16 0.21	<0.01 <0.01 <0.01 <0.01 <0.01	0.12 0.79 0.22 0.17 0.22
Brazil (Paraná), Ponta Grossa 2013 (Catissol) BIXAFEN_119, F13-006-04	0.067 0.062	-- 14	65 73	Seeds	15 20 25 29	<0.01 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01	<0.02 <0.02 <0.02 <0.02

Location, year (variety)	Application			Residues (mean), mg eq/kg				
Reference, Trial-ID	kg ai/ha	Interval in days	Max. growth stage	Sample	DALA	Bixafen	M21	Total
					33	<0.01	<0.01	<0.02
Brazil (São Paulo), Ituverava 2013 (SYN 042) BIXAFEN_119, F13-006-05	0.063 0.060	-- 14	65 79	Seeds	15 20 25 30 34	<0.01 <0.01 0.01 0.02 0.02	<0.01 <0.01 <0.01 <0.01 <0.01	<0.02 <0.02 0.02 0.03 0.03

DALA: days after last application

M21: bixafen-desmethyl, expressed as bixafen equivalents

Total: sum of bixafen and bixafen-desmethyl, expressed as bixafen equivalents

Maize forage and fodder

Residues of bixafen in maize forage and fodder in Northern America were investigated by Lenz C. (2016, BIXAFEN_092, see crop field trial data on maize grain). Forage samples were harvested after a nominal interval of 10 days after the first application in contrast to grain and stover samples, which received an additional treatment. In one trial (2014_RES-BAN1259-B), forage was unintentionally treated twice.

Samples were analysed for bixafen and bixafen-desmethyl by residue analytical method 01012 with a LOQ of 0.01 mg/kg. Samples were stored deep-frozen for a maximum of 175 days (6 months) for forage and for a maximum of 367 days (12 months) for stover prior to analysis.

Table 24 Residues of bixafen in maize forage

Location, year (variety)	Application			Residues (mean), mg eq/kg				
Reference, Trial-ID	kg ai/ha	Interval in days	Max. growth stage	Sample	DALA	Bixafen	M21	Total
USA (PA), Germansville 2014 (TA264-13VPR1B) BIXAFEN_092, 2014_RES-BAN1259-A	0.12	--	77	Forage	10	0.85, 0.95 (0.90)	0.12, 0.12 (0.12)	0.97, 1.1 (1.0)
USA (AL), Tallassee 2014 (1690YHR) BIXAFEN_092, 2014_RES-BAN1259-B	0.11 0.11	-- 20	79 85	Forage	30	0.66, 0.81 (0.74)	0.046, 0.047 (0.046)	0.71, 0.85 (0.78)
USA (IA), Elk Horn 2014 (Mycogen 2B709a) BIXAFEN_092, 2014_RES-BAN1259-C	0.11	--	85	Forage	0	0.94, 0.97 (0.96)	<0.01, <0.01 (<0.01)	0.95, 0.98 (0.96)
					5	0.27, 0.25 (0.26)	0.037, 0.032 (0.034)	0.31, 0.28 (0.29)
					10	0.10, 0.12 (0.11)	0.018, 0.016	0.12, 0.14 (0.13)

Location, year (variety) Reference, Trial-ID	Application			Residues (mean), mg eq/kg				
	kg ai/ha	Interval in days	Max. growth stage	Sample	DALA	Bixafen	M21	Total
							(0.017)	
					15	0.15, 0.12 (0.14)	0.024, 0.020 (0.022)	0.17, 0.14 (0.16)
					20	0.14, 0.12 (0.13)	0.021, 0.020 (0.020)	0.16, 0.14 (0.15)
USA (IL), Stewardson 2014 (Burrus 6F74AMX) BIXAFEN_092, 2014_RES-BAN1259-D	0.11	--	85	Forage	10	0.41, 0.42 (0.42)	0.10, 0.12 (0.11)	0.51, 0.53 (0.52)
USA (KS), Stilwell 2014 (DKC62-08RIB) BIXAFEN_092, 2014_RES-BAN1259-E	0.11	--	85	Forage	9	0.35, 0.41 (0.38)	0.048, 0.046 (0.047)	0.40, 0.46 (0.43)
USA (KS), Gardener 2014 (Producers Hybrid 7224VT3PRIB) BIXAFEN_092, 2014_RES-BAN1259-F	0.11	--	85	Forage	9	1.1, 1.1 (1.1)	0.078, 0.078 (0.078)	1.2, 1.2 (1.2)
USA (IA), Richland 2014 (P1498 AM) BIXAFEN_092, 2014_RES-BAN1259-G	0.11	--	85	Forage	0	0.97, 0.77 (0.87)	<0.01, <0.01 (<0.01)	0.98, 0.78 (0.88)
					5	0.35, 0.47 (0.41)	0.036, 0.041 (0.038)	0.38, 0.51 (0.45)
					10	0.19, 0.15 (0.17)	0.028, 0.022 (0.025)	0.22, 0.18 (0.20)
					14	0.21, 0.15 (0.18)	0.036, 0.019 (0.028)	0.25, 0.16 (0.20)
					20	0.15, 0.13 (0.14)	0.020, 0.026 (0.023)	0.17, 0.15 (0.16)
USA (WI), Oregon 2014 (DKC 49-29-RIB) BIXAFEN_092, 2014_RES-BAN1259-H	0.11	--	83	Forage	10	0.22, 0.13 (0.18)	0.13, 0.12 (0.12)	0.35, 0.25 (0.30)
USA (MN), York 2014 (P0876CHR) BIXAFEN_092, 2014_RES-BAN1259-I	0.11	--	73	Forage	10	0.068, 0.065 (0.066)	0.019, 0.014 (0.016)	0.087, 0.079 (0.083)
USA (MN), St. Cloud 2014 (DKC 38-03-RIB)	0.11	--	72	Forage	10	0.29, 0.44 (0.36)	0.043, 0.066	0.34, 0.51 (0.42)

Location, year (variety)	Application			Residues (mean), mg eq/kg				
Reference, Trial-ID	kg ai/ha	Interval in days	Max. growth stage	Sample	DALA	Bixafen	M21	Total
BIXAFEN_092, 2014_RES-BAN1259-J							(0.054)	
USA (KS),Stafford 2014 (P1105 AM) BIXAFEN_092, 2014_RES-BAN1259-K	0.11	--	75	Forage	9	0.13, 0.25 (0.19)	0.078, 0.12 (0.096)	0.21, 0.37 (0.29)
Canada (QC), St. Marc-sur-Richelieu 2014 (829RA) BIXAFEN_092, 2014_RES-BAN1259-L	0.11	--	79	Forage	10	0.65, 0.53 (0.57)	0.056, 0.049 (0.052)	0.71, 0.58 (0.64)
Canada (ON), Cambridge 2014 (3515 Maizex DBR) BIXAFEN_092, 2014_RES-BAN1259-M	0.11	--	85	Forage	11	0.29, 0.40 (0.35)	0.022, 0.027 (0.024)	0.32, 0.42 (0.37)
Canada (MB), High Bluff 2014 (Dekalb RR) BIXAFEN_092, 2014_RES-BAN1259-N	0.11	--	35	Forage	29	0.053, 0.060 (0.056)	0.01, <0.01 (0.01)	0.063, 0.070 (0.067)
USA (TX),Uvalde 2014 (DKC 69-43) BIXAFEN_092, 2014_RES-BAN1259-O	0.11	--	71	Forage	8	0.26, 0.28 (0.27)	0.11, 0.11 (0.11)	0.38, 0.39 (0.38)
Canada (BC), Okanagan Falls 2014 (HLR219) BIXAFEN_092, 2014_RES-BAN1259-P	0.11	--	36	Forage	11	0.31, 0.59 (0.45)	0.045, 0.062 (0.054)	0.35, 0.66 (0.50)

DALA: days after last application

M21: bixafen-desmethyl, expressed as bixafen equivalents

Total: sum of bixafen and bixafen-desmethyl, expressed as bixafen equivalents

Table 25 Residues of bixafen in maize fodder

Location, year (variety)	Application			Residues (mean), mg eq/kg				
Reference, Trial-ID	kg ai/ha	Interval in days	Max. growth stage	Sample	DALA	Bixafen	M21	Total
USA (PA), Germansville 2014 (TA264-13VPR1B) BIXAFEN_092, 2014_RES-BAN1259-A	0.12 0.11	-- 21	77 87	Stover	29	2.3, 2.4 (2.4)	0.17, 0.17 (0.17)	2.5, 2.6 (2.5)
USA (AL), Tallassee 2014 (1690YHR) BIXAFEN_092, 2014_RES-BAN1259-B	0.11 0.11	-- 20	79 85	Stover	30	1.6, 1.5 (1.6)	0.076, 0.12 (0.096)	1.7, 1.6 (1.6)

Location, year (variety) Reference, Trial-ID	Application			Residues (mean), mg eq/kg				
	kg ai/ha	Interval in days	Max. growth stage	Sample	DALA	Bixafen	M21	Total
USA (IA), Elk Horn 2014 (Mycogen 2V709a) BIXAFEN_092, 2014_RES-BAN1259-C	0.11 0.12	-- 20	85 87	Stover	20	0.50, 0.54 (0.52)	0.075, 0.092 (0.084)	0.57, 0.64 (0.60)
					25	0.56, 0.28 (0.42)	0.082, 0.049 (0.066)	0.64, 0.33 (0.48)
					30	0.48, 0.40 (0.44)	0.096, 0.089 (0.094)	0.58, 0.48 (0.53)
					35	0.43, 0.47 (0.45)	0.086, 0.075 (0.080)	0.52, 0.54 (0.53)
					40	0.50, 0.40 (0.45)	0.055, 0.078 (0.066)	0.56, 0.47 (0.52)
USA (IL), Stewardson 2014 (Burrus 6F74AMX) BIXAFEN_092, 2014_RES-BAN1259-D	0.11 0.11	-- 20	85 87	Stover	27	2.2, 1.7 (2.0)	0.26, 0.25 (0.26)	2.4, 2.0 (2.2)
USA (KS), Stilwell 2014 (DKC62-08RIB) BIXAFEN_092, 2014_RES-BAN1259-E	0.11 0.11	-- 20	85 85	Stover	30	1.9, 1.5 (1.7)	0.27, 0.24 (0.26)	2.1, 1.7 (1.9)
USA (KS), Gardener 2014 (Producers Hybrid 7224VT3PRIB) BIXAFEN_092, 2014_RES-BAN1259-F	0.11 0.11	-- 19	85 87	Stover	29	2.4, 3.6 (3.0)	0.17, 0.28 (0.22)	2.5, 3.9 (3.2)
USA (IA), Richland 2014 (P1498 AM) BIXAFEN_092, 2014_RES-BAN1259-G	0.11 0.11	-- 20	85 86	Stover	19	1.6, 1.8 (1.7)	0.095, 0.12 (0.11)	1.7, 1.9 (1.8)
					24	1.6, 1.7 (1.6)	0.087, 0.12 (0.10)	1.6, 1.9 (1.8)
					31	0.94, 1.1 (1.0)	0.073, 0.062 (0.068)	1.0, 1.2 (1.1)
					33	1.2, 1.5 (1.3)	0.079, 0.12 (0.10)	1.3, 1.7 (1.5)
					40	2.0, 1.7 (1.8)	0.17, 0.14 (0.16)	2.1, 1.9 (2.0)
USA (WI), Oregon 2014 (DKC 49-29-RIB) BIXAFEN_092, 2014_RES-BAN1259-H	0.11 0.11	-- 20	83 86	Stover	29	1.6, 2.2 (1.9)	0.28, 0.42 (0.35)	1.9, 2.7 (2.2)

Location, year (variety) Reference, Trial-ID	Application			Residues (mean), mg eq/kg				
	kg ai/ha	Interval in days	Max. growth stage	Sample	DALA	Bixafen	M21	Total
USA (MN), York 2014 (P0876CHR) BIXAFEN_092, 2014_RES-BAN1259-I	0.11 0.11	-- 20	73 85	Stover	30	0.81, 0.72 (0.76)	0.068, 0.054 (0.61)	0.88, 0.77 (0.82)
USA (MN), St. Cloud 2014 (DKC 38-03-RIB) BIXAFEN_092, 2014_RES-BAN1259-J	0.11 0.11	-- 21	72 83	Stover	29	1.7, 2.4 (2.0)	0.29, 0.31 (0.30)	2.0, 2.7 (2.4)
USA (KS), Stafford 2014 (P1105 AM) BIXAFEN_092, 2014_RES-BAN1259-K	0.11 0.11	-- 20	75 85	Stover	29	0.44, 0.53 (0.49)	0.19, 0.22 (0.20)	0.63, 0.75 (0.69)
Canada (QC), St. Marc-sur-Richelieu 2014 (829RA) BIXAFEN_092, 2014_RES-BAN1259-L	0.11 0.11	-- 19	79 85	Stover	30	2.4, 2.3 (2.4)	0.094, 0.082 (0.088)	2.5, 2.4 (2.4)
Canada (ON), Cambridge 2014 (3515 Maizex DBR) BIXAFEN_092, 2014_RES-BAN1259-M	0.11 0.11	-- 20	85 85	Stover	30	2.3, 1.7 (2.0)	0.21, 0.15 (0.18)	2.5, 1.9 (2.2)
Canada (MB), High Bluff 2014 (Dekalb RR) BIXAFEN_092, 2014_RES-BAN1259-N	0.11 0.12	-- 36	35 85	Stover	32	0.37, 0.27 (0.32)	0.051, 0.045 (0.048)	0.42, 0.31 (0.36)
USA (TX),Uvalde 2014 (DKC 69-43) BIXAFEN_092, 2014_RES-BAN1259-O	0.11 0.12	-- 21	71 87	Stover	28	2.6, 2.3 (2.4)	0.36, 0.35 (0.36)	2.9, 2.7 (2.8)
Canada (BC), Okanagan Falls 2014 (HLR219) BIXAFEN_092, 2014_RES-BAN1259-P	0.11 0.11	-- 26	36 65	Stover	29	1.2, 1.2 (1.2)	0.14, 0.10 (0.12)	1.3, 1.3 (1.3)

DALA: days after last application

M21: bixafen-desmethyl, expressed as bixafen equivalents

Total: sum of bixafen and bixafen-desmethyl, expressed as bixafen equivalents

Sorghum forage and fodder

Residues of bixafen in sorghum cultivated in the USA were investigated by Lenz C. (2016, BIXAFEN_094, see crop field trial data on sorghum grain). Forage samples were harvested after a nominal interval of 10 days after the first application in contrast to grain and stover samples, which received an additional treatment.

Samples were analysed for bixafen and bixafen-desmethyl by residue analytical method 01012 with a LOQ of 0.01 mg/kg. Samples were stored deep-frozen prior to analysis for a maximum of 209 days (7 months) for forage and for a maximum of 168 days (6 months) for stover.

Table 26 Residues of bixafen in sorghum forage

Location, year (variety)	Application			Residues (mean), mg eq/kg				
Reference, Trial-ID	kg ai/ha	Interval in days	Max. growth stage	Sample	DALA	Bixafen	M21	Total
USA (MO), Fisk 2014 (Mycogen M3838 C) BIXAFEN_94, 2014-RES-BAN1260-A	0.11	--	61	Forage	10	0.28, 0.23 (0.25)	0.20, 0.13 (0.15)	0.48, 0.35 (0.41)
USA (IA), Atlantic 2014 (Long-Tail Delight) BIXAFEN_94, 2014-RES-BAN1260-B	0.12	--	23	Forage	0	3.3, 2.9 (3.1)	0.069, 0.065 (0.067)	3.4, 3.0 (3.2)
					5	0.92, 1.0 (0.98)	0.53, 0.60 (0.56)	1.5, 1.6 (1.5)
					10	0.89, 0.70 (0.79)	0.81, 0.74 (0.78)	1.7, 1.4 (1.6)
					15	0.24, 0.25 (0.24)	0.69, 0.69 (0.69)	0.93, 0.94 (0.94)
					20	0.094, 0.089 (0.092)	0.46, 0.48 (0.47)	0.56, 0.57 (0.56)
USA (KS), Lenexa 2014 (K73-J6) BIXAFEN_94, 2014-RES-BAN1260-C	0.11	--	83	Forage	9	0.58, 0.69 (0.63)	0.17, 0.17 (0.17)	0.75, 0.86 (0.80)
USA (NE), York 2014 (VARA 1005964) BIXAFEN_94, 2014-RES-BAN1260-D	0.11	--	75	Forage	9	0.19, 0.23 (0.21)	0.10, 0.081 (0.091)	0.29, 0.31 (0.30)
USA (TX), East Bernard 2014 (DKS53-67) BIXAFEN_94, 2014-RES-BAN1260-E	0.11	--	57	Forage	9	1.3, 1.2 (1.2)	0.20, 0.23 (0.21)	1.5, 1.4 (1.4)
USA (TX), Uvalde 2014 (Pioneer 83G19) BIXAFEN_94, 2014-RES-BAN1260-F	0.12	--	51	Forage	9	0.32, 0.40 (0.37)	0.45, 0.55 (0.50)	0.76, 0.95 (0.86)
USA (ND), Carrington 2014 (LM 5009) BIXAFEN_94, 2014-RES-BAN1260-G	0.11	--	69	Forage	10	0.37, 0.23 (0.30)	0.13, 0.12 (0.12)	0.50, 0.35 (0.42)
USA (TX), Groom 2014 (Y373) BIXAFEN_94, 2014-RES-BAN1260-H	0.11	--	71	Forage	10	0.37, 0.19 (0.28)	0.086, 0.043 (0.064)	0.46, 0.24 (0.35)

Location, year (variety)	Application			Residues (mean), mg eq/kg				
Reference, Trial-ID	kg ai/ha	Interval in days	Max. growth stage	Sample	DALA	Bixafen	M21	Total
USA (TX), Wall 2014 (DKS49-45) BIXAFEN_94, 2014-RES-BAN1260-I	0.11	--	69	Forage	10	0.90, 0.98 (0.94)	0.22, 0.24 (0.23)	1.1, 1.2 (1.2)

DALA: days after last application

M21: bixafen-desmethyl, expressed as bixafen equivalents

Total: sum of bixafen and bixafen-desmethyl, expressed as bixafen equivalents

Table 27 Residues of bixafen in sorghum fodder

Location, year (variety)	Application			Residues (mean), mg eq/kg				
Reference, Trial-ID	kg ai/ha	Interval in days	Max. growth stage	Sample	DALA	Bixafen	M21	Total
USA (MO), Fisk 2014 (Mycogen M3838 C) BIXAFEN_94, 2014-RES-BAN1260-A	0.11 0.11	-- 20	61 85	Stover	30	0.50, 0.40 (0.45)	0.23, 0.17 (0.20)	0.73, 0.57 (0.65)
USA (IA), Atlantic 2014 (Long-Tail Delight) BIXAFEN_94, 2014-RES-BAN1260-B	0.12 0.11	-- 21	23 55	Stover	20	0.35, 0.44 (0.40)	1.6, 2.0 (1.8)	2.0, 2.4 (2.2)
					25	0.36, 0.27 (0.31)	1.7, 1.5 (1.6)	2.0, 1.8 (1.9)
					30	0.24, 0.18 (0.21)	1.4, 1.0 (1.2)	1.6, 1.2 (1.4)
					35	0.29, 0.29 (0.29)	1.5, 1.4 (1.4)	1.8, 1.7 (1.7)
					40	0.17, 0.18 (0.18)	1.1, 1.0 (1.1)	1.3, 1.2 (1.3)
USA (KS), Lenexa 2014 (K73-J6) BIXAFEN_94, 2014-RES-BAN1260-C	0.11 0.12	-- 19	83 87	Stover	29	0.37, 0.72 (0.54)	0.23, 0.44 (0.34)	0.60, 1.2 (0.90)
USA (NE), York 2014 (VARA 1005964) BIXAFEN_94, 2014-RES-BAN1260-D	0.11 0.11	-- 21	75 83	Stover	32	0.25, 0.31 (0.28)	0.075, 0.095 (0.085)	0.33, 0.40 (0.36)
USA (TX), East Bernard 2014 (DKS53-67) BIXAFEN_94, 2014-RES-BAN1260-E	0.11 0.11	-- 15	57 83	Stover	28	3.7, 3.9 (3.8)	0.58, 0.61 (0.60)	4.3, 4.5 (4.4)
USA (TX), Uvalde 2014 (Pioneer 83G19)	0.12 0.11	-- 22	51 87	Stover	28	0.66, 0.78 (0.72)	0.79, 0.94 (0.876)	1.5, 1.7 (1.6)

Location, year (variety)	Application			Residues (mean), mg eq/kg				
Reference, Trial-ID	kg ai/ha	Interval in days	Max. growth stage	Sample	DALA	Bixafen	M21	Total
BIXAFEN_94, 2014-RES-BAN1260-F								
USA (ND), Carrington 2014 (LM 5009) BIXAFEN_94, 2014-RES-BAN1260-G	0.11 0.11	-- 21	69 71	Stover	27	2.0, 1.4 (1.7)	0.27, 0.16 (0.22)	2.3, 1.5 (1.9)
USA (TX), Groom 2014 (Y373) BIXAFEN_94, 2014-RES-BAN1260-H	0.11 0.11	-- 20	71 87	Stover	30	1.8, 2.0 (1.9)	0.26, 0.18 (0.22)	2.1, 2.2 (2.1)
USA (TX), Wall 2014 (DKS49-45) BIXAFEN_94, 2014-RES-BAN1260-I	0.11 0.11	-- 20	69 87	Stover	29	0.91, 1.2 (1.0)	0.26, 0.29 (0.28)	1.2, 1.5 (1.3)

DALA: days after last application

M21: bixafen-desmethyl, expressed as bixafen equivalents

Total: sum of bixafen and bixafen-desmethyl, expressed as bixafen equivalents

Wheat forage, fodder and straw

Residues of bixafen in wheat cultivated in Northern America were investigated by Lenz C. (2016, BIXAFEN_095, see crop field trial data on wheat grain). Forage and hay samples were harvested after a nominal interval of 10 and 20 days, respectively, after the first application in contrast to grain and straw samples, which received an additional treatment.

Samples were analysed for bixafen and bixafen-desmethyl by residue analytical method 01012 with a LOQ of 0.01 mg/kg. Samples were stored deep-frozen prior to analysis for a maximum of 208 days (7 months) for grain, 438 days (14 months) for hay and 151 days (5 months) for straw.

Table 28 Residues of bixafen in wheat forage and hay

Location, year (variety)	Application			Residues (mean), mg eq/kg				
Reference, Trial-ID	kg ai/ha	Interval in days	Max. growth stage	Sample	DALA	Bixafen	M21	Total
USA (AL), Tallassee 2015 (Pioneer 26R41) BIXAFEN_095, 2014RES-BAN1261-A	0.11	--	25	Forage	10	0.71, 0.80 (0.75)	0.073, 0.097 (0.085)	0.78, 0.90 (0.84)
				Hay	20	0.94, 1.2 (1.1)	0.19, 0.25 (0.23)	1.1, 1.5 (1.3)
USA (MI), Fisk 2015 (Roane) BIXAFEN_095,	0.11	--	29	Forage	9	1.1, 0.97 (1.0)	0.11, 0.12 (0.12)	1.2, 1.1 (1.1)

Location, year (variety)	Application			Residues (mean), mg eq/kg				
Reference, Trial-ID	kg ai/ha	Interval in days	Max. growth stage	Sample	DALA	Bixafen	M21	Total
2014RES-BAN1261-B				Hay	20	0.67, 0.84 (0.75)	0.18, 0.19 (0.18)	0.85, 1.0 (0.94)
USA (IL), Stewardson 2014 (Becks 129) BIXAFEN_095,	0.11	--	33	Forage	9	1.7, 1.5 (1.6)	0.11, 0.099 (0.10)	1.8, 1.6 (1.7)
2014RES-BAN1261-C				Hay	19	0.91, 1.1 (0.98)	0.15, 0.16 (0.16)	1.1, 1.2 (1.1)
USA (KS), Gardener 2014 (Agromax 413) BIXAFEN_095,	0.11	--	29	Forage	10	1.1, 1.1 (1.1)	0.16, 0.18 (0.17)	1.2, 1.3 (1.2)
2014RES-BAN1261-D				Hay	20	2.5, 2.2 (2.4)	0.58, 0.57 (0.58)	3.0, 2.8 (2.9)
USA (ND), Northwood 2014 (Faller) BIXAFEN_095,	0.11	--	25	Forage	0	8.5, 8.5 (8.5)	<0.01, <0.01 (<0.01)	8.6, 8.5 (8.5)
2014RES-BAN1261-E					3	1.2, 1.1 (1.1)	0.099, 0.089 (0.094)	1.3, 1.2 (1.2)
					7	0.37, 0.37 (0.37)	0.067, 0.061 (0.064)	0.44, 0.43 (0.43)
					10	0.19, 0.21 (0.2)	0.050, 0.055 (0.052)	0.24, 0.27 (0.25)
					15	0.10, 0.098 (0.099)	0.039, 0.041 (0.040)	0.14, 0.14 (0.14)
				Hay	20	0.029, 0.042 (0.036)	0.011, 0.021 (0.016)	0.04, 0.063 (0.052)
USA (TX), Uvalde 2015 (Greer) BIXAFEN_095,	0.11	--	26	Forage	10	1.5, 1.4 (1.4)	0.073, 0.074 (0.073)	1.5, 1.5 (1.5)
2014RES-BAN1261-F				Hay	20	0.89, 1.1 (0.97)	0.12, 0.14 (0.13)	1.0, 1.2 (1.1)
USA (ND), Carrington 2015 (Propper) BIXAFEN_095,	0.11	--	25	Forage	10	1.3, 1.3 (1.3)	0.12, 0.13 (0.12)	1.4, 1.4 (1.4)
2014RES-BAN1261-G				Hay	21	1.2, 1.2 (1.2)	0.18, 0.20 (0.19)	1.4, 1.4 (1.4)
USA (ND), Jamestown 2014 (Prosper) BIXAFEN_095,	0.11	--	25	Forage	10	1.6, 1.9 (1.7)	0.27, 0.33 (0.30)	1.8, 2.2 (2.0)
2014RES-BAN1261-H				Hay	18	2.2, 2.9	0.56, 0.73	2.7, 3.7

Location, year (variety) Reference, Trial-ID	Application			Residues (mean), mg eq/kg				
	kg ai/ha	Interval in days	Max. growth stage	Sample	DALA	Bixafen	M21	Total
						(2.6)	(0.64)	(3.2)
USA (NB), York 2015 (Husker Genetics Overland), BIXAFEN_095, 2014RES-BAN1261-I2	0.11	--	25	Forage	8	2.5, 3.3 (2.9)	0.041, 0.044 (0.042)	2.5, 3.3 (2.9)
				Hay	19	2.0, 1.7 (1.9)	0.15, 0.12 (0.13)	2.2, 1.9 (2.0)
Canada (SK), Hanley 2014 (Cardale) BIXAFEN_095, 2014RES-BAN1261-J	0.11	--	31	Forage	9	1.8, 1.9 (1.8)	0.19, 0.20 (0.20)	1.9, 2.1 (2.0)
				Hay	21	2.7, 2.1 (2.4)	0.69, 0.49 (0.59)	3.4, 2.6 (3.0)
Canada (SK), Pike Lake 2014 (CDC Utmost) BIXAFEN_095, 2014RES-BAN1261-K	0.11	--	25	Forage	10	0.58, 0.49 (0.54)	0.10, 0.092 (0.096)	0.69, 0.58 (0.64)
				Hay	22	0.040, 0.050 (0.045)	0.017, 0.023 (0.020)	0.056, 0.073 (0.064)
Canada (AB), Taber 2014 (Carberry) BIXAFEN_095, 2014RES-BAN1261-L	0.11	--	30	Forage	10	0.38, 0.52 (0.45)	0.14, 0.16 (0.15)	0.52, 0.68 (0.60)
				Hay	21	0.047, 0.10 (0.074)	0.037, 0.060 (0.048)	0.084, 0.16 (0.12)
USA (KS), Lamed 2015 (LCS Wizard) BIXAFEN_095, 2014RES-BAN1261-M	0.11	--	25	Forage	0	11, 11 (11)	0.014, 0.013 (0.014)	11, 11 (11)
					3	6.7, 6.4 (6.6)	0.032, 0.033 (0.032)	6.7, 6.4 (6.6)
					6	3.5, 2.8 (3.1)	0.038, 0.033 (0.036)	3.5, 2.8 (3.2)
					9	3.1, 3.2 (3.2)	0.051, 0.049 (0.050)	3.2, 3.3 (3.2)
					13	1.7, 1.6 (1.6)	0.038, 0.033 (0.036)	1.7, 1.6 (1.6)
				Hay	20	0.94, 1.2 (1.1)	0.11, 0.16 (0.13)	1.1, 1.4 (1.2)
USA (OK), Hinton 2014 (Duster) BIXAFEN_095,	0.11	--	25	Forage	12	0.98, 0.90 (0.94)	0.22, 0.21 (0.22)	1.2, 1.1 (1.2)

Location, year (variety)	Application			Residues (mean), mg eq/kg				
Reference, Trial-ID	kg ai/ha	Interval in days	Max. growth stage	Sample	DALA	Bixafen	M21	Total
2014RES-BAN1261-N				Hay	20	1.0, 0.91 (0.96)	0.27, 0.26 (0.26)	1.3, 1.2 (1.2)
USA (TX), Claude 2015 (not reported) BIXAFEN_095,	0.11	--	27	Forage	10	0.84, 0.83 (0.84)	0.067, 0.065 (0.066)	0.91, 0.90 (0.90)
2014RES-BAN1261-O				Hay	20	1.5, 2.2 (1.8)	0.16, 0.21 (0.19)	1.7, 2.4 (2.0)
USA (TX), Wall 2015 (Coronado) BIXAFEN_095,	0.11	--	32	Forage	11	0.92, 0.77 (0.84)	0.069, 0.054 (0.062)	0.98, 0.83 (0.91)
2014RES-BAN1261-P				Hay	20	0.34, 0.47 (0.40)	0.068, 0.10 (0.086)	0.41, 0.57 (0.49)
USA (WS), Ephrata 2014 (Expresso) BIXAFEN_095,	0.11	--	25	Forage	12	0.11, 0.12 (0.12)	0.067, 0.072 (0.069)	0.18, 0.19 (0.19)
2014RES-BAN1261-Q				Hay	20	0.15, 0.12 (0.13)	0.092, 0.087 (0.90)	0.24, 0.21 (0.22)
Canada (MB), Brandon 2015 (not reported) BIXAFEN_095,	0.11	--	24	Forage	11	3.0, 3.3 (3.2)	0.21, 0.22 (0.21)	3.2, 3.5 (3.4)
2014RES-BAN1261-R2 Treatment: 04.05.15				Hay	21	0.80, 1.3 (1.0)	0.14, 0.23 (0.18)	0.95, 1.5 (1.2)
Canada (BC), Oliver 2014 (Carberry)	0.11	--	25	Forage	11	0.097, 0.14 (0.12)	0.047, 0.079 (0.063)	0.14, 0.22 (0.18)
BIXAFEN_095, 2014RES-BAN1261-S				Hay	18	0.14, 0.075 (0.11)	0.15, 0.091 (0.12)	0.29, 0.17 (0.23)
Canada (BC), Crawston 2014 (Common #1)	0.11	--	29	Forage	12	0.99, 0.83 (0.91)	0.25, 0.20 (0.22)	1.2, 1.0 (1.1)
BIXAFEN_095, 2014RES-BAN1261-T				Hay	18	1.4, 1.7 (1.5)	0.33, 0.38 (0.36)	1.7, 2.0 (1.9)
Canada (SK), Wakaw 2014 (Cardale) BIXAFEN_095,	0.11	--	31	Forage	12	0.61, 0.61 (0.61)	0.10, 0.099 (0.10)	0.71, 0.70 (0.71)
2014RES-BAN1261-U				Hay	21	0.70, 0.95 (0.82)	0.23, 0.29 (0.26)	0.93, 1.2 (1.1)
Canada (SK), Rosthem 2014 (Cardale) BIXAFEN_095,	0.11	--	31	Forage	10	0.63, 0.68 (0.66)	0.12, 0.11 (0.12)	0.75, 0.80 (0.77)
2014RES-BAN1261-V				Hay	19	0.73, 0.69	0.22, 0.22	0.94, 0.91

Location, year (variety)	Application			Residues (mean), mg eq/kg				
Reference, Trial-ID	kg ai/ha	Interval in days	Max. growth stage	Sample	DALA	Bixafen	M21	Total
						(0.71)	(0.22)	(0.93)
Canada (AB), Josephburg 2014 (Harvest)	0.11	--	31	Forage	12	0.57, 0.46 (0.51)	0.14, 0.12 (0.13)	0.71, 0.58 (0.64)
BIXAFEN_095, 2014RES-BAN1261-W				Hay	21	0.60, 0.58 (0.59)	0.24, 0.25 (0.24)	0.84, 0.82 (0.83)
Canada (AB), Gibbons 2014 (Utmost) BIXAFEN_095,	0.11	--	30	Forage	11	0.91, 1.0 (0.96)	0.099, 0.10 (0.10)	1.0, 1.1 (1.1)
2014RES-BAN1261-X				Hay	20	1.3, 0.85 (1.1)	0.22, 0.16 (0.19)	1.5, 1.0 (1.3)
Canada (MB), Brandon 2014 (Glen) BIXAFEN_095,	0.12	--	22	Forage	19	0.31, 0.35 (0.33)	0.12, 0.13 (0.12)	0.43, 0.48 (0.46)
2014RES-BAN1261-Y Treatment: 02.07.14				Hay	27	0.73, 0.66 (0.70)	0.33, 0.28 (0.30)	1.1, 0.94 (1.0)

DALA: days after last application

M21: bixafen-desmethyl, expressed as bixafen equivalents

Total: sum of bixafen and bixafen-desmethyl, expressed as bixafen equivalents

Table 29 Residues of bixafen in wheat straw

Location, year (variety)	Application			Residues (mean), mg eq/kg				
Reference, Trial-ID	kg ai/ha	Interval in days	Max. growth stage	Sample	DALA	Bixafen	M21	Total
USA (AL), Tallassee 2015 (Pioneer 26R41) BIXAFEN_095, 2014RES-BAN1261-A	0.11 0.11	-- 24	25 69	Straw	32	3.4, 3.7 (3.6)	0.26, 0.26 (0.26)	3.7, 4.0 (3.8)
USA (MI), Fisk 2015 (Roane) BIXAFEN_095, 2014RES-BAN1261-B	0.11 0.11	-- 24	29 71	Straw	30	1.2, 0.92 (1.1)	0.36, 0.25 (0.30)	1.6, 1.2 (1.4)
USA (IL), Stewardson 2014 (Becks 129) BIXAFEN_095, 2014RES-BAN1261-C	0.11 0.11	-- 24	33 77	Straw	30	0.33, 0.59 (0.46)	0.16, 0.36 (0.26)	0.49, 0.96 (0.72)
USA (KS), Gardener 2014 (Agromax 413) BIXAFEN_095, 2014RES-BAN1261-D	0.11 0.11	-- 32	29 75	Straw	30	1.1, 0.94 (1.0)	0.39, 0.32 (0.36)	1.5, 1.3 (1.4)
USA (ND), Northwood 2014 (Faller)	0.11 0.11	-- 42	25 71	Straw	27	1.1, 1.3 (1.2)	0.36, 0.36 (0.36)	1.4, 1.7 (1.5)

Location, year (variety) Reference, Trial-ID	Application			Residues (mean), mg eq/kg				
	kg ai/ha	Interval in days	Max. growth stage	Sample	DALA	Bixafen	M21	Total
BIXAFEN_095, 2014RES-BAN1261-E					30	1.8, 1.2 (1.5)	0.69, 0.39 (0.54)	2.5, 1.6 (2.0)
					35	1.7, 1.6 (1.6)	0.53, 0.51 (0.52)	2.2, 2.1 (2.2)
					41	1.3, 1.3 (1.3)	0.39, 0.49 (0.44)	1.7, 1.8 (1.7)
					44	1.6, 1.7 (1.6)	0.48, 0.48 (0.48)	2.0, 2.1 (2.1)
USA (TX), Uvalde 2015 (Greer) BIXAFEN_095, 2014RES-BAN1261-F	0.11 0.11	-- 73	26 77	Straw	35	2.9, 2.2 (2.6)	0.65, 0.72 (0.68)	3.6, 3.0 (3.3)
USA (ND), Carrington 2015 (Propper) BIXAFEN_095, 2014RES-BAN1261-G	0.11 0.12	-- 28	25 73	Straw	33	3.6, 2.4 (3.0)	0.74, 0.64 (0.69)	4.3, 3.0 (3.7)
USA (ND), Jamestown 2014 (Prosper) BIXAFEN_095, 2014RES-BAN1261-H	0.11 0.12	-- 18	25 65	Straw	33	1.5, 1.2 (1.3)	0.84, 0.75 (0.80)	2.3, 1.9 (2.1)
USA (NB), Grand Island 2014 (Proper HRS) BIXAFEN_095, 2014RES-BAN1261-I	0.11 0.11	-- 31	25 61	Straw	32	0.53, 0.53 (0.53)	0.35, 0.35 (0.35)	0.88, 0.88 (0.88)
USA (NB), York 2015 (Husker Genetics Overland) BIXAFEN_095, 2014RES-BAN1261-I2	0.11 0.11	-- 56	25 75	Straw	33	0.22, 0.23 (0.22)	0.13, 0.13 (0.13)	0.35, 0.36 (0.36)
Canada (SK), Hanley 2014 (Cardale) BIXAFEN_095, 2014RES-BAN1261-J	0.11 0.11	-- 50	31 83	Straw	31	1.4, 1.5 (1.4)	0.24, 0.29 (0.27)	1.6, 1.8 (1.7)
Canada (SK), Pike Lake 2014 (CDC Utmost) BIXAFEN_095, 2014RES-BAN1261-K	0.11 0.11	-- 46	25 75	Straw	34	1.3, 1.3 (1.3)	0.52, 0.55 (0.54)	1.8, 1.9 (1.8)
Canada (AB), Taber 2014 (Carberry) BIXAFEN_095, 2014RES-BAN1261-L	0.11 0.12	-- 35	30 75	Straw	35	1.2, 1.4 (1.3)	0.38, 0.38 (0.38)	1.6, 1.8 (1.7)
USA (KS), Lamed 2015 (LCS Wizard) BIXAFEN_095, 2014RES-BAN1261-M	0.11 0.11	-- 65	25 83	Straw	25	1.2, 1.7 (1.4)	0.14, 0.23 (0.19)	1.3, 2.0 (1.6)
					30	2.1, 2.2 (2.2)	0.22, 0.28 (0.25)	2.4, 2.4 (2.4)

Location, year (variety) Reference, Trial-ID	Application			Residues (mean), mg eq/kg				
	kg ai/ha	Interval in days	Max. growth stage	Sample	DALA	Bixafen	M21	Total
					35	2.3, 2.9 (2.6)	0.22, 0.29 (0.25)	2.5, 3.2 (2.8)
					41	1.5, 1.7 (1.6)	0.22, 0.25 (0.23)	1.8, 2.0 (1.9)
					46	1.8, 1.6 (1.7)	0.24, 0.22 (0.23)	2.0, 1.8 (1.9)
USA (OK), Hinton 2014 (Duster) BIXAFEN_095, 2014RES-BAN1261-N	0.11 0.11	-- 32	25 77	Straw	34	3.0, 4.1 (3.6)	0.51, 0.63 (0.57)	3.5, 4.8 (4.2)
USA (TX), Claude 2015 (not reported) BIXAFEN_095, 2014RES-BAN1261-O	0.11 0.11	-- 48	27 69	Straw	30	0.90, 1.0 (0.95)	0.13, 0.15 (0.14)	1.0, 1.2 (1.1)
USA (TX), Wall 2015 (Coronado) BIXAFEN_095, 2014RES-BAN1261-P	0.11 0.11	-- 46	32 77	Straw	35	1.0, 0.80 (0.90)	0.36, 0.29 (0.32)	1.4, 1.1 (1.2)
USA (WS), Ephrata 2014 (Expresso) BIXAFEN_095, 2014RES-BAN1261-Q	0.11 0.11	-- 45	25 77	Straw	35	1.2, 1.8 (1.5)	0.21, 0.29 (0.25)	1.5, 2.1 (1.8)
Canada (MB), Brandon 2015 (not reported) BIXAFEN_095, 2014RES-BAN1261-R2 Treatment: 04.05.15 & 21.07.15	0.11 0.11	-- 78	24 87	Straw	30	2.5, 2.5 (2.5)	0.58, 0.61 (0.59)	3.1, 3.1 (3.1)
Canada (BC), Oliver 2014 (Carberry) BIXAFEN_095, 2014RES-BAN1261-S	0.11 0.11	-- 26	25 85	Straw	35	2.0, 1.5 (1.8)	0.41, 0.32 (0.36)	2.4, 1.8 (2.1)
Canada (BC), Crawston 2014 (Common #1) BIXAFEN_095, 2014RES-BAN1261-T	0.11 0.12	-- 31	29 85	Straw	30	2.4, 3.1 (2.7)	0.18, 0.23 (0.21)	2.6, 3.3 (3.0)
Canada (SK), Wakaw 2014 (Cardale) BIXAFEN_095, 2014RES-BAN1261-U	0.11 0.11	-- 41	31 83	Straw	34	1.1, 1.0 (1.1)	0.30, 0.30 (0.30)	1.4, 1.3 (1.4)
Canada (SK), Rosthem 2014 (Cardale) BIXAFEN_095, 2014RES-BAN1261-V	0.11 0.11	-- 39	31 77	Straw	34	1.7, 1.5 (1.6)	0.46, 0.32 (0.39)	2.2, 1.8 (2.0)
Canada (AB), Josephburg 2014 (Harvest)	0.11 0.11	-- 40	31 75	Straw	35	4.0, 3.1 (3.6)	0.65, 0.51 (0.58)	4.7, 3.6 (4.2)

Location, year (variety) Reference, Trial-ID	Application			Residues (mean), mg eq/kg				
	kg ai/ha	Interval in days	Max. growth stage	Sample	DALA	Bixafen	M21	Total
BIXAFEN_095, 2014RES-BAN1261-W								
Canada (AB), Gibbons 2014 (Utmost) BIXAFEN_095, 2014RES-BAN1261-X	0.11 0.12	-- 41	30 75	Straw	32	2.3, 2.3 (2.3)	0.20, 0.25 (0.22)	2.5, 2.5 (2.5)
Canada (MB), Brandon 2014 (Glen) BIXAFEN_095, 2014RES-BAN1261-Y Treatment: 02.07.14 & 08.08.14	0.12 0.12	-- 37	22 75	Straw	25	1.2, 1.1 (1.2)	0.45, 0.40 (0.43)	1.7, 1.5 (1.6)

DALA: days after last application

M21: bixafen-desmethyl, expressed as bixafen equivalents

Total: sum of bixafen and bixafen-desmethyl, expressed as bixafen equivalents

Peanut hay

Residues of bixafen in peanuts cultivated in the USA were investigated by Lenz C. (2016, BIXAFEN_096, see crop field trial data on peanuts).

Samples were analysed for bixafen and bixafen-desmethyl by residue analytical method 01012 with a LOQ of 0.01 mg/kg. Samples were stored deep-frozen prior to analysis for a maximum of 133 days (4 months) for peanut hay.

Table 30 Residues of bixafen in peanut hay

Location, year (variety) Reference, Trial-ID	Application			Residues (mean), mg eq/kg				
	kg ai/ha	Interval in days	Max. growth stage	Sample	DALA	Bixafen	M21	Total
USA (AL), Tallassee 2014 (GA06) BIXAFEN_96, 2014RES-BAN1251-A	0.057 0.057 0.057 0.057	-- 14 13 15	73 79 85 87	Hay	14	2.8, 2.3 (2.6)	0.45, 0.35 (0.40)	3.3, 2.7 (3.0)
USA (AL), Tallassee 2014 (GA06) BIXAFEN_96, 2014RES-BAN1251-B	0.056 0.057 0.057 0.057	-- 13 15 14	71 77 83 87	Hay	14	1.7, 2.9 (2.3)	0.21, 0.33 (0.27)	1.9, 3.2 (2.5)
USA (AL), Auburn 2014 (GA06) BIXAFEN_96, 2014RES-BAN1251-C	0.057 0.057 0.057 0.057	-- 14 14 14	75 75 85 87	Hay	15	3.7, 3.3 (3.5)	0.60, 0.52 (0.56)	4.3, 3.8 (4.0)
USA (GA), Chula 2014 (GA09B) BIXAFEN_96, 2014RES-BAN1251-D	0.056 0.057 0.056 0.058	-- 14 14 14	75 75 79 85	Hay	-0	0.75, 0.64, 0.97, 0.98 (0.83)	0.37, 0.37, 0.49, 0.47 (0.42)	1.1, 1.0, 1.5, 1.4 (1.3)

Location, year (variety) Reference, Trial-ID	Application			Residues (mean), mg eq/kg				
	kg ai/ha	Interval in days	Max. growth stage	Sample	DALA	Bixafen	M21	Total
					0	3.4, 2.7 (3.1)	0.73, 0.58 (0.66)	4.1, 3.3 (3.9)
					6	0.70, 0.98 (0.84)	0.36, 0.32 (0.34)	1.1, 1.3 (1.2)
					14	0.93, 0.84 (0.88)	0.36, 0.37 (0.36)	1.3, 1.2 (1.2)
					21	0.83, 0.76 (0.80)	0.38, 0.38 (0.38)	1.2, 1.1 (1.2)
					28	0.80, 0.58 (0.69)	0.39, 0.27 (0.33)	1.2, 0.85 (1.0)
USA (GA), Abbeville 2014 (GA06G) BIXAFEN_96, 2014RES-BAN1251-E	0.056 0.056 0.056 0.056	-- 14 14 14	73 77 79 83	Hay	14	1.1, 1.3 (1.2)	0.34, 0.42 (0.38)	1.4, 1.7 (1.6)
USA (GA), Weston 2014 (GA06G) BIXAFEN_96, 2014RES-BAN1251-F	0.055 0.055 0.055 0.055	-- 14 14 14	72 74 76 80	Hay	14	4.0, 4.6 (4.3)	0.74, 0.92 (0.83)	4.7, 5.5 (5.1)
USA (NC), Seven Springs 2014 (Bailey) BIXAFEN_96, 2014RES-BAN1251-G	0.058 0.055 0.056 0.057	-- 14 14 14	81 82 84 87	Hay	14	4.0, 4.9 (4.4)	0.81, 0.90 (0.86)	4.8, 5.8 (5.3)
USA (NC), Albertson 2014 (Bailey) BIXAFEN_96, 2014RES-BAN1251-H	0.057 0.055 0.056 0.057	-- 14 14 14	72 80 83 86	Hay	14	3.2, 2.9 (3.0)	0.79, 0.70 (0.74)	3.9, 3.6 (3.8)
USA (FL), Center Hill 2014 (GA06G) BIXAFEN_96, 2014RES-BAN1251-I	0.054 0.057 0.058 0.057	-- 14 15 13	35 39 81 85	Hay	14	0.90, 1.2 (1.0)	0.27, 0.36 (0.31)	1.2, 1.5 (1.4)
USA (OK), Ringwood 2014 (Jupiter) BIXAFEN_96, 2014RES-BAN1251-J	0.057 0.056 0.056 0.056	-- 14 14 14	79 79 79 79	Hay	13	4.0, 3.8 (3.9)	1.0, 0.95 (0.98)	5.0, 4.8 (4.9)
USA (AR), Pocahontas 2014 (Georgia 09B) BIXAFEN_96, 2014RES-BAN1251-K	0.055 0.056 0.056 0.056	-- 14 13 14	71 75 84 87	Hay	14	3.3, 2.5 (2.9)	0.68, 0.54 (0.61)	4.0, 3.1 (3.5)
USA (IA), Richland 2014 (Bailey) BIXAFEN_96, 2014RES-BAN1251-L	0.056 0.056 0.056 0.056	-- 14 14 14	79 82 83 84	Hay	12	3.1, 3.3 (3.2)	0.32, 0.32 (0.32)	3.4, 3.6 (3.5)
USA (TX), East Bernard 2014 (Georgia 09B) BIXAFEN_96, 2014RES-BAN1251-M	0.056 0.056 0.056 0.056	-- 14 14 14	69 73 77 79	Hay	14	1.8, 2.4 (2.1)	0.46, 0.66 (0.56)	2.2, 3.1 (2.6)

Location, year (variety) Reference, Trial-ID	Application			Residues (mean), mg eq/kg				
	kg ai/ha	Interval in days	Max. growth stage	Sample	DALA	Bixafen	M21	Total
USA (OK), Hinton 2014 (TamNut 0L06) BIXAFEN_96, 2014RES-BAN1251-N	0.056 0.056 0.056 0.056	-- 15 13 14	73 79 79 85	Hay	16	0.81, 0.86 (0.84)	0.25, 0.26 (0.25)	1.1, 1.1 (1.1)
USA (TX), Wellington 2014 (TAMRUN) BIXAFEN_96, 2014RES-BAN1251-O	0.057 0.058 0.056 0.057	-- 14 14 14	79 81 84 86	Hay	14	0.062, 0.056 (0.059)	<0.01, <0.01 (<0.01)	0.067, 0.063 (0.065)

DALA: days after last application

M21: bixafen-desmethyl, expressed as bixafen equivalents

Total: sum of bixafen and bixafen-desmethyl, expressed as bixafen equivalents

FATE OF RESIDUES IN STORAGE AND PROCESSING

Residues after processing

The fate of bixafen and its metabolite M21 (bixafen-desmethyl) during processing of raw agricultural commodity (RAC) was investigated in additional studies on soya beans, potatoes, maize, sorghum, wheat, oilseed rape and peanuts using important processing procedures. The 2013 JMPR already evaluated the effect of processing for rapeseeds, barley and wheat. 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).

Soya beans

Study by Lenz C. (2016, BIXAFEN_120)

The magnitude of residues of bixafen (comprising parent bixafen, bixafen-desmethyl and the total residue bixafen calc.) in/on the processed fractions of soya bean was investigated in two plots in the USA. In the same study also residues of bixafen metabolite bixafen-desmethyl-pyrazole-4-carboxylic acid (M44) were determined.

Two trials were performed in Illinois, USA (DR001-13PA) and Iowa, USA (DR002-13PA) during the 2013 growing season. Four spray applications of an SC formulation (125 g bixafen/L) were made at an application rate of 2.5 L/ha (equivalent to 312.5 g ai/ha) for each application and a nominal interval of 14 days. The soya bean seed samples to be processed were sampled 27 and 32 days after the last application at a growth stage of BBCH 89 (fully ripe). Soya bean seed (RAC) were stored frozen (-18 °C) until analysis for a maximum of 12 months. All processed commodities were stored less than 30 days prior to analysis.

The samples were analysed for the parent bixafen and bixafen-desmethyl using method 01013 at an LOQ of 0.01 mg/kg for bixafen and bixafen-desmethyl and 0.02 mg/kg for the total bixafen residue

expressed as parent equivalent. Residue data for M44 (bixafen-desmethyl-pyrazole-4-carboxylic acid) were obtained using a separate analytical method (method 01366). Procedural recoveries for all analytes were demonstrated, being within acceptable ranges (70–120%, RSD <20%).

The effect of processing on bixafen residues in soya bean seed has been investigated for flour, milk and refined, bleached, and deodorized oil (from cold pressing and solvent extraction), as well as for the by-products aspirated grain fraction (AGF), hulls and meal.

Generation of aspirated grain fraction (AGF)

To generate aspirated grain fractions, each whole soya bean seed sample was divided into two batches. Each batch was 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). Each batch was moved for 120 minutes. Light impurities were classified using the following sieves: 2360 micron (8 mesh); 2000 micron (10 mesh); 1180 micron (16 mesh); 850 micron (20 mesh); and 425 micron (40 mesh). After classification of each sample, the material passing through the 2360 micron sieve was recombined to produce one aspirated grain fraction (AGF). For both samples, the material that passed through the 425 micron screen was greater than half the weight of the total material passing through the 2360 micron screen, so all the material passing through the 2360 micron screen was recombined. A representative sample was removed and the ash content was determined.

Processing of soya milk

Representative samples of soya bean were cleaned by aspiration and screening. Light impurities were removed from the whole soybean by aspiration. After aspiration, the samples were screened to separate small foreign particles (screenings) from the soybean seed sample. Whole cleaned soybeans were washed and soaked in water for a minimum of 12 hours. Soaked beans were ground and filtered to separate the liquid (soya milk) and solids. Soya milk was heated to 91–96 °C and held for 9–11 minutes. The resulting fractions were soya milk and okara.

Processing of refined, bleached, and deodorized oil (RBD oil) by solvent extraction

For production of hulls, toasted meal, defatted soya flour, crude oil, and processed oil, whole soya beans were fed into a roller mill to crack the hull and liberate the kernel. After hulling, the material was passed through the aspirator to separate hull and kernel. Kernel moisture content was determined. For kernel material less than 13.5%, the moisture was adjusted to 13.5% and allowed to temper for a minimum of twelve hours.

Moisture adjusted kernel material was heated to 71–79–°C and flaked in a flaking roll with a gap setting of 0.2–0.33 mm. A portion of the flakes were removed for direct solvent extraction without extrusion. Remaining flakes were extruded in a continuous processor, where they were turned into collets by direct steam injection and compression. Collets left the processor at 93–127 °C. After extrusion, the collets were ground in a disc mill and then dried in an oven at 66–82 °C for 30–40 minutes. Ground collets were used for solvent extraction.

Ground collets and flakes were placed in separate stainless steel batch extractors and submerged in 49–60 °C hexane. After 30 minutes, the miscella (crude oil and hexane) was drained and fresh hexane was added to repeat the cycle two more times. Miscella from ground collets and flakes was combined.

The solvent was removed from the extracted flakes with warm air. Dried flakes were ground in a pin mill. A representative sample of defatted soya flour was collected.

Extracted collets were toasted by direct steam injection onto the material until the product temperature reached 103–106 °C. Steam injection was stopped and the material heated to 104–116 °C and

held for 30–60 minutes. After toasting, the product was cooled to room temperature before screening. Material was hand screened with a 10 mesh sieve to collect the required fraction. A representative sample of toasted meal was collected and stored frozen until analysis.

Miscella was passed through a laboratory vacuum evaporator unit to separate the crude oil and hexane. Crude oil was heated to 91–96 °C to remove hexane and filtered. After filtration, a representative sample of crude oil was collected.

The content of free fatty acids (FFA) was determined for the crude oil. Based on the free fatty acids, a weighed amount of crude oil and 14° Baumé sodium hydroxide was placed in a water bath at 20–24 °C and mixed for 90 minutes at high speed, and then for 20 minutes at low speed and 63–67 °C. Neutralized oil was then centrifuged. Refined oil was decanted and filtered. Resulting fractions were alkali refined oil and soap-stock.

Alkali refined oil was heated to 40–50 °C and an activated bleaching earth added (1.0% by weight). The solution was stirred and placed under vacuum. Temperature was increased to 85–100 °C and held for 10–15 minutes. After the bleaching period, the temperature was reduced to 58–68 °C. The bleached oil was vacuum filtrated. The resulting fractions were bleached oil and spent bleaching earth/filter aid.

Bleached oil was placed in a steam bath for 28–32 minutes under vacuum and temperature held between 220–230 °C. During the cooling period a 0.5% citric acid solution was added (1 ml per 100 grams of deodorized oil). The resulting fractions were deodorized oil (RBD oil) and deodorizer distillates.

Processing of refined, bleached, and deodorized oil (RBD oil) from cold pressing

Cleaned soya bean samples were moisture adjusted to 16.0% and allowed to equilibrate overnight. Moisture adjusted seed was fed into a mechanical screw press to separate a portion of the crude oil from the presscake. Resulting fractions were crude oil and presscake (meal). Crude oil was processed into RBD oil using the same procedure as oil from solvent extraction. Presscake recovered from this processing step was weighed and discarded.

In the following table the residues of bixafen and its metabolites and the resulting processing factors for soya products are summarized. Bixafen-desmethyl was always found below the LOQ of 0.01 mg/kg, except for AGF (0.105–0.195 mg/kg). However, since the contribution to total bixafen residues are negligible, processing factors were only based on parent residues.

Table 31 Summary of bixafen and its metabolites in soya beans and processed commodities (Lenz, C., 2016, BIXAFEN_120) following treatment with 4 × 0.31 kg ai/ha (14-day interval, 27–32 DALA)

Location, Year (Variety)	Matrix	Bixafen in mg/kg[a]	PF bixafen (also applies to total bixafen)	M44/45	PF M44/45
Trial DR001-13PA, USA, Stewardson (IL) 2013 (38X1RR)	Soya beans (RAC)	0.247	--	0.022	--
	AGF	154	620	0.037	1.7
	Flour	0.031	0.13	0.020	0.91
	Hulls	0.691	2.8	0.042	1.9
	Meal	0.034	0.14	0.023	1.0
	Milk	<0.01	<0.04	<0.01	<0.45
	Refined oil (cold)	0.353	1.4	<0.01	<0.45
	Refined oil (solv.)	0.123	0.50	<0.01	<0.45
Trial DR002-13PA, USA, Atlantic (IA) 2013 (Croplan R2T2501)	Soya beans (RAC)	0.106	--	0.021	--
	AGF	50.1	470	0.011	0.52
	Flour	<0.01	<0.09	0.025	1.2
	Hulls	0.837	7.9	0.046	2.2
	Meal	0.012	0.11	0.024	1.1

Location, Year (Variety)	Matrix	Bixafen in mg/kg[a]	PF bixafen (also applies to total bixafen)	M44/45	PF M44/45
	Milk	<0.01	<0.09	<0.01	<0.48
	Refined oil (cold)	0.140	1.3	<0.01	<0.48
	Refined oil (solv.)	0.042	0.40	<0.01	<0.48

[a] means of triplicate samples

RAC: raw agricultural commodity

Total: sum of bixafen and bixafen-desmethyl, expressed as bixafen equivalents

AGF: Aspirated grain fractions

Cold: cold pressed oil

Solv.: Solvent extracted oil

Study by Lenz C. (2016, BIXAFEN_091)

Additionally, a parallel crop field trial at exaggerated rates on soya beans was conducted in the USA (see residue trials details under soya beans above). Seeds collected from this plot were processed into aspirated grain fractions, hulls, meal and refined oil. The samples were processed according to the same protocols as described in the previous study by Lenz C. (2016, BIXAFEN_120).

In the following table the residues of bixafen and its metabolites and the resulting processing factors for soya products are summarized. Bixafen-desmethyl was always found below the LOQ of 0.01 mg/kg, except for AGF (0.11 mg/kg). However, since the contribution to total bixafen residues are negligible, processing factors were only based on parent residues.

Table 32 Summary of bixafen in soya beans and processed commodities (Lenz, C., 2016, BIXAFEN_91) following treatment with 2 × 0.56–0.57 kg ai/ha (12-day interval, 7 DALA)

Location, Year (Variety)	Matrix	Bixafen in mg/kg[a]	PF bixafen (also applies to total bixafen)
Trial J, USA, Stewardson (IL) 2014 (Pioneer 93Y84)	Soya beans (RAC)	0.046	-
	AGF	14.1	310
	Hulls	0.11	2.4
	Meal	<0.01	<0.22
	Refined oil (solv.)	0.016	0.35

[a] means of triplicate samples

RAC: raw agricultural commodity

Total: sum of bixafen and bixafen-desmethyl, expressed as bixafen equivalents

AGF: Aspirated grain fractions

Cold: cold pressed oil

Solv.: Solvent extracted oil

Potatoes

Study by Lenz C. (2017, BIXAFEN_092)

A parallel crop field trial at exaggerated rates on potatoes was conducted in the USA (see residue trials details under potatoes above). Tubers collected from this plot were processed into wet peel, granules/flakes and chips/fries. The samples were processed according to the following protocols.

Generation of potato flakes and wet peel

Potato tubers were batch steam peeled followed by scrubbing for 15–30 seconds. The potato peel was collected from the peeling and scrubbing process. The peeled potatoes were inspected and hand trimmed to remove additional peel, rot, green or otherwise damaged potatoes. The collected peel was hydraulically pressed and blended with the cut trim waste collected.

The peeled potatoes were cut into slabs, spray-washed in cold tap water for about 30 seconds to remove free starch. The potato slabs were pre-cooked at about 70–77 °C for 20 minutes. After cooling to less than 32 °C for about 20 minutes, the pre-cooked potato slabs were steam-cooked at 94–100 °C for 40–42 minutes. An aliquot of cooked potato slabs was mashed using a modified meat grinder and mixed for about 37 seconds in a mixer with an emulsion of pre-weighed food additives. The remaining potato slabs were discarded.

The cooked mash was dried into a thin sheet and was initially broken into large flakes by hand. The flakes were then fed into a hammermill for uniform milling of the potato flakes. The moisture of the potato flakes was at 9.00% for the control and 5.08% for the treated flakes.

Generation of potato chips/fries

The aliquot of washed potatoes was peeled using a restaurant style peeler for 30 seconds. The peeled potatoes were inspected by hand and trimmed if necessary, to remove rot, green or otherwise damaged potato tissue. The peeled potatoes were cut into thin slices (~1.6 mm).

The potato slices were placed in a tub of hot water to remove free starch and drained over a screen to remove excess water and were then fried in oil at about 163–191 °C for about 80–105 seconds. The fried potato chips were drained, salted and inspected. Undesirable chips were removed.

Potato tubers and processed commodities were analysed for bixafen and bixafen-desmethyl by residue analytical method 01012 with an LOQ of 0.01 mg/kg.

Table 33 Summary of bixafen and its metabolites in potatoes and processed commodities (Lenz, C., 2017, BIXAFEN_092) following treatment with 4 × 0.27–0.28 kg ai/ha (6–8 day interval, 7 DALA)

Location, Year (Variety)	Matrix	Bixafen in mg/kg	PF bixafen (also applies to total bixafen)
Trial N, USA, American Falls (ID) 2014 (Russet Burbank)	Tubers (RAC)	<0.01	--
	Wet peel	0.012	NP
	Granules/flakes	<0.01	NP
	Chips/fries	<0.01	NP

RAC: raw agricultural commodity

NP: not possible, RAC <LOQ

Total: sum of bixafen and bixafen-desmethyl, expressed as bixafen equivalents

Maize grain

Study by Lenz C. (2016, BIXAFEN_121)

The magnitude of residues of bixafen (comprising parent bixafen, bixafen-desmethyl and the total residue bixafen calc.) in/on the processed fractions of maize grain was investigated in two plots in the USA.

Two trials were performed in Illinois, USA (DR003-13PA) and Iowa, USA (DR004-13PA) during the 2013 growing season. Two spray applications of an SC formulation (125 g bixafen/L), were made at an application rate of 2.5 L/ha (equivalent to 312.5 g ai/ha) for each application and a nominal interval of 14

days. The maize grain samples to be processed were sampled 13 and 20 days after the last application at a growth stage of BBCH 89 (fully ripe). Maize grain (RAC) was stored frozen (-18 °C) until analysis for a maximum of 9 months. All processed commodities were stored less than 30 days prior to analysis.

The samples were analysed for the parent bixafen and bixafen-desmethyl using method 01013 at a LOQ of 0.01 mg/kg for bixafen and bixafen-desmethyl and 0.02 mg/kg for the total bixafen residue expressed as parent equivalent. Procedural recoveries for all analytes were demonstrated, being within acceptable ranges (70–120%, RSD <20%).

The effect of processing on bixafen residues in maize grain has been investigated for flour, bran, germ, grits, meal and oil from dry milling and for germ, oil, starch and steep water from wet milling, as well as for the by-product aspirated grain fraction (AGF).

Generation of aspirated grain fraction (AGF)

The sample was processed according to a similar protocol as for the generation of AGF from soya beans (see above).

Dry milling - flour, bran, germ, grits, meal and oil

Representative samples of maize grain were cleaned by aspiration and screening. Representative samples of cleaned whole corn grain were moisture conditioned to 21% followed by disc milling to crack the kernel. Corn stock from the mill was dried in an oven for 30 minutes at 54–71 °C. Dried cornstock was screened on a 1/8-inch screen (3.2 mm) to separate bran, germ, and large grits from grits, meal and flour.

Material below 3.2 mm (grits, meal, and flour) was separated by sieving using the following two screens: 14 mesh (1.4 mm) and 62 mesh (0.25 mm) sieves. The fraction on top of 14 mesh sieve was "grits"; the fraction on top of the 62 mesh sieve was "meal" and the fraction through the 62 mesh sieve was "flour."

Material on top of the 1/8-inch screen (3.2 mm; bran, germ, and large grits) was screened again using a 13/64-inch screen (5.2 mm). Material above the screen was aspirated to remove hull from germ with attached hull and endosperm. Germ with attached hull and endosperm was passed through the disc mill and flaking roll and screened. Material above screen was aspirated to remove hull from the germ. Material through the 1/8-inch screen (3.2 mm) was added to the large grit fraction weight for mass balance purposes.

All material passing through the 12/64-inch screen (4.8 mm) was separated into germ and large grits. If necessary, germ may be milled, flaked, screened, and/or aspirated to remove endosperm.

The two germ fractions are combined and dried at 54–71 °C to a final moisture of 14–16%. Requested grits, meal, flour, and germ fractions were collected and placed into freezer storage.

Germ material was heated to 71-79°C in a mixer and held for 10 minutes. Following heating, the material was flaked at a gap setting of 0.18–0.25 mm. Flaked kernel material was placed in stainless steel batch extractors and submerged in hexane at 49–60 °C. After 30 minutes, the miscella (crude oil and hexane) was drained extraction was repeated twice. Flakes were desolventized with ambient air to remove residual hexane. Resulting fractions from solvent extraction were miscella and solvent extracted germ flakes. Miscella was passed through a laboratory vacuum evaporator to separate the crude oil and hexane. Crude oil was then heated to 91–96 °C for hexane removal, filtered, and collected for refining.

For alkali refining, the free fatty acid (FFA) content of the crude oil is determined. Based on the FFA, weighed amounts of crude oil and 16 degree Baume sodium hydroxide were placed in a water bath and mixed at high speed for 15 minutes at 20-24°C and for 12 minutes at low speed at 63–67 °C. Neutralized oil and soapstock were separated using centrifugation. Alkali refined oil samples were decanted and filtered prior to bleaching. The soapstock was discarded. Alkali refined oil was heated to 40–50 °C and an activated

bleaching earth added (1.0% by weight of oil). The solution was stirred and placed under vacuum. Temperature was increased to 85–100 °C for 10 to 15 minutes. Finally, the bleached oil was filtered. Resulting fractions were bleached oil and spent bleaching earth/filter aid.

Bleached oil was heated in a steam bath at 220–230 °C for 28–32 minutes under vacuum. The oil was then allowed to cool and citric acid (0.5%) was added at 1% of oil content. Resulting fractions were refined-bleached-deodorized oil (RBD oil) and deodorizer distillates. The oil samples were stored frozen until analysis.

Wet milling - germ, oil, starch and steep water

A representative sample of dried and cleaned maize grain was steeped in 49–54 °C sulfurous acid (0.1–0.2%) for 22–48 hours. After steeping, steepwater was drained and a representative fraction collected and placed into frozen storage. Steeped whole corn was passed through a disc mill and a majority of the germ and hull was removed using a water centrifuge. Germ and hull were dried at 74–91 °C to obtain a final moisture between 5–10%. After drying, the germ and hull were separated using aspiration and screening. Aliquots of wet milled germ were stored frozen until analysis.

Cornstock (without germ and hull) ground in the disc mill was screened at 325 mesh (50 µm) with bran (hull material) remaining on top of the screen which was discarded. Process water passing through the screen was separated into starch and gluten by centrifugation. The starch was dried in an oven at 54–71 °C until the moisture content was less than 15%. An aliquot of the starch was stored frozen until analysis.

Germ samples were moisture conditioned to 12%, heated to 88–104 °C in a mixer, flaked with a gap setting of 0.2 mm, and pressed to generate crude oil. Aliquots of crude oil and presscake were stored frozen until analysis.

Presscake was extracted three times with hexane at 49–60 °C and the miscella was combined. The spent presscake was desolventized with ambient air. Resulting fractions from solvent extraction were miscella and solvent extracted presscake (germ cake). The miscella was vacuum evaporated to remove the solvent from the crude oil which was finally heated to 91–96 °C to remove residues of hexane. The combined crude oils from expelling and solvent extraction were alkali refined, bleached, and deodorized utilizing the same procedure as for dry milling.

In the following table the residues of bixafen and the resulting processing factors for maize grain products are summarized. Bixafen-desmethyl was always found below the LOQ of 0.01 mg/kg. Since the contribution to total bixafen residues are negligible, processing factors were only based on parent residues.

Table 34 Summary of bixafen in maize grain and processed commodities (Lenz, C., 2016, BIXAFEN_121) following treatment with 2 × 0.31 kg ai/ha (14-day interval, 13–20 DALA)

Location, Year (Variety)	Matrix	Bixafen in mg/kg[a]	PF bixafen (also applies to total bixafen)
DR003-13PA, USA, Stewardson (IL) 2013 (P1184AM1)	Maize grain (RAC)	0.016	--
	AGF	2.9	180
	Bran (dry milling)	0.041	2.6
	Flour (dry milling)	<0.01	<0.63
	Germ (dry milling)	0.01	0.63
	Germ (wet milling)	0.01	0.63
	Grits (dry milling)	<0.01	<0.63
	Meal (dry milling)	<0.01	<0.63
	Refined oil (dry milling)	0.014	0.88
	Refined oil (wet milling)	0.027	1.7
	Starch (wet milling)	<0.01	<0.63
DR004-13PA, USA	Maize grain (RAC)	0.028	--

Location, Year (Variety)	Matrix	Bixafen in mg/kg[a]	PF bixafen (also applies to total bixafen)
Atlantic (IA) 2013 (Dekalb 62-98)	AGF	4.5	160
	Bran (dry milling)	0.102	3.6
	Flour (dry milling)	0.025	0.89
	Germ (dry milling)	0.021	0.75
	Germ (wet milling)	0.040	1.4
	Grits (dry milling)	<0.01	<0.36
	Meal (dry milling)	0.021	0.75
	Refined oil (dry milling)	0.026	0.93
	Refined oil (wet milling)	0.051	1.8
	Starch (wet milling)	<0.01	<0.36

[a] means of triplicate samples

RAC: raw agricultural commodity

Total: sum of bixafen and bixafen-desmethyl, expressed as bixafen equivalents

AGF: Aspirated grain fractions

Study by Lenz C. (2016, BIXAFEN_093)

Additionally, a parallel crop field trial at exaggerated rates on maize was conducted in the USA (see residue trials details under maize above). Maize grain collected from this plot was processed into aspirated grain fractions, starch, refined oil (wet), grits, flour, germ, meal and refined oil (dry). The samples were processed according to the same protocols as described in the previous study by Lenz C. (2016, BIXAFEN_121).

In the following table the residues of bixafen and the resulting processing factors for maize products are summarized. Bixafen-desmethyl was always found below the LOQ of 0.01 mg/kg. Since the contribution to total bixafen residues are negligible, processing factors were only based on parent residues.

Table 35 Summary of bixafen in maize grain and processed commodities (Lenz, C., 2016, BIXAFEN_092) following treatment with 2 × 0.56 kg ai/ha (20-day interval, 27 DALA)

Location, Year (Variety)	Matrix	Bixafen in mg/kg[a]	PF bixafen (also applies to total bixafen)
2014_RES-BAN1259-D, USA Stewardson (IL), 2014 (Burrus 6F74AMX)	Maize grain (RAC)	0.010	--
	AGF	1.7	160
	Flour (dry milling)	0.016	1.5
	Germ (dry milling)	<0.01	<0.93
	Grits (dry milling)	<0.01	<0.93
	Meal (dry milling)	0.015	1.4
	Refined oil (dry milling)	<0.01	<0.93
	Refined oil (wet milling)	0.020	1.9
	Starch (wet milling)	<0.01	<0.93

[a] means of triplicate samples

RAC: raw agricultural commodity

Total: sum of bixafen and bixafen-desmethyl, expressed as bixafen equivalents

AGF: Aspirated grain fractions

Sorghum

Study by Lenz C. (2016, BIXAFEN_094)

Two parallel crop field trial plots at exaggerated rates on sorghum were conducted in the USA (see residue trials details under sorghum above). Sorghum grain collected from one plot was processed into aspirated

grain fractions, sweet sorghum stalks from the other plot into syrup. The samples were processed according to the following protocols.

Generation of aspirated grain fraction (AGF)

The sample was processed according to a similar protocol as for the generation of AGF from soya beans (see above).

Sweet sorghum processing

Leaf material was removed from sweet sorghum stalks (RAC). Stalks were fed into a horizontal roll press to express the raw juice. Resulting products were raw juice and bagasse (fiber). During clarification, raw juice was screened through a 100 mesh sieve to remove material suspended in the juice. Screened juice was heated to 71–82 °C, removed from heat and allowed to settle for 1.5 hours. During this time period, any material that rose to the top was skimmed off the surface. After the settling period, juice was decanted to separate any material that settled to the bottom of the container from the juice.

Clarified juice was boiled at 97–108 °C until a 73–80°Brix density was reached. Density was measured with a refractometer. Any material that rose to the top during this process was removed by screening. The resulting sweet sorghum syrup was allowed to cool to 60–71 °C and filtered through a 45 mesh screen.

In the following table the residues of bixafen and the resulting processing factors for sorghum products are summarized.

Table 36 Summary of bixafen in sorghum grain and stalks and processed commodities thereof (Lenz, C., 2016, BIXAFEN_094) following treatment with 2 × 0.56 kg ai/ha (20-day interval, 30–31 DALA)

Location, Year (Variety)	Matrix	Bixafen in mg/kg	PF bixafen	Total in mg/kg	PF total
2014-RES- BAN1260-H Groom (TX), USA 2014 *Variety: Y373 (grain sorghum)* *Variety: M81-E (sweet sorghum)*	Sorghum grain (RAC)	0.69	--	0.70	--
	AGF	18	26	18	26
	Stalks (RAC)	1.8	--	2.1	--
	Syrup	0.30	0.17	0.31	0.15

RAC: raw agricultural commodity

Total: sum of bixafen and bixafen-desmethyl, expressed as bixafen equivalents

AGF: Aspirated grain fractions

Wheat

Study by Lenz C. (2016, BIXAFEN_095)

One parallel crop field trial at exaggerated rates on wheat was conducted in the USA (see residue trials details under wheat above). Wheat grain collected from the plot was processed into aspirated grain fractions, bran, flour, germ, middlings and shorts. The samples were processed according to the following protocols.

Generation of aspirated grain fraction (AGF)

The sample was processed according to a similar protocol as for the generation of AGF from soya beans (see above).

Processing of germ

Cleaned wheat grain was moisture adjusted (tempered) to 16%. After tempering for 1–1.5 hours, the grain was passed through a disc mill. Ground material was sieved on an 8 and 14 mesh sieve and a 34 mesh sieve. Material on top of the 34-mesh sieve was aspirated to remove bran from the germ and endosperm. Germ and endosperm were passed through a reduction mill. Flattened germ and reduced endosperm were separated by sifting. Recovered germ material was milled/sieved additional times to remove more endosperm.

Processing of wheat flour

Cleaned wheat was moisture conditioned (tempered) to 16.5–17.5% depending on the physical property of the wheat. Breaking of tempered wheat grain was accomplished by three break rolls. After passing through the break rolls, ground material was sieved over 140 µm and 800 µm sieves. Material exiting the break rolls passed over the 140 µm sieve first, followed by the 800 µm screen. Material passing through the 140 µm screen is "Break Flour", material passing through the 800 µm screen is middlings, with material exiting the end being bran (Coarse).

After sampling the middlings, the remainder was poured into the feed hopper of the reduction system. Reduction is achieved through two reduction rolls. After passing through the reduction rolls, the material was sieved through a 160 µm screen. Material passing through the screen is “reduction flour”, with the remainder being “shorts”. Break flour and reduction flour were combined.

The coarse bran is conveyed by beater bars over a 128 µm screen. Material passing through the screen is "Shorts" and is added to "Shorts" from the reduction mill. Material passing over the screen and exiting the end is "Bran."

In the following table the residues of bixafen and the resulting processing factors for wheat products are summarized.

Table 37 Summary of bixafen in wheat grain and processed commodities (Lenz, C., 2016, BIXAFEN_095) following treatment with 2 × 0.56 kg ai/ha (33-day interval, 30 DALA)

Location, Year (Variety)	Matrix	Bixafen in mg/kg	PF bixafen	Total in mg/kg	PF total
2014-RES-BAN1261-C Stewardson (IL), USA 2014 (Becks 129)	Wheat grain (RAC)	0.083	--	0.10	--
	AGF	17	200	18	180
	Bran	0.067	0.81	0.088	0.88
	Flour	0.010	0.12	0.020	0.20
	Middlings	0.015	0.18	0.025	0.25
	Shorts	0.027	0.33	0.038	0.38
	Germ	0.047	0.57	0.074	0.74

RAC: raw agricultural commodity

Total: sum of bixafen and bixafen-desmethyl, expressed as bixafen equivalents

AGF: Aspirated grain fractions

Oilseed rape

Study by Glaubitz J. (2013, BIXAFEN_122)

Winter rape seed samples (RAC) intended for processing originated from two supervised residue trials in Germany (12-3402-01) and France (12-3402-02) during the 2012 growing season. Two spray applications of an EC formulation containing bixafen (75 g/L) were made at an application rate of 2.4 L/ha (equivalent to 180 g ai/ha) for each application with a nominal interval of 14 days.

The rape seeds were sampled 32 or 40 days after the last application at a growth stage of BBCH 89 (fully ripe).

The samples were analysed for the parent bixafen and bixafen-desmethyl using method 01013 at a LOQ of 0.01 mg/kg for bixafen and bixafen-desmethyl each. Procedural recoveries for all analytes were demonstrated being within acceptable ranges (70–120%, RSD <20%).

Rape seeds (RAC) and processed commodities were stored frozen (-18 °C) until analysis for a maximum of 351 days.

The effect of processing in rape seed has been investigated for refined oil, as well as for the by-products pomace, meal and oil at different processing stages according to the following protocols.

Processing of winter rape seeds into crude oil - cold pressing and solvent extraction of oil

The seeds were cleaned by sieving. The cleaned rape seeds were dried in a hot-air stove at 80 °C for 30–120 min and conditioned to a water content of about 8%. The conditioned rape seeds were pressed in a screw press yielding oil and pomace.

An aliquot of the pomace was deep-frozen and pulverized to meal in a cutter with a low amount of dry ice. After sublimation of the dry ice, an aliquot of the press cake meal was extracted with n-hexane in a Soxhlet apparatus. The fractions resulting from the solvent extraction step were miscella (a mixture of n-hexane and solvent extracted oil) and extracted press cake meal. The miscella was distilled in a vacuum rotary evaporator, yielding the fraction oil, solvent extracted.

Both aliquots of oil fractions (oil, screwpressed and oil, solvent extracted) were mixed yielding the sample of crude oil. An aliquot of the solvent extracted press cake meal was steam-distilled for about 30 min in a special apparatus (= toasting) in order to remove the n-hexane. Subsequently, the material was dried in a hot-air stove for 30 min at 80–100 °C to a dry substance content of >86%, yielding the fraction extracted meal.

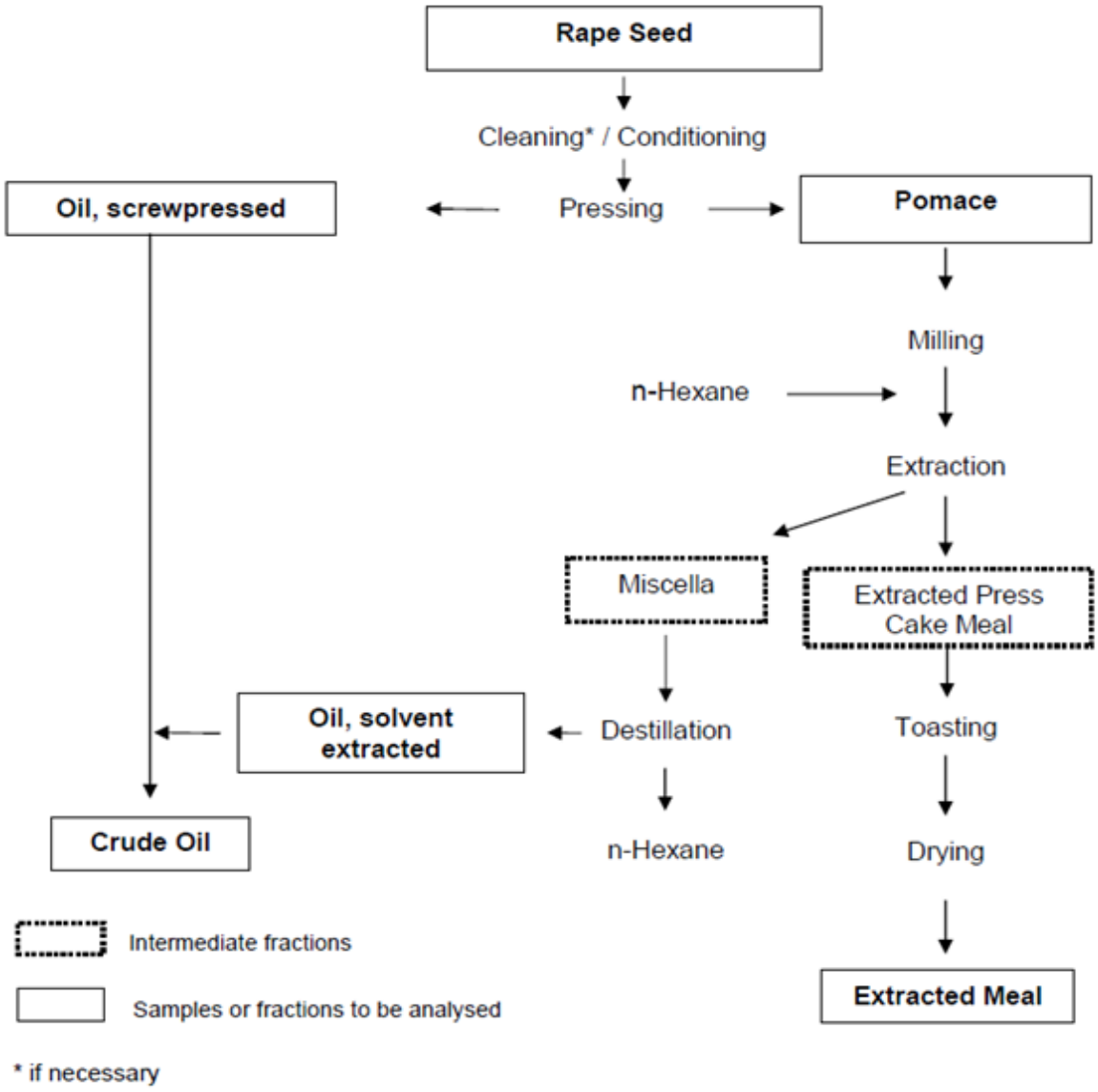


Figure 5 Processing flowchart for processing of rape seeds into pomace; oil, screwpressed; oil, solvent extracted; crude oil; meal

Refinement of crude oil into refined oil

Pre-clarification (hydration and desliming)

The crude oil was heated up to 60–70 °C while stirring. After addition of 10% water, the mixture of water and crude oil was heated up to 85–90 °C and then maintained for approx. 45 min. at 85 °C while stirring. Then the stirring was stopped and after phase separation the watery phase including mucilage was removed by centrifugation. To the oily phase approx. 1% of concentrated phosphorus acid was added. The mixture was heated up to approx. 85 °C and then maintained for approx. 45 min at that temperature while stirring. 10% water was added. After switching off the stirrer, the mixture of water and oily phase remained at approx. 85 °C until separation of the phases. Then the watery phase including flocculated precipitation was removed by centrifugation and an aliquot of crude oil, pre-clarified was sampled.

Neutralisation

The oily phase was heated up to approx. 90 °C while stirring. Then sodium hydroxide solution (amount depending on acidity of the oil phase) was added and the mixture was stirred for approx. 20 min. After addition of 10% water and further 5 min of stirring the stirrer was switched off and the phases were allowed to separate. After phase separation the watery phase was removed.

Washing

The remaining neutralised crude oil was washed by the addition of 10% water. The mixture was stirred for 20 min at 90 °C. After phase separation the aqueous phase was removed. These steps were repeated until the pH of the washing water was about 7. After washing, the acid-number of the oil was determined. The neutralisation and washing process were repeated until the acid number of the oil was ≤0.12.

Drying

The oil was heated up to approx. 95 °C while stirring. After addition of citric acid the oil was dried under vacuum until no more water escaped from the oil. Then an aliquot of the crude oil, neutralised was sampled.

Bleaching

The neutralised oil was transferred into a bleaching flask, where it was heated up to 90–100 °C while stirring. After addition of 1% podsol (referring to the used oil) the oil was bleached for 5 min without vacuum and 20 min with vacuum.

Filtration

The oil was transferred into a steaming flask via a glass frit covered with diatomaceous earth in order to remove the podsol.

Deodorization

The oil was heated up to 160 °C under vacuum while stirring. After the temperature reached 160 °C, steam was transferred through the oil in order to expel fatty acids, odour and taste influencing compounds as well as other volatile compounds. The mixture was heated to 205 °C and maintained at that temperature for about one hour. After the temperature cooled down to 160 °C the steam supply was stopped and then the oil was dried under vacuum until a temperature of ≤ 80 °C was reached. The refined oil was sampled.

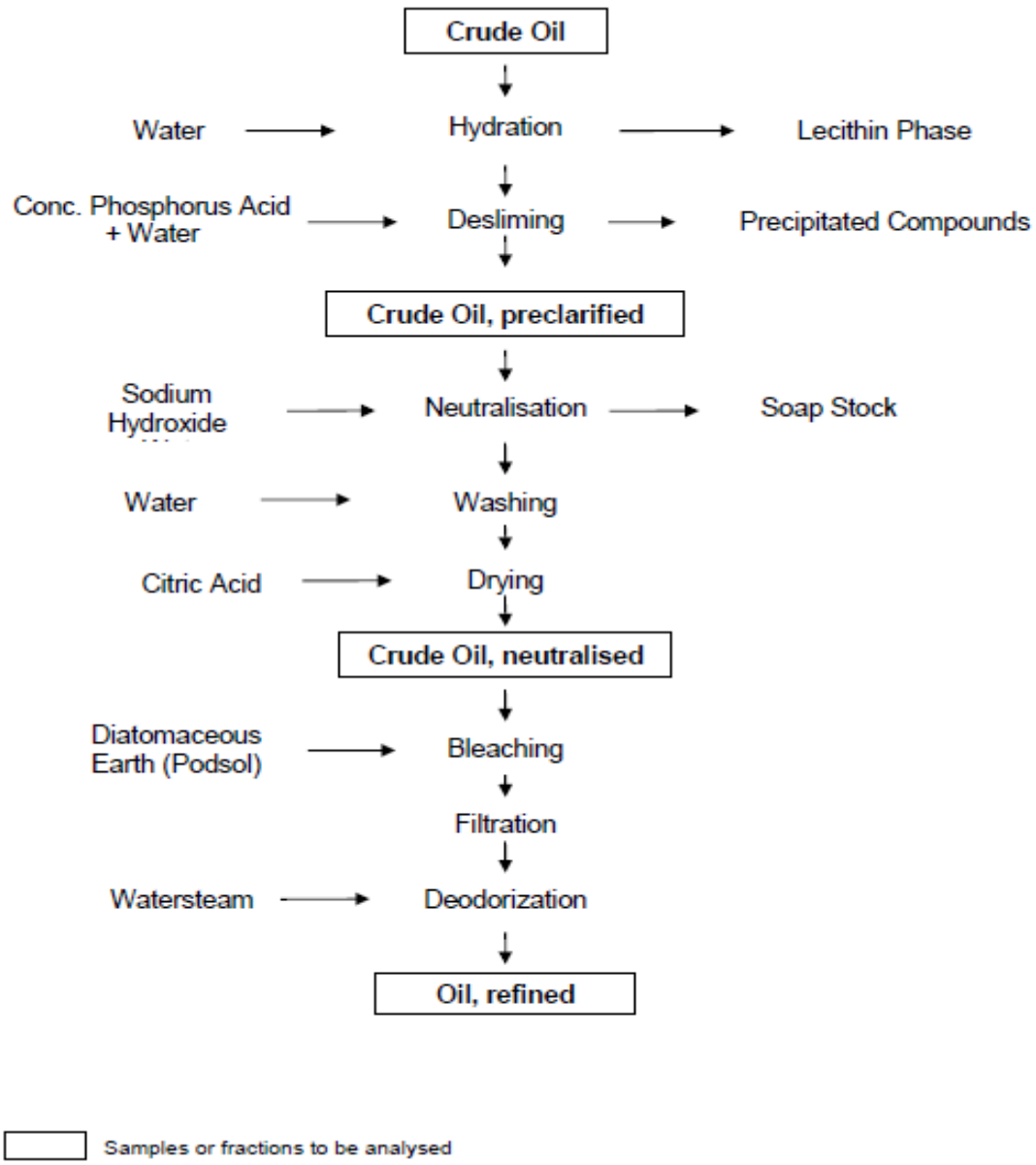


Figure 6 Processing flowchart for processing of rape, seeds: Refinement of crude oil

In the following table the residues of bixafen and the resulting processing factors for oilseed rape products are summarized.

Table 38 Summary of bixafen in oilseed rape and processed commodities (Glaubitz J. 2013, BIXAFEN_122) following treatment with 2 × 0.18 kg ai/ha (14-day interval, 32–40 DALA)

Location, Year (Variety)	Matrix	Bixafen in mg/kg	PF bixafen	M21 in mg/kg	Total in mg/kg	PF total
12-3402-01, Germany, Burscheid 2012 (Vision)	Seed (RAC)[1]	0.057	--	0.035	0.092	--
	Oil (screwpressed)	0.11	1.9	0.041	0.151	1.6
	Pomace	0.080	1.4	0.055	0.135	1.5
	Meal (extracted)	0.047	0.82	0.063	0.11	1.2
	Oil, solv. extracted)	0.14	2.5	0.021	0.161	1.8
	Oil, crude	0.14	2.5	0.028	0.168	1.8
	Oil, pre-clarified	0.13	2.3	0.027	0.157	1.7

Location, Year (Variety)	Matrix	Bixafen in mg/kg	PF bixafen	M21 in mg/kg	Total in mg/kg	PF total
	Oil, neutralized	0.13	2.3	<0.01	0.14	1.5
	Oil, refined	0.12	2.1	<0.01	0.13	1.4
12-3402-02, France (South), Velleron 2012 (Hybrilux)	Seed (RAC)[a]	0.025	--	<0.01	0.035	--
	Oil (screwpressed)	0.050	2.0	<0.01	0.060	1.7
	Pomace	0.046	1.8	<0.01	0.056	1.6
	Meal (extracted)	0.018	0.72	<0.01	0.028	0.80
	Oil, solv. extracted)	0.067	2.7	<0.01	0.077	2.2
	Oil, crude	0.060	2.4	<0.01	0.070	2.0
	Oil, pre-clarified	0.062	2.5	<0.01	0.072	2.1
	Oil, neutralized	0.060	2.4	<0.01	0.070	2.0
	Oil, refined	0.053	2.1	<0.01	0.063	1.8

[a] mean of two samples

RAC: raw agricultural commodity

M21: bixafen-desmethyl

Total: sum of bixafen and bixafen-desmethyl, expressed as bixafen equivalents

Peanuts

Study by Lenz C. (2016, BIXAFEN_096)

One parallel crop field trial at exaggerated rates on peanuts was conducted in the USA (see residue trials details under peanuts above). Nutmeat collected from the plot was processed into meal and refined oil. The samples were processed according to the following protocols.

Production of peanut oil and meal

Bulk peanut nutmeat samples were dried at 54–71 °C to a moisture content of 7–10% followed by moisture conditioning to 12%. The nutmeat was heated to 85–104 °C and pressed in an expeller to liberate a portion of the crude oil. Resulting products from expelling were crude oil and presscake. The presscake was ground in a disc mill prior to solvent extraction.

Ground presscake was placed in a stainless steel batch extractor and submerged in 49–60 °C hexane. After 30 minutes, the miscella (crude oil and hexane) was drained. The extraction was repeated twice and the extracts (miscella) combined. The remaining presscake was desolventised and an aliquot stored frozen until analysis. The miscella was vacuum evaporated to separate the crude oil and hexane. Crude oil was heated to 91–96 °C for hexane removal.

The combined crude oil recovered from expelling and solvent extraction was filtered and alkali refined. The free fatty acid (FFA) of the oil was determined. Depending on the FFA, a certain amount of crude oil and 16 degree Baume sodium hydroxide was mixed for 30 minutes at 20–24 °C at high speed followed by low speed at 63–67 °C for 12 minutes. The neutralized oil was centrifuged followed by bleaching, the soapstock was discarded.

For bleaching, the refined oil was heated to 40–50 °C, activated bleaching earth was added and the solution placed under vacuum. The temperature was increased to 85–100 °C and held for 10 to 15 minutes. After reaction, the bleached oil was filtered. Bleached oil was heated to 220–230 °C for 28–32 minutes under vacuum. During the cooling period a 0.5% citric acid solution was added.

In the following table the residues of bixafen the resulting processing factors for peanut products are summarized. Bixafen-desmethyl was always found below the LOQ of 0.01 mg/kg. Since the contribution to total bixafen residues are negligible, processing factors were only based on parent residues.

Table 39 Summary of bixafen in peanut nutmeat and processed commodities (Lenz, C., 2016, BIXAFEN_96) following treatment with 4 × 0.29 kg ai/ha (13–14-day interval, 14 DALA)

Location, Year (Variety)	Matrix	Bixafen in mg/kg	PF bixafen (also applies to total bixafen)
2014RES-BAN1251-A, USA, Tallassee (AL) 2014 (GA06)	Nutmeat (RAC) [a]	0.018	--
	Meal	<0.01	<0.57
	Refined oil	0.039	2.2

[a] means of triplicate samples

RAC: raw agricultural commodity

Total: sum of bixafen and bixafen-desmethyl, expressed as bixafen equivalents

In summary, the following processing factors were derived for bixafen and its metabolites:

Table 40 Summary of processing factors for bixafen and the total residue

	Bixafen		Total	
Matrix	Individual PF	Median or best estimate	Individual PF	Median or best estimate
Soya beans				
AGF	310, 470, 620	470	Covered by bixafen	
Flour	<0.09, 0.13	0.11	Covered by bixafen	
Hulls	2.4, 2.8, 7.9	2.8	Covered by bixafen	
Meal	0.11, 0.14, <0.22	0.14	Covered by bixafen	
Milk	<0.04, <0.09	0.065	Covered by bixafen	
Refined oil (cold)	1.3, 1.4	1.4	Covered by bixafen	
Refined oil (solv.)	0.35, 0.40, 0.50	0.40	Covered by bixafen	
Potatoes				
Wet peel	NP	NP	NP	NP
Granules/flakes	NP	NP	NP	NP
Chips/fries	NP	NP	NP	NP
Maize				
AGF	160, 160, 180	160	Covered by bixafen	
Bran (dry milling)	2.6, 3.6	3.1	Covered by bixafen	
Flour (dry milling)	<0.63, 0.89, 1.5	0.89	Covered by bixafen	
Germ (dry milling)	0.63, 0.75, <0.93	0.75	Covered by bixafen	
Germ (wet milling)	0.63, 1.4	1.0	Covered by bixafen	
Grits (dry milling)	<0.36, <0.63, <0.93	<0.63	Covered by bixafen	
Meal (dry milling)	<0.63, 0.75, 1.4	0.75	Covered by bixafen	
Refined oil (dry milling)	0.88, 0.93, <0.93	0.93	Covered by bixafen	
Refined oil (wet milling)	1.7, 1.8, 1.9	1.8	Covered by bixafen	
Starch (wet milling)	<0.36, <0.63, <0.93	<0.63	Covered by bixafen	
Sorghum				
AGF (grain sorghum)	26	26	26	26
Syrup (sweet sorghum)	0.17	0.17	0.15	0.15
Wheat				
AGF	200	200	180	180
Bran	JMPR 2013: 2.4, 2.6, 2.8, 3.5 (whiteflour bran) JMPR 2020: 0.81 Total: 0.81, 2.4, 2.6, 2.8, 3.5	2.6	JMPR 2013: 2.4, 2.5, 2.7, 4 (whiteflour bran) JMPR 2020: 0.88 Total: 0.88, 2.4, 2.5, 2.7, 4	2.5
Flour	JMPR 2013: 0.2, 0.2, 0.25, 0.5 (whiteflour) JMPR 2020: 0.12 Total: 0.12, 0.2, 0.2, 0.25, 0.5	0.20	JMPR 2013: 0.33, 0.33, 0.4, 0.66 (whiteflour) JMPR 2020: 0.20 Total: 0.20, 0.33, 0.33, 0.4, 0.66	0.33
Middlings	0.18	0.18	0.25	0.25
Shorts	0.33	0.33	0.38	0.38

	Bixafen		Total	
Matrix	Individual PF	Median or best estimate	Individual PF	Median or best estimate
Germ	JMPR 2013: 0.6, 0.75, 1.2, 1.5 JMPR 2020: 0.57 Total: 0.57, 0.6, 0.75, 1.2, 1.5	0.75	JMPR 2013: 0.8, 0.83, 1.3, 2.7 JMPR 2020: 0.74 Total: 0.74, 0.8, 0.83, 1.3, 2.7	0.83
Oilseed rape				
Oil (screwpressed)	JMPR 2013: <0.5, <1 JMPR 2020: 1.9, 2.0 Total: <0.5, <1, 1.9, 2.0	1.4	JMPR 2013: <0.66, <1 JMPR 2020: 1.6, 1.7 Total: <0.66, <1, 1.6, 1.7	1.3
Pomace	JMPR 2013: 0.5, 1 JMPR 2020: 1.4, 1.8 Total: 0.5, 1, 1.4, 1.8	1.2	JMPR 2013: 0.66, 1 JMPR 2020: 1.5, 1.6 Total: 0.66, 1, 1.5, 1.6	1.2
Meal (extracted)	JMPR 2013: <0.5, 2 JMPR 2020: 0.72, 0.82 Total: <0.5, 0.72, 0.82, 2	0.77	JMPR 2013: 0.66, 1.5 JMPR 2020: 0.8, 1.2 Total: 0.66, 0.8, 1.2, 1.5	1.0
Oil, solv. extracted)	JMPR 2013: <0.5, 2 JMPR 2020: 2.5, 2.7 Total: <0.5, 2, 2.5, 2.7	2.2	JMPR 2013: <0.66, 1.5 JMPR 2020: 1.8, 2.2 Total: <0.66, 1.5, 1.8, 2.2	1.6
Oil, crude	JMPR 2013: 0.5, 1 JMPR 2020: 2.4, 2.5 Total: 0.5, 1, 2.4, 2.5	1.7	JMPR 2013: 0.66, 1 JMPR 2020: 1.8, 2.0 Total: 0.66, 1, 1.8, 2.0	1.4
Oil, pre-clarified	JMPR 2013: <0.5, 1 JMPR 2020: 2.3, 2.5 Total: <0.5, 1, 2.3, 2.5	1.6	JMPR 2013: <0.66, 1 JMPR 2020: 1.7, 2.1 Total: <0.66, 1, 1.7, 2.1	1.4
Oil, neutralized	JMPR 2013: <0.5, 2 JMPR 2020: 2.3, 2.4 Total: <0.5, 2, 2.3, 2.4	2.2	JMPR 2013: <0.66, 1.5 JMPR 2020: 1.5, 2.0 Total: <0.66, 1.5, 1.5, 2.0	1.5
Oil, refined	JMPR 2013: <0.5, 2 JMPR 2020: 2.1, 2.1 Total: <0.5, 2, 2.1, 2.1	2.0	JMPR 2013: <0.66, 1.5 JMPR 2020: 1.4, 1.8 Total: <0.66, 1.4, 1.5, 1.8	1.4
Peanuts				
Meal	<0.57	<0.57	Covered by bixafen	
Refined oil	2.2	2.2	Covered by bixafen	

Total: sum of bixafen and bixafen-desmethyl, expressed as bixafen equivalents

NP: not possible, RAC <LOQ

Table 41 Summary of processing factors for M44/45

	M44/45	
Matrix	Individual PF	Median or best estimate
Soya beans		
AGF	0.52, 1.7	1.1
Flour	0.91, 1.2	1.0
Hulls	1.9, 2.2	2.0
Meal	1.0, 1.1	1.1
Milk	<0.45, <0.48	<0.47
Refined oil (cold)	<0.45, <0.48	<0.47
Refined oil (solv.)	<0.45, <0.48	<0.47

APPRAISAL

Bixafen (ISO common name) is a pyrazole-carboxamide fungicide used to control diseases on multiple crops. Bixafen inhibits fungal respiration by binding to mitochondrial respiratory complex II. It was considered for the first time by the 2013 JMPR for toxicology and residues, when an ADI of 0–0.02 mg/kg bw and an ARfD of 0.2 mg/kg bw were established. Bixafen was last reviewed for residues by the 2016 JMPR, where residues in rotational crops were evaluated and recommendations were made for maximum residue levels in plant and animal commodities.

The 2013 JMPR recommended the following residue definition for bixafen:

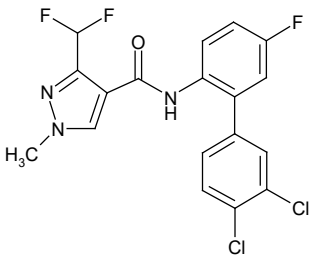
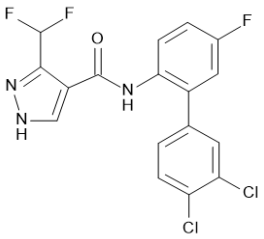
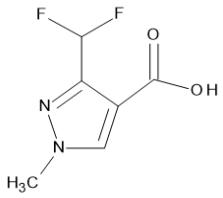
Definition of the residue for compliance with the MRL for plant commodities: *bixafen*

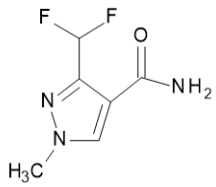
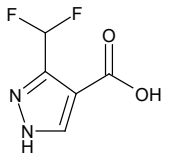
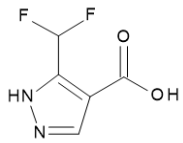
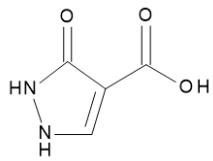
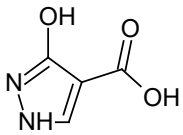
Definition of the residue for compliance with the MRL for animal commodities and for dietary risk assessment for plant and animal commodities: *sum of bixafen and N-(3',4'-dichloro-5-fluorobiphenyl-2-yl)-3-(difluoromethyl)-1H-pyrazole-4-carboxamide (bixafen-desmethyl), expressed as bixafen*

The residue is fat-soluble.

Bixafen was scheduled at the Fifty-first Session of the CCPR for the evaluation of additional uses by the 2020 JMPR Meeting, which was postponed to the 2021 Extra JMPR. The current Meeting received new information on use patterns for bixafen in pulses, potatoes, cereal grains and oilseed crops, supported by additional plant metabolism studies, field rotational crop studies, analytical methods and recovery data, supervised field trials and studies simulating typical processing conditions.

Table 1 List of compounds and abbreviations

Code Names	Chemical name	Structure	Where found
Bixafen BYF 00587 (AE1698406)	N-(3',4'-dichloro-5-fluorobiphenyl-2-yl)-3-(difluoromethyl)-1-methyl-1H-pyrazole-4-carboxamide (IUPAC) N-(3',4'-dichloro-5-fluoro[1,1'-biphenyl]-2-yl)-3-(difluoromethyl)-1-methyl-1*H*-pyrazole-4-carboxamide (CAS)		Plants Soil Livestock Rats
Bixafen-desmethyl (M21) (BCS-AA10008)	N-(3',4'-dichloro-5-fluorobiphenyl-2-yl)-3-(difluoromethyl)-1H-pyrazole-4-carboxamide		Plants Soil Livestock Rats
Bixafen-pyrazole-4-carboxylic acid (M42) (AE 1954999)	3-(difluoromethyl)-1-methyl-1H-pyrazole-4-carboxylic acid		Rotational crops Soil Livestock Rats

Code Names	Chemical name	Structure	Where found
Bixafen-pyrazole-4-carboxamide (M43)	3-(difluoromethyl)-1-methyl-1H-pyrazole-4-carboxamide		Rotational crops Rats
Bixafen-desmethyl-pyrazole-4-carboxylic acid (tautomer 1 & 2) (M44 & M45) (BCS-AA10651)	3-(difluoromethyl)-1H-pyrazole-4-carboxylic acid ⇔ 5-(difluoromethyl)-1*H*-pyrazole-4-carboxylic acid	 	Plants Rotational crops Rats
Bixafen-pyrazolone-4-carboxylic acid (M47)	3-oxo-2,3-dihydro-1H-pyrazole-4-carboxylic acid or 3-hydroxy-1H-pyrazole-4-carboxylic acid	 	Plants Rotational crops

Plant metabolism

The fate of bixafen in plants was evaluated by the 2013 Meeting following foliar spray application of [pyrazole-5-^{14}C]-bixafen or [dichlorophenyl-UL-^{14}C]-bixafen to soya beans or wheat. A detailed assessment of these studies is presented in the 2013 JMPR Report. The current Meeting received additional plant metabolism studies on potatoes and tomato.

The metabolism of both bixafen radiolabels in potatoes was investigated in parallel under semi-enclosed conditions (plant containers, natural sunlight, rain cover) with three foliar applications of 0.24 kg ai/ha each. The treatments were performed at the beginning of flowering (BBCH 61), at fruit development (BBCH 70) and 7 days before harvest (BBCH 97, leaves and stem dead). Potato leaves were harvested at an intermediate growth stage (BBCH 92, beginning of senescence) 28 days after the second application. Mature tubers were sampled 7 days after the final treatment.

Total radioactive residues (TRR) were comparable between both labels, representing 21.8–24.4 mg eq/kg for leaves and 0.002–0.003 mg eq/kg for tubers. All samples were extracted three times with a mixture of acetonitrile/water (4/1; v/v), which recovered ~98% of the TRR from the leaves and 85% of the TRR from the tubers (pyrazole-label only). Post extraction solids amounted 2.0–2.3% of the TRR for leaves (0.445–0.558 mg eq/kg) and 15.2% of the TRR for tubers (< 0.001 mg eq/kg).

The identification of the radioactive residues revealed unchanged bixafen as the predominant residue in potato leaves for both radiolabels (95.6–95.9% TRR). The predominant metabolite in leaves was bixafen-desmethyl, present at 0.6% of the TRR (0.121–0.135 mg eq/kg). No additional identified metabolites (M42, M43 and M47) or unidentified analytical fractions represented more than 0.4% of the TRR each.

For tubers, only the samples from the [pyrazole-^{14}C]-label were further investigated. TRR levels were low, allowing only limited identification of the residue. Parent bixafen was the predominant residue in tubers representing 31.4% of the TRR (0.001 mg eq/kg). Identified metabolites were M47 at the LOQ of 0.001 mg eq/kg (25.9% TRR) and the tautomeric mixture of M44 & M45 (12.5% TRR), M43 (9.0% TRR) and M42 (5.9% TRR), all present below 0.001 mg eq/kg.

For tomatoes, the metabolism of bixafen was investigated in a greenhouse by application of [pyrazole-5-^{14}C]-bixafen or [dichlorophenyl-UL-^{14}C]-bixafen under identical experimental conditions. The plants received three foliar applications with individual rates of about 0.21–0.23 kg ai/ha each at BBCH 78–80 (development of fruit: fruits have reached typical form and size), 21 days later at BBCH 84–85 (ripening of fruit: 40–50% of fruits show typical fully ripe colour) and further 14 days later at BBCH 87–89 (ripening of fruit: 70–100% of fruits show typical fully ripe colour). Samples of fruits and leaves were collected 3 days after the last treatment (DALT), however only the fruits were further investigated.

The fruits were surface-washed with dichloromethane and subsequently extracted three times with acetonitrile/water (4/1; v/v). TRR levels were 1.75 mg eq/kg for [pyrazole-^{14}C]-bixafen and 3.19 mg eq/kg for [dichlorophenyl-UL-^{14}C]-bixafen treated samples. Approximately 93–94% of the radioactivity was recovered in the surface wash and 5.6–6.9% of the TRR in the extract. Post extraction solids amounted to 0.1% of the TRR.

For both labels, in the combined surface washes and extracts, bixafen was the predominant residue representing a total of >99% of the TRR. The only identified metabolite was bixafen-desmethyl, present at 0.1% of the TRR (0.002–0.004 mg eq/kg). In addition, two minor unidentified analytical fractions were found at < 0.1–0.2% of the TRR (0.001–0.005 mg eq/kg).

The Meeting concluded that parent bixafen is the predominant residue in all plant parts (potato leaves and tubers, tomato fruits). The only other metabolite identified was bixafen-desmethyl at minor proportions (<1% TRR). In potato tubers, TRR levels were very low (0.002–0.003 mg eq/kg). Parent bixafen was also the predominant residue (31.4% TRR), however M47 was quantified as a major metabolite by proportion (25.9% TRR), but not based on its concentration (0.001 mg eq/kg). Further metabolites identified in tubers were M44 & M45, M43 and M42, all present below 0.001 mg eq/kg. Metabolites M21 (0.6%; 0.135 mg eq/kg), M43 (0.1%; 0.025 mg eq/kg), M42 (0.1%; 0.023 mg eq/kg) and M47 (< 0.1%; 0.006 mg eq/kg) were also identified in potato leaves.

Environmental fate (rotational crops)

The current Meeting received multiple additional field rotational crop studies, all conducted in Europe with one application of 1.13 kg ai/ha to bare soil. After plant back intervals (PBI) of 30, 120 and 365 days, various crops were planted/seeded and grown to commercial harvest. In parallel, initial residues in soil were measured directly after treatment.

All samples were analysed for bixafen and its desmethyl-metabolite (M21), M43 and M44/45. For strawberries and cabbage, residues were generally below the LOQs of 0.01 mg/kg at each PBI. Also, no residues of bixafen-desmethyl and M43 were quantified in any of the samples. For all other crops investigated, the results for residues of bixafen and M44/45 are summarized in the following Table.

Table 2 Overview of bixafen residues in rotational crops

Crop	Matrix	PBI	Residues (mg eq/kg)	
		(days)	Bixafen	M44/45
Leek	Stalks	30	4 × < 0.01	3 × < 0.01, 0.016
		120	4 × < 0.01	3 × < 0.01, 0.034
		365	4 × < 0.01	2 × < 0.01, 0.011, 0.014
Cabbage	Head	30	4 × < 0.01	3 × < 0.01, 0.01
		120	4 × < 0.01	4 × < 0.01
		365	4 × < 0.01	4 × < 0.01
Courgette	Fruit	30	4 × < 0.01	3 × < 0.01, 0.011
		120	4 × < 0.01	2 × < 0.01, 0.015, 0.023
		365	4 × < 0.01	3 × < 0.01, 0.018
Pea	Seed, dry	30	3 × < 0.01, 0.033	3 × < 0.02, 0.037
		120	4 × < 0.01	2 × < 0.02, 0.019, 0.11
		365	4 × < 0.01	3 × < 0.02, 0.061
	Vines	30	4 × < 0.01	2 × < 0.02, 0.030, 0.055
		120	4 × < 0.01	2 × < 0.02, 0.011, 0.082
		365	4 × < 0.01	3 × < 0.02, 0.047
Potato	Tubers	30	2 × < 0.01, 0.018, 0.058	< 0.02, 0.012, 0.061, 0.064
		120	3 × < 0.01, 0.019	0.016, 0.016, 0.050, 0.060
		365	4 × < 0.01	0.012, 0.015, 0.025, 0.038
Maize	Forage	30	4 × < 0.01	3 × < 0.01, 0.030
		120	4 × < 0.01	4 × < 0.01
		365	4 × < 0.01	4 × < 0.01
	Kernel	30	4 × < 0.01	3 × < 0.01, 0.063
		120	4 × < 0.01	4 × < 0.01
		365	4 × < 0.01	4 × < 0.01
	Stover	30	4 × < 0.01	3 × < 0.01, 0.017
		120	4 × < 0.01	3 × < 0.01, 0.025
		365	4 × < 0.01	3 × < 0.01, 0.023
Oilseed rape	Forage	30	4 × < 0.01	3 × < 0.01, 0.015
		120	4 × < 0.01	3 × < 0.01, 0.01
		365	4 × < 0.01	2 × < 0.01, 0.014, 0.042
	Seed, dry	30	4 × < 0.01	4 × < 0.01
		120	4 × < 0.01	4 × < 0.01
		365	4 × < 0.01	4 × < 0.01

Concerning the uptake of bixafen residues into rotational crops, the 2016 JMPR made a very conservative estimate for the soil plateau concentration equivalent to 0.93 kg ai/ha, which addresses many years of subsequent application at the maximum annual GAP rate (up to 0.25 kg ai/ha and year). This estimation also covers all GAPs evaluated by the current Meeting.

The 2016 JMPR concluded that small findings of bixafen in carrot roots (mean: 0.02 mg/kg, highest residue: 0.028 mg/kg) in field rotational crop studies addressing the estimated soil plateau concentrations were insignificant in view of the overall conservatism involved. However, additional field rotational crop

data submitted to the current Meeting suggest a potential transfer of parent bixafen into root crops and into pulse crops (not supported with data in 2016).

For peas, representing the pulses crop group, a highest residue of 0.033 mg/kg was found for bixafen in the 30 day crop rotation. The median for the total residue (sum of bixafen and bixafen-desmethyl, expressed as bixafen) was < 0.02 mg/kg.

New data on potato tubers showed quantifiable residues in two out of four field trials at the 30-day PBI and is complimented by three results in carrot roots as evaluated by the 2016 JMPR. The combined range of residues for root and tuber vegetables is: < 0.01, < 0.01, 0.014, 0.018, 0.019, 0.028 and 0.058 mg/kg for bixafen and < 0.02, < 0.02, 0.024, 0.028, 0.029, 0.038 and 0.068 mg/kg for the total bixafen residue.

In summary, the Meeting concluded that for root and tuber vegetables and pulse crops, the overall data available suggest a potential uptake of bixafen residues from soil. For all other crops that are potentially rotated, no significant uptake of bixafen residues from soil is expected.

The 2013 JMPR assessed the relevance of the tautomeric mixture M44/M45 against the TTC of a Cramer Class III compound. Based on field rotational crop data submitted to the 2016 JMPR and the additional data submitted to the current Meeting, the following concentrations of M44/M45 arising were estimated:

Table 3 Overview of M44/M45 residues in rotational crops

Commodity group	Field rotational crop commodity	M44/M45 in mg/kg (highest concentrations per trial from all PBIs)	M44/M45 STMR value in mg/kg
Bulb vegetables	Leek	< 0.01, < 0.01, 0.016, 0.034	0.013
Root and tuber vegetables	Carrot roots (2016 JMPR)	< 0.01, < 0.01, 0.044	
	Potato tuber	0.016, 0.016, 0.061, 0.064	
	Combined	< 0.01, < 0.01, 0.016, 0.016, 0.044, 0.061, 0.064	0.016
Leafy crops and brassica (extrapolated to stalk and stem vegetables)	Lettuce (2016 JMPR)	< 0.01, 0.017, 0.092	
	Cabbage	< 0.01, < 0.01, < 0.01, 0.01	
	Carrot leaves (2016 JMPR)	< 0.01, < 0.01, 0.18	
	Combined	< 0.01, < 0.01, < 0.01, < 0.01, < 0.01, < 0.01, 0.01, 0.017, 0.092, 0.18	0.01
Fruiting vegetables and strawberries (extrapolated to legume vegetables)	Courgettes	< 0.01, < 0.01, 0.015, 0.023	
	Strawberries	< 0.01, < 0.01, < 0.01, < 0.01	
	Combined	< 0.01, < 0.01, < 0.01, < 0.01, < 0.01, < 0.01, 0.015, 0.023	0.01
Pulses	Peas (dry)	< 0.01, < 0.01, 0.037, 0.082	0.0235
Cereal grains	Barley/wheat (2016 JMRP)	< 0.01, < 0.01, < 0.01	
	Maize	< 0.01, < 0.01, < 0.01, 0.063	
	Combined	< 0.01, < 0.01, < 0.01, < 0.01, < 0.01, < 0.01, 0.063	0.01
Oilseeds	Rape seed	< 0.01, < 0.01, < 0.01, < 0.01	0.01

Methods of residue analysis

The current Meeting received additional analytical methods for the determination of parent bixafen in plant commodities and additional concurrent recovery information for the methods 01012, 01013, 01633/M001 and 00952 evaluated by the 2013 JMPR.

For plant matrices, the new method 01367 (including ILV) was provided, based on the QuEChERs multi-method. Samples are extracted with acetonitrile in the presence of salts added for phase separation and cleaned by SPE. Bixafen is analysed by HLPC-MS/MS (ESI+) and the method was tested in high water, high oil, high protein, high acid and high starch matrices at an LOQ of 0.01 mg/kg.

The methods 01012, 01013 and 01633/M001 for plant commodities and method 00952 for soil were first evaluated by the 2013 JMPR. New recovery data submitted to the current Meeting confirmed the previous LOQs of 0.01 mg/kg for bixafen, bixafen-desmethyl (M21), M43 and M44/M45 in all matrices analysed within acceptable ranges.

The Meeting concluded that the presented methods were sufficiently validated and are suitable to measure bixafen, bixafen-desmethyl, M43 and M44/45 in plant commodities and/or soil.

Storage stability under frozen conditions

The current Meeting received additional information on the storage stability of bixafen and bixafen-desmethyl (M21) in plant matrices and of M44/M45 in soil.

In plant matrices, the storage stability of both bixafen and bixafen-desmethyl (M21) was demonstrated at -18 °C in orange fruits and dry bean seeds for at least 24 months.

In two different soil types, M44/M45 was also stable for up to 18 months under freezer storage conditions (-18 °C). At the last sampling interval of 24 months, M44/M45 was also stable in the clay loam investigated but showed significant degradation (< 70% remaining) in the silt loam soil.

The Meeting agreed that the demonstrated storage stability on various representative plant and animal commodities covered the residue sample storage intervals used in the field trials considered by the current Meeting.

Definition of the residue

The current Meeting received new data on the metabolism of bixafen following foliar treatment of potatoes and tomatoes.

Following foliar application to potatoes, bixafen was the predominant residue identified in all matrices. In leaves, unchanged parent bixafen represented 96% of the TRR (20.9–23.3 mg eq/kg). In tubers protected by soil, bixafen was still the major residue (31% TRR), but at low concentrations of 0.001 mg eq/kg. The only metabolite identified in leaves was bixafen desmethyl (M21; 0.6% TRR, 0.12–0.14 mg eq/kg). In tubers, M47 was found at 0.001 mg eq/kg (26% TRR), while M44 and M45, M43 and M42 were identified (5.9–12.5% TRR) at concentrations below t0.001 mg eq/kg.

In tomato fruits collected three days after the final treatment, more than 99% of the TRR was identified as unchanged parent bixafen (1.7–3.2 mg eq/kg). The only identified metabolite was bixafen-desmethyl (M21; 0.1% TRR, up to 0.004 mg eq/kg).

The Meeting confirmed its previous recommendation of bixafen for compliance with the MRL and the sum of bixafen and bixafen-desmethyl (M21) for dietary risk assessment for plant commodities.

Results of supervised residue trials on crops

The Meeting received supervised trial data for applications of bixafen on soya beans, potatoes, barley, maize (including sweet corn), sorghum, wheat, cotton, peanuts and sunflowers, respectively.

Residue values referred to as "total" are the sum of bixafen and M21 (bixafen-desmethyl), expressed as bixafen. Due to the general potential for uptake of residues from soil, LOQ values are always added to calculate the total residue concentration.

Soya beans (dry)

Bixafen is registered in the USA for use on soya beans with a maximum GAP involving two foliar spray applications of 0.077 kg ai/ha each (10-day interval) and a PHI of 21 days.

Supervised field trials conducted in Canada and the USA on soya beans were provided approximating the cGAP use pattern but at higher rates of 0.11 kg ai/ha per application. In absence of supervised field trial data matching the GAP within ±25%, the Meeting decided to scale the available data accordingly with a factor of 0.7 (0.077 kg ai/ha cGAP rate ÷ 0.11 kg ai/ha trial rate).

Unscaled residues of bixafen in soya beans were (n = 21): < 0.01(13), 0.01, 0.011, 0.012, 0.014, 0.014, 0.015, 0.024, 0.029 mg/kg.

Unscaled total residues of bixafen in soya beans were (n = 21): < 0.02(13), 0.020, 0.021, 0.022, 0.024, 0.024, 0.025, 0.034, 0.039 mg/kg.

Scaled (factor 0.7) residues of bixafen in soya beans were (n = 21): < 0.007(13), 0.007, 0.0077, 0.0084, 0.0098, 0.0098, 0.0105, 0.0168, 0.0203 mg/kg.

Scaled (factor 0.7) total residues of bixafen in soya beans were (n = 21): < 0.014(13), 0.014, 0.0147, 0.0154, 0.0168, 0.0168, 0.0175, 0.0238, 0.0273 mg/kg.

In field studies on succeeding crops a highest residue for bixafen in dry pea seeds of 0.033 mg/kg was found. The median total residue was < 0.02 mg/kg. Since soya beans can be both a primary and succeeding crop, the Meeting concluded that additional residues due to uptake via roots need to be taken into account.

The Meeting estimated a maximum residue level of 0.08 mg/kg (by adding the highest residue of 0.033 mg/kg for rotated pulse crops to each individual bixafen result for soya beans) and a STMR value of 0.034 mg/kg (by adding the median total residue of < 0.02 mg/kg for rotated pulse crops to the total median for soya beans) for bixafen in soya beans (dry).

Potatoes

Bixafen is registered in the USA for use on potatoes with a maximum GAP involving three foliar spray applications of 0.08 kg ai/ha each (10-day interval) and a PHI of 14 days.

Supervised field trials conducted in Canada and the USA on potatoes were provided involving four spray applications at <75% GAP rate and a shorter sampling and re-treatment interval of 7 days. The Meeting concluded that these trials do not approximate the cGAP. In addition, due to the number of deviations in the use and sampling intervals, the proportionality principle cannot be applied. No recommendation for the use of bixafen on potatoes can be given.

Barley

The 2016 JMPR assessed the use of bixafen on barley based on a GAP from the United Kingdom and recommended a maximum residue level of 0.4 mg/kg and a STMR value of 0.08 mg/kg for barley grain.

The current Meeting received new information that bixafen is registered in Brazil for the use on barley with a maximum GAP involving four foliar applications of 0.063 kg ai/ha each (15 days interval) and a PHI of 30 days.

Supervised field trials conducted in Brazil on barley were provided approximating the cGAP.

Residues of bixafen in barley grain were (n = 10): 0.11, 0.15, 0.17, 0.21, 0.30, 0.32, 0.53, 0.57, 0.58, 0.77 mg/kg.

Total residues of bixafen in barley grain were (n = 10): 0.12, 0.16, 0.18, 0.22, 0.32, 0.34, 0.54, 0.58, 0.59, 0.78 mg/kg.

The Meeting estimated a maximum residue level of 1.5 mg/kg and a STMR value of 0.33 mg/kg for bixafen in barley grain to replace its previous recommendations.

Maize

Bixafen is registered in the US for the use on maize with a maximum GAP involving two foliar spray applications of 0.077 kg ai/ha each (7 days interval, not after R4 stage/BBCH 83–85) and a PHI of 30 days.

Supervised field trials conducted in Canada and the USA on maize were provided involving two higher applications of 0.11 kg ai/ha at later growth stages, but at longer intervals of 19–36 days.

Residues of bixafen in maize grain were (n = 15): < 0.01(15) mg/kg.

Total residues of bixafen in maize grain were (n = 15): < 0.02(15) mg/kg

The Meeting noted that in these trials, treatment at rates higher than the cGAP and closer to harvest did not result in quantifiable residues in maize grain and the Meeting decided that they could be used to support the cGAP from the USA.

The Meeting estimated a maximum residue level of 0.01(*) mg/kg and a STMR value of 0.02 mg/kg for bixafen in maize grain.

Sweet corn

Bixafen is registered in Brazil for the use on maize (including sweet corn) with a maximum GAP involving two foliar spray applications of 0.063 kg ai/ha each (15 days interval) and a PHI of 15 days.

Supervised field trials conducted in Brazil on sweet corn were provided approximating the critical GAP.

Residues of bixafen in sweet corn cobs without husks were (n = 10): < 0.01(10) mg/kg.

Total residues of bixafen in sweet corn cobs without husks were (n = 10): < 0.02(10) mg/kg

The Meeting estimated a maximum residue level of 0.01(*) mg/kg, a STMR value of 0.02 mg/kg and a HR value of 0.02 mg/kg for bixafen in sweet corn (corn on the cob).

Sorghum

Bixafen is registered in the USA for the use on sorghum with a maximum GAP involving two foliar spray applications of 0.074 kg ai/ha each and a PHI of 30 days.

Supervised field trials conducted in the USA on sorghum were provided approximating the cGAP use pattern but at higher rates of 0.11 kg ai/ha per application. In the absence of supervised field trial data matching the GAP within ±25%, the Meeting decided to scale the available data accordingly with a factor of 0.7 (0.074 kg ai/ha cGAP rate ÷ 0.11 kg ai/ha trial rate).

Unscaled total residues of bixafen in sorghum grain were (n = 9): 0.077, 0.13, 0.17, 0.20, 0.28, 0.35, 0.49, 0.90, 1.9 mg/kg

Scaled (factor 0.7) residues of bixafen in sorghum grain were (n = 9): 0.0126, 0.077, 0.091, 0.098, 0.175, 0.175, 0.336, 0.553, 1.26 mg/kg.

Scaled (factor 0.7) total residues of bixafen in sorghum grain were (n = 9): 0.0539, 0.091, 0.119, 0.14, 0.196, 0.245, 0.343, 0.63, 1.33 mg/kg

The Meeting estimated a maximum residue level of 2 mg/kg and a STMR value of 0.196 mg/kg for bixafen in sorghum.

Wheat, rye and triticale

The 2016 JMPR assessed the use of bixafen on wheat, rye and triticale based on a GAP from the United Kingdom and recommended a maximum residue level of 0.05 mg/kg and a STMR value of 0.02 mg/kg for each of wheat, rye and triticale.

Bixafen is registered in the USA for use on wheat, rye and triticale with a maximum GAP involving two foliar spray applications of 0.075 kg ai/ha each (14 days interval) and a PHI of 30 days.

Supervised field trials on wheat conducted in Canada and the USA were provided, involving two applications of 0.11 kg ai/ha each with intervals of 18–73 days. Although the label recommends an early treatment as conducted in the supervised field trials, the label compliant cGAP allows two late treatments at a 14 days interval with a total rate not exceeding 0.15 kg ai/ha. Consequently, the Meeting concluded that the data do not approximate the cGAP registered in the USA.

In addition, bixafen is registered in Brazil for use on wheat with a maximum GAP involving four foliar spray applications of 0.063 kg ai/ha each (15 days interval) and a PHI of 30 days.

Supervised field trials conducted in Brazil on wheat were provided approximating the cGAP.

Residues of bixafen in wheat grain were (n = 10): 0.02, 0.05, 0.06, 0.06, 0.07, 0.10(3), 0.12, 0.17 mg/kg.

Total residues of bixafen in wheat grain were (n = 10): 0.03, 0.06, 0.07, 0.07, 0.08, 0.11, 0.11, 0.13, 0.14, 0.18 mg/kg.

Based on the Brazilian GAP for wheat only, the Meeting estimated a maximum residue level of 0.3 mg/kg and a STMR value of 0.095 mg/kg for bixafen to replace its previous recommendation on wheat grain.

Cotton

Bixafen is registered in Brazil for the use on cotton with a maximum GAP involving four foliar spray applications of 0.063 kg ai/ha each (15 days interval) and a PHI of 30 days.

Supervised field trials conducted in Brazil on cotton were provided approximating the cGAP.

Residues of bixafen in cotton seeds were (n = 10): < 0.01, < 0.01, 0.01, 0.01, 0.02, 0.02, 0.03, 0.06, 0.06, 0.16 mg/kg.

Total residues of bixafen in cotton seeds were (n = 10): < 0.02, < 0.02, 0.02, 0.02, 0.03, 0.03, 0.04, 0.07, 0.07, 0.17 mg/kg.

The Meeting estimated a maximum residue level of 0.3 mg/kg and a STMR value of 0.03 mg/kg for bixafen in cotton seeds.

Peanuts

Bixafen is registered in the USA for the use on peanuts with a maximum GAP involving two foliar spray applications of 0.10 kg ai/ha each (14 day interval) and a PHI of 14 days.

Supervised field trials on peanuts conducted in the USA were provided, involving four applications of approximately 0.056 kg ai/ha each with intervals of 13–15 days. The Meeting took note of the very slow metabolic degradation of bixafen residues in crops. Combined with low basipetal translocation of bixafen in plants, it was concluded that the splitting into four instead of two treatments had no impact on residues in peanut nutmeats, which are protected by soil. Since the total seasonal rate in the supervised field trials approximates the US GAP, the Meeting considered these trials as suitable to support a recommendation.

Residues of bixafen in peanut nutmeat were (n = 14): < 0.01(14) mg/kg.

Total residues of bixafen in peanut nutmeat seeds were (n = 14): < 0.02(14) mg/kg.

The Meeting estimated a maximum residue level of 0.01 mg/kg and a STMR value of 0.02 mg/kg for bixafen in peanuts.

Sunflower

Bixafen is registered in Brazil for the use on sunflowers with a maximum GAP involving two foliar spray applications of 0.063 kg ai/ha each (15 days interval) and a PHI of 30 days.

Supervised field trials conducted in Brazil on sunflowers were provided approximating the cGAP.

Residues of bixafen in sunflower seeds were (n = 10): < 0.01, < 0.01, 0.01, 0.02, 0.02, 0.05, 0.08, 0.21, 0.56, 1.7 mg/kg.

Total residues of bixafen in sunflower seeds were (n = 10): < 0.02, < 0.02, 0.02, 0.03, 0.03, 0.06, 0.09, 0.22, 0.57, 1.7 mg/kg.

The Meeting estimated a maximum residue level of 3 mg/kg and a STMR value of 0.045 mg/kg for bixafen in sunflower seeds.

Animal Feeds

Maize forage

Bixafen is registered in the US for the use on maize with a maximum GAP involving two foliar spray applications of 0.077 kg ai/ha each (7 days interval) and a PHI of 10 days for forage.

Supervised field trials conducted in Canada and the USA on maize forage were provided, mostly involving a single treatment at 0.11 kg ai/ha and one plot with two applications of 0.11 kg ai/ha, a longer re-treatment interval of 20 days and a longer sampling interval of 30 days.

The Meeting concluded that the available trials do not approximate the registered use in the USA.

Maize fodder

Bixafen is registered in the US for the use on maize with a maximum GAP involving two foliar spray applications of 0.077 kg ai/ha each (7 days interval, not after R4 stage/BBCH 83–85) and a PHI of 30 days for stover.

Supervised field trials conducted in Canada and the USA on maize were provided involving two higher applications of 0.11 kg ai/ha at a longer interval of 20 days. The Meeting noted that bixafen residues in maize forage decline trials decreased during the first 10 days and remained stable afterward. Therefore,

the deviation relating to the longer interval between applications in the field trials submitted was considered to be of low relevance for the final residue concentration at harvest. In absence of supervised field trial data matching the GAP within ±25%, the Meeting decided to scale the available data accordingly with a factor of 0.7 (0.077 kg ai/ha cGAP rate ÷ 0.11 kg ai/ha trial rate).

Unscaled residues of bixafen in maize stover were (n = 16): 0.32, 0.45, 0.49, 0.76, 1.2, 1.6, 1.7, 1.8, 1.9, 2.0(3), 2.4(3), 3.0 mg/kg.

Unscaled Total residues of bixafen in maize stover were (n = 16): 0.36, 0.53, 0.69, 0.82, 1.3, 1.6, 1.9, 2.0, 2.2(3), 2.4, 2.4, 2.5, 2.8, 3.2 mg/kg

Scaled residues of bixafen in maize stover were (n = 16): 0.224, 0.315, 0.343, 0.532, 0.84, 1.12, 1.19, 1.26, 1.33, 1.4(3), 1.68(3), 2.1 mg/kg.

Scaled Total residues of bixafen in maize stover were (n = 16): 0.252, 0.371, 0.483, 0.574, 0.91, 1.12, 1.33, 1.4, 1.54(3), 1.68, 1.68, 1.75, 1.96, 2.24 mg/kg

The Meeting estimated a maximum residue level of 5 mg/kg (DM, 89% dry-matter content), a median residue of 1.47 mg/kg (as received) and a highest residue of 2.24 mg/kg (as received) for bixafen in maize fodder.

Sorghum forage

Bixafen is registered in the US for the use on sorghum with a maximum GAP involving two foliar spray applications of 0.074 kg ai/ha each (7 days interval) and a PHI of 30 days for forage.

Supervised field trials conducted in the USA on sorghum forage were provided involving a single treatment at 0.11 kg ai/ha with a PHI of approximately 10 days.

The Meeting noted that the cGAP for bixafen also allows two applications on sorghum until the forage growth stage and concluded that the available trials do not approximate the registered use in the USA.

Sorghum fodder

Bixafen is registered in the US for the use on sorghum with a maximum GAP involving two foliar spray applications of 0.074 kg ai/ha each (7 days interval) and a PHI of 30 days.

Supervised field trials conducted in the USA on sorghum were provided involving two higher applications of 0.11 kg ai/ha at longer intervals of 15–22 days. In one decline trial conducted for sorghum forage, bixafen residues decreased significantly between day 10 (0.79 mg/kg) and day 20 (0.092 mg/kg) after the first treatment. The Meeting concluded that the longer interval in the field trials may result in a significant reduction of the residue concentrations at harvest and therefore does not reflect the critical use pattern for sorghum fodder.

Wheat forage

The 2016 JMPR evaluated the use of bixafen on small cereal grains based on a GAP from the United Kingdom and estimated a highest residue of 7.3 mg/kg and a median residue of 3.5 mg/kg for barley and wheat forage (as received) and decided to extrapolate the estimations to oats, rye and triticale forage.

The current Meeting received new GAP information for the use of bixafen on wheat from Brazil, however no residue data on forage were provided.

Bixafen is registered in the USA for the use on wheat, rye and triticale with a maximum GAP involving two foliar spray application of 0.075 kg ai/ha each (14 days interval) and a PHI of 10 days for forage and 20 days for hay.

Supervised field trials on wheat conducted in Canada and the USA were provided, involving single applications of 0.11 kg ai/ha. The cGAP allows two treatments at a 14 days interval with a total rate not exceeding 0.15 kg ai/ha including for forage. Consequently, the Meeting concluded that the data do not approximate the cGAP registered in the USA.

The Meeting confirmed its previous recommendations for bixafen in wheat forage.

Wheat straw

The 2016 JMPR evaluated the use of bixafen on small cereal grains based on a GAP from the United Kingdom and estimated a maximum residue level of 20 mg/kg (dry-weight basis, based on 89% DM content), a highest residue of 11 mg/kg (as received) and a median value of 2.2 mg/kg (as received) for barley and wheat, straw and fodder and decided to extrapolate the estimations to oats, rye and triticale straw and fodder.

The current Meeting received new GAP information for the use of bixafen on wheat from Brazil, however no residue data on straw were provided.

Bixafen is registered in the USA for use on wheat, rye and triticale with a maximum GAP involving two foliar spray applications of 0.075 kg ai/ha each (14 days interval) and a PHI of 30 days.

Supervised field trials on wheat conducted in Canada and the USA were provided, involving two applications of 0.11 kg ai/ha each with intervals of 18–73 days. Although the label recommends an early treatment as conducted in the supervised field trials, the label compliant cGAP allows two late treatments at a 14 day interval with a total rate not exceeding 0.15 kg ai/ha. Consequently, the Meeting concluded that the data do not approximate the cGAP registered in the USA.

The Meeting confirms its previous recommendations for bixafen in wheat straw.

Peanut hay

Bixafen is registered in the USA for use on peanuts with a maximum GAP involving two foliar spray applications of 0.10 kg ai/ha each (14 days interval). However, the label excludes utilisation of forage or hay for animal feed purposes.

Residues in rotational crops

Taking into account new data on field rotational crops, the current Meeting estimated a highest bixafen residue of 0.033 mg/kg for pulses. Median total bixafen residues were 0.02 mg/kg.

Based on these values, the Meeting estimated a maximum residue level of 0.04 mg/kg and a STMR value of 0.02 mg/kg for pulses, except soya beans (dry).

The current Meeting estimated a highest bixafen residue of 0.058 mg/kg for root and tuberous crops. Total bixafen residues were 0.028 mg/kg (median) and 0.068 mg/kg (highest residue).

Based on these values, the Meeting estimated a maximum residue level of 0.06 mg/kg, a STMR value of 0.028 mg/kg and a HR of 0.068 for root and tuber vegetables.

Fate of residues during processing

The fate of bixafen residues has been examined simulating commercial processing of soya beans, potatoes, maize grain, sorghum grain, wheat grain, oilseed rapeseeds and peanuts.

Estimated processing factors for the commodities considered at this Meeting are summarised below. In potatoes, residues in the raw agricultural commodity were below the LOQ. Consequently, no processing factors were derived for this commodity. For processing factors on other commodities, please refer to the evaluation document.

Table 4 Processing factors and processed commodities

Raw commodity	Processed commodity	Bixafen			Total residue		
		Individual processing factors	Mean or best estimate processing factor	Maximum residue level (mg/kg)	Individual processing factors	Mean or best estimate processing factor	STMR-P in mg/kg
Soya beans (dry) (STMR: 0.034 mg/kg, MRL: 0.08 mg/kg)	AGF	310, 470, 620	470	-	See bixafen	See bixafen	15.98
	Flour	< 0.09, 0.13	0.11	-			0.00374
	Hulls	2.4, 2.8, 7.9	2.8	0.3			0.0952
	Meal	0.11, 0.14, < 0.22	0.14	-			0.00476
	Milk	< 0.04, < 0.09	0.065	-			0.00221
	Refined oil (cold press)	1.3, 1.4	1.4	0.15			0.0476
	Refined oil (solvent ext.)	0.35, 0.40, 0.50	0.40	-			See cold press
Barley (STMR: 0.33 mg/kg, MRL: 1.5 mg/kg)	Brewer´s malt	0.33[a], 0.8[a], 0.92[a], 0.96[a]	0.86[a]	-	0.5[a], 0.91[a], 1[a], 1[a]	0.96[a]	0.317
	Beer	< 0.04[a],< 0.05[a], < 0.08[a], < 0.33[a]	< 0.065[a]	-	0.08[a], < 0.09[a], < 0.13[a], < 0.5[a]	< 0.11[a]	0.036
	Pearl barley	0.17[a], 0.2[a], 0.23[a], < 0.33[a]	0.22[a]	-	0.19[a], 0.23[a], 0.27[a], < 0.5[a]	0.25[a]	0.083
Maize (STMR: 0.02 mg/kg, MRL: 0.01* mg/kg)	AGF	160, 160, 180	160	-	See bixafen	See bixafen	3.2
	Bran (dry milling)	2.6, 3.6	3.1	0.03			0.062
	Flour (dry milling)	< 0.63, 0.89, 1.5	0.89	-			0.0178
	Germ (dry milling)	See wet milling	See wet milling	-			See wet milling
	Germ (wet milling)	0.63, 1.4	1.0	-			0.02
	Grits (dry milling)	< 0.36, < 0.63, < 0.93	< 0.63	-			0.0126
	Meal (dry milling)	< 0.63, 0.75, 1.4	0.75	-			0.015
	Refined oil (dry milling)	See wet milling	See wet milling	-			See wet milling
	Refined oil (wet milling)	1.7, 1.8, 1.9	1.8	0.02			0.036

Raw commodity	Processed commodity	Bixafen			Total residue		
		Individual processing factors	Mean or best estimate processing factor	Maximum residue level (mg/kg)	Individual processing factors	Mean or best estimate processing factor	STMR-P in mg/kg
	Starch (wet milling)	< 0.36, < 0.63, < 0.93	< 0.63	-			0.0126
Sorghum (STMR: 0.02 mg/kg, MRL: 0.01* mg/kg)	AGF (grain sorghum)	26	26	-	26	26	0.52
	Syrup (sweet sorghum)	0.17	0.17	-	0.15	0.15	0.0015
Wheat (STMR: 0.095 mg/kg, MRL: 0.3 mg/kg)	AGF	200	200	-	180	180	17.1
	Bran	0.81, 2.4[a], 2.6[a], 2.8[a], 3.5[a]	2.6	0.8	0.88, 2.4[a], 2.5[a], 2.7[a], 4[a]	2.5	0.2375
	Flour	0.12, 0.2[a], 0.2[a], 0.25[a], 0.5[a]	0.20	-	0.20, 0.33[a], 0.33[a], 0.4[a], 0.66[a]	0.33	0.03135
	Germ	0.57, 0.6[a], 0.75[a], 1.2[a], 1.5[a]	0.75	-	0.74, 0.8[a], 0.83[a], 1.3[a], 2.7[a]	0.83	0.07885
Oilseed rape (STMR: 0.02 mg/kg, MRL: 0.04 mg/kg, based on 2016 JMPR)	Meal (extracted)	< 0.5[a], 0.72, 0.82, 2[a]	0.77	-	0.66[a], 0.8, 1.2, 1.5[a]	1.0	0.02
	Oil, crude	0.5[a], 1[a], 2.4, 2.5	1.7	-	0.66[a], 1[a], 1.8, 2.0	1.4	0.028
	Oil, refined	< 0.5[a], 2[a], 2.1, 2.1	2.0	0.08	< 0.66[a], 1.4[a], 1.5, 1.8	1.4	0.028
Peanut (STMR: 0.02 mg/kg, MRL: 0.01 mg/kg)	Meal	< 0.57	< 0.57	-	See bixafen	See bixafen	0.0114
	Refined oil (edible)	2.2	2.2	0.03			0.044

[a] PF estimated by the 2013 JMPR

- no traded commodity or maximum residue level covered by raw agricultural commodity

In addition to the STMR-P values given in the table above, the Meeting estimated maximum residue levels of 0.3 mg/kg for soya bean hulls, 0.15 mg/kg for soya bean refined oil, 0.03 mg/kg for maize bran and 0.02 mg/kg for maize oil.

For wheat bran, the Meeting estimate a maximum residue level of 0.8 mg/kg to replace its previous recommendation of 0.15 mg/kg made by the 2016 JMPR.

For oilseed rape, new processing information were provided to the current Meeting. Based on the combined processing factors including those from the 2016 and 2020 JMPR, the Meeting confirms its previous maximum residue level recommendation of 0.08 mg/kg for bixafen in rapeseed oil, edible.

For peanut oil, edible, the Meeting estimated a maximum residue level of 0.03 mg/kg.

Residues in animal commodities

Farm animal feeding studies

No additional information on farm animals was provided to the current Meeting. Please refer to the 2013 JMPR Report and Evaluation.

Estimated maximum and mean dietary burdens of livestock and animal commodities maximum residue levels

Dietary burdens were calculated for beef cattle, dairy cattle, broilers and laying poultry based on feed items evaluated by the JMPR. The dietary burdens, estimated using the estimated using the most recent version of the OECD livestock dietary burden calculator, are presented in Annex 6 and summarised below.

Table 5 Estimated maximum and mean dietary burdens of farm animals

	Livestock dietary burden for the estimation of maximum residue levels and the dietary exposure, total bixafen, ppm of dry matter diet							
	US-Canada		EU		Australia		Japan	
	max.	mean	max.	Mean	max.	mean	max.	mean
Beef cattle	4.0	2.5	8.6	4.2	29 [a]	14 [c]	0.14	0.14
Dairy cattle	8.4	4.1	8.5	4.1	27 [b]	13 [d]	1.3	0.7
Poultry - broiler	0.18	0.18	0.25	0.21	0.17	0.17	0.16	0.16
Poultry - layer	0.18	0.18	3.2 [e]	1.6 [f]	0.17	0.17	0.14	0.14

[a] Highest maximum beef cattle burden suitable for MRL estimates and dietary exposure for mammalian meat

[b] Highest maximum dairy cattle burden suitable for MRL estimates for milk

[c] Highest mean beef cattle burden suitable for dietary exposure for mammalian meat

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

[e] Highest maximum broiler or laying hen burden suitable for MRL estimates and dietary exposure for poultry products and eggs

[f] Highest mean broiler or laying hen burden suitable for MRL estimates and dietary exposure for poultry products and eggs

Animal commodities maximum residue levels

In comparison to the estimations made by the 2016 JMPR, the dietary burden remained unchanged for beef and dairy cattle after including the uses considered by the current Meeting and increased insignificantly for laying hens (Max: 3.0 ppm → 3.2 ppm and mean: 1.5 ppm → 1.6 ppm).

Therefore, the Meeting confirms its previous recommendation for bixafen in animal commodities.

RECOMMENDATIONS

On the basis of the data obtained from supervised trials, the Meeting concluded that the residue levels listed below in Table 6 are suitable for establishing maximum residue limits and for IEDI and IESTI assessments.

Definition of the residue for compliance with MRL for plant commodities: *bixafen*

Definition of the residue for compliance with MRL for animal commodities and (for the estimation of dietary intake) for plant and animal commodities: *sum of bixafen and N-(3',4'-dichloro-5-fluorobiphenyl-2-yl)-3-(difluoromethyl)-1H-pyrazole-4-carboxamide (bixafen-desmethyl), expressed as bixafen*

The residue is fat-soluble.

Table 6 Maximum residue levels and dietary exposure

Commodity		Maximum residue level, mg/kg		STMR or STMR-P, mg/kg	Highest residue, mg/kg
CCN	Name	New	Previous		
GC 0640	Barley	1.5	0.4	0.33	
SO 0691	Cottonseed	0.3		0.03	
AS 0645	Maize fodder (dry)	5 (dw)		Median: 1.47 (as)	Highest: 2.24 (as)
GC 0645	Maize	0.01(*)		0.02	
AS 3490	Maize bran, unprocessed	0.03		0.062	
OR 0645	Maize oil, Edible	0.02		0.036	
SO 0697	Peanut	0.01		0.02	
OR 0697	Peanut oil. Edible	0.03		0.044	
VD 0070	Pulses, Group of (except Soya bean (dry))	0.04[a]		0.02 [a]	
VR 0075	Root and tuber vegetables, Group of	0.06[a]		0.028 [a]	0.068 [a]
GC 0651	Sorghum Grain	2		0.196	
VD 0541	Soya bean (dry)	0.08		0.034	
AB 0541	Soya bean hulls	0.3		0.0952	
OR 0541	Soya bean oil, Refined	0.15		0.0476	
SO 0702	Sunflower seed	3		0.045	
GC 0447	Sweet corn (corn on the cob) (kernels plus cob with husk removed)	0.01(*)		0.02	0.02
CF 0654	Wheat bran, Processed	0.8	0.15	0.2375	
GC 0654	Wheat	0.3	0.05	0.095	

[a] based on rotational crops

Table 7 Dietary exposure values only

Commodity		STMR-P, mg/kg
CCN	Name	
	Beer	0.036
	Brewer´s malt	0.317
	Maize, aspirated grain fractions	3.2
	Peanut meal	0.0114
	Pearl barley	0.083
	Rape seed meal	0.02
	Sorghum, aspirated grain fractions	0.52
	Soya bean, aspirated grain fractions	15.98
	Soya bean, flour	0.00374
	Soya bean, meal	0.00476
	Soya bean, milk	0.00221
	Wheat, aspirated grain fractions	17.1
CF 1211	Wheat, flour	0.03135
CF 1210	Wheat, germ	0.07885

Dietary risk assessment

Long-term dietary exposure

The ADI for bixafen is 0–0.02 mg/kg bw. The International Estimated Daily Intakes (IEDIs) for bixafen were estimated for the 17 GEMS/Food Consumption Cluster Diets using the STMR or STMR-P values estimated by the JMPR. The results are shown in Annex 3 of the 2021 Extra JMPR Report.

The IEDIs ranged from 4–10% of the maximum ADI. The Meeting concluded that long-term dietary exposure to residues of bixafen from uses considered by the JMPR is unlikely to present a public health concern.

Acute dietary exposure

The ARfD for bixafen is 0.2 mg/kg bw. The International Estimate of Short Term Intakes (IESTIs) for bixafen were calculated for the food commodities and their processed commodities for which HRs/HR-Ps or STMRs/STMR-Ps were estimated by the present Meeting and for which consumption data were available. The results are shown in Annex 4 of the 2021 Extra JMPR Report.

The IESTIs for children were 0–20% of the ARfD and for adults 0–20% of the ARfD. The Meeting concluded that the acute dietary exposure to residues of bixafen from the uses considered by the present Meeting is unlikely to present a public health concern.

Assessment of metabolites using the threshold of toxicological concern (TTC) approach

The tautomeric mixture M44/M45 could be assessed using the TTC approach (Cramer Class III threshold of 1.5 µg/kg bw per day) as concluded by the 2013 Meeting.

The current Meeting considered residues of M44/M45 in rotational crops resulting in a dietary exposure estimate of 0.31 µg/kg bw per day.

The Meeting concluded that the estimated dietary exposure to residues of the tautomeric mixture M44/M45 from uses considered by the JMPR is below the TTC for Cramer Class III compounds and is unlikely to present a public health concern. Should further uses be considered in the future, these conclusions may need to be re-evaluated.

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Code	Author	Year	Title, Institute, Report reference
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BIXAFEN_085	Freitag T.; Kowalski N.	2017	Determination of the residues of bixafen in/on potato and soil after spraying of bixafen EC 125 in the field in Belgium, the Netherlands, France (South) and Spain; Report No. 15-2512, Document No. M-606716-01-1, GLP, unpublished, 09 November 2017
BIXAFEN_086	Freitag T.; Kowalski N.	2017	Determination of the residues of bixafen in/on the field rotational crop pea and soil after spray application of bixafen EC 125 to bare soil in the field in Belgium, the Netherlands, Spain and Greece; Report No. 15-2513, Document No. M-606719-01-1, GLP, unpublished, 27 October 2017
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BIXAFEN_093	Lenz C.	2016	Residues of F9650 in Field Corn Forage, Grain, Stover and Field Corn Processed Commodities; Report No. 2014RES-BAN1259, Document No. M-574248-01-1, GLP, unpublished, 18 January 2016
BIXAFEN_094	Lenz C.	2016	Residues of F9650 in sorghum forage, grain, stover and sorghum processed commodities; Report No. 2014RES-BAN1260, Document No. M-574251-01-1, GLP, unpublished, 07 January 2016
BIXAFEN_095	Lenz C.	2016	Residues of F9650 in Wheat Forage, Hay, Grain, Straw and Wheat Processed Commodities; Report No. 2014RES-BAN1261, Document No. M-574253-01-1, GLP, unpublished, 20 January 2016
BIXAFEN_096	Lenz C.	2016	Residues of F9650 in peanut nutmeat, hay and peanut processed commodities; Report No. 2014RES-BAN1251, Document No. M-573881-01-1, GLP, unpublished, 15 January 2016

Code	Author	Year	Title, Institute, Report reference
BIXAFEN_097	Schoening, R.; van Berkum, S	2015	Storage stability of bixafen and its metabolite BYF00587-desmethyl in/on dry bean seed and orange fruit for 24 months, Report no MR-13/044, Document No M-532339-01-1, GLP, unpublished, 01 September 2015
BIXAFEN_098	Santiago L.	2016	Adendo 01: Estudo de validação das metodologias de análise de resíduos de trifloxystrobin e seu metabólito CGA321113, prothioconazole e seu metabólito JAU6476-desthio, bixafen e seu metabólito BYF00587-desmethyl, tebuconazole e cyproconazole em diversas matrizes; Report No. VM11-003, Document No. M-435708-02-1, GLP, unpublished, 26 October 2016
BIXAFEN_099	Resende G.	2015	Adendo 01: Estudo de validação da metodologia de análise de resíduos de trifloxystrobin e seu metabólito CGA321113, prothioconazole e seu metabólito JAU6476-desthio, bixafen e seu metabólito BYF00587-desmethyl em diversas matrizes; Report No. VM12-006, Document No. M-448030-02-1, GLP, unpublished, 03 September 2015
BIXAFEN_100	Schoening R., Masoudi R.	2013	Analytical method 01367 (based on "QuEChERS" Method) for the determination of bixafen in plant materials; Report No. MR-13/035, Document No. M-457376-01-1, GLP, unpublished, 19 June 2013
BIXAFEN_101	Konrad S.	2014	Independent laboratory validation of the analytical method 01367 (based on QuEChERS method) for the determination of bixafen in plant materials; Report No. 2013/0074/01, Document No. M-483710-01-1, GLP, unpublished, 14 February 2014
BIXAFEN_102	Hoffmann M.	2013	Determination of the residues of BYF 00587 and prothioconazole in/on rape, winter and the processed fractions (oil, screw-pressed; pomace; extracted meal; oil, solv. extracted; oil, crude; crude oil, preclarified; crude oil, neutralised and oil, refined) after spraying of Bixafen & Prothioconazole EC 225 in the field in the United Kingdom; Report No.: 09-3245, Document No. M-449117-02-1, GLP, unpublished, 13 March 2013, amended 10 April 2013
BIXAFEN_103	Koch V.	2016	Modification M002 of the analytical method 00952 for the determination of bixafen and the metabolite BYF00587-desmethyl and BYF00587-desmethyl-pyrazole-4-carboxylic acid in soil by HPLC-MS/MS; Report No. MR-15/136, Document No. M-546024-01-1, GLP, unpublished, 01 February 2016
BIXAFEN_104	Resende G.	2016	Adendo 02: Determinação de resíduos de Bixafen, Prothioconazole e Trifloxystrobin e seus respectivos metabólitos na cultura da cevada após a pulverização de Bixafen & Prothioconazole & Trifloxystrobin 450 SC juntamente com o adjuvante óleo metilado de soja em ensaios no Brasil; Report No. F12-029, Document No. M-448127-03-1, GLP, unpublished, 14 January 2016
BIXAFEN_105	Anonymous	2013	Summary sheet: Bixafen & prothioconazole & trifloxstrobin (125+175+150); 450 SC; barley; summary to Brazil Report F12-029; BBA (translation), Document No. M-461436-01-1, GLP, unpublished, 17 April 2013
BIXAFEN_106	Resende G.	2016	Adendo 02: Determinação de resíduos de Bixafen, Prothioconazole e Trifloxystrobin e seus respectivos metabólitos na cultura da cevada após aplicação de Bixafen & Prothioconazole & Trifloxystrobin 450 SC, juntamente com o adjuvante óleo metilado de soja em ensaios no Brasil;, Report No. F13-005, Document No. M-487620-03-1, GLP, unpublished, 14 January 2016
BIXAFEN_107	Resende G.	2016	Adendo 02 ao relatório final: Determinação de resíduos de Bixafen, Prothioconazole e Trifloxystrobin e seus respectivos metabólitos na cultura do milho após a pulverização de Bixafen & Prothioconazole & Trifloxystrobin 450 SC juntamente com o adjuvante óleo metilado de soja em ensaios no Brasil; Report No. F12-011, Document No. M-448104-03-1, GLP, unpublished, 10 March 2016
BIXAFEN_108	Anonymous	2016	Summary sheet: CGA 279202 & BYF 00587 & JAU 6476; SC 450; maize/corn; summary to Brazil Report F12-011; BBA sheet (translation); Document No. M-451489-02-1, GLP, unpublished, 10 March 2016

Code	Author	Year	Title, Institute, Report reference
BIXAFEN_109	Resende G.	2016	Adendo 02 ao relatório final: Determinação de resíduos de Bixafen, Prothioconazole e Trifloxystrobin e seus respectivos metabólitos na cultura do Milho, após aplicação de Bixafen & Prothioconazole & Trifloxystrobin 450 SC, juntamente com o adjuvante óleo metilado de soja em ensaios no Brasil; Report No. F13-007, Document No. M-465976-03-1, GLP, unpublished,22 March 2016
BIXAFEN_110	Anonymous	2016	Summary sheet bixafen & prothioconazole & trifloxystrobin 450 SC , maize; summary to Brazil Report F13-007, BBA sheet (translation), Document No. M-478876-01-1, GLP, unpublished, 01 December 2013
BIXAFEN_111	Resende G.	2016	Adendo 02 ao relatório final: Determinação de resíduos de Bixafen, Prothioconazole e Trifloxystrobin e seus respectivos metabólitos na cultura do trigo após a pulverização de Bixafen & Prothioconazole & Trifloxystrobin 450 SC juntamente com o adjuvante óleo metilado de soja em ensaios no Brasil; Report No. F12-028, Document No. M-448133-03-1, GLP, unpublished, 10 March 2016
BIXAFEN_112	Anonymous	2013	Summary sheet bixafen & prothioconazole & trifloxystrobin 450 SC, wheat, summary to Brazil Report F12-028, BBA sheet (translation); Document No. M-461434-01-1, GLP, unpublished, 17 April 2013
BIXAFEN_113	Resende G.	2016	Adendo 01 ao relatório final: Determinação de resíduos de Bixafen, Prothioconazole e Trifloxystrobin e seus respectivos metabólitos na cultura do trigo após aplicação de Bixafen & Prothioconazole & Trifloxystrobin 450 SC, juntamente com o adjuvante óleo metilado de soja em ensaios no Brasil; Report No. F13-009, Document No. M-486614-02-1, GLP, unpublished, 10 March 2016
BIXAFEN_114	Resende G.	2016	Adendo 02 ao relatorio final - Determinação de resíduos de bixafen, prothioconazole e trifloxystrobin e seus respectivos metabólitos na cultura do algodão após a pulverização de bixafen & prothioconazole & trifloxystrobin 450 SC juntamente com o adjuvante óleo metilado de soja em ensaios no Brasil; Report No. F12-010, Document No. M-448074-03-1, GLP, unpublished, 10 March 2016
BIXAFEN_115	Anonymous	2013	Summary sheet bixafen & prothioconazole & trifloxystrobin 450 SC , cotton, summary to Brazil Report F12-010, BBA sheet (translation), Document No. M-461432-01-1, GLP, unpublished, 15 April 2013
BIXAFEN_116	Resende G.	2016	Adendo 02 ao relatorio final - Determinação de resíduos de bixafen, prothioconazole e trifloxystrobin e seus respectivos metabólitos na cultura do algodão, após aplicação de bixafen & prothioconazole & trifloxystrobin 450 SC, juntamente com o adjuvante óleo metilado de soja em ensaios no Brasil; Report No. F13-004, Document No. M-487608-03-1, GLP, unpublished, 22 March 2016
BIXAFEN_117	Resende G.	2016	Adendo 02 ao relatorio final - Determinação de resíduos de bixafen, prothioconazole e trifloxystrobin e seus respectivos metabólitos na cultura do girassol após a pulverização de bixafen & prothioconazole & trifloxystrobin 450 SC juntamente com o adjuvante óleo metilado de soja em ensaios no Brasil; Report No. F12-030, Document No. M-449336-03-1, GLP, unpublished, 10 March 2016
BIXAFEN_118	Anonymous	2013	Summary sheet bixafen & prothioconazole & trifloxystrobin 450 SC, sunflower, summary to Brazil Report F12-030, BBA sheet (translation), Document No. M-461437-01-1, GLP, unpublished, 17 April 2013
BIXAFEN_119	Resende G.	2016	Adendo 01 ao relatorio final - Determinação de resíduos de bixafen, prothioconazole e trifloxystrobin e seus respectivos metabólitos na cultura do girassol, após aplicação de bixafen & prothioconazole & trifloxystrobin 450 SC, juntamente com o adjuvante óleo metilado de soja em ensaios no Brasil; Report No. F13-006, Document No. M-487633-02-1, GLP, unpublished, 10 March 2016
BIXAFEN_120	Lenz C., Fischer D.R.	2016	BIX+PTZ+TFS 450 SC (bixafen + prothioconazole + trifloxystrobin; 125+175+150) - Magnitude of the Residue in/on Soybean Processed Commodities; Report No. RADRL042, Document No. M-544496-01-1, GLP, unpublished, 12 January 2016

Code	Author	Year	Title, Institute, Report reference
BIXAFEN_121	Lenz C., Fischer D.R.	2016	BIX+PTZ+TFS 450 SC (bixafen + prothioconazole + trifloxystrobin; 125+175+150) - Magnitude of the Residue in/on Field Corn Processed Commodities; Report No. RADRN042, Document No. M-546874-01-1, GLP, unpublished, 09 February 2016
BIXAFEN_122	Glaubitz J., Czeja C.	2013	Determination of the residues of BYF 00587 and prothioconazole in/on rape, winter and the processed fractions (oil, screwpressed; pomace; extracted meal; oil, solv. extracted; oil, crude; crude oil, preclarified; crude oil, neutralised and oil, refined) after spray application of bixafen & prothioconazole EC 225 in the field in Germany and Southern France; Report No. 12-3402, Document No. M-474270-01-1, GLP, unpublished, 18 December 2013

CHLORPYRIFOS (017)

First draft prepared by Dr M Lee, Andong National University, Republic of Korea

EXPLANATION

Chlorpyrifos is a non-systemic organophosphorous insecticide with contact, stomach and respiratory action. It was first evaluated by the JMPR in 1972 and the last periodic reviews were in 1999 for toxicology and in 2000 for residues. In 2004 and 2006, the JMPR evaluated additional uses.

The 1999 JMPR confirmed an ADI of 0–0.01 mg/kg bw for chlorpyrifos and established an ARfD of 0.1 mg/kg bw. In 2000, the JMPR defined the residue definition as *chlorpyrifos* for compliance with the MRL and dietary risk assessment for plant and animal commodities.

The residue is fat-soluble.

Chlorpyrifos was scheduled at the Fifty-first Session of the CCPR for evaluation of additional uses by the 2020 JMPR, which was postponed to the 2021 Extra JMPR. The current Meeting received information on GAP and residue trials on eggplant (brinjal).

RESIDUE ANALYSIS

Analytical methods

The fruits of the eggplant samples were homogenized at 3,000 rpm for 2 minutes. Representative 15 g homogenized samples were subjected to QuEChERS sample preparation method: add acetonitrile (containing 1% acetic acid) and salts (magnesium sulphate, sodium acetate), shake, centrifuge, and then clean-up of the organic layer by dispersive solid-phase extraction. Determination of chlorpyrifos was conducted by GC-ECD or GC-FPD. Matrix-matched standard solutions at six different concentrations, showing linearity at $r^2 \geq 0.99$, were used. Recovery tests were performed at three fortification levels (n=3 at 0.01, 0.05 and 0.10 mg/kg). Mean recovery value at each fortification level/test ranged from 80–109% (RSDs, ≤8%). The LOQ values for chlorpyrifos by GC-ECD or GC-FPD were 0.01 mg/kg. Table 1 shows the results of recovery test.

Table 1 Recovery test results for chlorpyrifos in eggplant

Trial location	Fortification level, mg/kg	Individual values, %	Mean value, %	RSD, %	Instrument used
Anand	0.01	94.0, 98.5, 95.2	96	2	GG-FPD
	0.05	112, 109, 105	109	3	
	0.10	89.9, 77.1, 80.1	82	8	
Hisar	0.01	91.6, 92.5, 91.2	92	1	GG-ECD
	0.05	94.6, 92.5, 95.2	94	2	
	0.10	90.1, 88.1, 83.1	87	4	
Kanpur	0.01	84.9, 86.7, 86.4	86	1	GG-ECD
	0.05	86.3, 88.2, 89.5	88	2	
	0.10	88.6, 87.7, 90.7	89	2	
Solan	0.01	104, 96.0, 88.0	96	8	GG-FPD
	0.05	106, 110, 97.6	105	6	
	0.10	100, 104, 99.0	101	2	
Bangalore	0.01	88.6, 86.7, 90.7	89	2	GG-ECD
	0.05	90.5, 92.4, 90.6	91	1	
	0.10	89.6, 86.6, 90.7	89	2	
Vellayani	0.01	93.4, 91.5, 94.2	93	1	GG-FPD

Trial location	Fortification level, mg/kg	Individual values, %	Mean value, %	RSD, %	Instrument used
	0.05	93.2, 90.7, 89.1	91	2	
	0.10	78.6, 80.3, 81.1	80	2	

All LOQ values, <0.01 mg/kg

USE PATTERN

The Meeting received the GAP information on eggplant from India. The information is summarised in Table 2.

Table 2 Registered use of chlorpyrifos on eggplant in India

Crop	Formulation	Application			PHI (days)
		Method	kg ai/ha	Dilution in water, L/ha	
Eggplant	20% EC (21.50% w/w)	Foliar spray	0.20	500-1,000	Not specified

Number of sprays and an application interval: not specified

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

The Meeting received residue trials on eggplant conducted in India. The detailed information are summarised in Table 3 as below.

Fruiting vegetables, other than Cucurbits

Eggplant

Supervised field trials (six trials) on eggplant were conducted during 2014–2015 in India under All India Network Project on Pesticide Residues [Sharma, K.K., 2019]. Each trial composed of three plots for treatment and one control plot. Chlorpyrifos (20% EC form.) was applied with foliar spray (500 L/ha) at a rate of 0.20 kg ai/ha. Two applications were made with an interval of 10 days at fruiting stage. Eggplant samples (1 kg) were taken at 0, 1, 3, 5, (7), (10), (15) days after the last application. Samples, separately collected from each plot, were extracted immediately (within 24 hours) after sampling and analysed. In all control samples, chlorpyrifos was not detected.

Table 3 Residue concentration of chlorpyrifos from residue trials on eggplant in India (replicate plots)

Location, Year (Variety)	Application			DALA	Chlorpyrifos, mg/kg			
	n	kg ai/ha	Int. days		Individual value			Mean
GAP: India		0.20		PHI, not specified				
Anand, 2014 (Doli-5)	2	0.20	10	0	0.22	0.23	0.22	0.22
				1	0.05	0.04	0.03	0.040
				3	0.01	0.01	0.01	0.010
				5	0.01	0.01	0.01	0.010
				7	<0.01	<0.01	<0.01	<0.01
				10	<0.01	<0.01	<0.01	<0.01
Hisar, 2014 (BR112/ Pusa Syamal)	2	0.20	10	0	1.1	0.89	0.99	0.98

Location, Year (Variety)	Application			DALA	Chlorpyrifos, mg/kg			
	n	kg ai/ha	Int. days		Individual value			Mean
				1	0.67	0.68	0.63	0.66
				3	0.38	0.37	0.36	0.37
				5	0.14	0.16	0.15	0.15
				7	0.08	0.09	0.10	0.090
				10	<0.01	<0.01	<0.01	<0.01
Kanpur, 2014 (Azad B-3)	2	0.20	10	0	1.4	1.5	1.4	1.4
				1	1.3	1.2	1.2	1.2
				3	0.60	0.59	0.59	0.59
				5	0.15	0.14	0.15	0.15
				7	0.06	0.05	0.06	0.060
				10	<0.01	<0.01	<0.01	<0.01
				15	<0.01	<0.01	<0.01	<0.01
Solan, 2015 (Pusa purple long)	2	0.20	10	0	1.1	1.1	1.1	1.1
				1	0.67	0.63	0.67	0.66
				3	0.33	0.34	0.35	0.34
				5	0.10	0.09	0.09	0.090
				7	0.05	0.05	0.05	0.050
				10	<0.01	<0.01	<0.01	<0.01
Bangalore, 2014 (Mayco-11)	2	0.20	10	0	0.14	0.15	0.12	0.14
				1	0.10	0.10	0.10	0.10
				3	0.03	0.03	0.03	0.030
				5	0.01	0.01	0.01	0.010
				7	<0.01	<0.01	<0.01	<0.01
				10	<0.01	<0.01	<0.01	<0.01
Vellayani, 2014 (-)	2	0.20	10	0	0.67	0.68	0.66	0.67
				1	0.30	0.33	0.32	0.32
				3	0.14	0.17	0.13	0.15
				5	<0.01	<0.01	<0.01	<0.01

20% EC formulation was applied.

APPRAISAL

Chlorpyrifos is a non-systemic organophosphorus insecticide with contact, stomach and respiratory action. It was first evaluated by the JMPR in 1972 and the last periodic reviews were in 1999 for toxicology and in 2000 for residues. In 2004 and 2006, the JMPR evaluated additional uses.

The 1999 JMPR confirmed an ADI of 0–0.01 mg/kg bw for chlorpyrifos and established an ARfD of 0.1 mg/kg bw. In 2000, the JMPR defined the residue definition as chlorpyrifos for compliance with the MRL and dietary risk assessment for plant and animal commodities. The residue is fat-soluble.

Chlorpyrifos was scheduled at the Fifty-first Session of the CCPR for evaluation of additional uses by the 2020 JMPR, which was postponed to the 2021 Extra JMPR. The Meeting received information on GAP and residue trials on eggplant.

Methods of analysis

Chlorpyrifos residues in eggplant were analysed by a new method using QuEChERS sample preparation method and GC-ECD or GC-FPD. Recovery test results showed recoveries of 80–109%. The LOQ was 0.01 mg/kg. The analytical method used in the eggplant residue trials was considered sufficiently validated.

Stability of residues in stored analytical samples

Residue analysis was performed within 24 hours after sample collection.

Results of supervised residue trials on crops

Fruiting vegetables, other than Cucurbits

Eggplant

Chlorpyrifos is registered for use on eggplant in India with the critical GAP involving an application rate of 0.20 kg ai/ha with no specified PHI (the maximum number of foliar sprays and minimum re-treatment intervals are not specified).

Six decline trials (including a sample collection time of 0 days) were conducted in India during 2014–2015 at a rate of 0.20 kg ai/ha with 2 applications at a 10-day interval. The residue concentrations of chlorpyrifos in eggplant were (n = 6): 0.14, 0.22, 0.67, 0.98, 1.1 and 1.4 mg/kg (highest value of 1.5 mg/kg from replicate plots).

Communication by the sponsor indicated that the local agricultural practice involves re-treatment intervals of 3–6 days. Residues declined with a median half-life of 1.63 days (1st order) and the modelled residue at a spray interval of 3 days and 2 applications differed by more than 25% from the supervised field trials. Therefore, the Meeting concluded that the supervised field trials were conducted at a significantly longer re-treatment interval and cannot be used for the estimation of a maximum residue level.

REFERENCES

Author	Year	Study title, Institute
K.K. Sharma	2019	Data/Information for Fixation of MRL of Chlorpyrifos on Brinjal. All India Network Project on Pesticide Residues, ICAR-Indian Agricultural Research Institute, New Delhi-110012, India

CLOFENTEZINE (156)

First draft prepared by Mr C. Sieke, Federal Institute for Risk Assessment, Germany

EXPLANATION

Clofentezine (3,6-bis (2-chlorophenyl)-1,2,4,5-tetrazine) (IUPAC) is an acaricide used for the control of red spider mites on a wide range of crops. It was evaluated for the first time by the 1986 JMPR and re-evaluated for residues several times up to 1992. Clofentezine underwent periodic review of the toxicology in 2005, where an ADI of 0–0.02 mg/kg bw was established and an ARfD was deemed unnecessary. The most recent periodic review of the residues was conducted by the 2007 JMPR.

The 2007 JMPR recommended the following residue definition for clofentezine:

Definition of the residue for compliance with the MRL and dietary risk assessment for plant commodities: *clofentezine.*

Definition of the residue for compliance with the MRL and dietary risk assessment for animal commodities: *sum of clofentezine, and all metabolites containing the 2-chlorobenzoyl moiety, expressed as clofentezine.*

The residue is fat-soluble.

Clofentezine was scheduled at the Fifty-first Session of the CCPR for evaluation of additional uses by the 2020 JMPR, which was postponed to the 2021 Extra JMPR. The current Meeting received new information on use patterns for clofentezine in hops, supervised field trials supported by additional plant metabolism studies, analytical methods and storage stability data.

METABOLISM AND ENVIRONMENTAL FATE

One additional metabolism study on lettuce was submitted using [tetrazine-3-^{14}C]-clofentezine. The position of the label is presented in the following figure:

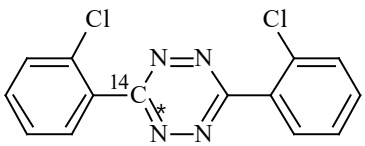
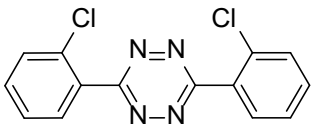
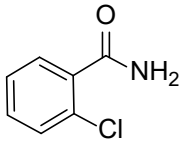
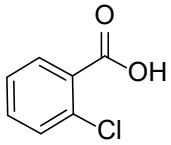
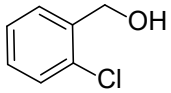
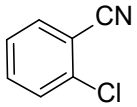
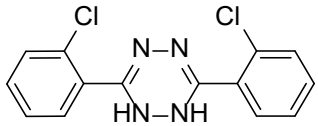
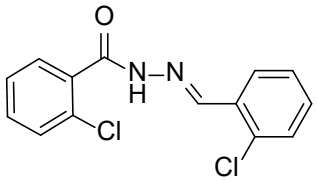


Figure 1 [tetrazine-3-^{14}C]-clofentezine

Chemical names, structures and code names of metabolites and degradation products of clofentezine discussed within this document are shown below. For a complete list of metabolites, please refer to the 2007 JMPR Monograph.

Table 1 Metabolites of clofentezine discussed within this document

Code Names	Chemical name	Structure
Clofentezine Parent (NC 21 314)	3,6-bis (2-chlorophenyl)-1,2,4,5-tetrazine	

Code Names	Chemical name	Structure
2-Chlorobenzamide	2-Chlorobenzamide	
2-Chlorobenzoic acid	2-Chlorobenzoic acid	
2-Chlorobenzyl alcohol	2-Chlorobenzyl alcohol	
2-Chlorobenzonitrile	2-Chlorobenzonitrile	
(Di-hydro-clofentezine)	3,6-bis(2-chlorophenyl)-1,2-dihydro-1,2,4,5-tetrazine	
(Hydrazide-hydrazone)	2-chlorobenzoic acid (2-chlorobenzylidene) hydrazide	

Plant metabolism

The Meeting received a new plant metabolism study with clofentezine on lettuce treated with [tetrazine-3-^{14}C]-clofentezine.

Lettuce

The metabolic fate of [tetrazine-3-^{14}C]-clofentezine (Kang, S., 2017, 14116.6100) in lettuce (*Variety "Salad Bowl"*) was investigated under outdoor conditions in California (USA). The crop was cultivated in containers and treated with a single foliar application of 0.28 kg ai/ha at BBCH 39 (rosette development complete). Untreated control plots were conducted in parallel. Lettuce leaves were harvested at the stage of commercial harvest 21 days after treatment and stored at -20 °C until analysis (up to 10 days).

Lettuce samples were homogenised with dry ice using a food processor. The total radioactive residue (TRR) in each sample was determined by combustion analysis.

A sample of treated lettuce was extracted by homogenisation with acetonitrile followed by sonication and shaking before being centrifuged to separate the supernatant from the post-extraction solids (PES). The extraction procedure was repeated using acetonitrile/water (1:1 v/v). The radioactivity in the acetonitrile and acetonitrile/water extracts was determined by liquid scintillation counting (LSC) before the extracts were combined. Radioactivity in the combined extracts was determined by LSC, and the radioactivity in the post-extraction solids (PES) was quantified by combustion.

A portion of the combined extract was concentrated to dryness by rotary evaporation and reconstituted in acetone/water (95:5, v/v). After centrifugation, the sample was analysed by reverse phase HPLC using an in-line radioactivity detector (HPLC-RAM).

Characterization and identification of components of the residue was made initially by comparison of HPLC-RAM sample retention times with a co-injected reference standard mix. Compounds were then characterized by reverse phase HPLC co-chromatography with reference standards and normal phase TLC against reference standards.

TRR levels found in lettuce leaves are summarized in Table 2.

Table 2 Total radioactivity in lettuce leaves following application of [tetrazine-3-^{14}C]-clofentezine (1 × 0.28 kg ai/ha)

Matrix	DAT	Control TRR in mg eq/kg	Treated TRR in mg eq/kg
TRR by extraction + combustion	21	<0.0006	0.243
TRR by combustion	21	<0.003	0.248

The total radioactive residue (TRR) in the treated mature lettuce foliage was 0.243 mg/kg, the majority of which was solvent extractable (0.225 mg/kg (90.8% TRR). The unextracted residue accounted for <10% TRR, (0.0184 mg/kg, 7.43% TRR), and therefore no further work was done on the post-extraction solids. The distribution of the radioactive residues in the solvent extracts and post-extraction solids for the treated lettuce sample is shown in Table 3.

Table 3 Distribution of radioactive residues in lettuce leaves treated with [^{14}C]-clofentezine

Matrix	TRR in mg eq/kg	%TRR
Extractable fraction	0.225	90.8
Acetonitrile extract	*0.203*	*82.1*
Acetonitrile/water (1:1) extract	*0.021*	*8.66*
Post-extraction solids	0.0184	7.43
Total recovery	0.243	98.2

The identification of residues in treated lettuce leaves is given in Table 4. Unchanged clofentezine was the only component of the residue present in the solvent extracts, accounting for 0.225 mg/kg (90.8% TRR). No other regions of radioactivity matched reference substances in the HPLC or TLC chromatograms.

Table 4 Distribution of radioactivity in lettuce leaves after foliar application ^{14}C-clofentizine (1 × 0.28 kg ai/ha)

Fraction	TRR Lettuce leaves mg eq/kg	% TRR
Clofentezine	0.225	90.8
2-chlorobenzamide (Ref. compound used)	*ND*	*NA*
2-chlorobenzoic acid (Ref. compound used)	*ND*	*NA*
2-chlorobenzyl alcohol (Ref. compound used)	*ND*	*NA*

Fraction	TRR Lettuce leaves mg eq/kg	% TRR
2-chlorobenzonitrile (Ref. compound used)	*ND*	*NA*
Di-hydro-clofentezine (Ref. compound used)	*ND*	*NA*
Hydrazide-hydrazone (Ref. compound used)	*ND*	*NA*
Total solvent extractable	0.225	90.8

In summary, no metabolic degradation of clofentezine was observed in lettuce within 21 days after harvest. Also, formation of unextracted residues was very limited (<10% TRR).

RESIDUE ANALYSIS

Analytical methods

For the analysis of clofentezine and its metabolite 2-chlorobenzonitrile (2-CBN) new analytical methods for plant matrices were submitted. Additionally, the metabolism study on lettuce allows conclusions on the extraction efficiency. In the following table an overview of the new methods and of the additional validation data is presented.

Table 5 Overview of analytical methods for clofentezine residues

Method	Matrix	Extraction	Clean-Up	Detection, LOQ
11735	Dry hops cones	Acetonitrile	SPE with NH2 and SCX SPE columns	HPLC-MS/MS (ESI+) Clofentezine m/z: 303→138 (quant.) m/z: 305→102 (conf.) LOQ: 0.02 mg/kg GC-MS 2-CBN m/z 137 (quantifier) m/z 102 (qualifier) m/z 139 (qualifier) LOQ: 0.05 mg/kg
14116.6103	Dry hops cones	Acetonitrile	SPE with NH2 and SCX SPE columns	HPLC-MS/MS (ESI+) Clofentezine m/z: 303→138 (quant.) m/z: 305→140 (conf.) LOQ: 0.01 mg/kg

Extraction efficiency

The lettuce metabolism study presented above (Kang, S., 2017, 14116.6100) used acetonitrile, followed by acetonitrile/water (1:1, v/v), as the extraction solvents. The first extraction with acetonitrile extracted 82.1% TRR, all of which was identified as clofentezine. Acetonitrile was therefore shown to efficiently extract clofentezine from a representative leafy crop matrix. No 2-CBN was detected in the lettuce

metabolism study at the PHI of 21 days, and therefore the extractability of 2-chlorobenzonitrile could not be evaluated.

Plant materials

Method 11735 (Barney, 2019, Report no. IR-4 PR No. 11735)

Analyte: Clofentezine, 2 Chlorobenzonitrile (2-CBN)

Principle: LC-MS/MS (clofentezine), GC-MS (2-CBN)

LOQs: Clofentezine: 0.02 mg/kg in dry hop cones

2-CBN: 0.05 mg/kg in dry hop cones

Description: Samples (1 g) were extracted with acetonitrile (10 mL) by homogenising/shaking for 1 minute using a Geno/Grinder (1000 rpm), followed by filtration using a SPE reservoir equipped with a filter frit, applying mild vacuum if needed. The filter cake was extracted using a second portion of acetonitrile (10 mL), and the volume of the combined filtrates adjusted to 20 mL with acetonitrile.

For clofentezine analysis, a 2 mL aliquot of the extract was applied to stacked pre-conditioned NH2 and SCX SPE columns, and the columns washed with acetonitrile (10 mL), collecting the eluant. The NH2 SPE column was then removed, and the SCX column washed with acetonitrile (5 mL). The combined eluant was concentrated and diluted with acetonitrile/water (90:10, v/v) for LC-MS/MS analysis in positive ion mode, using a Zorbax Eclipse Plus phenyl-hexyl column (2.1 × 100 mm, 3.5 µm).

Clofentezine mass transitions: m/z 303 → 138 (quantification),

m/z 305 → 102 (confirmation)

For 2-chlorobenzonitrile analysis, a 2 mL aliquot of the extract was applied to stacked pre-conditioned NH2, ENVI-Carb and SCX SPE columns, and the columns washed with acetonitrile (3 mL), collecting the eluant. The NH2 SPE column was removed, and the stacked ENVI-Carb and SCX columns washed with acetonitrile (2 mL). The ENVI-Carb SPE column was then removed, and the SCX column washed with acetonitrile (2 mL). The combined eluant was concentrated and diluted with acetonitrile for GC-MS analysis, using an Agilent HP-5ms UI column (30 m × 0.25 mm i.d., 0.5 µm film thickness).

2-Chlorobenzonitrile ions: m/z 137 (quantifier)

m/z 102 (qualifier)

m/z 139 (qualifier)

Table 5 Procedural recovery data for method PR No. 11735

Matrix	Analyte	Fortification mg/kg	n	Range Recovery (%)	Mean recovery (%)	% RSD	Reference
Hop, dry cones	Clofentezine	0.02	7	61-89	72	12.9	Barney, 2019, Report no. IR-4 PR No. 11735
		0.1	17	65-99	74	10.5	
		1	2	92-97	95	3.7	
		10	8	74-91	85	7.2	
Hop, dry cones	2-CBN	0.05	9	91-106	98	5.9	Barney, 2019, Report no. IR-4 PR No. 11735
		0.1	3	103-107	105	1.9	
		1	20	81-99	88	5.6	
		10	3	81-86	83	3.0	

Method 14116.6103 (Jutson, 2019, Report no. 14116.6103; McInerney, K., 2019, 100130852)

Analyte: Clofentezine

Principle: LC-MS/MS

LOQ: Clofentezine: 0.01 mg/kg in dry hop cones

Description: Samples (1 g) were extracted with acetonitrile (10 mL) by shaking for 1 minute using a Geno/Grinder (1000 rpm), centrifuged (10 minutes at 4000 rpm) and the supernatant transferred into a clean tube. The pellet was extracted using a second portion of acetonitrile (10 mL), centrifuged and the supernatant combined with the first extract.

A 2 mL aliquot of the extract was applied to a pre-conditioned MCX SPE cartridge and allowed to flow through under gravity before eluting with acetonitrile (4 mL) under gravity. The total eluant was applied to NH2 SPE cartridge and allowed to flow through under gravity, the tube was washed with acetonitrile (1 mL), the washings applied to the cartridge and eluted under gravity. The sample volume was adjusted to 10 mL with ultra-pure water and formic acid (0.01 mL) added. Samples were analysed by LC-MS/MS in positive ion mode, using a Phenomenex Synergi Polar-RP column (2 × 50 mm, 2.5 µm).

Clofentezine mass transitions: m/z 303 → 138 (quantification),

m/z 305 → 140 (confirmation)

Table 7 Procedural recovery data for method 14116.6103

Matrix	Analyte	Fortification mg/kg	n	Range Recovery (%)	Mean recovery (%)	% RSD	Reference
Hop, dry cones	Clofentezine m/z 303 → 138	0.01	5	81-89	84	3.9	Jutson, 2019, Report no. 14116.6103
		0.1	5	84-90	87	3.2	
Hop, dry cones	Clofentezine m/z 305 → 140	0.01	5	74-91	81	9.0	
		0.1	5	81-91	86	5.4	
Hop, dry cones	Clofentezine m/z 303 → 138	0.01	5	71-73	72	1.5	Independent lab validation, McInerney, K., 2019, 100130852
		0.1	5	70-73	72	2.1	
Hop, dry cones	Clofentezine m/z 305 → 140	0.01	5	70-81	74	5.9	
		0.1	5	70-71	70	0.8	

USE PATTERN

For the purpose of estimating new maximum residue levels, use pattern information for clofentezine in hops from the USA were provided.

Table 18 List of uses of clofentezine considered by the current Meeting

Crop or cropgroup	Country	Rate	Number of treatments	Pre-harvest interval (PHI)
Hops	USA	0.28 kg ai/ha (Concentrations:	1	21 days

Crop or cropgroup	Country	Rate	Number of treatments	Pre-harvest interval (PHI)
		0.15 kg ai/hL boom sprayer 0.06 kg ai/hL airblast)		

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

Residue levels were reported as measured. Application rates were always reported as clofentezine 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. Residue values from the trials conducted according to the maximum GAP that have been used for the estimation of maximum residue levels are underlined. Growth stages are reported according to the BBCH scale (https://www.julius-kuehn.de/media/Veroeffentlichungen/bbch%20epaper%20en/bbch%20skala%20englisch.html).

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 percent recovery.

Hops

Residues of clofentezine in hops cultivated in the USA were investigated in a study by Barney W. (2019, IR-4 PR No. 11735).

A total of 9 supervised trials were carried out on hops in the USA during 2016 and 2018. Two of the trials were decline trials and the other seven were at harvest trials. The two trials conducted in Washington state in 2018 (trials WA3 and WA4) were located 5.3 km apart and treated at the same date. Although sites had different soil types (sandy loam and loam soils) and different hop varieties were used, the Meeting considered these trials as not sufficiently different to be independent.

In all trials, applications were made using backpack mist blower or airblast spray equipment. Samples were collected 20–22 days after application. In the decline trials, additional samples were taken at 15–16, 25–26, 30–31 and 35–37 days after application. Duplicate samples of wet cones were collected from each control and treated plot. The wet cones were dried at 60 ± 7 °C to a moisture content of approximately 8–12% (3.6–4.6% for treated samples from trial OR312, 0-5.1% for control samples from trial OR5, 8.2–16.5% for samples from trial ID6) before sampling of dry cones.

Samples of dry cones were maintained frozen at generally -20 °C up to analysis. Accompanying storage stability investigations on dry hop cones concluded that clofentezine is not stable in spiked samples. Average recoveries represented 67% (day 0), 28% (86 days), 11% (182 days) and <2% (368 days) of the fortified concentrations of 2 mg/kg. No concurrent recoveries were conducted.Compared to initial concentrations at day zero, the percent remaining was 40% (86 days), 16% (182 days) and <3% (386 days).

In addition, the decline of residues over frozen storage in two field trial samples was investigated. During the first analysis 383–386 days after harvest and re-analysis at 751–754 days after harvest, 125% and 107% of the first measurement were recovered.

The metabolite 2-chlorobenzonitrile was demonstrated to be stable for at least 368 days under frozen storage conditions using fortified samples.

The maximum interval from harvest to analysis was 404 days for the four trials conducted in 2016. In five additional trials conducted in 2018, analysis was performed close to sample collection. The maximum interval from harvest to analysis was 27 days for the 2018 trials.

Samples were analysed for clofentezine by HPLC-MS/MS and 2-chlorobenzonitrile by GC-MS using method 11735. The method has been validated with LOQs of 0.02 mg/kg for clofentezine and 0.05 mg/kg for 2-chlorobenzonitrile in dry hop cones (see Section analytical methods).

Table 19 Residues of clofentezine in hops

Location, year (variety)	Application	Residues (mean), mg eq./kg				
Trial-ID	kg ai/ha	kg ai/hL	Sample	DALA	Clofentezine	2-CBN
Wisconsin, USA, 2016 (Crystal) 11735.16-WI410 Storage interval: 384 d	0.279	0.037	Hop, dry cones	21	1.3, 1.0 (1.2)	<0.05, <0.05 (<0.05)
Idaho, USA, 2016 (Newport) 11735.16-ID170 Storage interval: 386 & 754 d	0.280	0.030	Hop, dry cones	21	386 d storage: 0.66, 1.4 (1.0) 754 d storage: 0.90, 1.6 (1.2)	386 d storage: 0.14, 0.21 (0.18)
Oregon, USA, 2016 (Nugget) 11735.16-OR312 Storage interval: 383 & 751 d	0.286	0.034	Hop, dry cones	22	383 d storage: 2.0, 2.5 (2.2) 751 d storage: 2.2, 2.6 (2.4)	383 d storage: 0.14, 0.17 (0.16)
Washington, USA, 2016 (Tomahawk) 11735.16-WA392 Storage interval: 404 d	0.280	0.030	Hop, dry cones	15 21 26 30 35	1.2, 1.2 (1.2) 0.55, 0.55 (0.55) 0.32, 0.33 (0.32) 0.42, 0.50 (0.46) 0.29, 0.16 (0.22)	2× <0.05 2× <0.05 2× <0.05 2× <0.05 2× <0.05
Wisconsin, USA, 2018 (Cascade) 11735.19-WI2 Storage interval: 15 d	0.287	0.051	Hop, dry cones	21	1.7, 2.3 (2.0)	0.13, 0.17 (0.15)
Washington, USA, 2018 (Galena) 11735.19-WA3 Storage interval: 7 d	0.280	0.031	Hop, dry cones	20	2.5, 2.8 (2.7)	0.12, 0.12 (0.12)
Washington, USA, 2018 (Nugget) 11735.19-WA4 Storage interval: 7 d	0.282	0.020	Hop, dry cones	20	2.5, 2.6 (2.6)	0.13, 0.14 (0.14)

Location, year (variety)	Application	Residues (mean), mg eq./kg				
Trial-ID	kg ai/ha	kg ai/hL	Sample	DALA	Clofentezine	2-CBN
Oregon, USA, 2018 (Nugget) 11735.19-OR5 Storage interval: 20 d	0.284	0.030	Hop, dry cones	22	2.4, 2.5 (2.4)	<0.05, 0.13 (0.09)
Idaho, USA, 2018 (Super Galena) 11735.19-ID6 Storage interval: 27 d	0.279	0.040	Hop, dry cones	16 22 25 31 37	2.3, 2.5 (2.4) 1.3, 1.8 (1.6) 1.7, 1.7 (1.7) 0.39, 0.33 (0.36) 0.32, 0.42 (0.37)	0.15, 0.22 (0.18) <0.05, 0.11 (0.08) 0.14, 0.17 (0.16) <0.05, 0.14 (0.10) 2× <0.05

2-CBN: 2-chloro-benzonitrile
DALA: days after last application

APPRAISAL

Clofentezine (3,6-bis (2-chlorophenyl)-1,2,4,5-tetrazine) (IUPAC) is an acaricide used for the control of red spider mites on a wide range of crops. It was evaluated for the first time by the 1986 JMPR and re-evaluated for residues several times up to 1992. Clofentezine underwent periodic review of the toxicology in 2005, where an ADI of 0–0.02 mg/kg bw was established and an ARfD was deemed unnecessary. The most recent periodic review of the residues was conducted by the 2007 JMPR.

The 2007 JMPR recommended the following residue definition for clofentezine:

Definition of the residue for compliance with the MRL and dietary risk assessment for plant commodities: *clofentezine.*

Definition of the residue for compliance with the MRL and dietary risk assessment for animal commodities: *sum of clofentezine, and all metabolites containing the 2-chlorobenzoyl moiety, expressed as clofentezine.*

The residue is fat-soluble.

Clofentezine was scheduled at the Fifty-first Session of the CCPR for evaluation of additional uses by the 2020 JMPR, which was postponed to the 2021 Extra JMPR. The current Meeting received new information on use patterns for clofentezine in hops, supervised field trials supported by additional plant metabolism studies, analytical methods and storage stability data.

Plant metabolism

The fate of clofentezine in plants was last evaluated by the 2007 JMPR following foliar spray application to lemons, apples, peaches and grapes. A detailed assessment of these studies is presented in the Report of the 2007 JMPR. The current Meeting received an additional plant metabolism study on lettuce.

The metabolism of [tetrazine-3-^{14}C]-clofentezine in lettuce was investigated under outdoor conditions by the application of one foliar spray at 0.28 kg ai/ha. The treatment was performed at BBCH 39 (rosette development complete). Mature lettuce leaves were sampled 21 days after the treatment.

Total radioactive residues (TRR) were 0.243 mg eq/kg (combustion measurement). The sample was extracted with acetonitrile, followed by a mixture of acetonitrile/water (1/1; v/v), which recovered ~91% of the TRR from the leaves. Post extraction solids amounted 7.4% of the TRR.

The identification of the radioactive residues revealed unchanged clofentezine as the only residue in lettuce, representing 100% of the radioactivity in the extract and 90.2% of the TRR.

Methods of residue analysis

The current Meeting received additional analytical methods for the determination of parent clofentezine and its metabolite 2-CBN (2-Chlorobenzonitrile) in hops.

Methods 11735 and 14116.6103 both rely on liquid extraction with acetonitrile, followed by solid-phase extraction for clean-up. Parent clofentezine is measured via LC-MS/MS (ESI+), while its metabolite 2-CBN is quantified by GC-MS. Following validation experiments, LOQs of 0.02 mg/kg and 0.05 mg/kg were demonstrated for clofentezine and 2-CBN, respectively.

Storage stability

The current Meeting received information on the storage stability of clofentezine and 2-CBN in dry hops, investigated together with the supervised field trial samples.

In fortified hops samples (homogenized dry cones) clofentezine was quickly degraded. Average percentages remaining compared to initial concentrations (normalized to 100%) at day 0 were 40% (86 days), 16% (182 days) and < 3% (386 days). No concurrent recovery tests have been conducted.

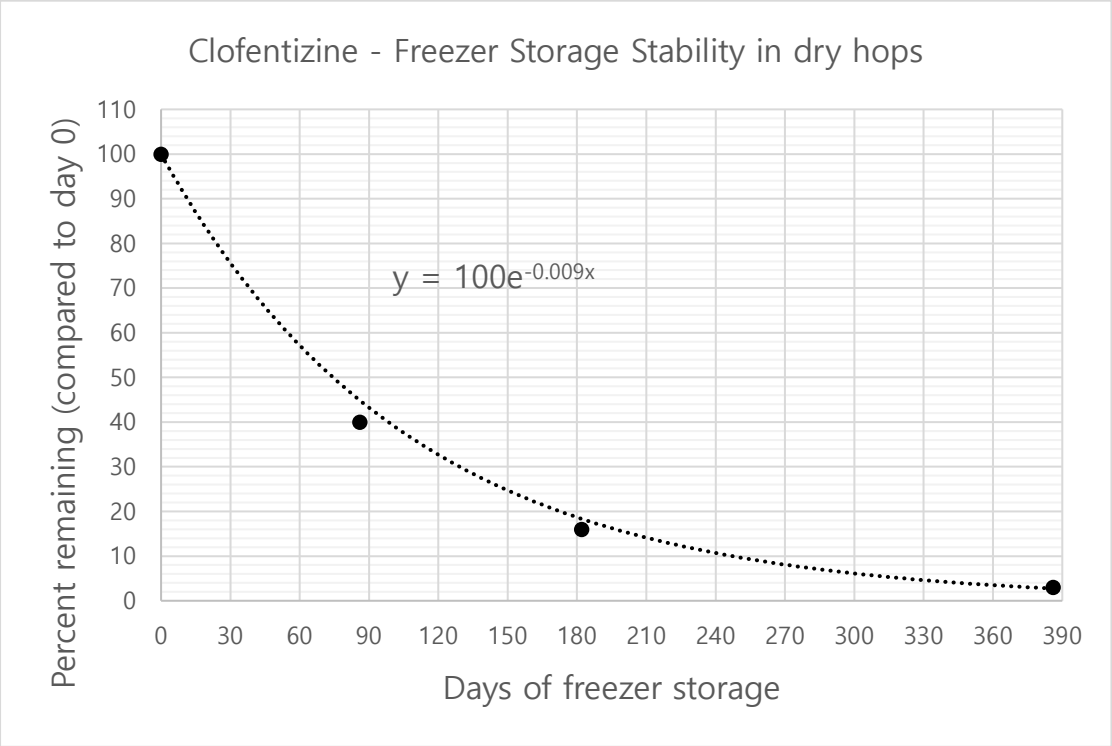


Figure 1 1st order decline of clofentezine residues in dry hops under freezer storage conditions

In addition, incurred residues in two field trial samples were investigated. In the re-analysis at 751–754 days after harvest, 125% and 107% of the first measurement (made 383–386 days after harvest) were recovered. Based on the decline kinetics in the storage stability samples, over 90% of the clofentezine

present at harvest would have dissipated by the time the first analysis was made. Without an analysis much closer to harvest, the Meeting could not use the analysis from the field trial samples to ascertain stability of incurred residues during storage.

The Meeting concluded that clofentezine in dry hops cones is not stable during freezer storage. According to the decline observed in stored fortified samples, analysis of the frozen field trial samples should be conducted within one month to recover at least 70% of the initial residue concentration.

For 2-CBN, no decline was observed in fortified samples. The Meeting concluded, that 2-CBN is stable in dry hops cones for at least one year.

Definition of the residue

The current Meeting received new data on the metabolism of clofentezine following foliar treatment of lettuce.

Following foliar application to lettuce, parent clofentezine was the only residue identified and represented > 90% of the TRR.

The Meeting therefore confirmed its previous residue definition for compliance with the MRL and dietary risk assessment for plant commodities.

Results of supervised residue trials on crops

The Meeting received supervised trial data for applications of clofentezine on hops from the USA.

Hops, dry cones

Clofentezine is registered in the USA for the use on hops with a maximum GAP involving one foliar spray of 0.28 kg ai/ha and a PHI of 21 days.

Supervised field trials conducted in the USA on hops were provided approximating the critical GAP. However, several trial samples had been stored longer than one month and degradation of the residue during storage cannot be excluded. Also, two of the trials (11735.19-WA3 & 11735.19-WA4) were conducted in close proximity and on the same dates, these trials were therefore not considered as independent by the Meeting.

Residues of clofentezine in hops, dry cones from independent trials, where analysis was conducted within one month of harvest, were (n = 4): 1.7, 2.0, 2.4, 2.7 mg/kg.

The Meeting estimated a maximum residue level of 7 mg/kg and an STMR of 2.2 mg/kg for clofentezine in hops, dry cones.

Residues in animal commodities

The additional use on hops does not affect the dietary burden of livestock animals. No re-evaluation of clofentezine residues in animal commodities was conducted.

RECOMMENDATIONS

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

Definition of the residue for compliance with the MRL and dietary risk assessment for plant commodities: *clofentezine.*

Definition of the residue for compliance with the MRL and dietary risk assessment for animal commodities: *sum of clofentezine, and all metabolites containing the 2-chlorobenzoyl moiety, expressed as clofentezine.*

The residue is fat-soluble.

Table 1 Recommendations for residues of clofentezine from the 2021 Extra JMPR

Commodity		Recommended maximum residue level mg/kg		STMR or STMR-P, mg/kg
CCN	Name	New	Previous	
DH 1100	Hops, Dry	7	-	2.2

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for clofentezine is 0–0.02 mg/kg bw. The International Estimated Daily Intakes (IEDIs) for clofentezine were estimated for the 17 GEMS/Food Consumption Cluster Diets using the STMR or STMR-P values estimated by the JMPR. The results are shown in Annex 3 of the 2021 Extra JMPR Report.

The IEDIs ranged from 0–4% of the maximum ADI. The Meeting concluded that long-term dietary exposure to residues of clofentezine from uses considered by the JMPR is unlikely to present a public health concern.

Acute dietary exposure

The 2005 JMPR decided that an ARfD for clofentezine was unnecessary. The Meeting therefore concluded that the acute dietary exposure to residues of clofentezine from the use considered by the Meeting is unlikely to present a public health concern.

REFERENCES

Code	Author	Year	Title, Institute, Report reference
14116.6100	Kang, S.	2017	Metabolism of [14C]-Clofentezine in Lettuce, Report No. 14116.6100, GLP: yes; Unpublished, 08 Aug 2017
141166.6103	Jutson, J.I.	2019	Validation of an Analytical Method for the Determination of Clofentezine in Hops Report No. 141166.6103, GLP: yes; Unpublished, 18 Apr 2019
100130852	McInerney, K.	2019	Independent Laboratory Validation Clofentezine in Hops, Report No. 100130852, GLP: yes; Unpublished, 19 Jul 2019
IR-4 PR No. 11735	Barney, W.	2019	Clofentezine: Magnitude of the Residue on Hops, Report No. IR-4 PR No. 11735, GLP: yes; Unpublished, 01 Apr 2019

CLOTHIANIDIN (238)

First draft prepared by Dr Doherty, the Environmental Protection Agency, United States of America

APPRAISAL

Clothianidin (ISO common name) is a broad-spectrum, neonicotinoid insecticide with registered uses on multiple crops. Clothianidin is a major metabolite of thiamethoxam (245). Thiamethoxam was scheduled at the Fifty-first Session of the CCPR for evaluation of additional uses by the 2020 JMPR, which was postponed to the 2021 Extra JMPR. The Meeting also considered residues of clothianidin arising from the uses of thiamethoxam. The current Meeting received information on analytical methods, field trials and processing studies to support estimation of maximum residue levels in commodities of persimmon, barley, rice, sorghum, sweet corn and wheat.

Clothianidin was evaluated for the first time by the 2010 JMPR, which established an acceptable daily intake (ADI) of 0–0.1 mg/kg bw and an acute reference dose (ARfD) of 0.6 mg/kg bw. Clothianidin underwent subsequent evaluations by the JMPR in 2011, 2012 and 2014.

The definition of the residue for compliance with the MRLs and for dietary risk assessment for animal and plant commodities is *clothianidin*.

The residue is not fat-soluble.

Methods of analysis

The Meeting received method validation and concurrent recovery data for use of Method AG-765 (reviewed by the 2010 JMPR) and the related Method GRM.009.02A, an unnamed method for analysis of residues in Japanese persimmon, as well as the QuEChERS multiresidue method. All methods were demonstrated to have adequate performance for recovery of clothianidin, with an LOQ of 0.01 mg/kg (0.02 mg/kg in Japanese persimmon).

Storage stability

No new information on the stability of clothianidin residues during frozen storage was provided. The 2010 JMPR had determined that residues of clothianidin are stable for 1–2 years under frozen (-18 °C) conditions for a large range of commodities, including the high-water and high-starch commodities considered by the current Meeting. The stability of residues in crops under consideration by the present Meeting is considered to be adequately demonstrated for the periods that field trial samples had been stored prior to analysis.

Results of supervised residue trials on crops

The Meeting received data from supervised residue trials generated following application of thiamethoxam to crops and GAP information for thiamethoxam on persimmon, wheat, barley, rice, sorghum (grain and sweet) and sweet corn. In making recommendations for maximum residues levels, the Meeting first determined whether a Codex MRL has been adopted based on the recommendation of previous JMPRs for residues of clothianidin, from the use of clothianidin on the crop. Under the assumption that growers are unlikely to use both thiamethoxam and clothianidin, both at the critical GAP, during any one growing season, the Meeting made a recommendation based on which source (application of clothianidin, *per se*, or residues arising from use of thiamethoxam) resulted in the highest residue estimates.

Japanese Persimmon

The critical thiamethoxam GAP for Japanese persimmon is from the Republic of Korea and consists of three applications, on a 10-day interval, of a 2000-fold dilution of the formulation (equivalent to 5 g thiamethoxam/hL). Applications are made to the point of run-off and there is a 7-day PHI.

Residues of clothianidin arising from use of thiamethoxam in independent trials provided to the current Meeting and approximating the critical GAP were (n = 2): < 0.02 (2) mg/kg.

The 2014 JMPR evaluated residues in Japanese persimmon in trials approximating the critical GAP. Residues of clothianidin arising from use of thiamethoxam in independent trials were (n = 2): < 0.02 (2) mg/kg.

The Meeting decided to combine data from the two sets of trials (n = 4): < 0.02 (4) mg/kg.

The maximum expected residue of clothianidin from the use of thiamethoxam on Japanese persimmon is < 0.02 mg/kg. The Meeting noted that a Codex MRL already exists for residues of clothianidin in Japanese persimmon, via the Group of Pome Fruits, at 0.4 mg/kg (STMR = 0.1 mg/kg, HR = 0.2 mg/kg). The Meeting confirmed its previous recommendation.

Wheat

Labels were provided for registrations of thiamethoxam in Mexico (2 foliar applications at 56 g ai/ha, 7-day RTI, 14-day PHI) and the USA (seed treatment at 52 g ai/100 kg seed + 2 foliar applications at 96 g ai/ha, 5-day RTI, 30-day PHI). Residue decline data indicated that for those two GAPs, the rate is the determining factor in residues at harvest rather than the RTI or the PHI. Therefore, the Meeting decided that the GAP from the USA is the critical GAP.

Trials in the USA approximated the US GAP for application rate and timing, but with a significantly shorter interval between the last application and harvest (14 days). The Meeting decided that the data are not suitable to support recommendations based on the US GAP. The Meeting agreed to consider the GAP from Mexico.

The Meeting determined that the seed treatment application used in the trials was not likely to contribute significantly to residues at harvest and decided that the US trials can be adjusted using proportionality to approximate the GAP from Mexico.

The Meeting noted that the results from the trial in Grand Island, NE (Trial C33-0170) are inconsistent within the residue decline data (< 0.01 mg/kg in the 7, 10, 14 and 17 DALA samples and 5.0 mg/kg in the 21 DALA sample) and in terms of the full set of available field trials (residue range = 0.015 to 0.18 mg/kg). The Meeting agreed not to consider data from that trial in any residue recommendations.

Residues (unscaled) of clothianidin arising from use of thiamethoxam in independent trials were (n = 16): < 0.01 (7), 0.012, 0.017, 0.018, 0.023, 0.026, 0.029, 0.034, 0.13 and 0.15 mg/kg.

When scaled (scaling factors range from 0.57 to 0.59), residues were (n = 16): < 0.01 (7), < <u>0.01</u>, <u>0.01</u>, 0.011, 0.013, 0.015, 0.017, 0.019, 0.075 and 0.087 mg/kg.

The Meeting noted that the Codex MRL for clothianidin in wheat grain of 0.02 mg/kg does not accommodate the expected clothianidin residues arising from the use of thiamethoxam considered by the current Meeting. Therefore, the Meeting used the data listed above to estimate a new maximum residue level of 0.15 mg/kg and an STMR of 0.01 mg/kg for wheat grain to replace its previous recommendation.

Based on the registered use including triticale, the Meeting decided to extrapolate the residue estimates to triticale.

Barley

Labels were provided for registrations of thiamethoxam in Mexico (2 foliar applications at 56 g ai/ha, 7-day RTI, 14-day PHI) and the USA (seed treatment at 52 g ai/100 kg seed + 2 foliar applications at 96 g ai/ha, 5-day RTI, 30-day PHI). Residue decline data indicated that for those two GAPs, the rate is the determining factor in residues at harvest rather than the RTI or the PHI. Therefore, the Meeting decided that the GAP from the USA is the critical GAP.

Trials in the USA approximated the US GAP for application rate and timing, but with a significantly shorter interval between the last application and harvest (14 days). The Meeting decided that the data are not suitable to support recommendations based on the US GAP. The Meeting agreed to consider the GAP from Mexico.

The Meeting determined that the seed treatment application used in the trials was not likely to contribute significantly to residues at harvest and decided that the US trials can be adjusted using proportionality to approximate the GAP from Mexico.

Residues (unscaled) of clothianidin arising from use of thiamethoxam in independent trials were (n = 12): < 0.01 (3), 0.015, 0.022, 0.025, 0.028, 0.041, 0.044, 0.049, 0.053 and 0.072 mg/kg.

When scaled (scaling factors range from 0.57 to 0.61), residues were (n = 12): < 0.01 (4), 0.013, 0.014, 0.016, 0.024, 0.026, 0.028, 0.031 and 0.042 mg/kg.

The Meeting noted that the Codex MRL for clothianidin in barley grain of 0.04 mg/kg does not accommodate the expected clothianidin residues arising from the use of thiamethoxam considered by the current Meeting. Therefore, the Meeting used the data listed above to estimate a new maximum residue level of 0.07 mg/kg and an STMR of 0.015 mg/kg for barley grain to replace its previous recommendation.

Based on the registered uses including oats and barley being an example representative commodity of the subgroup that includes oats, the Meeting decided to extrapolate the residue estimates to oats.

Rice

The critical thiamethoxam GAP for rice is from Japan and consists of two applications of a 1000x dilution in up to 1500 L/ha (equivalent to 97.5 g ai/ha) with a PHI of 14 days; a re-treatment interval is not specified.

Residues of clothianidin arising from use of thiamethoxam in husked rice from independent trials approximating the critical GAP were (n = 11): 0.07, 0.09, 0.11 (2), 0.12, 0.14, 0.16 (3), 0.18 and 0.26 mg/kg. Of those, two trials were done in paddy rice with residues of 0.12 and 0.14 mg/kg.

The Meeting noted that the Codex MRL for clothianidin in rice of 0.5 mg/kg (STMR = 0.145 mg/kg) accommodates the expected clothianidin residues arising from the use of thiamethoxam on upland and paddy rice considered by the current Meeting. Therefore, the Meeting confirmed its previous recommendations for residues of clothianidin in rice.

Grain sorghum

The critical thiamethoxam GAP for grain sorghum is from the USA and consists of three applications: a seed treatment at 2.97 g ai/100 kg seed followed by two in-season foliar applications, each at 96 g ai/ha, on a 7-day interval, with a PHI of 14 days.

Trials were conducted in the USA using the in-season applications only. The Meeting determined that residues of thiamethoxam coming from the seed treatment use were not likely to contribute significantly to residues at harvest.

Residues of clothianidin arising from use of thiamethoxam in grain sorghum from independent trials approximating the critical GAP were (n = 11): < 0.01 (4), 0.015, 0.018, 0.029, 0.031, 0.039, 0.046 and 0.078 mg/kg.

The Meeting noted that the Codex MRL for clothianidin in sorghum grain of 0.01 (*) mg/kg does not accommodates the expected clothianidin residues arising from the use of thiamethoxam considered by the current Meeting. Therefore, the Meeting used the data listed above to estimate a new maximum residue level of 0.15 mg/kg and an STMR of 0.018 mg/kg for sorghum grain to replace its previous recommendation.

Sweet corn

The critical thiamethoxam GAP for sweet corn is from the USA and consists of three applications: a seed treatment at 52 g ai/100 kg seed followed by two in-season foliar applications, each at 96 g ai/ha, on a 5-day interval, with a PHI of 1 day.

Residues of clothianidin arising from use of thiamethoxam in sweet corn (kernel + cob with husk removed) from independent trials approximating the critical GAP were (n = 12): < 0.01 (12) mg/kg.

The Meeting noted that a Codex MRL has been adopted for clothianidin residues in sweet corn (corn on the cob) at 0.01 (*) mg/kg (STMR, HR = 0.01 mg/kg). Furthermore, The Meeting noted that the only crop in the Subgroup of sweet corns is sweet corn and that the representative commodity is sweet corn (corn on the cob, kernels plus cob with husk removed) and agreed to estimate a new maximum residue level of 0.01(*) mg/kg, an STMR of 0.01 mg/kg and an HR of 0.01 mg/kg for residues of clothianidin in the Subgroup of sweet corns (Subgroup 020F) and withdrew its previous recommendation for sweet corn (corn on the cob).

Grasses for sugar or syrup production

Sweet sorghum

The critical thiamethoxam GAP for sweet sorghum is from the USA and consists of three applications: a seed treatment at 2.97 g ai/100 kg seed followed by two in-season foliar applications, each at 96 g ai/ha, on a 7-day interval, with a PHI of 14 days.

Trials were conducted using the in-season applications only. The Meeting determined that residues of clothianidin coming from thiamethoxam seed treatment use were not likely to contribute significantly to residues at harvest. Residues of clothianidin arising from use of thiamethoxam in sweet sorghum cane from independent trials approximating the critical GAP were (n = 4): 0.039, 0.062, 0.067 and 0.20 mg/kg.

The Meeting estimated a maximum residue level of 0.4 mg/kg, an HR of 0.2 mg/kg and an STMR of 0.0645 mg/kg for sorgo or sorghum, sweet.

Residues in animal feeds

Forage of cereal grains

Wheat

The critical thiamethoxam GAP for forage is from the USA (seed treatment at 52 g ai/100 kg seed + 2 foliar applications at 96 g ai/ha, 5-day RTI, 7-day PHI).

Residues of clothianidin arising from use of thiamethoxam in forage from trials in the USA approximating the US GAP were (n = 17): < 0.011, < 0.018, 0.017, 0.024 (2), 0.026, 0.043, 0.054, 0.060, 0.072, 0.078, 0.10, 0.13, 0.16, 0.17, 0.19 and 0.21 mg/kg.

The registration in the USA includes barley, oat, rye, triticale and wheat. The residue data (as received) for wheat forage listed above have a median residue of 0.06 mg/kg and a highest residue of 0.21 mg/kg. The Meeting agreed to extrapolate the median and highest residue estimates from wheat forage to forage of barley, oat, rye and triticale.

Sorghum

The critical thiamethoxam GAP for sorghum is from the USA and consists of three applications: a seed treatment at 2.97 g ai/100 kg seed followed by two in-season foliar applications, each at 96 g ai/ha, on a 7-day interval, with a PHI of 14 days.

Trials were conducted using the in-season applications only. The Meeting determined that residues of clothianidin coming from thiamethoxam seed treatment use were not likely to contribute significantly to residues at harvest. Residues of clothianidin arising from use of thiamethoxam in sorghum forage from independent trials approximating the critical GAP were (n = 10): 0.099, 0.10, 0.11, 0.14, 0.17, 0.21, 0.22, 0.26 (2) and 0.29 mg/kg (as received).

The Meeting estimated a median residue of 0.19 mg/kg (as received) and a highest residue of 0.29 mg/kg (as received) in sorghum forage to replace the previous estimates.

Sweet corn

The critical thiamethoxam GAP for sweet corn is from the USA and consists of three applications: a seed treatment at 52 g ai/100 kg seed followed by two in-season foliar applications, each at 96 g ai/ha, on a 7-day interval, with a PHI of 1 day for forages.

Residues of clothianidin arising from use of thiamethoxam in sweet corn forage from independent trials approximating the critical GAP were (n = 12): 0.017, 0.029, 0.037, 0.038, 0.044, 0.078, 0.09 (2), 0.1, 0.12 (2) and 0.22 mg/kg (as received).

The Meeting estimated a median residue of 0.084 mg/kg (as received) and a highest residue of 0.22 mg/kg (as received) in sweet corn forage to replace the previous estimates.

Hay of cereal grains

Wheat

The critical thiamethoxam GAP for hay is from the USA (seed treatment at 52 g ai/100 kg seed + 2 foliar applications at 96 g ai/ha, 5-day RTI, 7-day PHI).

Residues of clothianidin arising from use of thiamethoxam in wheat hay from trials in the USA approximating the US GAP were (n = 17): 0.015, 0.034, 0.058 (2), 0.064, 0.080, 0.091, 0.15, 0.20 (2), 0.23, 0.26, 0.31, 0.40, 0.43, 0.60 and 0.72 mg/kg.

Barley

The critical thiamethoxam GAP for hay is from the USA (seed treatment at 52 g ai/100 kg seed + 2 foliar applications at 96 g ai/ha, 5-day RTI, 7-day PHI).

Residues of clothianidin arising from use of thiamethoxam in barley hay from trials in the USA approximating the US GAP were (n = 12): 0.014, 0.027, 0.048, 0.053, 0.063, 0.067, 0.072, 0.089, 0.096, 0.10, 0.21 and 0.23 mg/kg.

The registration in the USA includes barley, oat, rye, triticale and wheat. The Meeting determined via a Mann-Whitney U Test that the residue data listed above for wheat and barley hay are not statistically different and agreed to combine the data. Residues of clothianidin in wheat and barley hay arising from thiamethoxam treatment approximating the US GAP were (n = 29): 0.014, 0.015, 0.027, 0.034, 0.048, 0.053, 0.058 (2), 0.063, 0.064, 0.067, 0.072, 0.080, 0.089, 0.091, 0.096, 0.10, 0.15, 0.20 (2), 0.21, 0.23 (2), 0.26, 0.31, 0.40, 0.43, 0.60 and 0.72 mg/kg.

Based on the registered uses and on the data for wheat and barley hay and on an OECD standard dry-matter content of 88%, the Meeting estimated a maximum residue level of level of 1 mg/kg (dw), a median residue of 0.09 mg/kg (as received) and a highest residue of 0.72 mg/kg (as received) in hay of wheat, barley, oat, rye and triticale.

Straw of cereal grains

Wheat

Labels were provided for registrations of thiamethoxam in Mexico (2 foliar applications at 56 g ai/ha, 7-day RTI, 14-day PHI) and the USA (seed treatment at 52 g ai/100 kg seed + 2 foliar applications at 96 g ai/ha, 5-day RTI, 30-day PHI).

For wheat straw, the trials in the USA did not match the USA GAP due to the straw being harvested 14 DALA rather than 30 days specified for the PHI. Therefore, the Meeting decided that the data are not suitable to support recommendations based on the US GAP. The Meeting agreed to consider the GAP from Mexico. As noted for wheat grain, the Meeting determined that residues of thiamethoxam coming from the seed treatment use were not likely to contribute significantly to residues at harvest and that residues from the US trials can be adjusted using proportionality to approximate the GAP from Mexico.

Residues (unscaled) of clothianidin arising from use of thiamethoxam in wheat straw from independent trials were (n = 17): 0.016, 0.021, 0.028, 0.031, 0.071, 0.072, 0.084, 0.087, 0.090, 0.097, 0.10, 0.12, 0.24, 0.26, 0.29, 0.37 and 0.46 mg/kg.

When scaled (scaling factors range from 0.57 to 0.59), residues were (n = 17): < 0.01, 0.012, 0.016, 0.018, 0.041 (2), 0.049, 0.050, 0.052, 0.056, 0.059, 0.070, 0.14, 0.15, 0.17, 0.21 and 0.27 mg/kg.

Barley

Labels were provided for registrations of thiamethoxam in Mexico (2 foliar applications at 56 g ai/ha, 7-day RTI, 14-day PHI) and the USA (seed treatment at 52 g ai/100 kg seed + 2 foliar applications at 96 g ai/ha, 5-day RTI, 30-day PHI).

For barley straw, the trials in the USA did not match the USA GAP due to the straw being harvested 14 DALA rather than 30 days specified for the PHI. Therefore, the Meeting decided that the data are not suitable to support recommendations based on the US GAP. The Meeting agreed to consider the GAP from Mexico. As noted for barley grain, the Meeting determined that residues of thiamethoxam coming from the seed treatment use were not likely to contribute significantly to residues at harvest and that residues from the US trials can be adjusted using proportionality to approximate the GAP from Mexico.

Residues (unscaled) of clothianidin arising from use of thiamethoxam in barley straw from independent trials were (n = 12): < 0.01, 0.022, 0.032, 0.034, 0.041, 0.044, 0.053, 0.059, 0.067, 0.074, 0.085 and 0.11 mg/kg.

When scaled (scaling factors range from 0.57 to 0.59), residues were (n = 12): < 0.01, 0.013, 0.019, 0.020, 0.024, 0.026, 0.030, 0.034, 0.039, 0.042, 0.049 and 0.064 mg/kg.

The registration in Mexico includes barley, oat, triticale and wheat. Noting that residues appear to be similar between the straw data for wheat and barley, the Meeting agreed to explore a common set of residue estimates for these straw commodities. A Mann-Whitney U-Test indicated that the residue distributions from wheat and barley are significantly different. The OECD MRL calculator suggested maximum residue levels of 0.4 mg/kg (median = 0.054 mg/kg) for wheat straw and 0.1 mg/kg for barley (median = 0.028 mg/kg), all on an as-received basis.

The Meeting noted that the maximum residue level, median residue and highest residue estimates in hay are greater than those in straw and agreed to apply those estimates to straw. Furthermore, noting that the straw commodities of different cereal crops are generally indistinguishable from each other when in trade, the Meeting agreed to make recommendations for the straw of cereals included on the Mexico label (barley, oat, triticale, wheat).

Based on OECD standard dry-matter content of 88%, the Meeting estimated a maximum residue level of 1 mg/kg (dw), a median residue of 0.09 mg/kg (as received) and a highest residue of 0.72 mg/kg (as received) in straw and fodder of wheat, barley, oat and triticale.

The above recommendations replace the previous recommendations, each at 0.2 mg/kg, for barley straw and fodder and wheat straw and fodder.

Rice

The critical thiamethoxam GAP for rice is from Japan and consists of two applications of a 1000× dilution in up to 1500 L/ha (equivalent to 97.5 g ai/ha) with a PHI of 14 days; a re-treatment interval is not specified.

Residues of clothianidin arising from use of thiamethoxam in rice straw from independent trials approximating the critical GAP were (n = 11): 0.01 (2), 0.02 (2), 0.03 (3), 0.04, 0.05, 0.10 and 0.13 mg/kg.

Based on OECD dry-matter content of 88%, the Meeting estimated a maximum residue level of 0.2 mg/kg (dw), a median residue of 0.030 mg/kg (as received) and a highest residue of 0.13 mg/kg (as received) for residues of clothianidin in rice straw and fodder, dry.

Sorghum

The critical thiamethoxam GAP for sorghum is from the USA and consists of three applications: a seed treatment at 2.97 g ai/100 kg seed followed by two in-season foliar applications, each at 96 g ai/ha, on a 7-day interval, with a PHI of 14 days.

Trials were conducted using the in-season applications only. The Meeting determined that residues of clothianidin coming from thiamethoxam seed treatment use were not likely to contribute significantly to residues at harvest. Residues of clothianidin arising from use of thiamethoxam in sorghum stover from independent trials approximating the critical GAP were (n = 10): 0.038, 0.045, 0.067, 0.068, 0.077, 0.10, 0.17, 0.18, 0.20 and 0.46 mg/kg (as received).

The Meeting noted that the established Codex MRL for residues of clothianidin in sorghum straw and fodder (dry) of 0.01(*) mg/kg does not accommodate the expected clothianidin residues arising from the use of thiamethoxam considered by the current Meeting. Therefore, the Meeting used the data above

to estimate a maximum residue level of 0.8 mg/kg (dw), a median residue of 0.0885 mg/kg (as received) and a highest residue of 0.46 mg/kg (as received) in sorghum straw and fodder, dry to replace the previous estimates.

Sweet corn

The critical thiamethoxam GAP for sweet corn is from the USA and consists of three applications: a seed treatment at 52 g ai/100 kg seed followed by two in-season foliar applications, each at 96 g ai/ha, on a 7-day interval, with a PHI of 26 days.

Residues of clothianidin arising from use of thiamethoxam in sweet corn stover from independent trials approximating the critical GAP were (n = 4): < 0.01 (3) and 0.021 mg/kg (as received).

Based on OECD standard dry-matter content of 83%, the Meeting estimated a maximum residue level of 0.05 mg/kg (dw), a median residue of 0.01 mg/kg (as received) and a highest residue of 0.021 mg/kg (as received) in sweet corn stover.

Fate of residues during processing

The Meeting received data showing the effect of processing wheat grain into aspirated grain fractions, bran, flour, middlings, shorts and germ; rice into hulls, bran and polished rice; grain sorghum into aspirated grain fractions and flour; and sweet sorghum into syrup. Processing factors and residue estimates are summarized below. The Meeting noted the conclusion of the 2010 Meeting that thiamethoxam was "essentially stable during the hydrolysis conditions simulating food processing conditions." On that basis, the Meeting concluded that clothianidin is not likely to have formed and the processing factors directly reflected the changing concentrations of clothianidin during the processing of cereal grain RACs into processed commodities.

Table 1 Processing factors and residue estimates for clothianidin in wheat, sorghum and sweet sorghum commodities

Raw commodity	Residue in RAC, mg/kg			Processed commodity	Processing Factors		Residue in processed commodity, mg/kg		
	Max	STMR	HR		Individual	Best estimate	Max-P	STMR-P	HR-P
Wheat grain	3	0.01	--	Aspirated fractions	3.8, 10	6.9	--	0.069	--
				Bran	1.8, 1.9	1.85	5.55	0.0185	--
				Flour	0.6, 0.69	0.645	--	0.00645	--
				Middlings	0.78, 0.81	0.795	--	0.00795	--
				Shorts	0.89, 0.91	0.90	--	0.009	--
				Germ	1.8, 1.8	1.8	5.4	0.018	--
Sorghum grain	0.15	0.018	--	Aspirated fractions	8.2, 17	12.6	--	0.227	--
				Flour	0.55, 0.72	0.635	--	0.0114	--
Sorghum cane	0.4	0.0645	--	Syrup	0.92	0.92	--	0.059	--

Based on the maximum residue estimates above and the OECD rounding classes, the Meeting estimated maximum residue levels of 6 mg/kg for wheat bran and 6 mg/kg for wheat germ. The Meeting decided to apply the processing factor for wheat bran (1.85) to barley, giving a maximum residue level of 0.15 mg/kg. The estimated STMR-Ps for the processed commodities considered by the Meeting are shown in Table 1. Furthermore, the Meeting decided to apply the processing factors for wheat bran (1.85) and flour (0.645) to barley and triticale, giving STMRs as follows:

Barley bran = 0.028 mg/kg,

Barley flour = 0.0097 mg/kg,

Triticale flour = 0.00645 mg/kg.

For rice commodities, the processing study provided residue analysis for rice grain, rice hulls, rice bran and polished rice, but not for husked rice. The Meeting derived a processing factor for husked rice by mathematically reconstituting the commodity based on residues in bran, residues in polished rice and their respective material balances reported in the processing study. Processing factors describing residues relative to the rice RAC (grain) and from husked rice are shown in Table 2.

Table 2 Processing factors and residue estimates for clothianidin in rice commodities

Raw commodity	Residue in starting commodity, mg/kg			Processed commodity	Processing Factors		Residue in processed commodity, mg/kg		
	Max	STMR	HR		Individual	Best estimate	Max-P	STMR-P	HR-P
Rice grain	See text below			Husked rice	0.38, 0.81	0.595	--	--	--
				Hulls	3.5, 5.0	4.25	--	--	--
				Bran	0.79, 1.4	1.1	--	--	--
				Polished rice	0.32, 0.68	0.5	--	--	--
Husked Rice	0.5 [a]	0.145 [a]		Hulls	6.24, 9.17	7.7	3.8	1.1	--
				Bran	1.81, 2.08	1.95	0.97	0.28	--
				Polished rice	0.833, 0.857	0.845	0.42	0.12	--

[a] 2010 JMPR

For rice, the Meeting estimated maximum residue levels of 4 mg/kg for rice hulls, 1 mg/kg for rice bran and 0.5 mg/kg for polished rice. Furthermore, the Meeting used the residue estimates and the processing factor from rice grain to husked rice to derive residue estimates for rice grain. The Meeting estimated a maximum residue level of 0.9 mg/kg and a median residue of 0.3 mg/kg for rice grain.

Residues in animal commodities

The Meeting has added feed items and their associated residues to the dietary burden calculation used by the 2014 JMPR. Dietary burden calculations are provided in Annex 6; the dietary burden estimates are summarized below.

Table 3 Estimated maximum and mean dietary burdens of farm animals

Animal	Dietary burden estimates, ppm							
	Canada-US		European Union		Australia		Japan	
	Maximum	Mean	Maximum	Mean	Maximum	Mean	Maximum	Mean
Beef cattle	0.478	0.239	0.976	0.258	1.184 [a]	0.883 [c]	0.148	0.087
Dairy cattle	0.616	0.378	0.793	0.292	1.143 [b]	0.863 [d]	0.394	0.266
Broiler poultry	0.12	0.115	0.199	0.069	0.239	0.239	0.036	0.036
Laying hen	0.115	0.115	0.313 [e]	0.134	0.239	0.239 [f]	0.08	0.08

[a] Highest maximum cattle dietary burden suitable for MRL estimates for mammalian tissues.

[b] Highest maximum dairy cattle dietary burden suitable for MRL estimates for milk

[c] Highest mean 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 and eggs

[f] Highest mean poultry dietary burden suitable for STMR estimates for poultry tissues and eggs

Farm animal feeding studies

Cattle

A cattle-feeding study with clothianidin was reviewed by the 2010 JMPR. Animals were dosed at 0.27, 0.8, or 2.6 ppm in the diet. Clothianidin residues were < 0.02 mg/kg in all samples. Based on the results of the feeding study with clothianidin and on the dietary burden, residues of clothianidin from consumption of clothianidin residues in feeds are expected to be < 0.02 mg/kg in all animal commodities.

Clothianidin residues in mammalian commodities may also arise from consumption of thiamethoxam in treated feeds. In a feeding study with thiamethoxam reviewed by the 2010 JMPR, animals were dosed at 2, 6, or 20 ppm in the diet. Clothianidin residues were as follows:

Table 4 Clothianidin residue levels in animal commodities from 2, 6 and 20 ppm dosage groups

Matrix	2 ppm in feed		6 ppm in feed		20 ppm in feed	
	Maximum	Mean	Maximum	Mean	Maximum	Mean
Muscle	< 0.01 mg/kg	< 0.01 mg/kg	< 0.01 mg/kg	< 0.01 mg/kg	< 0.01 mg/kg	< 0.01 mg/kg
Fat	Not analysed				< 0.01	< 0.01
Kidney	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
Liver	0.049	0.039	0.14	0.12	0.38	0.27
Milk	0.006	< 0.005	0.02	0.014	0.07	0.043

The dietary burdens for thiamethoxam relevant for estimating residues of clothianidin from thiamethoxam in animal commodities (see section on thiamethoxam) and the anticipated residues are shown in Table 5.

Table 5 Anticipated residues of clothianidin from thiamethoxam in animal commodities

Thiamethoxam feeding study	Feed level (ppm) for milk/egg residues	Residues (mg/kg) in milk	Feed level (ppm) for tissue residues	Clothianidin Residues (mg/kg)			
				Muscle	Liver	Kidney	Fat
Maximum residue level beef or dairy cattle							
Feeding study	20	0.043	20	< 0.01	0.38	< 0.01	< 0.01
Dietary burden and high residue	20.6	0.044	20.6	< 0.01	0.39	< 0.01	< 0.01
STMR beef or dairy cattle							
Feeding study	6 20	0.014 0.043	6 20	< 0.01 < 0.01	0.12 0.27	< 0.01 < 0.01	-- < 0.01
Dietary burden and residue estimate	18.8	0.041	18.8	< 0.01	0.257	< 0.01	< 0.01

The Meeting estimated new maximum residue levels for liver at 0.4 mg/kg and milks at 0.05 mg/kg to replace its previous recommendations and confirmed its previous recommendations of 0.02(*) mg/kg for meat (from mammals other than marine mammals); edible offal (mammalian), except liver; and mammalian fat.

Poultry

Residues of clothianidin in poultry commodities may occur following consumption of feed bearing residues of clothianidin as well as consumption of feed bearing residues of thiamethoxam. Poultry-feeding studies with clothianidin or thiamethoxam are not available. In the laying hen clothianidin metabolism study, test animals were dosed at 134 ppm in the feed. In the laying hen thiamethoxam metabolism study, test animals

were dosed at either 98 or 112 ppm in the feed. The residues of clothianidin observed in those studies, as well as the estimated residues of clothianidin based on the ratio of the dose from the metabolism study and either the maximum or mean dietary burden for poultry are shown in Table 6.

Table 6 Estimated residues of clothianidin in poultry commodities following consumption of clothianidin or thiamethoxam in the diet

	Metabolism study		Maximum		Mean	
Test substance →	Clothianidin	Thiamethoxam	Clothianidin	Thiamethoxam	Clothianidin	Thiamethoxam
Dose/dietary burden	134 ppm	98 ppm	0.313 ppm	11.8 ppm	0.151 ppm	11.8 ppm
Commodity	Observed clothianidin residue, mg/kg		Estimated clothianidin residue, mg/kg		Estimated clothianidin residue, mg/kg	
Muscle	0.0465	0.0119	0.00011	0.0014	0.000052	0.0014
Fat + Skin	0.00742	0.0277	0.000017	0.0033	0.0000084	0.0033
Liver	0.165	3.071	0.00039	0.37	0.00019	0.37
Eggs	0.142	0.0514	0.00033	0.0062	0.00016	0.0062

The Meeting confirmed its previous recommendations of 0.01(*) mg/kg for poultry meat, poultry fat and eggs and made a new recommendation of 0.4 mg/kg for poultry edible offal, with an HR and STMR both at 0.37 mg/kg, to replace its previous recommendation.

RECOMMENDATIONS

On the basis of the data obtained from supervised trials, the Meeting concluded that the residue levels listed below for clothianidin from the 2021 Extra JMPR are suitable for establishing maximum residue limits and for IEDI and IESTI assessments.

Definition of the residue for compliance with the MRL and dietary risk assessment for plant and animal commodities: *clothianidin.*

The residue is not fat-soluble.

Table 7 Recommendations for residues of clothianidin from the 2021 Extra JMPR

CCN	Crop/Commodity	Recommended maximum residue level mg/kg		STMR or STMR-P, mg/kg	HR or HR-P, mg/kg
		New	Previous		
CF 0640	Barley bran, processed	0.15 c,T		0.028 c,T	
	Barley hay	1 (dw)		Median: 0.09 (as)	Highest: 0.72 (as)
AS 0640	Barley straw and fodder, Dry	1 (dw) c,T	0.2	Median: 0.09 (as) c,T	Highest: 0.72 (as) c,T
GC 0640	Barley	0.07 c,T	0.04	0.015 c,T	
PE 0112	Eggs	0.01(*)	0.01(*)	0.0062	0.0062
MO 0099	Liver of cattle, goats, pigs & sheep	0.4	0.2	0.257	0.39
ML 0106	Milks	0.05	0.02	0.041	
AS 0647	Oat straw and fodder, Dry	1 (dw) c,T		Median: 0.09 (as) c,T	Highest: 0.72 (as) c,T
GC 0647	Oats	0.07 c,T		0.015 c,T	
	Oat hay	1 (dw)		Median: 0.09 (as)	Highest: 0.72 (as)
PO 0111	Poultry, edible offal of	0.4	0.1	0.37	0.37
PF 0111	Poultry fats	0.01(*)	0.01(*)	0.0033	0.0033

CCN	Crop/Commodity	Recommended maximum residue level mg/kg		STMR or STMR-P, mg/kg	HR or HR-P, mg/kg
		New	Previous		
PM 0110	Poultry meat	0.01(*)	0.01(*)	0.0014	0.0014
CM 1206	Rice bran, Unprocessed	1 C,t		0.28 C,t	
CM 1207	Rice hulls	4 C,t		1.1 C,t	
AS 0649	Rice straw and fodder, Dry	0.2 (dw) C,t		Median: 0.03 (as) C,t	Highest: 0.13 (as) C,t
GC 0649	Rice	0.9 C,t		0.3 C,t	
CM 1205	Rice, Polished	0.5 C,t		0.12 C,t	
	Rye hay	1 (dw)		Median: 0.09 (as)	Highest: 0.72 (as)
AS 0651	Sorghum straw and fodder, Dry	0.8 (dw) c,T	0.01	Median: 0.0885 (as) c,T	Highest: 0.46 (as) c,T
GC 0651	Sorghum Grain	0.15 c,T	0.01(*)	0.018 c,T	
GS 0658	Sorgo or Sorghum, Sweet	0.4 T		0.0645 T	0.2 T
GC 2090	Sweet Corns, Subgroup of	0.01(*) C,t		0.01 C,t	0.01 C,t
GC 0447	Sweet corn (Corn-on-the-cob) (kernels plus cob with husk removed)	W	0.01(*)		
AS 0447	Sweet corn fodder	0.05 (dw) C,t		Median: 0.01 (as) c,T	Highest: 0.021 (as) c,T
GC 0653	Triticale	0.15 c,T		0.01 c,T	
	Triticale hay	1 (dw)		Median: 0.09 (as)	Highest: 0.72 (as)
AS 0653	Triticale straw and fodder, Dry	1 (dw) c,T		Median: 0.09 (as) c,T	Highest: 0.72 (as) c,T
CF 0654	Wheat bran, Processed	6 c,T		0.0185 c,T	
CF 1210	Wheat germ	6 c,T	0.02(*)	0.018 c,T	
AS 0654	Wheat straw and fodder, Dry	1 (dw) c,T	0.2	Median: 0.09 (as) c,T	Highest: 0.72 (as) c,T
GC 0654	Wheat	0.15 c,T	0.02	0.01 c,T	
	Wheat hay	1 (dw)		Median: 0.09 (as)	Highest: 0.72 (as)

CCN	Crop/Commodity	MRL, mg/kg		STMR or STMR-P, mg/kg	HR or HR-P, mg/kg
		New	Previous		
For dietary risk assessment and/or dietary burden calculations					
	Wheat and triticale flour	--	--	0.00645	--
	Wheat aspirated grain fractions	--	--	0.069	--
	Barley flour			0.0097	
	Sorghum (grain) flour	--	--	0.0114	--
	Sorghum aspirated grain fractions	--	--	0.227	--
	Sorghum (sweet) syrup	--	--	0.059	--
	Triticale hay	1 (dw)	--	0.09 (as)	0.72 (as)
	Wheat forage	--	--	0.06 (as)	0.21 (as)
	Barley forage	--	--	0.06 (as)	0.21 (as)
AF 0647	Oat forage (green)	--	--	0.06 (as)	0.21 (as)
AF 0650	Rye forage (green)	--	--	0.06 (as)	0.21 (as)
	Triticale forage	--	--	0.06 (as)	0.21 (as)
AF0651	Sorghum forage (green)	--	--	0.19 (as)	0.29 (as)
	Sweet corn forage	--	--	0.084 (as)	0.22 (as)
	Poultry muscle			0.0014	0.0014
	Poultry fat + skin			0.0033	0.0033
PE 0112	Eggs			0.0062	0.0062

T = based on thiamethoxam use only; c,T or C,t = combined clothianidin and thiamethoxam use

(as) – as received; (dw) – dry weight

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for clothianidin is 0–0.1 mg/kg bw. The International Estimated Daily Intakes (IEDIs) for clothianidin were estimated for the 17 GEMS/Food Consumption Cluster Diets using the STMR or STMR-P values estimated by the JMPR. The results are shown in Annex 3 of the 2021 Extra JMPR Report.

The IEDIs ranged from 0–2% of the maximum ADI. The Meeting concluded that long-term dietary exposure to residues of clothianidin from uses considered by the JMPR is unlikely to present a public health concern.

Acute dietary exposure

The ARfD for clothianidin is 0.6 mg/kg bw. The International Estimate of Short-Term Intakes (IESTIs) for clothianidin were calculated for the food commodities and their processed commodities for which HRs/HR-Ps or STMRs/STMR-Ps were estimated by the present Meeting and for which consumption data were available. The results are shown in Annex 4 of the 2021 Extra JMPR Report.

The IESTIs varied from 0–1% of the ARfD for children and for the general population. The Meeting concluded that acute dietary exposure to residues of clothianidin from uses considered by the present Meeting is unlikely to present a public health concern.

CYPERMETHRINS (including alpha- and zeta-cypermethrin) (118)

First draft prepared by Dr M Lee, Andong National University, Republic of Korea

EXPLANATION

Cypermethrin is a non-systemic pyrethroid insecticide with contact and stomach action. Cypermethrin was first evaluated by the JMPR in 1979 and periodic reviews were conducted in 2006 for toxicology and in 2008 for residues. Further evaluations for additional uses were conducted in 2009, 2011 and 2019.

For cypermethrins (including alpha- and zeta-cypermethrin), a group ADI of 0-0.02 mg/kg bw and a group ARfD of 0.04 mg/kg were established by the 2006 JMPR. In 2008, the JMPR established a residue definition of *cypermethrin (sum of isomers)* for compliance with the MRL and dietary risk assessment for plant and animal commodities.

The residue is fat-soluble.

Cypermethrin was scheduled at the Fifty-first Session of the CCPR for evaluation of additional uses by the 2020 JMPR, which were postponed to the 2021 Extra JMPR. The Meeting received information on GAP and residue trials on eggplant (brinjal).

RESIDUE ANALYSIS

Analytical methods

The eggplant samples were homogenized at 3,000 rpm for 2 minutes. Representative 15 g homogenized samples were subjected to QuEChERS sample preparation method: homogenize after adding acetonitrile, centrifuge after adding sodium chloride, remove moisture of the organic layer using anhydrous sodium sulphate, and then clean-up by dispersive solid-phase extraction. Determination of cypermethrin was conducted by GC-ECD. Matrix-matched standard solutions at seven different concentrations, showing linearity at $r^2 \geq 0.99$, were used. Recovery tests were performed at three fortification levels (n=3 at 0.05, 0.25 and 0.5 mg/kg). Mean recovery values at each fortification level/test ranged from 83–105% (RSDs, ≤7%). The LOQ values for cypermethrin were 0.05 mg/kg. Table 1 shows the results of the recovery test.

Table 1 Recovery test results for cypermethrin in eggplant

Trial location	Fortification level, mg/kg	Individual values, %	Mean value, %	RSD, %
Hisar	0.05	84.5, 88.4, 86.1	86	2
	0.25	88.1, 89.1, 88.4	89	1
	0.5	91.3, 91.2, 85.8	89	4
Kanpur	0.05	94.0, 87.0, 86.0	89	5
	0.25	96.0, 89.0, 94.0	93	4
	0.5	102, 100, 104	102	2
Solan	0.05	112, 104, 98.0	105	7
	0.25	113, 100, 102	105	7
	0.5	100, 103, 105	103	2
Vellayani	0.05	93.0, 86.0, 85.0	88	5
	0.25	88.0, 86.0, 93.0	89	4
	0.5	99.0, 97.0, 95.0	97	2
Ludhiana	0.05	90.5, 93.2, 92.5	92	2
	0.25	88.6, 86.3, 93.4	89	4
	0.5	81.8, 86.1, 79.9	83	4
Hyderabad	0.05	100, 104, 104	103	2
	0.25	101, 103, 102	102	1

Trial location	Fortification level, mg/kg	Individual values, %	Mean value, %	RSD, %
	0.5	97.6, 96.8, 99.0	98	1
Coimbatore	0.05	103, 100, 106	103	3
	0.25	104, 100, 103	102	2
	0.5	111, 102, 103	105	5

All LOQ values, <0.05 mg/kg

USE PATTERN

The Meeting received GAP information on eggplant from India. The information is summarised in Table 2.

Table 2 Registered use of cypermethrin on eggplant in India

Crop	Formulation	Application			PHI (days)
		Method	kg ai/ha	Quantity of water, L/ha	
Eggplant	10% EC	Foliar spray	0.050-0.070	200-800	3

Number of sprays and an application interval: not specified

RESULTS OF SUPERVISED RESIDUE TRIALS ON CROPS

The Meeting received residue trials on eggplant conducted in India. The detailed information are summarised in Table 3 below.

Fruiting vegetables, other than Cucurbits

Eggplant

Supervised field trials (seven trials) on eggplant were conducted during 2015–2016 in India under All India Network Project on Pesticide Residues [Sharma, K.K., 2019]. Each trial composed of three plots for treatment and one control plot. Cypermethrin (10% EC form.) was applied with foliar spray (500 L/ha) at a rate of 0.050 kg ai/ha. Two applications were made with an interval of 10 days at fruiting stage. Eggplant samples (1 kg) were taken at 0, 1, 3, (5), (7), (10), (15) days after the last application. Samples, separately collected from each plot, were extracted immediately (within 24 hours) after sampling and analysed. In all control samples, cypermethrin was not detected.

Table 3 Residue concentrations of cypermethrin from residue trials on eggplant in India (replicate plots)

Location, Year (Variety)	Application			DALA	Cypermethrin, mg/kg			
	n	kg ai/ha	Int. days		Individual value			Mean
GAP: India		0.050-0.070		PHI, 3 days				
Hisar, 2015 (Pusa Syamal)	2	0.05	10	0	0.80	0.57	0.68	0.68
				1	0.68	0.65	0.66	0.66
				3	0.50	0.64	0.57	0.57
				5	0.50	0.36	0.42	0.43
				7	0.28	0.22	0.25	0.25
				10	0.06	0.08	0.06	0.070
				15	<0.05	<0.05	<0.05	<0.05
Kanpur, 2016 (Azad T-3)	2	0.05	10	0	1.3	1.2	1.2	1.2
				1	0.37	0.39	0.39	0.38

Location, Year (Variety)	Application			DALA	Cypermethrin, mg/kg			
	n	kg ai/ha	Int. days		Individual value			Mean
				3	0.10	0.09	0.09	0.090
				5	<0.05	<0.05	<0.05	<0.05
				7	<0.05	<0.05	<0.05	<0.05
Solan, 2015 (Pusa purple long)	2	0.05	10	0	0.49	0.48	0.49	0.49
				1	0.25	0.26	0.25	0.25
				3	0.12	0.11	0.10	0.11
				5	0.07	0.07	0.06	0.070
				7	<0.05	<0.05	<0.05	<0.05
Vellayani, 2016 (-)	2	0.05	10	0	0.35	0.32	0.30	0.32
				1	0.16	0.16	0.18	0.17
				3	<0.05	<0.05	<0.05	<0.05
Ludhiana, 2016 (Punjab Neelam)	2	0.05	10	0	0.26	0.26	0.28	0.27
				1	0.10	0.11	0.11	0.11
				3	<0.05	<0.05	<0.05	<0.05
Hyderabad, 2015 (Utkarsha, Hybrid)	2	0.05	10	0	0.28	0.29	0.30	0.29
				1	0.23	0.22	0.22	0.22
				3	0.18	0.18	0.18	0.18
				5	0.08	0.08	0.07	0.08
				7	<0.05	<0.05	<0.05	<0.05
Coimbatore, 2016 (Dhruva)	2	0.05	10	0	0.20	0.19	0.19	0.19
				1	0.12	0.11	0.13	0.12
				3	0.10	0.10	0.10	0.10
				5	<0.05	<0.05	<0.05	<0.05
				7	<0.05	<0.05	<0.05	<0.05

10% EC formulation was applied.

APPRAISAL

Cypermethrin is a non-systemic pyrethroid insecticide with contact and stomach action. Cypermethrin was first evaluated by the JMPR in 1979 and periodic reviews were conducted in 2006 for toxicology and in 2008 for residues. Further evaluations for additional uses were conducted in 2009, 2011 and 2019.

For cypermethrins (including alpha- and zeta-cypermethrin), a group ADI of 0–0.02 mg/kg bw and a group ARfD of 0.04 mg/kg bw were established by the 2006 JMPR. In 2008, the JMPR established a residue definition of *cypermethrin (sum of isomers)* for compliance with the MRL and dietary risk assessment for plant and animal commodities. The residue is fat-soluble.

Cypermethrin was scheduled at the Fifty-first Session of the CCPR for evaluation of additional uses by the 2020 JMPR, which was postponed to the 2021 Extra JMPR. The Meeting received information on GAP and residue trials on eggplant.

Methods of analysis

Analysis of cypermethrin in eggplant samples was conducted by a new method using QuEChERS sample preparation and GC-ECD. Recovery test results showed recoveries of 83–105%. The LOQ was 0.05 mg/kg. The analytical method used in the eggplant residue trials was considered sufficiently validated.

Stability of residues in stored analytical samples

Residue analysis was performed within 24 hours after sample collection.

Results of supervised residue trials on crops

Fruiting vegetables, other than Cucurbits

Eggplant

The critical GAP for the use of cypermethrin on eggplants in India is foliar spraying with 0.07 kg ai/ha and a 3-day PHI (the maximum number of sprays and minimum re-treatment intervals are not specified).

Seven residue trials on eggplant (decline trials) were conducted in India during 2015–2017, at a rate of 0.050 kg ai/ha with 2 applications at a 10-day interval and a PHI of 3 days. The residue concentrations of cypermethrin in eggplant were (n = 7): < 0.05, < 0.05, 0.090, 0.10, 0.11, 0.18 and 0.57 mg/kg (highest value of 0.64 mg/kg from replicate plots). The trials did not match the application rate of the critical GAP.

Communication by the sponsor indicated that the local agricultural practice involves re-treatment intervals of 3–6 days. Residues declined with a median half-life of 2.35 days (1st order) and the modelled residue at a spray interval of 3 days and 2 applications differed by more than 25% compared to the supervised field trials. The Meeting concluded that the supervised field trials were conducted at a significantly longer re-treatment interval in combination with lower application rates and cannot be used for the estimation of a maximum residue level.

REFERENCES

Author	Year	Study title, Institute
K.K. Sharma	2019	Data/Information for Fixation of MRL of Cypermethrin on Brinjal. All India Network Project on Pesticide Residues, ICAR-Indian Agricultural Research Institute, New Delhi-110012, India

CYPRODINIL (207)

First draft prepared by Dr A Leahigh, the Environmental Protection Agency, United States of America

EXPLANATION

Cyprodinil is a broad-spectrum fungicide belonging to the anilinopyridine group used to control a range of pathogens including *Tapesia yallundae*, *Botrytis spp.*, *Alternaria spp.*, and *Rhynchospium secalis*. Cyprodinil was first evaluated by the JMPR in 2003 when an ADI of 0–0.03 mg/kg bw was established. The 2003 JMPR determined that an ARfD was unnecessary. The residue definition for both plants and animal commodities, for both compliance with the MRL and dietary risk assessment, is *cyprodinil*.

The residue is fat-soluble.

The Forty-third Codex Alimentarius Commission (2020) approved the new work proposals including the priority list of pesticides for evaluation by the current Meeting. The priority list included cyprodinil for evaluation of additional uses on peas, beans, and ginseng. The current Meeting received information on supervised field trials in beans, peas, and ginseng, processing studies for ginseng processed commodities as well as information on analytical methods and storage stability.

RESIDUE ANALYSIS

Analytical methods

Relevant analytical methods for the determination of residues of cyprodinil in food and feedstuffs of plant origin were previously evaluated by the 2013, 2015, and 2017 JMPR.

Method REM 141.10

Method REM 141.10 was evaluated by the 2017 JMPR. The validation data included barley grain (high starch content), lettuce (high water content), orange (high acid content) and dry crops (barley straw).

In field trials considered by the current Meeting the method was used for the determination of cyprodinil residues in fresh beans with pods and remaining plant. Procedural recoveries using this method are presented in Table 1 and demonstrate its satisfactory performance in the studies relied on in this evaluation. The analytical method has been validated for the analysis of cyprodinil in a wide range of crops and is therefore considered to be valid for the analysis of beans (whole plant, green with pods and remaining plant) samples with a validated LOQ of 0.01 mg/kg.

Table 1 Summary of concurrent/procedural recoveries of cyprodinil in beans, green, with pods.

Matrix	Analyte	Fortification level [mg/kg]	n	Individual recoveries [%]	Range of recoveries [%]	Mean recovery [%]	RSD [%]	Reference
Method REM 141.10								
Beans, green with pod	Cyprodinil	0.01	4	80, 79, 92, 82	79-92	83	7	H.P. Yozgatli N. Breyer (2018, Report Number S17-03822)
		0.10	3	72, 70, 70	70-72	70	2	
		2.5	1	72	NA	NA	NA	
Remaining Plant		0.010	5	80, 80, 88, 93, 72	72-93	83	8	
		0.10	3	77, 66, 71	66-77	71	6	
		2.0	1	69	NA	NA	NA	

Matrix	Analyte	Fortification level [mg/kg]	n	Individual recoveries [%]	Range of recoveries [%]	Mean recovery [%]	RSD [%]	Reference
		20	1	67	NA	NA	NA	

Method CGA 219417 (AG-631B)

Method CGA 219417 is a modification of Method AG-631B, which was evaluated by the 2013 and 2015 JMPR and found to be suitable for the analysis of cyprodinil in multiple crops, including dry beans (high protein) as well as high water and high acid commodities. The methods are considered equivalent for the determination of cyprodinil residues via LC-MS/MS.

Information on the performance of Method CGA 219417 was provided to the current Meeting and was used for the determination of cyprodinil residues in dry beans.

The samples were extracted once by shaking with a methanol/water mixture at room temperature. After centrifugation, an aliquot was removed and acidified. The extract was passed through a SCX SPE column and eluted with a methanol/dilute ammonia solution, and concentrated. The extract was diluted in methanol/water and analysed by LC-MS/MS. The method was validated with a LOQ of 0.02 mg/kg and a LOD of 0.0065 mg/kg. Procedural recoveries using this method are presented in Table 2 and demonstrate its satisfactory performance in the studies relied on in this evaluation.

Table 2 Summary of concurrent/procedural recoveries of cyprodinil in dry, edible beans

Matrix	Analyte	Fortification level [mg/kg]	n	Individual recoveries [%]	Range of recoveries [%]	Mean recovery [%]	RSD [%]	Reference
Method CGA 219417								
Dry, edible beans	Cyprodinil	0.0065	3	79, 102, 118	79-118	100	20	N. Trout (2006. Residue Project No. CER 04164/06)
		0.0200	3	92, 101, 112	92-112	102	10	
		0.100	2	113, 115	113-115	114	1	
		0.200	2	80, 112	80-112	96	NA	

Method GRM010.02A

Method GRM010.02A is a modification of method REM 141.01 and was evaluated by the 2017 JMPR and found to be suitable for the determination of cyprodinil residues in potato, almond nutmeat and almond hulls. Briefly, plant samples are homogenized and extracted with methanol/water (80:20; v/v). Aliquots of the extracts are cleaned-up using solid phase extraction cartridges (HLB phase). Cyprodinil is eluted in 0.1% ammonium acetate/acetonitrile (40:60; v/v). Final determination is by HPLC-MS/MS.

In comparison, using method REM 141.01, plant samples are homogenized and extracted with methanol/water (70:30; v/v). Aliquots of the extracts are acidified and cleaned-up using solid phase extraction cartridges (SCX phase). Cyprodinil is eluted in methanol/35% ammonia (95:5; v/v); the eluate is evaporated and dissolved in mobile phase. Final determination is by LC-MS/MS.

In field trials considered by the current Meeting this method was used for the determination of cyprodinil residues in dry peas. Procedural recoveries using this method are presented in Table 3 and

demonstrate its satisfactory performance in the studies relied on in this evaluation. The method has an LOQ of 0.01 mg/kg.

Table 3 Summary of concurrent/procedural recoveries of cyprodinil in dry peas

Matrix	Analyte	Fortification level [mg/kg]	n	Individual recoveries [%]	Range of recoveries [%]	Mean recovery [%]	RSD [%]	Reference
Method GRM010.02A								
Concurrent Recovery								
Dry Pea	Cyprodinil	0.010	3	83, 83, 85	83-85	84	1	K. Sagan (2017. Report Number TK0256751)
		0.10	3	83, 85, 91	83-91	86	5	
Method Validation								
Dry Pea	Cyprodinil	0.010	3	89, 92, 86	86-92	89	3	K. Sagan (2017. Report Number TK0256751)
		0.10	3	90, 90, 91	90-91	90	<1	
		0.20	3	84, 84, 85	84-85	84	0.69	

Method JLND2020RS002

The analytical method (Study report number: JLND2020RS002-A Zhiguang Hou, 2020) for cyprodinil in ginseng, dried ginseng and red ginseng was provided as a new method to the current Meeting.

Residues of cyprodinil were extracted from fresh ginseng samples with acetonitrile and distilled water by homogenizing for 2 min and from dried ginseng and red ginseng samples with acetonitrile and distilled water by shaking for 1 h. After filtering by Buchner funnel under vacuum conditions, the organic and aqueous components were separated by the addition of sodium chloride and shaking vigorously. An aliquot of the organic layer was transferred to a centrifuge tube containing PSA and anhydrous $MgSO_4$. After dispersive solid-phase extraction, the purified extract was filtered prior to analysis by LC-MS/MS. Residues of cyprodinil were determined by LC-MS/MS monitoring ion transitions at m/z 226.1→107.9 (quantitation) and m/z 226.1→132.9 (confirmation). The method has an LOQ of 0.01 mg/kg.

The method was validated for fresh, dried, and red ginseng (Table 4).

Table 4 Summary of method validation of cyprodinil in fresh ginseng, dried ginseng and red ginseng

Matrix	Analyte	Fortification level [mg/kg]	n	Individual recoveries [%]	Range of recoveries [%]	Mean recovery [%]	RSD [%]	Reference
Method JLND2020RS002								
Fresh Ginseng	Cyprodinil	0.010	5	90, 100, 100, 95, 101	90-101	97	5	JLND2020RS002, Zhiguang Hou, 2020
		0.050	5	92, 98, 89, 90, 104	90-104	95	7	
		0.50	5	109, 100, 109, 109, 107	100-109	107	4	
		5.0	5	103, 103, 104, 99, 101, 102	99-104	102	2	
Dried Ginseng		0.010	5	108, 83, 99, 99, 73, 92	73-108	92	15	
		0.050	5	106, 96, 91, 92, 86, 94	91-106	94	8	
		0.50	5	87, 89, 85, 82, 78, 84	78-89	84	5	
		5.0	5	109, 106, 101, 99, 99, 103	99-109	103	4	

Matrix	Analyte	Fortification level [mg/kg]	n	Individual recoveries [%]	Range of recoveries [%]	Mean recovery [%]	RSD [%]	Reference
Red Ginseng		0.010	5	87, 80, 102, 92, 75, 87	75-102	87	12	
		0.050	5	105, 100, 97, 84, 85, 94	84-105	94	10	
		0.50	5	83, 80, 86, 88, 81, 84	80-88	84	4	
		5.0	5	84, 82, 76, 79, 73, 79	73-84	79	6	

Stability of residues in stored analytical samples

Ginseng, Dried Ginseng, and Red Ginseng

The study (JLND2020RS002-S, Zhiguang Hou, 2020) was conducted to determine the stability of cyprodinil in fresh ginseng, dried ginseng, and red ginseng under frozen storage.

Aliquots of untreated control fresh ginseng, dried ginseng and red ginseng were fortified in duplicate with cyprodinil at 1.0 mg/kg. Storage stability samples were stored in a freezer at ≤-18 °C. Samples were analysed approximately 0, 2, 3, 6, and 9 months using a validated LC-MS/MS method described in JLND2020RS002.

Storage stability results of cyprodinil in fresh ginseng, dried ginseng, and red ginseng are summarized in Table 5. Mean remaining concentrations of cyprodinil in fresh ginseng, dried ginseng and red ginseng sample were 81–119% over the range of storage durations. Therefore, residues of cyprodinil are considered to be stable in fresh, dried, and red ginseng during frozen storage for at least 277 days.

Table 5 Summary of Stability of cyprodinil residues in Fresh Ginseng, Dried Ginseng and Red Ginseng Matrices Following Frozen Storage (≤18 °C)

Fortification [mg/kg]	Temperature [°C]	Storage [days]	Concentration [mg/kg]	Mean remaining [%] [a]	Procedural recovery [%]	Reference
Ginseng						
1.0	-18	0	1.01, 0.99	100	100, 100	JLND2020RS002-S
1.0		76	0.88, 0.92	90	103, 102	
1.0		91	0.86, 0.87	86	93, 92	
1.0		191	0.78, 0.84	81	93, 94	
1.0		277	1.08, 1.09	109	90, 85	
Dried ginseng						
1.0	-18	0	1.01, 0.97	100	96, 98	JLND2020RS002-S
1.0		76	0.83, 0.83	84	90, 90	
1.0		91	0.89, 0.88	89	86, 90	
1.0		191	0.81, 0.92	87	88, 100	
1.0		277	1.00, 1.06	104	86, 85	
Red ginseng						
1.0	-18	0	0.87, 0.89	100	91, 90	JLND2020RS002-S
1.0		76	0.99, 0.93	110	100, 100	
1.0		91	0.99, 0.99	113	92, 94	
1.0		191	0.78, 0.87	94	102, 94	

1.0		277	1.05, 1.04	119	98, 76	

[a] Calculated with reference to Day 0 recoveries.

USE PATTERN

Labels were provided for use of cyprodinil on legume vegetables in Europe (Latvia and Spain), on pulses in Canada, and on ginseng in China.

Table 6 Registered Use of Cyprodinil on Legume Vegetables, Pulses, and Ginseng provided to the 2021 Extra JMPR

Crop	Country	Formulation	Application		Spray				PHI [days]
			Method	Max [g ai/ha]	Max [g ai/hL]	L/ha	Max No.	Interval [days]	
Group 014 – Legume vegetables									
Green legumes (fresh, with pods)	Spain (Greenhouse)	Switch 62.5 WG	Foliar	375	100	1000	2	14	3
Peas, beans (fresh, with and without pods)	Latvia (Out-door)	Switch 62.5 WG	Foliar	375	94	400-800	3	10-14	14
Group 015 – Pulses									
CROP SUBGROUP 6C: Dried shelled pea and bean [a] (except soybean)	Canada	Switch 62.5 WG	Foliar	365	209	175-225	3	7	7
Ginseng									
Ginseng	China	50% WG	Foliar	450	NS	NS	2	NS	28

[a] chickpea (garbanzo bean) (*Cicer arietinum*), beans (*Lupinus* spp. including grain lupin, sweet lupin, white lupin, white sweet lupin), beans (*Phaseolus* spp. including field bean, kidney bean, lima bean (dry), navy bean, pinto bean, tepary bean,), broad bean (fava bean) (*Vicia faba*), beans (*Vigna* spp. adzuki bean, black-eyed pea, catjang, cowpea, Crowder pea, moth bean, mung bean, rice bean, southern pea, urd bean); Guar (*Cyamopsis tetragonoloba*); Lablab bean or Hyacinth bean (*Lablab purpureus*); Lentil (*Lens esculenta*); Pea (*Pisum* spp.) (includes field pea);Pigeon pea (*Cajanus cajan)*; Bean (*Phaseolus* spp.) (includes lima bean, snap bean and wax bean); Bean (*Vigna* spp.) (includes blackeyed pea, asparagus bean); Broad bean (fava bean) (*Vicia faba*)

RESULTS OF SUPERVISED RESIDUE TRIALS ON CROPS

The Meeting received data from supervised residue trials conducted on fresh beans (with pods), fresh peas (with pods), dry beans, dry peas, and ginseng.

The field trial reports included concurrent recovery data at levels reflecting those observed in the field trial samples; dates from critical events during the study, including application, harvest, storage, and analysis; and 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.

When residues were not quantifiable they are shown as below the LOQ (e.g. < 0.01 mg/kg). Residues, application rates and spray concentrations have generally been rounded to two significant figures or, for residues near the LOQ, to one significant figure so as not to represent spuriously high precision. Results have not been corrected for concurrent method recoveries. No residues of cyprodinil were measured at or above 50% of the limit of quantification (LOQ) in any of the control and reagent blank samples used in this study.

The results of the trials are summarised in Table 7, 8, 9 and 10, where residues relevant to the estimation of a maximum residue level are underlined.

Supervised trials for cyprodinil:

Class	Commodity	Tables
Legume vegetables	Beans with pods	Table 7
Pulses	Dry beans	Table 8
	Dry peas	Table 9
Ginseng	Ginseng	Table 10

Beans with pods

Eight supervised residue trials with cyprodinil on beans (fresh with pods) were conducted in Southern European countries (all within the SEU zone) in 2017 (S17-03822; Yozgatli, H P; Breyer, N; 2018). In all trials cyprodinil was applied as a WG formulation containing 375 g ai/kg. The test substance was applied three times as a foliar application at a nominal rate of 375 g ai/ha, with application intervals of 9–11 days. All applications were made at BBCH 51–79.

Samples of beans with pods were collected from all trials 14 days after the final application. Additional samples were collected from four trial sites prior to the final application (-0) and then 3, 7, and 10–11 days after the final application, to provide residue decline data. Samples were collected and stored frozen prior to analysis. Samples were stored for up to a maximum of 380 days (*ca.* 12 months) prior to analysis.

Samples were analysed for residues of cyprodinil using the validated analytical method REM 141.10 with an LOQ of 0.01 mg/kg. The performance of the method was verified by procedural recoveries of cyprodinil in the range of 70–120%.

The residues of cyprodinil in beans (fresh with pods) in Europe are summarised in Table 7.

Table 7 Residues of cyprodinil in beans (fresh with pods) in Europe.

GLP and Trial Details	Crop (Variety)	Country (Region)	Application Rate (g ai/ha)	Spray Volume (L/ha)	PHI (days)	RTI (days)	Crop Part	Residue Found (Uncorrected) Cyprodinil (mg/kg)
GAP	Beans (fresh with pods)	Latvia	3× 375	400-800	14	Oct-14		
Report: S17-03822	Bean	Italy	386	412	0	-	Beans with pods	0.39
Trial: S16-03822-01	(Shubert)	(Ravenna)	397	423	3	11		0.67

GLP and Trial Details	Crop (Variety)	Country (Region)	Application Rate (g ai/ha)	Spray Volume (L/ha)	PHI (days)	RTI (days)	Crop Part	Residue Found (Uncorrected) Cyprodinil (mg/kg)
GAP	Beans (fresh with pods)	Latvia	3× 375	400-800	14	Oct-14		
			358	382	7 11 14	10		0.46 0.48 0.25
Report: S17-03822 Trial: S17-03822-02	Bean (Kylie)	Spain (Soria)	351 366 348	374 391 371	0 3 7 11 14	- 10 11	Beans with pods	13 1.4 2.1 0.04 0.03
Report: S17-03822 Trial: S17-03822-03	Bean (Manosi)	France (Tarn et Garonne) (S France)	367 368 376	392 393 401	0 3 7 10 14	- 9 10	Beans with pods	0.05 0.59 0.42 0.19 0.13
Report: S17-03822 Trial: S17-03822-04	Beans (Plaja)	Bulgaria (Lovech)	380 382 377	405 408 403	0 3 7 10 14	- 9 11	Beans with pods	0.03 0.21 0.1 0.06 0.04
Report: S17-03822 Trial: S17-03822-05	Beans (SV1286GW)	Greece (Pella)	392 385 346	627 616 553	14	- 10 11	Beans with pods	0.01
Report: S17-03822 Trial: S17-03822-06	Beans (Primel)	Spain (Cadiz)	350 365	560 778	14	- 10	Beans with pods	0.04

GLP and Trial Details	Crop (Variety)	Country (Region)	Application Rate (g ai/ha)	Spray Volume (L/ha)	PHI (days)	RTI (days)	Crop Part	Residue Found (Uncorrected) Cyprodinil (mg/kg)
GAP	Beans (fresh with pods)	Latvia	3× 375	400-800	14	Oct-14		
			373	796		11		
Report: S17-03822 Trial: S17-03822-07	Beans (Koala)	Greece (Thessaloniki)	383 389 388	613 622 620	14	- 10 10	Beans with pods	0.03
Report: S17-03822 Trial: S17-03822-08	Beans (Gina)	Bulgaria (Pazardzhik)	399 391 384	426 417 409	14	- 9 10	Beans with pods	0.06

Pulses

Supervised trials on dry beans in Canada

Five supervised residue trials with cyprodinil on dry beans were conducted in Canada during 2006 (Report TK0256751; Sagan, K; 2017). All trials were conducted under field conditions. In all trials cyprodinil was applied as a WG formulation containing 375 g ai/kg. The test substance was applied three times as a foliar application to duplicate treatment plots at a nominal rate of 366 g ai/ha, with nominal application intervals of 7 days. All applications were made at BBCH 74–97. The Meeting determined that Trials 175, 176, and 177 were dependent based on proximity.

Samples of beans were collected from all trials 7 days after the final application. Samples were collected and stored frozen prior to analysis. Samples were stored for up to a maximum of 35 days (*ca.* 1 month) prior to analysis.

Samples were analysed for residues of cyprodinil using a modified version of the validated analytical method CGA 219417 (AG-631B) with an LOQ of 0.02 mg/kg. The performance of the method was verified by procedural recoveries of cyprodinil in the range of 70–120%.

The residues of cyprodinil in dry beans in Canada are summarised in Table 8.

Supervised trials on dry peas in Canada

Seven supervised residue trials with cyprodinil on dry peas were conducted in Canada during 2015 (Report TK0256751; Sagan, K; 2017). All trials were conducted under field conditions. In all trials cyprodinil was applied as a WG formulation containing 375 g ai/kg. The test substance was applied three times as a foliar application at a nominal rate of 366 g ai/ha, with nominal application intervals of 5–8 days. All applications were made at BBCH 75–89.

Samples of peas were collected from all trials 6–7 days after the final application. Additional samples of pea were collected from two sites at 3–4, 9–10 and 13 days after the final application to provide residue decline data. Samples were collected and stored frozen prior to analysis. Samples were stored for up to a maximum of 8.2 months prior to analysis.

Samples were analysed for residues of cyprodinil using a modified version of the validated analytical method GRM010.02A with a LOQ of 0.01 mg/kg. The performance of the method was verified by procedural recoveries of cyprodinil in the range of 70–120%.

The residues of cyprodinil in dry peas in Canada are summarised in Table 9.

Table 8 Residues of cyprodinil in dry beans in Canada.

GLP and Trial Details	Crop (Variety)	Country (Region)	Application Rate (g ai./ha)	Spray Volume (L/hA)	PHI (days)	RTI (days)	Crop Part	Residue Found (Uncorrected) Cyprodinil (mg/kg)
GAP	Dry beans and peas	Canada	3× 365	175-225	7	7		
Report: CER04164/06 Trial: T174	Dry Beans (OAC Thunder)	Canada (Thorndale, ON)	371 360 367	200 200 200	7	- 6 8	Seed	<0.02, <0.02 (<0.02)
Report: CER04164/06 Trial: T175	Dry beans (Envoy)	Canada (Elm Creek, MB)	362 365 368	200 200 200	7	- 6 8	Seed	<0.02, <0.02 (<0.02)
Report: CER04164/06 Trial: T176	Dry Beans (Envoy)	Canada (Barnsley, MB)	361 367 366	200 200 200	7	- 7 7	Seed	<0.02, <0.02 (<0.02)
Report: CER04164/06 Trial: T177	Dry Beans (Pinto)	Canada (Elm Creek, MB)	368 371 367	200 200 200	7	- 7 7	Seed	0.027, 0.023 (0.025)
Report: CER04164/06 Trial: T178	Dry Beans (Pinto)	Canada (Taber, AB)	362 351 356	200 200 200	7	- 7 7	Seed	0.047, 0.029 (0.038)

Table 9 Residues of cyprodinil in dry peas in Canada

GLP and Trial Details	Crop (Variety)	Country (Region)	Application Rate (g ai/ha)	Spray Volume (L/hA)	PHI (days)	RTI (days)	Crop Part	Residue Found (Uncorrected) Cyprodinil (mg/kg)
GAP	Dry beans and peas	Canada	3× 365	175-225	7	7		
Report: TK0256751 Trial: T769-D	Dry Peas (CDC Amarillo)	Canada (Zealandia, SK)	355 363 364	200 200 200	3 6 9 13	- 7 7	Seed	0.085 (0.080) 0.063 0.065

GLP and Trial Details	Crop (Variety)	Country (Region)	Application Rate (g ai/ha)	Spray Volume (L/hA)	PHI (days)	RTI (days)	Crop Part	Residue Found (Uncorrected) Cyprodinil (mg/kg)
GAP	Dry beans and peas	Canada	3× 365	175-225	7	7		
Report: TK0256751 Trial: T770-D	Dry Peas (CDC Amarillo)	Canada (Delisle, SK)	351 373 381	200 200 200	4 6 10 13	- 7 7	Seed	0.081 (0.048) 0.043 0.039
Report: TK0256751 Trial: T771	Dry Peas (Meadow)	Canada (Dundern, SK)	371 380 368	200 200 200	7	- 6 7	Seed	(0.037)
Report: TK0256751 Trial: T772	Dry Peas (CDC Amarillo)	Canada (Moon lake, SK)	377 363 364	200 200 200	6	- 6 8	Seed	(0.12)
Report: TK0256751 Trial: T774	Dry Peas (CDC Amarillo)	Canada (Blaine Lake, SK)	360 359 362	200 200 200	7	- 7 7	Seed	(0.054)
Report: TK0256751 Trial: T775	Dry Peas (CDC Amarillo)	Canada (Hague, SK)	371 379 361	200 200 200	7	- 8 7	Seed	(0.047)
Report: TK0256751 Trial: T776	Dry Peas (Meadow)	Canada (Glenboro, MB)	367 371 366	200 200 200	6	- 7 5	Seed	(0.11)

Ginseng

Five supervised residue trials with cyprodinil on ginseng were conducted in China during 2020 (Study report number: JLND2020RS002-A Zhiguang Hou, 2020). All trials were conducted under field conditions. In all trials cyprodinil was applied as a WG formulation containing 450 g ai/ha The test substance was applied two times as a foliar application to three treatment plots at a nominal rate of 450 g ai/ha, with nominal application intervals of 7 days, with a PHI of 28 days at each site. An additional two treatment plots were established to determine residue decline in Baishan, Huanren, and Fusong. Two independent samples were collected at each plot.

The samples from all trials were shipped to the laboratory for sample preparation and stored deep frozen in polyethylene plastic within 8 hours. All field samples were shipped to the analytical laboratory in good condition. The field sub samples were stored in a freezer at -18 °C or below until preparation. The samples were prepared following the instructions for the Residue Analytical Method JLND2020RS002.

The residues of cyprodinil in ginseng in China are summarised in Table 10.

Table 10 Residues of cyprodinil in ginseng in China

Study Details	Crop	Country (Region)	Plot	Application Rate (g ai/ha)	Water (L/ha)	PHI (days)	RTI (days)	Residue Found (Uncorrected) Cyprodinil (mg/kg)		
GAP	Fresh Ginseng	China		2× 450	NS	28	7	Sample 1	Sample 2	Average
JLND2020RS00201	Fresh Ginseng	China [Yanji, Jilin Province]	1	453,452	604,602	21	7	0.046	0.028	0.037
			2	453,453	604,604	28	7	0.018	0.020	0.019
			3	453,453	604,604	35	7	0.029	0.019	0.024
JLND2020RS00202	Fresh Ginseng	China [Baishan, Jilin Province]	1	449,452	598,602	14	7	0.035	0.014	0.025
			2	452,453	602,604	21	7	0.038	0.027	0.033
			3	449,453	598,604	28	7	0.023	0.019	0.021
			4	452,446	602,594	35	7	0.028	0.027	0.028
			5	446,447	594,596	42	7	0.206	0.023	0.115
JLND2020RS00203	Fresh Ginseng	China [Huanren, Liaonign Province]	1	447,452	596,602	14	7	0.051	0.065	0.058
			2	447,447	596,596	21	7	0.10	0.14	0.120
			3	447,453	596,604	28	7	0.026	0.014	0.020
			4	449,449	598,598	35	7	0.039	0.035	0.037
			5	452,453	602,604	42	7	0.071	0.032	0.052
JLND2020RS00204	Fresh Ginseng	China [Fusong, Jilin Province]	1	447,450	596,600	14	7	0.011	0.011	0.011
			2	449,447	598,596	21	7	0.045	0.034	0.034
			3	447,452	596,602	28	7	0.010	0.010	0.017
			4	449,453	598,604	35	7	0.023	0.019	0.020
			5	453,453	604,604	42	7	0.021	0.014	0.014
JLND2020RS00205	Fresh Ginseng	China [Ji'an, Jilin, Province]	1	447,452	596,602	21	7	0.025	0.064	0.045
			2	452,450	602,600	28	7	0.030	0.033	0.032
			3	450,453	600,604	35	7	0.022	0.010	0.016

Feed commodities

In trials on beans with pods conducted in Europe (reported previously), the analysis of feed samples (bean plants) was included. The results are shown in Table 11.

Table 11 Results from supervised trials conducted with cyprodinil on beans with pods in Europe

GLP and Trial Details	Crop (Variety)	Country (Region)	Application Rate (g ai/ha)	Spray Volume (L/hA)	PHI (days)	RTI (days)	Crop Part	Residue Found (Uncorrected) Cyprodinil
GAP	Beans (fresh with pods)	Latvia	3× 375	400-800	14	Oct-14		(mg/kg)
Report: S17-03822 Trial: S16-03822-01	Bean (Shubert)	Italy (Ravenna)	386	412	14	-	Remaining Plant	0.56
			397	423		11		
			358	382		10		

GLP and Trial Details	Crop (Variety)	Country (Region)	Application Rate (g ai/ha)	Spray Volume (L/hA)	PHI (days)	RTI (days)	Crop Part	Residue Found (Uncorrected) Cyprodinil
GAP	Beans (fresh with pods)	Latvia	3× 375	400-800	14	Oct-14		(mg/kg)
Report: S17-03822 Trial: S17-03822-02	Bean (Kylie)	Spain (Soria)	351 366 348	374 391 371	14	- 10 11	Remaining Plant	0.32
Report: S17-03822 Trial: S17-03822-03	Bean (Manosi)	France (Tarn et Garonne) (S France)	367 368 376	392 393 401	14	- 9 10	Remaining Plant	0.06, 0.07 (mean: 0.07)
Report: S17-03822 Trial: S17-03822-04	Beans (Plaja)	Bulgaria (Lovech)	380 382 377	405 408 403	14	- 9 11	Remaining Plant	0.05
Report: S17-03822 Trial: S17-03822-05	Beans (SV1286GW)	Greece (Pella)	392 385 346	627 616 553	14	- 10 11	Remaining Plant	0.08
Report: S17-03822 Trial: S17-03822-06	Beans (Primel)	Spain (Cadiz)	350 365 373	560 778 796	14	- 10 11	Remaining Plant	0.29
Report: S17-03822 Trial: S17-03822-07	Beans (Koala)	Greece (Thessaloniki)	383 389 388	613 622 620	14	- 10 10	Remaining Plant	0.05
Report: S17-03822 Trial: S17-03822-08	Beans (Gina)	Bulgaria (Pazardzhik)	399 391 384	426 417 409	14	- 9 10	Remaining Plant	0.12

FATE OF RESIUDES IN STORAGE AND DURING PROCESSING

In Processing

Ginseng

A study was submitted investigating the effect of ginseng processing on cyprodinil residues (Study report Number: JLND2020RS002, Zhiguang Hou, 2020).

Fresh ginseng samples were taken from each field site and portions were processed into dried ginseng and red ginseng with the simulation of industrial practices being followed as closely as possible. Two independent samples were collected at each plot. Dried ginseng was produced by baking samples of fresh ginseng for 24 h at 55 °C. Red ginseng was produced by steaming the fresh ginseng for ca. 2.5–3 h, cooling, drying for 6 h at 70 °C, infiltrating the dried product with water, and then drying a final time for 24 h at 55 °C.

Samples were analysed for residues of cyprodinil using Method JLND2020RS002, described above.

Table 12 Residues of cyprodinil in dried ginseng and red ginseng in China

Study Details	Crop	Country (Region)	Plot	Application Rate (g ai/ha)	Water (L/ha)	PHI (days)	RTI (days)	Residue Found (Uncorrected) Cyprodinil (mg/kg)		
GAP	Fresh Ginseng	China		2× 450	NS	28	7	Sample 1	Sample 2	Average
JLND2020 RS00201	Dried Ginseng	China [Yanji, Jilin Province]	1	453,452	604,602	21	7	0.035	0.032	0.034
			2	453,453	604,604	28	7	0.040	0.041	0.041
			3	453,453	604,604	35	7	0.031	0.034	0.033
JLND2020 RS00201	Red Ginseng	China [Yanji, Jilin Province]	1	453,452	604,602	21	7	0.033	0.028	0.031
			2	453,453	604,604	28	7	<0.01	0.051	0.031
			3	453,453	604,604	35	7	<0.01	<0.01	<0.01
JLND2020 RS00202	Dried Ginseng	China [Baishan, Jilin Province]	1	449,452	598,602	14	7	0.060	0.058	0.059
			2	452,453	602,604	21	7	0.084	0.092	0.088
			3	449,453	598,604	28	7	0.076	0.077	0.077
			4	452,446	602,594	35	7	0.11	0.11	0.110
			5	446,447	594,596	42	7	0.082	0.069	0.076
JLND2020 RS00202	Red Ginseng	China [Baishan, Jilin Province]	1	449,452	598,602	14	7	0.035	0.029	0.032
			2	452,453	602,604	21	7	0.062	0.054	0.058
			3	449,453	598,604	28	7	0.031	0.10	0.066
			4	452,446	602,594	35	7	0.058	0.046	0.052
			5	446,447	594,596	42	7	0.057	0.17	0.114
JLND2020 RS00203	Dried Ginseng	China [Huanren, Liaonign Province]	1	447,452	596,602	14	7	0.21	0.21	0.210
			2	447,447	596,596	21	7	0.47	0.41	0.440
			3	447,453	596,604	28	7	0.068	0.073	0.071
			4	449,449	598,598	35	7	0.19	0.18	0.185
			5	452,453	602,604	42	7	0.22	0.21	0.215

Study Details	Crop	Country (Region)	Plot	Application Rate (g ai/ha)	Water (L/ha)	PHI (days)	RTI (days)	Residue Found (Uncorrected) Cyprodinil (mg/kg)		
GAP	Fresh Ginseng	China		2× 450	NS	28	7	Sample 1	Sample 2	Average
JLND2020 RS00203	Red Ginseng	China [Huanren, Liaonign Province]	1	447,452	596,602	14	7	0.62	1.3	0.960
			2	447,447	596,596	21	7	1.2	0.77	0.985
			3	447,453	596,604	28	7	1.4	1.3	1.350
			4	449,449	598,598	35	7	0.34	0.56	0.450
			5	452,453	602,604	42	7	0.26	0.68	0.470
JLND2020 RS00204	Dried Ginseng	China [Fusong, Jilin Province]	1	447,450	596,600	14	7	0.046	0.036	0.041
			2	449,447	598,596	21	7	0.062	0.062	0.062
			3	447,452	596,602	28	7	0.057	0.059	0.058
			4	449,453	598,604	35	7	0.052	0.054	0.053
			5	453,453	604,604	42	7	0.052	0.043	0.048
JLND2020 RS00204	Red Ginseng	China [Fusong, Jilin Province]	1	447,450	596,600	14	7	0.029	0.026	0.028
			2	449,447	598,596	21	7	0.025	0.050	0.038
			3	447,452	596,602	28	7	0.011	0.023	0.017
			4	449,453	598,604	35	7	0.064	0.032	0.048
			5	453,453	604,604	42	7	0.059	0.094	0.077
JLND2020 RS00205	Dried Ginseng	China [Ji'an, Jilin, Province]	1	447,452	596,602	21	7	0.18	0.17	0.175
			2	452,450	602,600	28	7	0.089	0.090	0.090
			3	450,453	600,604	35	7	0.063	0.054	0.059
JLND2020 RS00205	Red Ginseng	China [Ji'an, Jilin, Province]	1	447,452	596,602	21	7	0.19	0.080	0.135
			2	452,450	602,600	28	7	0.058	0.048	0.053
			3	450,453	600,604	35	7	<0.01	0.032	0.021

APPRAISAL

Cyprodinil is a broad-spectrum fungicide belonging to the anilinopyridine group used to control a range of pathogens including *Tapesia yallundae*, *Botrytis spp.*, *Alternaria spp.*, and *Rhynchospium secalis*. Cyprodinil was first evaluated by the JMPR in 2003 when an ADI of 0–0.03 mg/kg bw was established. The 2003 JMPR determined that an ARfD was unnecessary. The residue definition for both plants and animal commodities, for both compliance with the MRL and dietary risk assessment, is cyprodinil. The residue is fat-soluble.

The Forty-third Codex Alimentarius Commission (2020) approved the new work proposals including the priority list of pesticides for evaluation by the current Meeting. The priority list included cyprodinil for evaluation of additional uses on peas, beans, and ginseng. The current Meeting received information on supervised field trials in beans, peas, and ginseng, processing studies for ginseng processed commodities as well as information on analytical methods and storage stability.

Methods of residue analysis

The analytical methods (REM 141.10, CGA 219417 and GRM010.02A) used in the legume and pulse trials received by the Meeting were considered the same as those evaluated by previous meetings (the 2013, 2015 and 2017 JMPR). These methods were validated for the analysis of cyprodinil in beans (whole plant,

green with pods and remaining plant) and dry peas with a validated LOQ of 0.01 mg/kg and in dry beans at a LOQ of 0.02 mg/kg.

The current Meeting received method validation data for use of Method JLND2020RS002. The method was demonstrated to have adequate performance for recovery of cyprodinil in ginseng and ginseng processed commodities. A LOQ of 0.01 mg/kg was validated for ginseng and its processed fractions.

Stability of pesticide residues in stored analytical samples

The stability of residues of cyprodinil in stored frozen samples was previously evaluated by the 2003, 2013, 2015, and 2017 JMPR. Stability for at least 2 years has been demonstrated for high-water, high-starch, and high-acid commodities and for at least 9 months in high-oil commodities. Samples of legume vegetables were stored frozen for up to ca. 1 year, which is covered by the available data. Although data specific to a high-protein commodity have not been evaluated, the Meeting decided the conclusion could be extrapolated to pulses, which were stored for up to 1 month (dry beans) or up to 8.2 months (dry peas). New storage stability data were provided for cyprodinil in ginseng and its processed fractions. The Meeting concluded that cyprodinil is stable at -18 °C for at least 277 days for ginseng, dried ginseng, and red ginseng, which covered the longest storage intervals of field sampling and laboratory analysis in ginseng supervised trials.

Results of supervised residue trials on crops

The Meeting received GAP information for cyprodinil on legume vegetables in Europe (Latvia and Spain), on pulses in Canada, and on ginseng and its processed fractions in China. Supervised residues trials were provided for beans (with pods), peas and beans (dried), and ginseng.

Peas (with pods)

The registered GAP in Spain for the use of cyprodinil on protected peas with pods is for two applications at up to 375 g ai/ha with application intervals of 14 days and a minimum PHI of 3 days. The Meeting received information concerning a GAP in Latvia for cyprodinil on unprotected peas with pods involving three applications at up to 375 g ai/ha with application intervals of 10–14 days and a minimum PHI of 14 days.

No residue trials on peas with pods were available to support the estimation of a maximum residue level for peas with pods. The Meeting noted that beans with pods is considered a representative crop for Codex subgroup 14B (Peas with pods) and that residue trial data on beans could be used to support a recommendation for peas with pods.

In eight supervised residue trials matching the Latvian GAP, residues of cyprodinil were (n = 8): 0.01, 0.03 (2), 0.04 (2), 0.06, 0.13, and 0.25 mg/kg.

In trials approximating the GAP from Spain evaluated by the 2017 JMPR, residues of cyprodinil in beans with pods were (n = 9): 0.34, 0.47, 0.54, 0.58, 0.60, 0.61, 0.75, 0.83, and 1.2 mg/kg.

The Meeting agreed that the GAP in Spain is the critical GAP and decided to extrapolate the recommendation of the 2017 JMPR for the Subgroup of beans with pods to the Subgroup of peas with pods.

The Meeting estimated a maximum residue level of 2 mg/kg and a STMR of 0.60 mg/kg for the Subgroup peas with pods.

Pulses

Beans (dry)

A Codex MRL has been established for residues of cyprodinil in dry beans at 0.2 mg/kg.

The registered GAP in Canada for the use of cyprodinil on dry beans and peas is for three applications at up to 365 g ai/ha with application intervals of 7 days and a minimum PHI of 7 days.

Five supervised residue trials with cyprodinil on beans were conducted in Canada and matching the Canadian GAP. The residues in dried beans were (n = 5): < 0.02, < 0.02, < 0.02, 0.025, and 0.038 mg/kg.

The Meeting noted that the residues in dried beans from the Canadian GAP are accommodated by the existing MRL; however, the Meeting agreed to extrapolate the recommendation made by the 2013 JMPR for beans, dry to the Subgroup of dry beans. The Meeting estimated a maximum residue level of 0.2 mg/kg and a STMR of 0.03 mg/kg for residues of cyprodinil in the Subgroup of dry beans and withdrew its previous recommendation.

Peas (dry)

The critical GAP for the use of cyprodinil on dry peas in Canada consists of three applications at up to 365 g ai/ha with application intervals of 7 days and a PHI of 7 days.

Seven supervised residue trials with cyprodinil on peas were conducted in Canada matching the critical GAP. Residues of cyprodinil in dried peas were (n = 7): 0.037, 0.047, 0.048, 0.054, 0.080, 0.11, and 0.12 mg/kg.

The Meeting estimated a maximum residue level of 0.2 mg/kg and a STMR of 0.054 mg/kg for residues of cyprodinil in the Subgroup of dry peas.

Ginseng

The critical GAP for the use of cyprodinil on ginseng in China consists of two foliar applications at up to 450 g ai/ha with application intervals of 7 days and a minimum PHI of 28 days.

Five supervised residue trials matching the critical GAP were conducted in China. Residues of cyprodinil on ginseng were (n = 5): 0.034, 0.037, 0.045, 0.115, and 0.120 mg/kg.

The Meeting estimated a maximum residue level for cyprodinil in ginseng of 0.3 mg/kg and a STMR of 0.045 mg/kg.

Residues in animal Feeds

Beans and Peas (vines)

The registered GAP in Spain for the use of cyprodinil on protected beans and peas (with pods) is for two applications at up to 375 g ai/ha with application intervals of 14 days and a minimum PHI of 3 days. The Meeting received information concerning a GAP in Latvia for cyprodinil on unprotected beans and peas (with pods) for three applications at up to 375 g ai/ha with application intervals of 10–14 days and a minimum PHI of 14 days.

No trials were available matching the GAP from Spain. Residues of cyprodinil in bean plants from trials matching the Latvian GAP were (n = 8): 0.05 (2), 0.07, 0.08, 0.12, 0.29, 0.32, 0.56 mg/kg. The meeting estimated a median residue of 0.1 mg/kg and highest residue of 0.56 mg/kg for bean vines and pea vines.

Fate of residues during processing

The Meeting received data showing the effect of processing ginseng into dried ginseng and red ginseng.

In the supervised residue trials with cyprodinil on ginseng discussed above, samples of ginseng were processed into dried ginseng and red ginseng. The Meeting selected the higher of the dried or red

ginseng residues for making residue estimates. Residues of cyprodinil in processed ginseng were (n = 5): 0.041, 0.062, 0.114, 0.175, and 1.4 mg/kg.

The Meeting estimated a maximum residue level of 3 mg/kg in dried and red ginseng and a STMR-P of 0.114 mg/kg.

Farm animal dietary burdens

The potential dietary burdens of cyprodinil in animals diets were calculated from the highest and median residue levels of animal feeds, estimated using the most recent version of the OECD livestock dietary burden calculator. The maximum and mean dietary burdens were estimated for beef and dairy cattle, laying hens and broiler hens in Australia, Europe, Japan, and USA-Canada.

Dietary burdens for cyprodinil are presented in Annex 6 of the 2021 Extra JMPR Report and summarised in Table 2, below, for all uses of cyprodinil on feed items evaluated by this and previous Meetings.

Table 2 Estimated maximum and mean dietary burdens of farm animals for Cyprodinil (ppm)

	USA-Canada		EU		Australia		Japan	
	Max	Mean	Max	Mean	Max	Mean	Max	Mean
Beef cattle	1.084	0.470	15.12	1.934[b]	6.667	1.476	0.702	0.702
Dairy cattle	2.499	1.030	14.82	1.518	25.32[a]	1.804[c]	0.382	0.382
Poultry – broiler	0.690	0.690	1.342	0.672	0.200	0.200	0.079	0.079
Poultry - layer	0.554	0.554	2.265[d]	0.756[e]	0.200	0.200	0.160	0.160

[a] Highest maximum beef or dairy cattle dietary burden used for mammalian milk and tissue MRL calculations.

[b] Highest mean beef or dairy cattle dietary burden used for mammalian tissue STMR estimates.

[c] Highest mean dairy cattle dietary burden used for mammalian milk STMR estimates.

[d] Highest maximum poultry dietary burden used for poultry tissue and egg MRL calculations.

[e] Highest mean poultry dietary burden used for poultry tissue and egg STMR estimates.

Residues in animal commodities

Maximum residue level and STMR estimates for poultry and ruminant commodities from previous meetings were based on maximum / mean dietary burdens of 23.3 / 1.8 ppm in cattle and 4.1 / 0.76 ppm in poultry. The current Meeting concluded that the animal dietary burden had changed insignificantly (or had decreased) and confirmed the previous recommendations.

RECOMMENDATIONS

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

Definition of the residue for compliance with the MRL and dietary risk assessment for plant and animal commodities: *cyprodinil.*

The residue is fat-soluble.

Table 3 Recommendations for residues of cyprodinil from the 2021 Extra JMPR

CCN	Crop/Commodity	Recommended maximum residue level mg/kg		STMR or STMR-P (mg/kg)
		New	Previous	
VR 0604	Ginseng	0.3	--	0.045
DV 0604	Ginseng, dried including red ginseng	3	--	0.114
VP 2061	Peas with pods, Subgroup of	2	-	0.60
VD 0071	Beans (dry)	W	0.2	-
VD 2065	Dry beans, Subgroup of (except soya beans)	0.2	--	0.03
VD 2066	Dry peas, Subgroup of	0.2	-	0.054

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The 2003 JMPR set an ADI of 0–0.03 mg/kg bw for cyprodinil. The International Estimated Daily Intakes (IEDIs) for cyprodinil were estimated for the 17 GEMS/Food Consumption Cluster Diets using the STMR or STMR-P values estimated by the JMPR. The results are shown in Annex 3 of the 2021 Extra JMPR Report.

The IEDIs ranged from 7–70% of the maximum ADI. The Meeting concluded that long-term dietary exposure to residues of cyprodinil from uses considered by the JMPR is unlikely to present a public health concern.

Acute dietary exposure

The 2003 JMPR decided that an ARfD was unnecessary. Therefore, the Meeting concluded that the acute dietary exposure to residues of cyprodinil from the uses considered is unlikely to present a public health concern.

REFERENCES

Code	Author	Year	Title, Institute, Report reference
VV-470881	Yozgatli, H.P Breyer, N	2018	Cyprodinil and Fludioxonil - Residue Study on Fresh Beans with Pods in Italy, Spain Southern France, Bulgaria and Greece in 2017
VV-512169	Sagan, K	2017	Fludioxonil/Cyprodinil WG (A9219B) & Diquat SL (A1412H) Magnitude of the Residues in or on Dry Pea (Representative Commodities for Crop Group 6C) Canada 2015
VV-486159	Tout, N	2006	Fludioxonil and Cyprodinil – Residue Levels on Dry Edible Beans from Trials Conducted with SWITCH 62.5WG in Canada during 2006
JLND2020RS002	Hou, Z	2020	Magnitude of the Residue of Cyprodinil in fresh Ginseng, dried Ginseng and red Ginseng
JLND2020RS002-S	Hou, Z	2020	Storage Stability of Cyprodinil in Ginseng For Up to 9 Months of Frozen Storage
JLND2020RS002-A	Hou, Z	2020	Method Performance Validation for the Determination of Residues of Cyprodinil in fresh Ginseng, dried Ginseng and red Ginseng by LC MS/MS

DIFENOCONAZOLE (224)

First draft prepared by Dr M Doherty, the Environmental Protection Agency, United States of America

EXPLANATION

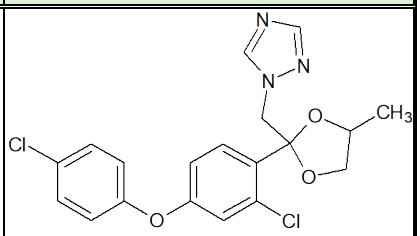
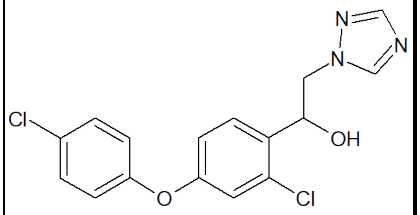
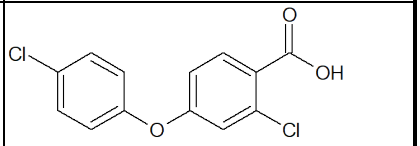
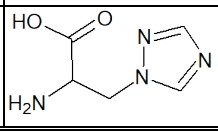
Difenoconazole (ISO common name) is a broad-spectrum conazole (triazole) fungicide used for the control of diseases in multiple crops. Its fungicidal mode of action is as a sterol demethylation inhibitor. Difenoconazole was evaluated for the first time by JMPR 2007 when an acceptable daily intake (ADI) of 0–0.01 mg/kg bw and an acute reference dose (ARfD) of 0.3 mg/kg bw were established. In 2007, 2010, 2013, 2015, and 2017, the JMPR evaluated the compound for residues and recommended a number of maximum residue levels.

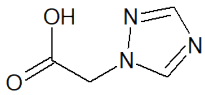
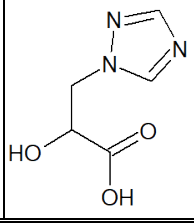
The definition of the residue for both compliance with the MRL and dietary risk assessment is *difenoconazole* for plant commodities, and *the sum of difenoconazole and 1-[2-chloro-4-(4-chloro-phenoxy)-phenyl]-2-(1,2,4-triazol)-1-yl-ethanol (CGA205375), expressed as difenoconazole*, for animal commodities.

The residue is fat-soluble.

Difenoconazole was scheduled at the Fifty-first Session of the CCPR for evaluation of additional uses by the 2020 JMPR, which was postponed to the 2021 Extra JMPR. The current Meeting received storage stability, GAP information, field trials, and processing studies to support maximum residue level estimations in commodities of cotton, cranberry, guava, and tea. In addition, the current Meeting also received seed treatment and confined rotational crop metabolism studies.

Table 1 Metabolites of difenoconazole referenced in this document

Common or code name	Chemical name molecular formula molar mass, g/mol	Structure
Difenoconazole	1-({2-[2-chloro-4-(4-chlorophenoxy)phenyl]-4-methyl-1,3-dioxolan-2-yl}methyl)-1*H*-1,2,4-triazole $C_{19}H_{17}Cl_2N_3O_3$ 406.26	
CGA205375	1-[2-chloro-4-(4-chlorophenoxy)phenyl]-2-(1*H*-1,2,4-triazol-1-yl)ethan-1-ol $C_{16}H_{13}Cl_2N_3O_2$ 350.20	
CGA189138	2-chloro-4-(4-chlorophenoxy)benzoic acid $C_{13}H_8Cl_2O_3$ 283.11	
CGA131013 Triazolylalanine	2-amino-3-(1*H*-1,2,4-triazol-1-yl)propanoic acid $C_5H_8N_4O_2$ 156.14	

Common or code name	Chemical name molecular formula molar mass, g/mol	Structure
CGA142856 Triazolylacetic acid	(1*H*-1,2,4-triazol-1-yl)acetic acid $C_4H_5N_3O_2$ 127.10	
CGA205369 Triazolyllactic acid	2-hydroxy-3-(1*H*-1,2,4-triazol-1-yl)propanoic acid $C_5H_7N_3O_2$ 157.13	

METABOLISM AND ENVIRONMENTAL FATE

The Meeting received a study investigating the metabolism of difenoconazole in spring wheat following seed treatment as well as a study investigating residues in rotational lettuce, radish, sorghum, and wheat grown under confined field conditions.

Crop Metabolism

In a metabolism study using spring wheat (Gärtner and Herrchen, 2019, Study Report SYN-018/6-40), wheat seeds were treated with either [chlorophenoxy-U-^{14}C]-difenoconazole (CP) or [triazolyl-U-^{14}C]-difenoconazole (TZ), each at a rate of 32.3 mg ai/100 g seeds. Treated seeds were grown in containers in a greenhouse. Forage was harvested at BBCH 14–21, hay at BBCH 56–65, and straw and grain at maturity.

Total radioactive residues were determined by combustion analysis with liquid scintillation counting. For samples with TRR of at least 0.01 mg eq/kg, residues were extracted twice with acetonitrile:water (4:1, v/v). A third extraction was done with either the same solvent if the second extraction liberated >10% TRR or with acetonitrile:water (1:1, v/v). A fourth extraction (acetonitrile:water, 1:1, v/v) was done if the total from previous extractions was <90% TRR. For grain grown from TZ-treated seed, additional extractions were made with acetonitrile:water (3:7, v/v, extractions 5 and 6) and with acetonitrile alone (extraction 7). Note that in the case of forage, the water content of the matrix was considered during the preparation of the extraction solvent to ensure the desired ratio of acetonitrile to water in the extract. An additional extraction procedure was used for the CP straw sample whereby the sample was extracted under reflux for 2 hours with methanol:35% NH_4OH (8:2, v/v).

Some samples of forage, hay, and straw underwent various hydrolysis treatment to release conjugated forms of the metabolites. Hydrolysis treatments included alkaline digestion (NaOH, pH 11, 48 h), pectinase or cellulase treatment, and/or acid hydrolysis (6 mol/L HCl, 100 °C, 5 h).

Residues were characterized and identified by TLC and by radio-HPLC with UV detection. Storage stability was determined by comparison of patterns of radioactive residues by TLC made at the initial extraction (within 6 months of harvest) and at the end of the study (*ca.* 3.4 years).

Table 2 Summary of total radioactive residues in spring wheat grown from [^{14}C]difenoconazole-treated seeds.

Radiolabel	Matrix	Extracted (E) Radioactivity		Non-extracted (NE) Radioactivity		TRR (E + NE)	TRR (combustion)
		%TRR [s]	mg eq/kg	%TRR [a]	mg eq/kg	mg eq/kg	mg eq/kg
Chlorophenoxy	Forage	88.3	0.131	11.7	0.017	0.148 [b]	0.097
	Hay	79.9	0.024	20.1	0.006	0.030	0.030
	Straw	64.1	0.069	35.9	0.038	0.107	0.106
	Grain	--					0.005
Triazolyl	Forage	90.8	0.198	9.3	0.020	0.218 [c]	0.180
	Hay	89.7	0.100	10.4	0.012	0.113	0.106
	Straw	78.3	0.216	21.6	0.059	0.275	0.307
	Grain	91.8	0.370	8.1	0.033	0.402	0.422

[a] Calculated on the basis of TRR (E + NE)

[b] An alternate sample had TRR of 0.104 mg eq/kg; however, the workup for analysis was not successful.

[c] An alternate sample had TRR of 0.190 mg eq/kg; however, the workup for analysis was not successful.

The extractability of residues into acetonitrile/water ranged from 64 to 78% in straw, from 80 to 90% in hay, and approximately 90% in forage and grain. Extractability was ca. 105% from a separate pair of forage samples, and the investigator noted that there were difficulties in quantitatively transferring the forage subsamples from the storage container to the extraction vessel due to thawing of the samples during the process.

Table 3 Summary of the nature of the residues in spring wheat following seed-treatment application of [^{14}C-**chlorophenoxy]**difenoconazole (32.3 mg ai/100 g seed).

	Forage		Hay		Straw	
TRR, mg eq/kg	0.148		0.030		0.107	
Identification	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Difenoconazole	2.0	0.003	3.3	0.001	5.6	0.006
CGA205375 (free and glycoside)	47	0.070	60	0.018	12	0.013
-free	6.1	0.009	10	0.003	12	0.013
-glycoside	41	0.061	50	0.015	ND	ND
Characterized	12	0.018	6.7	0.002	18	0.019
-TLC origin	2.7	0.004	3.3	0.001	1.9	0.002
-"unassigned"	3.4	0.005	--	--	--	--
-unknowns	3.4	0.005	--	--	--	--
-Unknown A	--	--	--	--	5.6	0.006
-free	--	--	--	--	ND	ND
-glycoside	--	--	--	--	5.6	0.006
-Unknown B	--	--	--	--	10	0.011
-other	2.7	0.004	3.3	0.001	2.8	0.003
PES	11	0.017	20	0.006	36	0.038
-Cellulose	--				19	0.020
-Lignin					4.6	0.0049

	Forage		Hay		Straw	
TRR, mg eq/kg	0.148		0.030		0.107	
Identification	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
-Hemicellulose					2.3	0.0024
-Acetone fraction					3.6	0.0039
-Total					30	0.032
Total	73	0.108	90	0.027	71	0.076

ND = Not detected; -- = not analysed

Table 4 Summary of the nature of the residues in spring wheat following seed-treatment application of [^{14}C-**triazolyl]**difenoconazole (32.3 mg ai/100 g seed).

	Forage		Hay		Straw		Grain	
TRR, mg eq/kg	0.218		0.113		0.275		0.402	
Identification	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Difenoconazole	ND	ND	ND	ND	1.5	0.004	ND	ND
CGA205375 (free and glycoside)	61	0.133	23	0.026	11	0.030	ND	ND
-free	7.8	0.017	3.5	0.004	11	0.030	--	--
-glycoside	53	0.116	19	0.022	ND	ND	--	--
1,2,4-triazolylacetic acid	9.2	0.020	30	0.034	41	0.113	33	0.131
CGA131013	6.4	0.014	16	0.018	11	0.031	27	0.108
Characterized	1.8	0.004	2.7	0.003	6.2	0.017	0.25	0.001
-TLC origin	--	--	--	--	--	--	--	--
-"unassigned"	ND	ND	ND	ND	--	--	--	--
-unknowns	--	--	--	--	6.2	0.017	--	--
-other	1.8	0.004	2.7	0.003	--	--	0.25	0.001
PES	9.2	0.020	11	0.012	21	0.059	8.2	0.033
-Cellulose	--				14	0.038	--	
-Lignin					2.2	0.0060		
-Hemicellulose					4.0	0.011		
-Acetone fraction					2.6	0.0072		
-Total					23	0.062		
Total	88	0.191	82	0.093	92	0.254	68	0.273

ND = Not detected; -- = not analysed

Difenoconazole was not a major residue in wheat matrices grown from treated seed. The principal extracted residue in forage extract was the CGA205375, totalling 47% TRR (CP; 0.070 mg eq/kg) and 61% TRR (TZ; 0.13 mg eq/kg); the majority of the residue was present as a glycoside conjugate. In other matrices from the CP-treated crop, no other residues occurred as major residues. Following treatment with TZ-difenoconazole, the major residues were 1,2,4-triazolylacetic acid (hay: 30% TRR, 0.034 mg eq/kg; straw: 41% TRR, 0.113 mg eq/kg; and grain: 33% TRR, 0.131 mg eq/kg) and CGA131013 (hay: 16% TRR, 0.018 mg eq/kg; straw: 11% TRR, 0.031 mg eq/kg; and grain: 27% TRR, 0.108 mg eq/kg)

Further workup of the straw post-extraction solids showed the majority of the unextracted residues to be associated with natural plant constituents, primarily cellulose.

Confined Rotational Crops

In a confined rotational crop study using leafy vegetable, grain, and root vegetable crops, difenoconazole, uniformly labelled in the 4-chlorophenyl moiety, was applied to bare soil at a rate of *ca*. 0.52 kg ai/ha (Findak and Herczog, 2010, Study Report T009582-07). Lettuce, radish, sorghum, and wheat were planted as rotational crops at intervals of 30, 60, 120, and 270 days after treatment. Lettuce was harvested as immature (BBCH 43) and mature (BBCH 49) leaves, radish leaves and roots were harvested at maturity (BBCH 49), sorghum was harvested as forage (whole plants except roots, BBCH 19) and as grain and stover at maturity (BBCH 89), and wheat was harvested as forage (BBCH 29), hay (BBCH 85), grain, and straw (both at BBCH 89). Soil samples were collected following application of the test solution, just prior to planting, and at harvest from the final plant-back from the wheat plot.

Total radioactive residues in all plant and soil samples were determined by combustion-LSC. Crop samples with TRR ≥ 0.01 mg eq/kg were extracted twice with acetonitrile:water (4:1, v/v). The solids were then extracted with acetonitrile:water (1:1, v/v). The PES was analysed by combustion-LSC. Radioactive residues were determined/characterized by HPLC with a radioanalytical detector and by TLC. Storage stability was investigated by comparing residue profiles in radish leaf extract from the initial analysis and after 230 days in frozen storage, which covers the storage duration of the analytical samples. The distribution of radioactive residues remained the same between the two analyses.

Table 5 Summary of total radioactive residues in confined rotational crops following treatment with [chlorophenoxy-U-^{14}C]difenoconazole to bare soil at ca. 0.52 kg ai/ha

Matrix	Plant-back Interval, days	Extracted (E) Radioactivity		Non-extracted (NE) Radioactivity		TRR (E + NE)	TRR (combustion)
		%TRR [a]	mg eq/kg	%TRR [a]	mg eq/kg	mg eq/kg	mg eq/kg
Immature lettuce	30	81	0.038	19	0.009	0.047	0.052
	60	95	0.018	5.3	0.001	0.019	0.020
	120	79	0.015	21	0.004	0.019	0.017
	270	64	0.007	36	0.004	0.011	0.012
Mature lettuce	30	80	0.066	20	0.017	0.083	0.095
	60	60	0.003	40	0.002	0.005	0.008
	120	76	0.013	24	0.004	0.017	0.017
	270	--	--	--	--	--	0.005
Radish leaves	30	81	0.022	19	0.005	0.027	0.026
	60	79	0.019	21	0.005	0.024	0.021
	120	80	0.067	20	0.017	0.084	0.088
	270	65	0.017	35	0.009	0.026	0.026
Radish roots	30	77	0.020	23	0.006	0.026	0.028
	60	81	0.017	19	0.004	0.021	0.022
	120	79	0.022	21	0.006	0.028	0.029
	270	79	0.015	21	0.004	0.019	0.019
Sorghum forage [b]	30	60	0.009	40	0.006	0.015	0.015
Sorghum stover [b]	30	59	0.041	41	0.028	0.069	0.066
Sorghum grain [b]	30	20	0.002	80	0.008	0.010	0.009
Wheat forage	60	71	0.005	29	0.002	0.007	0.008
	120	56	0.005	44	0.004	0.009	0.012
	270	50	0.005	50	0.005	0.010	0.011

Matrix	Plant-back Interval, days	Extracted (E) Radioactivity		Non-extracted (NE) Radioactivity		TRR (E + NE)	TRR (combustion)
		%TRR [a]	mg eq/kg	%TRR [a]	mg eq/kg	mg eq/kg	mg eq/kg
Wheat hay	60	57	0.008	43	0.006	0.014	0.018
	120	37	0.007	63	0.012	0.019	0.020
	270	39	0.007	61	0.011	0.018	0.015
Wheat straw	60	63	0.033	37	0.019	0.052	0.055
	120	57	0.028	43	0.021	0.049	0.051
	270	59	0.017	41	0.012	0.029	0.030
Wheat grain	60	--	--	--	--	--	0.004
	120	--	--	--	--	--	0.004
	270	--	--	--	--	--	0.006

[a] Calculated on the basis of extracted + non-extracted radioactivity

[b] Sorghum commodities were only analysed at the 30-day PBI due to low levels of radioactivity.

Table 6 Summary of the nature of the residues in rotational lettuce following bare soil application of [^{14}C-**chlorophenoxy]**difenoconazole (0.52 kg ai/ha)

	30-DAT planting		60-DAT planting		120-DAT planting		270-DAT planting	
Identification	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Immature Leaf	TRR→	0.047	TRR→	0.019	TRR→	0.019	TRR→	0.011
Extracted	81	0.038	95	0.018	79	0.015	64	0.007
Difenoconazole	55	0.026	82	0.016	69	0.013	42	0.005
CGA205375	5.5	0.003	11	0.002	9.9	0.002	2.8	0.0003
CGA189138	4.6	0.002	--	ND	--	ND	17	0.002
RT = 26.80 min	5.7	0.003	--	ND	--	ND	--	ND
RT = 28.07 min	9.8	0.005	--	ND	--	ND	--	ND
PES	19	0.009	5.3	0.001	21	0.004	36	0.004
Total	100	0.047	100	0.019	100	0.019	100	0.011
Mature Leaf	TRR→	0.082	TRR→	0.005	TRR→	0.017	TRR→	0.005 [a]
Extracted	80	0.066	60	0.003	76	0.013	No workup due to low radioactivity	
Difenoconazole	48	0.040	No workup due to low radioactivity		69	0.012		
CGA205375	4.8	0.004			6.6	0.001		
CGA189138	12	0.010			--	ND		
RT = 3.87 min	8.3	0.007			--	ND		
RT = 28.07 min	6.2	0.005			--	ND		
PES	20	0.017	40	0.002	24	0.004		
Total	100	0.082	100	0.005	100	0.017		

ND = Not detected

[a] based on combustion analysis

Table 7 Summary of the nature of the residues in rotational radish following bare soil application of [^{14}C-**chlorophenoxy**]difenoconazole (0.52 kg ai/ha)

	30-DAT planting		60-DAT planting		120-DAT planting		270-DAT planting	
Identification	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Leaves	TRR→	0.027	TRR→	0.024	TRR→	0.084	TRR→	0.025
Extracted	81	0.022	79	0.019	80	0.067	65	0.017
Difenoconazole	28	0.008	58	0.014	72	0.060	51	0.013
CGA205375	22	0.006	23	0.005	7.9	0.007	15	0.004
RT = 3.67 min	6.9	0.002	--	ND	--	ND	--	ND
RT = 29.3 min	7.8	0.002	--	ND	--	ND	--	ND
RT = 32.1 min	2.7	0.001	--	ND	--	ND	--	ND
RT = 32.8 min	4.1	0.001	--	ND	--	ND	--	ND
PES	19	0.005	21	0.005	20	0.017	35	0.009
Total	100	0.027	100	0.024	100	0.084	100	0.025
Roots	TRR→	0.026	TRR→	0.021	TRR→	0.028	TRR→	0.018
Extracted	77	0.020	81	0.017	79	0.022	79	0.015
Difenoconazole	31	0.008	53	0.011	60	0.017	45	0.008
CGA205375	7.6	0.002	22	0.005	14	0.004	24	0.004
CGA205374	--	ND	1.4	0.0003	--	ND	2.8	0.001
CGA189138	--	ND	3.4	0.001	--	ND	--	ND
RT = 17.0 min	5.3	0.001	--	ND	--	ND	--	ND
RT = 17.9 min	11	0.003	--	ND	--	ND	--	ND
RT = 23.5 min	--	ND	--	ND	4.8	0.001	--	ND
RT = 25.5 min	2.8	0.001	--	ND	--	ND	--	ND
RT = 26.4 min	2.4	0.001	--	ND	--	ND	--	ND
RT = 27.9 min	3.2	0.001	--	ND	--	ND	--	ND
RT = 29.3 min	8.6	0.002	--	ND	--	ND	--	ND
RT = 29.9 min	--	ND	--	ND	--	ND	8.4	0.002
RT = 32.8 min	3.6	0.001	--	ND	--	ND	--	ND
PES	23	0.006	19	0.004	21	0.006	21	0.004
Total	100	0.026	100	0.021	100	0.028	100	0.018

ND = Not detected

Table 8 Summary of the nature of the residues in rotational sorghum forage and stover following bare soil application of [^{14}C-**chlorophenoxy**]difenoconazole (0.52 kg ai/ha). TRR in grain < 0.01 mg eq/kg

	30-DAT planting		60-DAT planting		120-DAT planting		270-DAT planting	
Identification	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Forage	TRR→	0.015	TRR→	Not analysed	TRR→	Not analysed	TRR→	Not analysed
Extracted	60	0.009	Not extracted or further analysed					
Difenoconazole	9.9	0.001						
CGA205375	2.6	0.0003						
RT = 3.73 min	11	0.002						

	30-DAT planting		60-DAT planting		120-DAT planting		270-DAT planting	
Identification	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
RT = 26.9 min	36	0.005						
PES	40	0.006						
Total	100	0.015						
Stover	TRR→	0.069	TRR→	Not analysed	TRR→	Not analysed	TRR→	Not analysed
Extracted	59	0.041	Not extracted or further analysed					
Difenoconazole	--	ND						
CGA205375	--	ND						
RT = 17.8 min	3.1	0.002						
RT = 18.5 min	19	0.013						
RT = 21.0 min	14	0.010						
RT = 26.9 min	22	0.015						
PES	41	0.028						
Total	100	0.069						

ND = Not detected

Table 9 Summary of the nature of the residues in rotational wheat straw following bare soil application of [^{14}C-**chlorophenoxy**]difenoconazole (0.52 kg ai/ha). TRR in grain < 0.01 mg eq/kg. Rotational wheat forage and hay were extracted but not further analysed to determine residues due to low extracted radioactivity

	60-DAT planting		120-DAT planting		270-DAT planting	
Identification	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Straw	TRR→	0.052	TRR→	0.049	TRR→	0.029
Extracted	63	0.033	57	0.028	59	0.017
Difenoconazole	7.1	0.004	12	0.006	6.6	0.002
CGA205375	40	0.021	38	0.019	39	0.011
RT = 3.4 min	--	ND	5.7	0.003	--	ND
RT = 20.3-20.5 min	17	0.009	1.9	0.001	13	0.004
PES	37	0.019	43	0.021	41	0.012
Total	100	0.052	100	0.049	100	0.029

ND = Not detected

Difenoconazole was a major residue in rotational lettuce and radish matrices, but not in animal feed commodities of rotational sorghum or wheat; residues were too low in sorghum and wheat grain to identify or characterize residues. The principal residues in sorghum forage and stover were unidentified compounds, with none exceeding 0.015 mg eq/kg. The principal residue in wheat straw was CGA205375 and occurred at levels (roughly 0.01–0.02 mg eq/kg) ca. 3- to 5-fold greater than difenoconazole. As the triazole moiety of difenoconazole was not radiolabeled, the fate of that portion of the molecule could not be monitored in this study.

RESIDUE ANALYSIS

Analytical methods

Method REM 147.08

This method (reflux with methanol: conc. ammonium hydroxide; C18 SPE clean-up; LC-MS/MS analysis) was used for the determination of difenoconazole residues in cotton, cranberry, and tea residues studies. The method was evaluated previously by the JMPR (2007, 2010, 2015, 2017) and found to be acceptable.

QuEChERS

Field trial samples of guava were analysed for residues of difenoconazole using a standard QuEChERS method.

Concurrent recovery data for guava, cranberry, cotton, and tea are summarized below.

Table 10 Summary of validation (V) and concurrent (C) recovery of difenoconazole from cotton, cranberry, and tea

Matrix [Method]	Fortification level [mg/kg]	Individual recoveries [%]	Range of recoveries [%]	Mean recovery [%]	RSD [%]	Reference
Guava (fruit) [QuEChERS]	0.01	C: 93, 109 V: 83, 84, 93, 94, 109, 119	83-119	100	14	PR 11605
	0.1	C: 73, 77, 96, 98, 100, 122, 123 V: 101, 105, 107	73-123	100	16	
	1.0	V: 99, 102, 103	99-103	101	2	
Cranberry (fruit) [REM 147.08]	0.01	82, 82, 84, 84, 84, 84, 89, 89, 91	82-91	85	4	PR 10828
	0.10	91, 91, 94, 96, 96, 98	91-98	94	3	
	1.00	76, 87, 92, 93, 96	76-96	89	9	
Cotton (undelinted seed) [REM 147.08]	0.01	V: 80, 99, 100	80-100	93	12	TK0211638
	0.01	C: 82, 85, 87, 90, 96, 96, 99, 100, 102, 106, 106	82-106	95	9	
	1.00	V: 103, 103, 116	103-116	107	7	
	5.0	C: 86, 86, 90, 96, 99, 100, 103, 108, 112, 116	86-116	100	10	
Cotton (gin by-products) [REM 147.08]	0.01	V: 88, 106, 107	88-107	100	11	
	0.01	C: 82, 97, 102	82-102	94	11	
	1.00	V: 97, 100, 101	97-101	99	2	
	5.0	C: 88, 92, 101	88-101	94	7	
	8.0	C: 94	94	94	--	
Cotton (meal) [REM 147.08]	0.01	C: 92	92	92	--	
	0.1	C: 94	94	94	--	

Matrix [Method]	Fortification level [mg/kg]	Individual recoveries [%]	Range of recoveries [%]	Mean recovery [%]	RSD [%]	Reference
Cotton (seed hulls) [REM 147.08]	0.01	C: 90, 91	90-91	90	0.8	
	5.0	C: 96	96	96	--	
Cotton (refined oil) [REM 147.08]	0.01	C: 73, 91	73-91	82	15	
	5.0	C: 91	91	91	--	
Tea (dried leaves) [REM 147.08]	0.01	C: 72, 84	84-72	78	--	T010388-05-REG
		C: 84, 84, 85, 89, 97	84-97	88	6	JP2016C029
	0.5	C: 70, 85, 89, 89, 96	70-96	86	11	
		C: 75, 77, 77, 78	75-78	77	2	10043
	0.8	C: 89, 96	89-96	92	--	
	2.0	C: 90	90	90	--	T010388-05-REG
	8	C: 88, 91	88-91	90	--	10043
	10.0	C: 88	88	88	--	T010388-05-REG
	20	C: 85, 86, 86, 91, 91	85-91	88	3	JP2016C029
Tea (fresh leaves) [REM 147.08]	0.01	C: 95	95	95	--	T010388-05-REG
	0.5	C: 90	90	90	--	

Method Meth-160, Revision #2

Residues of 1,2,4-triazole, triazolylalanine, triazolylacetic acid, and/or triazolyllactic acid were analysed using Morse Labs Method Meth-160, Revision #2. In this method, residues of the analytes are extracted with methanol:water (8:2, v/v). The extract is mixed with celite, filtered, and an aliquot taken for separate analysis of each analyte. For 1,2,4-triazole, residues are derivatized using dansyl chloride. The dansyl derivative is partitioned into ethyl acetate, and the sample is evaporated to dryness. For triazolylalanine, residues are derivatized with 3 mol/L HCl in n-butanol followed by heptafluorobutyric anhydride, and the sample is evaporated to dryness. For triazolylacetic acid and triazolyllactic acid, residues are purified using a C18 solid-phase extraction cartridge and then esterified using HCl/butanol; following derivatization, the sample is evaporated to dryness. For all analytes, residues are reconstituted in acetonitrile:water (3:7, v/v) prior to analysis by HPLC-MS/MS.

Table 11 Summary of validation (V) and concurrent (C) recovery of triazole metabolites from cotton and tea

Matrix	Analyte	Fortification level [mg/kg]	Individual recoveries [%]	Range of recoveries	Mean recovery [%]	RSD [%]	Reference
Cotton (undelinted seed)	1,2,4-triazole	0.01	V: 117, 113, 117	113-117	116	2.0	TK0211638
			C: 92, 105, 119, 96, 90, 96, 94, 86, 96, 98	86-119	97	9.4	
		0.1	C: 93, 104, 99, 96, 93, 99, 98, 95, 98, 100	93-104	98	3.5	

Matrix	Analyte	Fortification level [mg/kg]	Individual recoveries [%]	Range of recoveries	Mean recovery [%]	RSD [%]	Reference
		1.0	V: 91, 89, 95	89-95	92	3.3	
	Triazolylalanine	0.01	C: 83	83	83	--	
		0.1	V: 90, 85, 88	85-90	88	2.9	
			C: 92, 74, 81, 89, 90, 97, 92, 84, 89	74-97	88	7.9	
		0.8	C: 88, 79, 87, 85, 93, 82, 86, 103, 78, 83	78-103	86	8.5	
		1.0	V: 86, 87, 87	86-87	87	0.7	
			C: 113	113	113	--	
	Triazolylacetic acid	0.01	V: 117, 105, 100	100-117	107	8.1	
			C: 102, 106, 104, 112, 107, 109, 105, 100, 108, 107, 109	100-112	106	3.2	
		0.1	C: 104, 107, 104, 106, 106, 98, 103, 101, 101, 102, 102	98-107	103	2.6	
		1.0	V: 99, 102, 133	99-133	111	16.9	
Cotton (gin by-products)	1,2,4-triazole	0.01	V: 111, 119, 120	111-120	117	4.2	
			C: 81, 113, 88, 101	81-113	96	14.8	
		0.1	C: 92, 95	92-95	94	--	
		1.0	V: 95, 92, 90	90-95	92	2.7	
	Triazolylalanine	0.01	V: 105, 93, 110	93-110	103	8.5	
			C: 85, 75, 74, 80	74-85	79	6.5	
		0.1	C: 75, 90	75-90	83	--	
		1.0	V: 112, 118, 111	111-118	114	3.3	
	Triazolylacetic acid	0.01	V: 93, 101, 103	93-103	99	5.3	
			C: 115, 94, 91, 90	90-115	98	12.1	
		0.1	C: 101, 91	91-101	96	--	
		1.0	V: 97, 97, 96	96-97	97	0.6	
Cotton (meal)	1,2,4-triazole	0.01	C: 96	96	96	--	
		0.1	C: 98, 94	94-98	96	--	
	Triazolylalanine	0.1	C: 107, 91, 121	91-121	106	14.1	
		0.8	C: 88, 90	88-90	89	--	
	Triazolylacetic acid	0.01	C: 100	100	100	--	
		0.1	C: 107, 102	102-107	105	--	
Cotton (seed hulls)	1,2,4-triazole	0.01	C: 95	95	95	--	
		0.1	C: 95, 98	95-98	97	--	
	Triazolylalanine	0.8	C: 80, 110	80-110	95	--	
	Triazolylacetic acid	0.01	C: 92	92	92	--	
		0.1	C: 107, 101	101-107	104	--	

Matrix	Analyte	Fortification level [mg/kg]	Individual recoveries [%]	Range of recoveries	Mean recovery [%]	RSD [%]	Reference
Cotton (refined oil)	1,2,4-triazole	0.01	C: 91	91	91	--	
		0.1	C: 101, 98	98-101	100	--	
	Triazolylalanine	0.1	C: 89, 89	89-89	89	--	
		0.8	C: 90	90	90	--	
	Triazolylacetic acid	0.01	C: 102	102	102	--	
		0.1	C: 111, 106	106-111	109	--	
Tea (dried leaves)	1,2,4-triazole	0.01	C: 108, 103, 91, 83, 80	80-108	93	13.1	JP2016C029
		0.5	C: 101, 96, 95, 94, 92	92-101	96	3.5	
	Triazolylalanine	0.05	C: 74, 73, 72, 72, 70	70-74	72	2.1	
		2.5	C: 71, 71, 71, 70, 70	70-71	71	0.8	
	Triazolylacetic acid	0.05	C: 85, 84, 81, 79, 75	75-85	81	5.0	
		2.5	C: 88, 88, 85, 85, 84	84-88	86	2.2	
	Triazolyllactic acid	0.05	C: 100, 94, 86, 78, 76	76-100	87	11.8	
		2.5	C: 90, 88, 85, 85, 83	83-90	86	3.2	
Tea infusion	1,2,4-triazole	0.01	C: 117, 111, 110, 105, 100	100-117	109	5.9	
		0.5	C: 98, 98, 97, 97, 97	97-98	97	0.6	
	Triazolylalanine	0.05	C: 102, 99, 98, 98, 96	96-102	99	2.2	
		2.5	C: 99, 97, 94, 93, 92	92-99	95	3.1	
	Triazolylacetic acid	0.05	C: 112, 112, 111, 106, 102	102-112	109	4.1	
		2.5	C: 102, 101, 101, 101, 98	98-102	101	1.5	
	Triazolyllactic acid	0.05	C: 116, 115, 112, 109, 108	108-116	112	3.2	
		2.5	C: 102, 102, 101, 99, 95	95-102	100	3.0	

The analytical methods appear to be suitable for analysis of difenoconazole and have been validated to an LOQ of 0.01 mg/kg in all investigated commodities. Similarly, the methods used for analysis of 1,2,4-triazole are considered suitable and have been validated to an LOQ of 0.01 mg/kg. The LOQs for the other triazole metabolites vary depending on the matrix and range from 0.05 to 0.1 mg/kg.

Stability of residues in stored analytical samples

Orange and dried bean

The stability of difenoconazole was evaluated in dried beans (high protein) and whole oranges (high acid) during two years of frozen storage (G. Andrews and G. Fowle, 2016, Report TK0208888). Samples of homogenized oranges and dried broad beans were fortified with difenoconazole at 0.2 mg/kg and placed into frozen (-20 °C) storage. Samples were analysed for residues of difenoconazole after 0, 3, 6, 9, 13, 18, and 24 months of frozen storage using residue analytical method REM 147.08.

The results (Table 12) indicate that residues are stable in high protein and high acid commodities during frozen storage for at least 2 years.

Table 12 Summary of stability data for residues of difenoconazole in oranges and dried beans stored under frozen conditions

Matrix	Storage time, days	Fortification level, mg/kg	Procedural recovery, %	Residue remaining, mg/kg [mean]	Mean % remaining [a]
Orange	0	0.2	89	0.19, 0.19, 0.18 [0.18]	100
	95	0.2	79	0.15, 0.16 [0.16]	83
	190	0.2	96	0.21, 0.21 [0.21]	112
	273	0.2	95	0.20, 0.20 [0.20]	107
	382	0.2	94	0.18, 0.19 [0.18]	99
	553	0.2	92	0.18, 0.19 [0.18]	99
	732	0.2	94	0.18, 0.22 [0.20]	107
Dried beans	0	0.2	89	0.19, 0.19, 0.19 [0.19]	100
	95	0.2	92	0.16, 0.18 [0.17]	89
	190	0.2	105	0.22, 0.23 [0.22]	118
	273	0.2	97	0.20, 0.21 [0.20]	108
	382	0.2	98	0.20, 0.20 [0.20]	105
	553	0.2	100	0.19, 0.20 [0.20]	103
	732	0.2	100	0.20, 0.20 [0.20]	105

[a] Normalized to the Day 0 result

Guava

As part of the supervised residue trials (Report 11605), concurrent storage stability samples were established by fortifying homogenized control samples with 0.1 mg/kg difenoconazole. The fortified samples were placed into frozen (-20 °C) storage and analysed after durations of 187 and 398 days using a suitable (see Table 10) QuEChERS multiresidue method.

Although no Day-0 data were provided, the results (Table 13) indicate that residues are stable in guava during frozen storage for at least 398 days.

Table 13 Summary of stability data for residues of difenoconazole in guava stored under frozen conditions

Matrix	Storage time, days	Fortification level, mg/kg	Residue remaining, mg/kg [mean]	Mean % remaining
Guava	187	0.1	0.096, 0.104, 0.106 [0.102]	102
	398	0.1	0.0815, 0.094, 0.091 [0.088]	88

Tea

Storage stability data were reported for residues of difenoconazole, 1,2,4-triazole, triazolylalanine, triazolylacetic acid, and triazolyllactic acid in tea (Report JP2016C029). Samples of control dry tea leaves were fortified with analytes each at 0.5 mg/kg. Although no Day-0 data were provided, the results (Table 14) indicate that residues are stable in dry tea leaves during frozen storage for at least *ca*. 400 days.

Table 14 Summary of stability data for residues of difenoconazole and triazole metabolites in dry tea leaves stored under frozen conditions

Analyte	Fortification level, mg/kg	Concurrent recovery, %	Storage time, days	% remaining	Mean % remaining
Difenoconazole	0.5	78	372	82	82
			395	86	
			408	84	
			407	76	
1,2,4-Triazole	0.5	92	376	80	83
			399	82	
			411	86	
			412	84	
Triazolylalanine	0.5	77	372	70	72
			395	70	
			407	75	
			408	72	
Triazolylacetic acid	0.5	91	372	86	87
			395	87	
			407	86	
			408	88	
Triazolyllactic acid	0.5	82	372	82	82
			395	83	
			407	78	
			408	85	

USE PATTERN

Registered labels describing the use of difenoconazole were provided to the current Meeting for guava, cranberry, cotton, and tea (Table 15).

Table 15 Registered uses of difenoconazole provided to the current Meeting

Use site	Country	Formulation		Application						PHI, days
		Conc.	Type	Timing	Method	Rate	Water	Number	Interval, days	
Guava	EG	125 g ai/L	SC	PHI	Broadcast spray	50 mL/ 100 L (6.25 g ai/100 L)	400-600 L/fed (950-1430 L/ha)	3	7-14	7
	US	250 g ai/L	EC	Disease development	Broadcast spray	128 g/ha (516 g/ha/yr)	ground: n.s. aerial: ≥94 L/ha	n.s.	7-14	0
Cranberry	US	250 g ai/L	EC	Disease development	Broadcast spray	128 g/ha (381 g/ha/yr)	ground: n.s. aerial: ≥47 L/ha	n.s.	7-14	30

Use site	Country	Formulation		Application						PHI, days
		Conc.	Type	Timing	Method	Rate	Water	Number	Interval, days	
Cotton	US	250 g ai/L	EC	Disease development	Broadcast spray	128 g/ha (381 g/ha/yr)	ground: n.s. aerial: ≥47 L/ha	n.s.	14-21	45
Tea	JP	10%	WP	PHI	Broadcast spray	2000X Dilution	2000-4000 L/ha	2	n.s.	7

RESULTS OF SUPERVISED RESIDUE TRIALS ON CROPS

The Meeting received data from supervised residue trials conducted on guava, cranberry, cotton, and tea.

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 method described above for plant commodities.

The field trial study designs included control plots. Measured residues from control plots were <LOQ and are not included in the summary tables in this evaluation.

When calculating average residues, values below the LOQ were assumed to be at the LOQ, and average residues are denoted as being <LOQ only when all samples from a plot were <LOQ. In the summary tables, residue values leading to maximum residue estimations are underlined. The highest individual values selected for estimating acute dietary risks are bolded.

Supervised trials for difenoconazole:

Category	Crop	Table
Tropical fruit, edible peel	Guava	Table 16
Low growing berries	Cranberry	Table 17
Oilseeds	Cotton (undelinted seed)	Table 18
		Table 19
Teas		Table 20
Animal Feeds		Table 21
		Table 22

Guava

Six field trials were conducted in Egypt during the 2015 season (DeFrancesco, J. 2016, Report 11605). Treatment consisted of three foliar applications of ca. 62.5 g difenoconazole/ha, on a 6- to 8-day interval. Harvest occurred 7 days after the last application.

Following harvest, samples (≥ 12 marketable sized fruits) were bagged and put into coolers with 'blue ice' for transport to the analytical facility. Upon arriving at the facility, samples were put into frozen storage (within 3 hours of harvest). Prior to analysis, samples were homogenized in the presence of dry ice and returned to frozen storage. Samples were stored for a maximum of 403 days prior to analysis.

Concurrent storage stability samples indicate that residues of difenoconazole are stable for at least the maximum storage duration of the samples.

Samples were analysed for residues of difenoconazole using a QuEChERS multiresidue method (AOAC Official Method 2007.01). Concurrent recovery data indicate that the method is suitable. Residues of the common triazole metabolites (1,2,4-triazole, triazolylalanine, and triazolylacetic acid) were not reported.

Minor deviations from good laboratory practice (GLP) were noted. These are not expected to have a significant impact on the results reported in the trials.

Table 16 Results of difenoconazole residue trials in **guava** in Egypt

Location; year (Trial ID)	Crop Variety	Application				DALA	Residues (mg/kg) [Mean]	Study report
		No. [interval, days]	Dilution, mL prod/ L spray	Rate, g ai/ha	L/ha		Difenoconazole	
Critical GAP (US)	--	3 [7]	--	128	--	0		
Alternative GAP (EG)	--	3 [7]	50 (6.25 g ai/100 L)	--	950-1430	7	--	Spray to runoff (equivalent to 59- 89 g ai/ha)
Moshtohor; 2015 (EG01)	Etmany	1 2 [7] 3 [7]	50 50 50	63.3 64.9 62.6	1010 1036 999	7	0.046, 0.059 [0.053]	11605
Qalama; 2015 (EG02)	Ghoneimy	1 2 [7] 3 [7]	50 50 50	62.8 62.4 64.7	1002 997 1032	0	0.081, 0.13 [0.11]	
						3	0.040, 0.039 [0.040]	
						7	0.018, 0.028 [0.023]	
						10	0.027, 0.038 [0.033]	
						14	0.021, 0.026 [0.024]	
Salhia; 2015 (EG03)	Etmany	1 2 [6] 3 [7]	50 50 50	61.8 64.0 63.0	987 1023 1006	7	**0.095**, 0.059 [0.077]	
Al-Khanka; 2015 (EG04)	Balady	1 2 [7] 3 [8]	50 50 50	51.6 66.1 66.5	986 1058 1060	6	0.010, 0.016 [0.013]	
Nawa; 2015 (EG05)	Etmany	1 2 [7] 3 [7]	50 50 50	62.0 64.0 65.7	986 1022 1048	8	0.018, 0.017 [0.018]	
Manzala; 2015 (EG06)	Banaty	1 2 [7] 3 [7]	50 50 50	64.5 63.3 61.4	1033 1014 983	8	0.014, 0.054 [0.034]	

Cranberry

Five field trials were conducted in the USA during the 2013 season (Homa, K. 2015, Report PR 10828). Treatment consisted of three foliar applications by backpack sprayer of ca. 128 g difenoconazole/ha, on a 6 to 7 day interval. A non-ionic surfactant or crop oil concentrate was used at all trial locations. Harvest occurred 29–30 days after the last application and was done by hand or by cranberry scoop.

Following harvest, samples (≥ 0.92 kg) were bagged and put into frozen storage within 2.25 hours prior to transport to the analytical facility. Samples were shipped frozen to the facility and were immediately placed in frozen storage. Prior to analysis, samples were homogenized in the presence of dry ice and returned to frozen storage. Samples were stored for a maximum of 161 days prior to analysis. Storage stability data for residues of difenoconazole or the common triazole metabolites were not provided in this study.

Samples were analysed for residues of difenoconazole and the triazole metabolites using Method REM 147.08 and Meth-160, Revision 2, respectively, noted above. Concurrent recovery data indicate that both methods are suitable.

Minor deviations from GLP were noted. These are not expected to have a significant impact on the results reported in the trials.

Table 17 Results of difenoconazole residue trials in **cranberry** in the USA

Location; year (Trial ID)	Crop Variety	Application			DALA	Residues (mg/kg) [Mean]	Study report
		No. [interval, days]	Rate, g ai/ha	L/ha		Difenoconazole	Remarks
Critical GAP (US)	--	n.s. [7]	128 (381/year max)	ground: n.s. aerial: ≥47 L/ha	30	--	
East Wareham, MA, US; 2013 (MA02)	Stevens	1 2 [7] 3 [7]	130 130 130	623 623 623	30	0.21, 0.19 [0.20]	PR 10828
Chatsworth, NJ, US; 2013 (NJ14)	Stevens	1 2 [7] 3 [7]	131 132 132	263 262 268	29	0.20, 0.19 [0.20]	
Warrenton, OR, US; 2013 (OR21)	Pilgrim	1 2 [6] 3 [6]	132 129 128	574 563 558	29	0.13, 0.14 [0.14]	
Warrens, WI, US; 2013 (WI19) [a]	Stevens	1 2 [7] 3 [7]	130 129 133	250 247 261	20	0.19, 0.28 [0.24]	
					23	0.23, 0.27 [0.25]	
					29	0.17, 0.17 [0.17]	
					37	0.16, 0.11 [0.14]	
					41	0.12, 0.12 [0.12]	
Warrens, WI, US; 2013 (WI20) [a]	Stevens	1 2 [7] 3 [7]	132 132 130	442 441 443	29	**0.26**, 0.22 [0.24]	

[a] Trials WI19 and WI20 were conducted at the same location with the same variety. Applications were initiated on the same date. Different spray volumes and adjuvants (WI19: non-ionic surfactant, WI20: crop oil concentrate) were used for each trial.

Residues of the common triazole metabolites were <0.01 mg/kg in all samples.

Cotton

Twelve field trials were conducted in the USA during the 2014 season (McDonald, T.J. 2016, Report TK0211638). Treatment consisted of three foliar applications of ca. 128 g difenoconazole/ha, on a 10-day interval. A non-ionic surfactant or crop oil concentrate was used at all trial locations. Samples of undelinted seed were taken 45 days after the last application.

Following harvest, samples (sample size not specified) were ginned to separate seed from gin by-products. Samples were bagged and put into frozen storage prior to transport to the analytical facility. Samples were shipped frozen to the facility and were immediately placed in frozen storage. Prior to analysis, samples were cryogenically homogenized and returned to frozen storage. Samples were stored for a maximum of 12.6 months prior to difenoconazole analysis or 13.1 months prior to triazole metabolite analysis. Storage stability data for residues of difenoconazole or the common triazole metabolites were not provided.

Samples were analysed for residues of difenoconazole using Method REM 147.08, noted above, and for the triazole metabolites using Method Meth-160, Revision #2. Concurrent recovery data indicate that both methods are suitable.

Table 18 Residues of difenoconazole in **undelinted cotton seed** from residue trials in cotton in the USA

Location; year (Trial ID)	Crop Variety	Application				DALA	Residues (mg/kg) [Mean]	Study report
		No. [interval, days]	Rate, g ai/ha	L/ha	Formulation/ Adjuvant [a]		Difenoconazole	Remarks
Critical GAP (US)	--	n.s. [14]	128	>47 [b]	--	45	--	
Weston, GA, US; 2014 (TK0211638-01)	449	1 2 [9] 3 [9]	128 128 128	206 178 178	SC / NIS	35	0.083	TK0211638
						40	0.057	
						45	0.314, 0.289 [0.302]	
						50	0.156	
						55	0.133	
		1 2 [9] 3 [9]	128 128 131	206 178 178	EC / COC	35	0.133	
						40	0.119	
						45	0.214, 0.159 [0.186]	
						50	0.100	
						55	0.114	
Cheneyville, LA, US; 2014 (TK0211638-02)	Phytogen 33 WRF	1 2 [9]	127 124	47 37	SC / NIS	44	<0.01, <0.01 [<0.01]	

Location; year (Trial ID)	Crop Variety	Application				DALA	Residues (mg/kg) [Mean]	Study report
		No. [interval, days]	Rate, g ai/ha	L/ha	Formulation/ Adjuvant [a]		Difenoconazole	Remarks
		3 [10]	137	47				
		1 2 [9] 3 [10]	119 132 136	47 37 47	EC / COC	44	<0.01, <0.01 [<0.01]	
Fisk, MO, US; 2014 (TK0211638-03)	PHY 333	1 2 [9] 3 [10]	130 130 130	187 187 187	SC / NIS	44	<0.01, <0.01 [<0.01]	
		1 2 [9] 3 [10]	128 128 129	187 187 187	EC / COC	44	0.026, 0.011 [0.018]	
Greenville, MS, US; 2014 (TK0211638-04)	Stoneville 4946 GLB2	1 2 [15] 3 [9]	130 132 130	196 206 196	SC / NIS	35	<0.01	
						40	<0.01	
						45	<0.01, <0.01 [<0.01]	
						50	<0.01	
						56	<0.01	
		1 2 [15] 3 [9]	125 132 125	196 206 196	EC / COC	35	<0.01	
						40	<0.01	
						45	<0.01, <0.01 [<0.01]	
						50	<0.01	
						56	<0.01	
Uvalde, TX, US; 2014 (TK0211638-05)	Phytogen 499 WRF	1 2 [10] 3 [11]	130 129 128	159 168 159	SC / NIS	44	0.036, 0.058 [0.047]	
		1 2 [10] 3 [11]	125 131 129	150 178 168	EC / COC	44	0.046, 0.039 [0.042]	
Hinton, OK, US; 2014 (TK0211638-06)	NG1511 B2RF/ A1020045	1 2 [9] 3 [12]	132 124 130	243 215 178	SC / NIS	45	0.011, <0.01 [0.010]	
		1 2 [9] 3 [12]	131 128 129	243 215 178	EC / COC	45	0.011, 0.014 [0.012]	
Groom, TX, US; 2014 (TK0211638-07)	FM2011 B2F	1 2 [12] 3 [10]	129 129 129	206 206 215	SC / NIS	47	0.036, 0.035 [0.036]	

Location; year (Trial ID)	Crop Variety	Application				DALA	Residues (mg/kg) [Mean]	Study report
		No. [interval, days]	Rate, g ai/ha	L/ha	Formulation/ Adjuvant [a]		Difenoconazole	Remarks
		1 2 [12] 3 [10]	130 128 129	206 206 215	EC / COC	47	0.033, 0.036 [0.034]	
San Angelo, TX, US; 2014 (TK0211638-08)	FM2334 GLT	1 2 [8] 3 [11]	127 130 127	178 178 168	SC / NIS	48	<0.01, <0.01 [<0.01]	
		1 2 [8] 3 [11]	127 129 129	178 178 168	EC / COC	48	<0.01, <0.01 [<0.01]	
Levelland, TX, US; 2014 (TK0211638-09)	FM9063 B2F	1 2 [10] 3 [11]	129 131 127	187 196 187	SC / NIS	45	0.011, 0.010 [0.010]	
		1 2 [10] 3 [11]	133 133 130	187 187 187	EC / COC	45	0.028, 0.021 [0.024]	
Porterville, CA, US; 2014 (TK0211638-10)	Phytogen 339	1 2 [10] 3 [10]	128 130 129	37 37 37	SC / NIS	45	0.104, 0.178 [0.141]	
		1 2 [10] 3 [10]	125 132 130	37 47 37	EC / COC	45	0.088, 0.085 [0.086]	
Kerman, CA, US; 2014 (TK0211638-11)	Phy 802 RF Pima	1 2 [10] 3 [11]	129 128 129	187 187 187	SC / NIS	45	<0.01, <0.01 [<0.01]	
		1 2 [10] 3 [11]	129 129 128	187 187 187	EC / COC	45	<0.01, <0.01 [<0.01]	
Paso Robles, CA, US; 2014 (TK0211638-12)	DP358RF (Pima)	1 2 [9] 3 [11]	127 133 129	234 243 234	SC / NIS	45	0.051, 0.031 [0.041]	
		1 2 [9] 3 [11]	128 136 130	234 243 234	EC / COC	45	0.030, 0.024 [0.027]	

[a] NIS = Non-ionic surfactant, COC = crop oil concentrate

[b] For aerial applications; not specified for ground applications

Residues of **1,2,4-triazole** were <0.01 mg/kg in all cotton seed samples.

Table 19 Residues of triazolylalanine and triazolylacetic acid in **undelinted cotton seed** from residue trials in cotton in the USA

Location; year (Trial ID)	Crop Variety	Application				DALA	Residues (mg/kg) [Mean]		Study report Remarks
		No. [interval, days]	Rate, g ai/ha	L/ha	Formulation/ Adjuvant [a]		Triazolyl-alanine	Triazolyl-acetic acid	
Critical GAP (US)	--	n.s. [14]	128	>47 [b]	--	45	--	--	
Weston, GA, US; 2014 (TK0211638-01)	449	1 2 [9] 3 [9]	128 128 128	206 178 178	SC / NIS	35	0.424	<0.01	TK0211638
						40	0.329	<0.01	
						45	0.504, 0.386 [0.445]	<0.01, <0.01 [<0.01]	
						50	0.357	<0.01	
						55	0.432	<0.01	
		1 2 [9] 3 [9]	128 128 131	206 178 178	EC / COC	35	0.553	0.018	
						40	0.473	0.012	
						45	0.590, 0.607 [0.599]	0.014, 0.014 [0.014]	
						50	0.553	0.013	
						55	0.530	<0.01	
Cheneyville, LA, US; 2014 (TK0211638-02)	Phytogen 33 WRF	1 2 [9] 3 [10]	127 124 137	47 37 47	SC / NIS	44	0.189, 0.169 [0.179]	<0.01, <0.01 [<0.01]	
		1 2 [9] 3 [10]	119 132 136	47 37 47	EC / COC	44	0.206, 0.200 [0.203]	<0.01, <0.01 [<0.01]	
Fisk, MO, US; 2014 (TK0211638-03)	PHY 333	1 2 [9] 3 [10]	130 130 130	187 187 187	SC / NIS	44	0.613, 0.466 [0.540]	0.019, 0.013 [0.016]	
		1 2 [9] 3 [10]	128 128 129	187 187 187	EC / COC	44	0.325, 0.366 [0.346]	<0.01, <0.01 [<0.01]	
Greenville, MS, US; 2014 (TK0211638-04)	Stoneville 4946 GLB2	1 2 [15] 3 [9]	130 132 130	196 206 196	SC / NIS	35	0.259	<0.01	
						40	0.263	<0.01	
						45	0.293, 0.229 [0.261]	<0.01, <0.01 [<0.01]	
						50	0.175	<0.01	
						56	0.263	<0.01	
		1	125	196	EC / COC	35	0.241	<0.01	

Location; year (Trial ID)	Crop Variety	Application				DALA	Residues (mg/kg) [Mean]		Study report Remarks
		No. [interval, days]	Rate, g ai/ha	L/ha	Formulation/ Adjuvant [a]		Triazolyl-alanine	Triazolyl-acetic acid	
		2 [15] 3 [9]	132 125	206 196					
						40	0.227	<0.01	
						45	0.239, 0.231 [0.235]	<0.01, <0.01 [<0.01]	
						50	0.291	<0.01	
						56	0.272	<0.01	
Uvalde, TX, US; 2014 (TK0211638-05)	Phytogen 499 WRF	1 2 [10] 3 [11]	130 129 128	159 168 159	SC / NIS	44	0.103, 0.111 [0.107]	<0.01, 0.01 [<0.01]	
		1 2 [10] 3 [11]	125 131 129	150 178 168	EC / COC	44	<0.1, 0.109 [0.109]	<0.01, <0.01 [<0.01]	
Hinton, OK, US; 2014 (TK0211638-06)	NG1511 B2RF/ A1020045	1 2 [9] 3 [12]	132 124 130	243 215 178	SC / NIS	45	0.153, 0.172 [0.163]	<0.01, <0.01 [<0.01]	
		1 2 [9] 3 [12]	131 128 129	243 215 178	EC / COC	45	0.218, 0.216 [0.217]	<0.01, <0.01 [<0.01]	
Groom, TX, US; 2014 (TK0211638-07)	FM2011 B2F	1 2 [12] 3 [10]	129 129 129	206 206 215	SC / NIS	47	<0.1, <0.1 [<0.1]	<0.01, <0.01 [<0.01]	
		1 2 [12] 3 [10]	130 128 129	206 206 215	EC / COC	47	<0.1, <0.1 [<0.1]	<0.01, <0.01 <0.01]	
San Angelo, TX, US; 2014 (TK0211638-08)	FM2334 GLT	1 2 [8] 3 [11]	127 130 127	178 178 168	SC / NIS	48	0.211, 0.179 [0.195]	<0.01, <0.01 [<0.01]	
		1 2 [8] 3 [11]	127 129 129	178 178 168	EC / COC	48	0.149, 0.154 [0.152]	<0.01, <0.01 [<0.01]	
Levelland, TX, US; 2014 (TK0211638-09)	FM9063 B2F	1 2 [10] 3 [11]	129 131 127	187 196 187	SC / NIS	45	0.164, 0.152 [0.158]	<0.01, <0.01 [<0.01]	
		1 2 [10] 3 [11]	133 133 130	187 187 187	EC / COC	45	0.114, 0.117 [0.116]	<0.01, <0.01 [<0.01]	
Porterville, CA, US; 2014 (TK0211638-10)	Phytogen 339	1 2 [10] 3 [10]	128 130 129	37 37 37	SC / NIS	45	0.239, 0.208 [0.224]	<0.01, <0.01 [<0.01]	

Location; year (Trial ID)	Crop Variety	Application				DALA	Residues (mg/kg) [Mean]		Study report Remarks
		No. [interval, days]	Rate, g ai/ha	L/ha	Formulation/ Adjuvant [a]		Triazolyl-alanine	Triazolyl-acetic acid	
		1 2 [10] 3 [10]	125 132 130	37 47 37	EC / COC	45	0.150, 0.238 [0.194]	<0.01, <0.01 [<0.01]	
Kerman, CA, US; 2014 (TK0211638-11)	Phy 802 RF Pima	1 2 [10] 3 [11]	129 128 129	187 187 187	SC / NIS	45	0.286, 0.323 [0.305]	<0.01, <0.01 [<0.01]	
		1 2 [10] 3 [11]	129 129 128	187 187 187	EC / COC	45	0.365, 0.427 [0.396]	<0.01, <0.01 [<0.01]	
Paso Robles, CA, US; 2014 (TK0211638-12)	DP358RF (Pima)	1 2 [9] 3 [11]	127 133 129	234 243 234	SC / NIS	45	0.143, 0.146 [0.145]	<0.01, <0.01 [<0.01]	
		1 2 [9] 3 [11]	128 136 130	234 243 234	EC / COC	45	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	

[a] NIS = Non-ionic surfactant, COC = crop oil concentrate

[b] For aerial applications; not specified for ground applications

Tea

Two study volumes (Jones, S. 2008, Report T010388-05-REG; Takahashi, Y. 2018, Report JP2016C029) of supervised residue trials in tea were submitted to the 2020 JMPR, as well as two analysis reports of difenoconazole residues in tea (30 Sept. 1993, File A7491J_10043 and 27 Aug. 1993, File A7491J_10059). The sample storage durations and conditions for all studies are covered by the available storage stability data.

Report T010388-05-REG

Four trials were conducted during 2006 in Taiwan Province of China. At each site, two applications of difenoconazole were made on a 7-day interval, each at 200 g ai/ha. Harvest occurred 0, 3, 8, 10, and/or 14 DALA, depending on the site. Samples consisted of approximately 1 kg of tea leaves. Following harvest, leaves were either placed into frozen storage (fresh leaves) or dried and then placed into frozen storage. Samples were stored for a maximum of 398 days prior to extraction. Residue analysis was done using Method REM 147.08.

Report JP2016C029

Four trials were conducted in Japan during the 2016 season. At each site, 2 applications of a 2000X dilution of a water-dispersible granule formulation containing 10% difenoconazole were made using approximately 4000 L spray/ha (equivalent to ca. 200 g/ha) on a 7-day interval. Tea leaves were harvested 7, 14, and 21 DALA. Samples consisted of at least 200 g of leaves. Leaves were dried, pulverised, and put into frozen storage prior to analysis; storage durations were up to 390 days. Residues of difenoconazole were analysed using Method REM 147.08.

A7491J_10043 and A7491J_10059

Two trials were conducted in Japan during the 1993 season under either open-field conditions (A7491J_10043) or shaded conditions (A7491J_10059). For the shaded conditions, the conditions were reported as 'plain-shaded' at the Mie location and 'shaded with super (#610)' at the Miyazaki location. For both locations, the shade was put in place 10 days before harvest. In all cases, a 1:2000 dilution of a wettable powder formulation (10% ai) was applied at a spray rate of 2000 L/ha (equivalent to 100 g ai/ha). At each site, either a single application or two applications, on a 7-day interval, were made to tea. Tea leaves were harvested 7, 14, and 21 DALA. Sample weights were reported as ranging from ca. 230 g to 500 g for the open-field study; weights were not reported for the shade study. Samples were stored under frozen conditions for up to 135 days (open-field study) or 93 days (shade study).

Table 20 Residues of difenoconazole in dried **tea leaves** from residue trials in tea in Japan and Taiwan Province of China

Location; year (Trial ID)	Crop Variety	Application				DALA	Residues (mg/kg)*	Study report
		No. [interval, days]	Rate, g ai/ha	L/ha	Commodity		Difeno-conazole	Remarks
Critical GAP (JP)	--	2 [n.s.]	2000× dilution	4000		7	--	10% ai formulation: equivalent to 5 g ai/hL or 200 g ai/ha
Mie Agric. Tech. Center, Tsubaisocho, Kameyama City, JP; 1993	Okumidori	1	2000×	2000	Dried leaves	7 (open)	3.88	A7491J_10043 (open) A7491J_10059 (shade)
						(shade)	3.20	
						14 (open)	4.69	
						(shade)	4.28	
						21 (open)	0.45	
						(shade)	0.44	
		1 [--] 2 [7]	2000× 2000×	2000 2000		7 (open)	7.87	
						(shade)	7.48	
						14 (open)	2.74]	
						(shade)	2.74	
						21 (open)	0.48	
						(shade)	0.43	
Miyazaki Agric. Exp. Station, Kawamani-micho, Koyugun, JP; 1993	Yabukita	1	2000×	2000	Dried leaves	7 (open)	6.80	
						(shade)	6.44	
						14 (open)	1.31	
						(shade)	1.22	
						21 (open)	0.12	
						(shade)	0.13	
		1 [--]	2000×	2000		7 (open)	5.22	

Location; year (Trial ID)	Crop Variety	Application				DALA	Residues (mg/kg)*	Study report
		No. [interval, days]	Rate, g ai/ha	L/ha	Commodity		Difeno-conazole	Remarks
		2 [7]	2000×	2000				
						(shade)	5.31	
						14 (open)	2.82	
						(shade)	3.31	
						21 (open)	0.14	
						(shade)	0.08	
Liouguei, TW; 2006 (TW-FR-06-0009) [a]	TC27	1 [--] 2 [7]	200 200	2000 2000	Dried leaves	0	55.5	T010388-05-REG Application in March/April
						3	6.87	
						8	1.92	
						10	1.50	
						14	0.77	
Liouguei, TW; 2006 (TW-FR-06-0010) [a]	TC27	1 [--] 2 [7]	200 200	2000 2000	Dried leaves	8	1.86	Application in March/April
						14	0.89	
					Fresh leaves	8	0.72	
						14	0.32	
Liouguei, TW; 2006 (TW-FR-06-0011) [b]	TC12	1 [--] 2 [7]	200 200	2000 2000	Dried leaves	0	28.6	Application in Nov.
						3	4.60	
						8	1.60	
						10	1.57	
						14	1.04	
Liouguei, TW; 2006 (TW-FR-06-0012) [b]	TC12	1 [--] 2 [7]	200 200	2000 2000	Dried leaves	8	1.57	Application in Nov.
						14	1.12	
					Fresh leaves	8	0.93	
						14	0.49	
Ibaraki, JP; 2016	Yabukita	1 [--] 2 [7]	2000× 2000×	3940 3940	Dried leaves	7	8.08	JP2016C029
						14	1.22	
						21	0.09	
Saitama, JP; 2016	Hokumei	1 [--] 2 [7]	2000× 2000×	4000 4000	Dried leaves	7	11.6	
						14	2.79	
						21	0.68	
Kagoshima, JP; 2016	Yamato-midori	1 [--] 2 [7]	2000× 2000×	4020 4020	Dried leaves	7	2.08	
						14	0.81	
						21	0.16	

Location; year (Trial ID)	Crop Variety	Application				DALA	Residues (mg/kg)*	Study report
		No. [interval, days]	Rate, g ai/ha	L/ha	Commodity		Difeno-conazole	Remarks
Miyazaki, JP; 2016	Yabikita	1 [--] 2 [7]	2000× 2000×	3970 3970	Dried leaves	7	4.38	
						14	0.83	
						21	0.01	

* Individual values are of duplicate analyses of the same sample.

[a] Same trial location and critical dates; these trials are not independent.

[b] Same trial location and critical dates; these trials are not independent.

Residues of **1,2,4-triazole** were <0.01 mg/kg in all tea samples.

Table 21 Residues of triazolylalanine, triazolylacetic acid, and triazolyllactic acid in **tea leaves** from residue trials in Japan

Location; year (Trial ID)	Crop Variety	Application				DALA	Residues (mg/kg)*			Study report
		No. [interval, days]	Rate, g ai/ha	L/ha	Com-modity		Triazolyl-alanine	Triazolyl-acetic acid	Triazolyl-lactic acid	Remarks
Critical GAP (JP)	--	2 [n.s.]	2000X dilution	2000-4000		7	--	--	--	10% ai formulation: equivalent to 5 g ai/hL or 200 g ai/ha
Ibaraki, JP; 2016	Yabukita	2 [7]	2000X	3940	Dried leaves	7	0.16 (0.26 c)	0.06 (0.06 c)	0.46] (0.42 c)	JP2016C029
						14	0.16	0.06	0.51	
						21	0.29	0.10	0.52	
Saitama, JP; 2016	Hokumei	2 [7]	2000X	4000	Dried leaves	7	0.46 (0.20 c)	0.12 (0.07 c)	0.59 (0.33 c)	
						14	0.32	0.09	0.44	
						21	0.35	0.12	0.65	
Kagoshima, JP; 2016	Yamatomidori	2	2000X	4020	Dried leaves	7	0.54 (0.66 c)	0.12 (0.15 c)	0.22 (0.22 c)	
						14	0.49	0.12	0.22	
						21	0.58	0.14	0.28	
Miyazaki, JP; 2016	Yabikita	2	2000X	3970	Dried leaves	7	<0.05	<0.05	0.38 (0.21 c)	
						14	<0.05	<0.05	0.32	
						21	0.06	<0.05	0.36	

* Individual values are of duplicate analyses of the same sample.

Note: values denoted with 'c' are residues in control samples.

Animal feeds

Cotton

In the cotton field trials described above, residues in cotton gin by-products were reported for trials designated as TL0211638-06, -07, and -08. Results from those trials are summarized below.

Table 22 Residues of difenoconazole in **cotton gin by-products** from residue trials in cotton in the USA

Location; year (Trial ID)	Crop Variety	Application				DALA	Residues (mg/kg) [Mean]	Study report
		No. [interval, days]	Rate, g ai/ha	L/ha	Formulation/ Adjuvant [a]		Difenoconazole	Remarks
Critical GAP (US)	--	n.s. [14]	128	>47 [b]	--	45	--	
Hinton, OK, US; 2014 (TK0211638-06)	NG1511 B2RF/ A1020045	1 2 [9] 3 [12]	132 124 130	243 215 178	SC / NIS	45	0.992, 0.944 [0.968]	TK0211638
		1 2 [9] 3 [12]	131 128 129	243 215 178	EC / COC	45	1.02, 1.43 [1.22]	
Groom, TX, US; 2014 (TK0211638-07)	FM2011 B2F	1 2 [12] 3 [10]	129 129 129	206 206 215	SC / NIS	47	5.35, 6.49 [2.95]	
		1 2 [12] 3 [10]	130 128 129	206 206 215	EC / COC	47	6.61, 4.54 [5.58]	
San Angelo, TX, US; 2014 (TK0211638-08)	FM2334 GLT	1 2 [8] 3 [11]	127 130 127	178 178 168	SC / NIS	48	1.08, 1.06 [1.07]	
		1 2 [8] 3 [11]	127 129 129	178 178 168	EC / COC	48	1.69, 1.31 [1.50]	

[a] NIS = Non-ionic surfactant, COC = crop oil concentrate

[b] For aerial applications; not specified for ground applications

Table 23 Residues of triazolylalanine and triazolylacetic acid in **cotton gin by-products** from residue trials in cotton in the USA

Location; year (Trial ID)	Crop Variety	Application				DALA	Residues (mg/kg) [Mean]		Study report
		No. [interval, days]	Rate, g ai/ha	L/ha	Formulation/ Adjuvant [a]		Triazolyl-alanine	Triazolyl-acetic acid	Remarks
Critical GAP (US)	--	n.s. [14]	128	>47 [b]	--	45	--	--	
Hinton, OK, US; 2014 (TK0211638-06)	NG1511 B2RF/ A1020045	1 2 [9] 3 [12]	132 124 130	243 215 178	SC / NIS	45	0.016, 0.017 [0.016]	0.017, 0.016 [0.016]	TK0211638

Location; year (Trial ID)	Crop Variety	Application				DALA	Residues (mg/kg) [Mean]		Study report Remarks
		No. [interval, days]	Rate, g ai/ha	L/ha	Formulation/ Adjuvant [a]		Triazolyl-alanine	Triazolyl-acetic acid	
		1 2 [9] 3 [12]	131 128 129	243 215 178	EC / COC	45	0.014, 0.024 [0.019]	0.022, 0.025 [0.024]	
Groom, TX, US; 2014 (TK0211638-07)	FM2011 B2F	1 2 [12] 3 [10]	129 129 129	206 206 215	SC / NIS	47	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	
		1 2 [12] 3 [10]	130 128 129	206 206 215	EC / COC	47	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	
San Angelo, TX, US; 2014 (TK0211638-08)	FM2334 GLT	1 2 [8] 3 [11]	127 130 127	178 178 168	SC / NIS	48	<0.01, <0.01 [<0.01]	0.019, 0.021 [0.020]	
		1 2 [8] 3 [11]	127 129 129	178 178 168	EC / COC	48	<0.01, <0.01 [<0.01]	0.018, 0.019 [0.018]	

[a] NIS = Non-ionic surfactant, COC = crop oil concentrate

[b] For aerial applications; not specified for ground applications

FATE OF RESIUDES DURING PROCESSING

Cotton

In a study investigating difenoconazole residues in processed cotton commodities (T. J. McDonald, 2016, Report TK0211638), cottonseed samples from the TK0211368-03 and TK0211368-05 field trials described above were processed into meal, hulls, and refined oil (e.g., Figure 1) using simulated commercial practices. Upon receipt at the processing facility, the undelinted cottonseed samples were placed into frozen (ca. -25 °C) storage, where they remained for up to 12.5 months prior to processing. Residues of difenoconazole were analysed using Method REM 147.08, and residues of the triazole metabolites were analysed using Method Meth-160, Revision #2.

Gin by-products. Samples were cleaned using a stick extractor to remove gin-by-products. The seed was then ginned to remove most of the lint. The lint and gin-by-products were weighed and discarded. The seed was mechanically delinted to ca. 3% lint remaining. The resulting linters and motes were weighed and discarded. The delinted seed was cracked, and the kernels were separated from the hulls by screening. Hull material was placed into frozen storage; kernels were adjusted by addition of water to a moisture content of 13.5%. Kernels were heated to 80–90°C for ca. 30 minutes and then flaked. The flakes were fed into a processor where they were heated with steam to ca. 95–125 °C and extruded as collets. The collets were ground and dried (65–82 °C, 30–40 minutes). The dried collets were extracted three times with hexane (49–60 °C). The resulting solids were processed into toasted meal and the miscella was processed into oil.

Meal. Solvent was evaporated from the solids. The desolventized material was sieved, with material passing the sieve collected and adjusted to a moisture content of 12–15%. The resulting meal was toasted (ca 105–115 °C, 45 minutes). The toasted meal was placed into frozen storage prior to residue analysis.

Oil. A vacuum evaporator (ca. 95 °C) was used to separate the miscella into crude oil and hexane. The crude oil was processed with NaOH to separate refined oil from soapstock. The refined oil was treated with bleaching earth and the resulting bleached oil was deodorized under vacuum (220–230 °C) and by addition of citric acid. The refined-bleached-deodorized (RBD) oil was placed into frozen storage prior to residue analysis.

Table 24 Residues of difenoconazole in **cotton processed commodities** from residue trials in cotton in the USA

Trial ID	Commodity	Residues (mg/kg) [Mean]	Processing factor [a]	Study report
		Difenoconazole		*Remarks*
TK0211638-03	Seed (pre-processing)	0.0919, 0.108, 0.141 [0.114]	--	TK0211638
	Meal	0.0101	0.089	
	Hulls	0.0491	0.43	
	RBD oil	ND	<0.088	
TK0211638-05	Seed (pre-processing)	0.140, 0.163, 0.149 [0.151]	--	
	Meal	<0.01	<0.066	
	Hulls	<0.01	<0.066	
	RBD oil	ND	<0.066	

[a] Values reported as ND were assumed to be <0.01 mg/kg for purposes of calculating processing factors.

Table 25 Residues of 1,2,4-triazole, triazolylalanine, and triazolylacetic acid in **cotton processed commodities** from residue trials in cotton in the USA

Trial ID	Commodity	Residues (mg/kg) [Mean]			Processing factors [a]			Study report
		1,2,4-triazole	Triazolyl-alanine	Triazolyl-acetic acid	1,2,4-triazole	Triazolyl-alanine	Triazolyl-acetic acid	Remarks
TK0211638-03	Seed (pre-processing)	<0.01, <0.01, <0.01 [<0.01]	0.840, 0.757, 0.910 [0.836] (0.441 c)	0.0215, 0.0191, 0.0234 [0.0213] (0.0115 c)	--	--	--	TK0211638
	Meal	0.0124	0.921 (0.500 c)	0.0336 (0.0151 c)	>1.24	1.07	1.89	
	Hulls	<0.01	0.503 (0.261 c)	0.0143	nc	0.61	1.46	
	RBD oil	<0.01	ND	ND	nc	nc	nc	
TK0211638-05	Seed (pre-processing)	<0.01, <0.01, <0.01 [<0.01]	0.318, 0.329, 0.335 [0.327]	<0.01, 0.0101, 0.0104 [0.0102]	--	--	--	
	Meal	0.0113	0.500	0.0256	>1.13	1.53	2.51	
	Hulls	<0.01	<0.1	<0.01	nc	<0.31	<0.98	
	RBD oil	<0.01	ND	ND	nc	nc	nc	

Note: values denoted with 'c' are residues in control samples.

[a] Values reported as either <0.01 mg/kg or ND were assumed to be 0.01 mg/kg for purposes of calculating processing factors. Factors are based on results that have been corrected for residues in control samples.

nc = not calculated

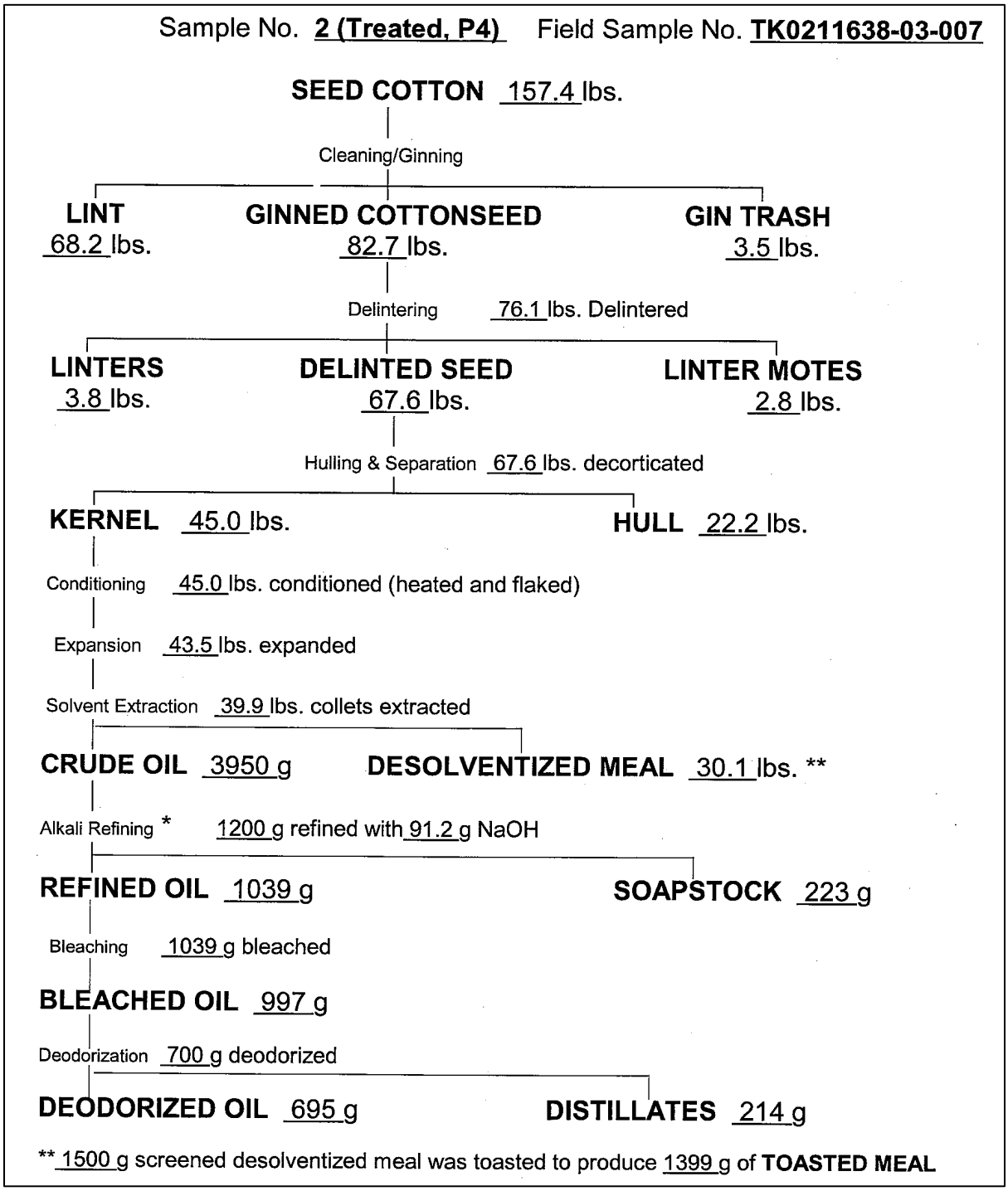


Figure 1 Processing scheme and material balance for cottonseed sample TK0211638-03.

Tea

Samples of dried tea leaves from field trials described in Report JM2019C029 were processed into tea infusion and analysed for residues of difenoconazole, 1,2,4-triazole, triazolylalanine, triazolylacetic acid, and triazolyllactic acid. Tea infusions were produced by soaking 9 g of dried tea leaves in 540 mL of boiling water for five minutes.

Table 26 Residues of difenoconazole in **tea infusion**

Location; year (Trial ID)	Crop Variety	Application				DALA	Residues (mg/kg) *	Study report
		No. [interval, days]	Rate, g ai/ha	L/ha	Commodity		Difeno-conazole	Remarks
Critical GAP (JP)	--	2 [n.s.]	2000X dilution	2000-4000		7	--	10% ai formulation: equivalent to 5 g ai/hL or 200 g ai/ha
Ibaraki, JP; 2016	Yabukita	2 [7]	2000X	3940	Tea infusion	7	0.74	JP2016C029
						14	0.12	
						21	0.01	
Saitama, JP; 2016	Hokumei	2 [7]	2000X	4000	Tea infusion	7	1.04	
						14	0.20	
						21	0.07	
Kagoshima, JP; 2016	Yamatomidori	2	2000X	4020	Tea infusion	7	0.14	
						14	0.05	
						21	0.01	
Miyazaki, JP; 2016	Yabikita	2	2000X	3970	Tea infusion	7	0.44	
						14	0.05	
						21	<0.01	

* Residues in tea infusion are reported on a dry-leaf basis

Residues of **1,2,4-triazole** were <0.01 mg/kg in all tea infusion samples.

Table 27 Residues of triazolylalanine, triazolylacetic acid, and triazolyllactic acid in **tea infusion**

Location; year (Trial ID)	Crop Variety	Application				DALA	Residues (mg/kg) *			Study report
		No. [interval, days]	Rate, g ai/ha	L/ha	Com-modity		Triazolyl-alanine	Triazolyl-acetic acid	Triazolyl-lactic acid	Remarks
Critical GAP (JP)	--	2 [n.s.]	2000X dilution	2000-4000		7	--	--	--	10% ai formulation: equivalent to 5 g ai/hL or 200 g ai/ha
Ibaraki, JP; 2016	Yabukita	2 [7]	2000X	3940	Tea infusion	7	0.13 (0.26 c)	0.06	0.36 (0.35 c)	JP2016C029
						14	0.12	0.06	0.36	
						21	0.24	0.10	0.43	

Location; year (Trial ID)	Crop Variety	Application				DALA	Residues (mg/kg) *			Study report Remarks
		No. [interval, days]	Rate, g ai/ha	L/ha	Com-modity		Triazolyl-alanine	Triazolyl-acetic acid	Triazolyl-lactic acid	
Saitama, JP; 2016	Hokumei	2 [7]	2000X	4000	Tea infusion	7	0.52 (0.18 c)	0.14	0.62 (0.29 c)	
						14	0.30	0.10	0.40	
						21	0.30	0.12	0.55	
Kagoshima, JP; 2016	Yamatomidori	2	2000X	4020	Tea infusion	7	0.50 (0.64 c)	0.13 (0.15 c)	0.20 (0.21 c)	
						14	0.42	0.11	0.20	
						21	0.48	0.13	0.23	
Miyazaki, JP; 2016	Yabikita	2	2000X	3970	Tea infusion	7	<0.05	<0.05	0.24 (0.16 c)	
						14	<0.05	<0.05	0.19	
						21	<0.05	<0.05	0.22	

* Residues in tea infusion are reported on a dry-leaf basis

Note: values denoted with 'c' are residues in control samples.

Table 28 Processing factors for residues of difenoconazole in **tea infusion**

Location; year (Trial ID)	Crop Variety	Difenoconazole residues (mg/kg)			Processing factor †	Study report Remarks
		Dry leaves	Infusion (dry-leaf)	Infusion (as consumed) *		
Ibaraki, JP; 2016	Yabukita	8.08	0.74	0.0123	0.0015	JP2016C029
		1.22	0.12	0.002	0.0016	
		0.09	0.01	0.000167	0.0019	
Saitama, JP; 2016	Hokumei	11.6	1.04	0.0173	0.0015	
		2.79	0.20	0.00333	0.0012	
		0.68	0.07	0.00117	0.0017	
Kagoshima, JP; 2016	Yamatomidori	2.08	0.14	0.00233	0.0011	
		0.81	0.05	0.000833	0.0010	
		0.16	0.01	0.000167	0.0010	
Miyazaki, JP; 2016	Yabikita	4.38	0.44	0.00733	0.0017	
		0.83	0.05	0.000833	0.0010	
		0.01	<0.01	<0.000167	<0.017	

* Residues in tea infusion were reported on a dry-leaf basis. To make the infusion, 9 g dried tea were mixed with 540 mL boiling water; therefore, to express the residue on an as-consumed basis, the residues in the infusion (dry leaf basis) were divided by 60.

† Residue in infusion (as consumed) ÷ residue in dry leaves

APPRAISAL

Difenoconazole (ISO common name) is a broad-spectrum conazole (triazole) fungicide used for the control of diseases in multiple crops. Its fungicidal mode of action is as a sterol demethylation inhibitor. Difenoconazole was evaluated for the first time by JMPR 2007 when an acceptable daily intake (ADI) of 0–0.01 mg/kg bw and an acute reference dose (ARfD) of 0.3 mg/kg bw were established. In 2007, 2010, 2013, 2015, and 2017, the JMPR evaluated the compound for residues and recommended a number of maximum residue levels.

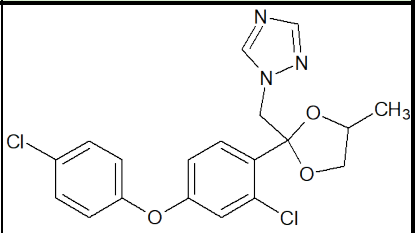
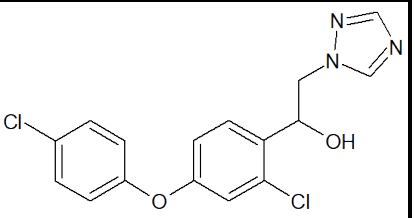
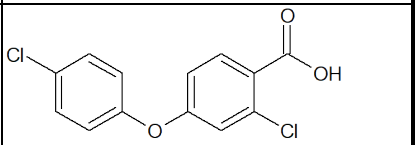
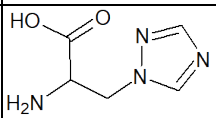
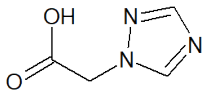
The definition of the residue for both compliance with the MRL and dietary risk assessment for plant commodities is: *difenoconazole*, and

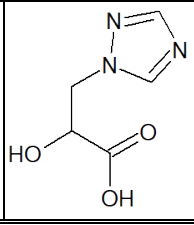
The definition of the residue for both compliance with the MRL and dietary risk assessment for animal commodities is: *the sum of difenoconazole and 1-[2-chloro-4-(4-chloro-phenoxy)-phenyl]-2-(1,2,4-triazol)-1-yl-ethanol (CGA205375), expressed as difenoconazole*.

The residue is fat-soluble.

Difenoconazole was scheduled at the Fifty-first Session of the CCPR for evaluation of additional uses by the 2020 JMPR, which was postponed to the 2021 Extra JMPR. The current Meeting received storage stability, GAP information, field trials, and processing studies to support maximum residue level estimations in commodities of cotton, cranberry, guava, and tea. In addition, the current Meeting also received seed treatment and confined rotational crop metabolism studies.

The following compounds are discussed in this document:

Common or code name	Chemical name molecular formula molar mass, g/mol	Structure
Difenoconazole	1-({2-[2-chloro-4-(4-chlorophenoxy)phenyl]-4-methyl-1,3-dioxolan-2-yl}methyl)-1*H*-1,2,4-triazole $C_{19}H_{17}Cl_2N_3O_3$ 406.26	
CGA205375	1-[2-chloro-4-(4-chlorophenoxy)phenyl]-2-(1*H*-1,2,4-triazol-1-yl)ethan-1-ol $C_{16}H_{13}Cl_2N_3O_2$ 350.20	
CGA189138	2-chloro-4-(4-chlorophenoxy)benzoic acid $C_{13}H_8Cl_2O_3$ 283.11	
CGA131013 Triazolylalanine	2-amino-3-(1*H*-1,2,4-triazol-1-yl)propanoic acid $C_5H_8N_4O_2$ 156.14	
CGA142856 Triazolylacetic acid	(1*H*-1,2,4-triazol-1-yl)acetic acid $C_4H_5N_3O_2$ 127.10	

Common or code name	Chemical name molecular formula molar mass, g/mol	Structure
CGA205369 Triazolyllactic acid	2-hydroxy-3-(1*H*-1,2,4-triazol-1-yl)propanoic acid $C_5H_7N_3O_2$ 157.13	

Plant metabolism

Wheat (seed treatment)

In a study submitted to the current Meeting, wheat grain was treated with either [^{14}C-chlorophenoxy] or [^{14}C-triazolyl]difenoconazole, each at ca. 32 mg ai/100 g seed. Treated seeds were container-grown in a greenhouse, and samples of forage (BBCH 14–21), hay (BBCH 56–65), and straw and grain (maturity) were analysed for TRR and extracted to determine the metabolic profile of the residue. Total radioactive residues were about 2- to 3-fold higher from the triazolyl treatment in forage (0.18 vs. 0.097 mg eq/kg), hay (0.11 vs. 0.03 mg eq/kg), and straw (0.31 vs. 0.11 mg eq/kg) samples, and approximately 80-fold higher in grain (0.42 vs. 0.005 mg eq/kg). The extractability of residues into acetonitrile/water ranged from 64 to 78% in straw, from 80 to 90% in hay, and approximately 90% in forage and grain. In total, approximately 70 to 96% of the unextracted radioactive residues in straw consisted of cellulose, lignin, and hemicellulose. Analyses of these components was not undertaken for other commodities.

In terms of specific residues, concentrations of parent difenoconazole were low in all commodities, ranging from not detected to 0.006 mg/kg. Following treatment with chlorophenoxy-labelled compound, the major residue was CGA 205375 (forage: 47% TRR, 0.07 mg eq/kg; hay: 60% TRR, 0.018 mg eq/kg; straw: 12% TRR, 0.013 mg eq/kg), with ca. 85% of the residue in forage and hay occurring as the glycoside-conjugated form (which was not detected in straw). Grain samples from the chlorophenoxy-labelled treatment were not extracted due to low radioactivity. Treatment with triazolyl-labelled difenoconazole resulted, again, in CGA 205375 (free and conjugated) as a major residue in forage, hay, and straw (forage: 61% TRR, 0.133 mg eq/kg; hay: 23% TRR, 0.026 mg eq/kg; straw: 11% TRR, 0.03 mg eq/kg), but was not detected in grain; the residue consisted primarily (ca. 85%) in a glycoside-conjugate form in forage and hay. Other major residues observed following treatment with triazolyl-labelled compound were triazolylalanine (CGA131013) (forage: 6.4% TRR, 0.014 mg eq/kg; hay 16% TRR, 0.018 mg eq/kg; straw: 11% TRR, 0.031 mg eq/kg; grain: 27% TRR, 0.11 mg eq/kg) and 1,2,4-triazolylacetic acid (forage: 9.2% TRR, 0.020 mg eq/kg; hay: 30% TRR, 0.034 mg eq/kg; straw: 41% TRR, 0.113 mg eq/kg; grain: 33% TRR, 0.13 mg eq/kg). Unidentified compounds were reported as being ≤10% TRR from either treatment in all matrices; however, up to approximately 30% of the radioactivity was not accounted for.

The Meeting noted that metabolism of difenoconazole by plants following foliar (tomato, potato, grape, oilseed rape) and seed-treatment (wheat) applications was evaluated by the 2007 JMPR. Overall, the residues were described as low amounts of difenoconazole and higher amounts of the metabolite CGA 205375. The 2007 Meeting noted that 'The presence of triazolylalanine and triazolylacetic acid was suggested but not confirmed for extracts of the mature grain (triazole label).' The results of the current study are generally in good agreement with those from the studies reviewed by the 2007 Meeting. The Meeting decided that the new study does not impact the established residue definitions for difenoconazole.

Environmental fate

Confined rotational crops

In the study submitted to the current Meeting, difenoconazole was labelled only in the chlorophenyl moiety. The study was conducted by treating bare soil at a rate of ca. 0.52 kg ai/ha (approximately 0.5X the maximum seasonal rate for most crops). Lettuce, radish, sorghum, and wheat were planted into treated soil 30, 60, 120, and 270 days after the application. Total radioactive residues at the 30-day plant-back interval (PBI) ranged from 0.01 mg eq/kg to 0.083 mg eq/kg. In the majority of analysed crop matrices, TRR levels showed little to no decline with increasing plant-back intervals (PBI) of 30–270 days. The exceptions were lettuce and wheat straw, where residues from the 270-day PBI were ca. 2- to 4-fold lower than those at the 30-day PBI. TRRs in wheat grain were very low (0.004–0.006 mg eq/kg) and did not decline with increasing PBI. In most crops, the proportion of unextracted residues (acetonitrile:water; 4:1, v/v) increased with longer PBIs.

In immature and mature lettuce, radish leaves, and radish roots, difenoconazole was the principal residue at all PBIs. Difenoconazole was a minor residue in sorghum forage and wheat straw and was not detected in sorghum stover. CGA 205375 and CGA 189138 were minor residues in lettuce and sorghum samples. CGA 205375 was a major residue in radish leaves and roots (generally 15–22% TRR) but occurred at low levels (< 0.007 mg eq/kg). CGA 205375 was also a major residue in wheat straw (ca. 40% TRR, ca. 0.02 mg eq/kg).

The 2007 Meeting also reviewed confined rotational crop studies (phenyl and triazolyl label; lettuce, wheat, maize, sugar beet, mustard, turnips). The TRR from those studies were generally low (< 0.01 mg eq/kg) following treatment with the phenyl label and higher (0.01–0.34 mg eq/kg) from the triazolyl label. The predominant residues were triazolylalanine (10–66% TRR), triazolylacetic acid (0–39% TRR), and triazolyllactic acid (0–54% TRR), depending on the matrix.

The 2007 JMPR had noted that the confined rotational crops studies indicated that the water-soluble and mobile metabolites triazolylalanine, triazolylacetic acid and triazolyllactic acid would be the predominant residues in rotational crops. Limited field rotational crop studies evaluated by the 2007 JMPR showed absence of residues of triazolylalanine and difenoconazole above their respective LOQs in carrots and spinach as following crops. While the available confined rotational crop studies indicate that difenoconazole may occur in rotational crops, the current Meeting concludes, based on a comparison of residues in confined and field rotational crop studies, that residues of difenoconazole above the LOQ are unlikely to occur.

Methods of analysis

The Meeting received method validation and concurrent recovery data for use of the QuEChERS multiresidue method to analyse residues in guava and concurrent recovery data for use of Method REM 147.08 on cranberry, cotton, and tea commodities. Method REM 147.08 was previously reviewed by the 2007, 2010, 2015, and 2017 Meetings. Both methods were demonstrated to have adequate performance for recovery of difenoconazole, with an LOQ of 0.01 mg/kg.

The Meeting also received method validation and concurrent recovery data for Method Meth-160, Revision #2. This method was used for analysis of 1,2,4-triazole, triazolylalanine, and triazolylacetic acid in cotton and tea commodities. All three analytes were validated with an LOQ of 0.01 mg/kg in cotton seed and gin by-products. Data were insufficient (too few replicates) to establish a validated LOQ in cotton meal, seed hulls, and refined oil; nevertheless, the Meeting noted that recoveries of all three analytes were good

(≥ 89%) at the lowest fortification levels (0.01 mg/kg for 1,2,4-triazole and triazolylacetic acid, 0.1 mg/kg for triazolylalanine).

The Meeting decided that the methods used for data collection of metabolite residues are fit for purpose for all analytes.

Stability of pesticide residues in stored analytical samples

Storage stability data were provided for difenoconazole residues in oranges, dried beans, guava, and tea. In addition, storage stability data were provided for 1,2,4-triazole; triazolylalanine, triazolylacetic acid, and triazolyllactic acid residues in tea.

Residues of all analytes were shown to be stable as follows:

Difenoconazole

Oranges: at least 732 days (24 months),

Dried beans: at least 732 days (24 months),

Guava: at least 398 days (13 months),

Dry tea leaves: at least 372 days (12 months),

1,2,4-triazole; triazolylalanine, triazolylacetic acid, and triazolyllactic acid

Dry tea leaves: at least 407–412 days (13 months).

The Meeting confirms its previous conclusions that residues of difenoconazole are 'stable at approximately -20 °C for two years in matrices with high oil content (cotton seed, cotton seed meal, cotton seed oil (soya bean only one year)), high water content matrices (potatoes, tomatoes (banana, lettuce only one year)), high starch matrices (wheat grain) and wheat forage and straw'.

Results of supervised residue trials on crops

The Meeting received data from supervised residue trials and GAP information on guava, cranberry, cotton, and tea.

Guava

Labels were provided for use of difenoconazole on guava in Egypt and the USA. On a per-hectare basis, the US label appears to be the critical GAP (3 × 128 g ai/ha, 7-day RTI, 0-day PHI versus 3 × 89 g ai/ha, 7-day RTI, 7-day PHI). No field trials matching the US GAP were available; therefore, the Meeting considered the GAP from Egypt.

The Egyptian GAP consists of three applications of 6.25 g ai/hL, using 950–1430 L/ha (equivalent to 59–89 g ai/ha), on a 7-day interval, with a PHI of 7 days.

Residues of difenoconazole in independent trials conducted in Egypt and matching the Egyptian GAP were (n = 6): 0.013, 0.018, 0.033, 0.034, 0.053, and 0.077 mg/kg.

The Meeting estimated a maximum residue level of 0.15 mg/kg, a STMR of 0.0335 mg/kg and a HR of 0.095 mg/kg (from a single sample) for guava.

Cranberry

The critical GAP for cranberry is from the USA and consists of three applications each at 128 g ai/ha, on a 7-day interval, with a PHI of 30 days with a maximum seasonal rate of 381 g ai/ha.

Residues of difenoconazole in independent trials approximating the critical GAP were (n = 4): 0.14, 0.20 (2), and 0.24 mg/kg.

The Meeting estimated a maximum residue level of 0.6 mg/kg, a STMR of 0.2 mg/kg, and a HR of 0.26 mg/kg (from a single sample) for cranberry.

Cotton seed

The critical GAP for cotton is from the USA and consists of three applications each at 128 g ai/ha, on a 14-day interval, with a PHI of 45 days with a maximum seasonal rate of 381 g ai/ha.

Residues of difenoconazole in cotton seed from independent trials approximating the critical GAP were (n = 12): < 0.01 (4), 0.012, 0.018, 0.024, 0.036, 0.041, 0.047, 0.14, and 0.30 mg/kg.

The Meeting estimated a maximum residue level of 0.4 mg/kg and a STMR of 0.021 mg/kg for cotton seed.

Tea

The critical GAP for tea is from Japan and consists of two applications of a 2000-fold dilution (5 g ai/100 L) of the formulated product with a 7-day PHI; the minimum re-treatment interval is not specified.

The Meeting considered the three sets of supervised field trials (Japan, 1993, 2016 and Taiwan Province of China, 2006), field trial use patterns matched the critical GAP. Residues of difenoconazole in independent trials were (n = 8): 1.6, 1.9, 2.1, 4.4, 5.3, 7.9, 8.1, and 12 mg/kg in dried tea leaves.

The Meeting estimated a maximum residue level of 20 mg/kg and a STMR of 4.85 mg/kg for tea, green, black (black, fermented and dried).

Residues in animal feeds

Cotton gin by-products

The critical GAP for cotton is from the USA and consists of three applications each at 128 g ai/ha, on a 14-day interval, with a PHI of 45 days with a maximum seasonal rate of 381 g ai/ha.

Residues of difenoconazole in cotton gin by-products from independent trials approximating the critical GAP were (n = 3): 1.2, 0.50, and 5.6 mg/kg.

The Meeting agreed that three trials were not sufficient to make a recommendation for residues of difenoconazole in cotton gin by-products.

Fate of residues during processing

The Meeting received data showing the effect of processing cotton seed into meal, hulls, and refined oil. In addition, the Meeting received data on residues in tea infusion created from treated dried tea leaves. Processing factors and residue estimates are summarized below.

Table 1 Processing factors and residue estimates for difenoconazole

Raw commodity	Residue in RAC, mg/kg			Processed commodity	Processing Factors		Residue in processed commodity, mg/kg		
	Max	STMR	HR		Individual	Best estimate	Max-P	STMR-P	HR-P
Cotton seed	0.4	0.021	--	Meal	< 0.066, 0.089	0.089	--	0.0019	--
				Hulls	< 0.066, 0.43	0.43	--	0.0090	--

Raw commodity	Residue in RAC, mg/kg			Processed commodity	Processing Factors		Residue in processed commodity, mg/kg		
	Max	STMR	HR		Individual	Best estimate	Max-P	STMR-P	HR-P
				RBD oil [a]	< 0.066, < 0.088	< 0.066	--	< 0.0014	--
Tea	20	4.85	--	Tea infusion	0.0010 (3), 0.0011, 0.0012, 0.0015 (2), 0.0016, 0.0017 (2), 0.0019, < 0.017	0.0015	--	0.0072	

[a] Refined Bleached Deodorized

Residues of difenoconazole did not concentrate in any of the processed commodities considered by the Meeting; therefore, the Meeting did not estimate maximum residues for those commodities. Residues for estimating animal dietary burden and for dietary risk assessment are provided in the table above.

Residues in animal commodities

Of the uses considered by the Meeting, only cotton meal, cotton hulls, and cotton gin by-products are significant animal feed items. The Meeting was not able to make residue recommendations for cotton gin by-products. A comparison of the most recent burden estimates used for residue estimation (2013 JMPR) with those estimated by this meeting using current practices are shown below.

Table 2 Dietary burden (ppm) comparison from the 2013 JMPR and the current Meeting

Animal	Maximum		Mean	
	2013 JMPR	Current	2013 JMPR	Current
Beef cattle	17.9	17.9	15.3	15.0
Dairy cattle	14.9	14.7	12.4	12.1
Poultry (broiler and layer)	1.89	1.20	1.11	0.80

As the dietary burdens were essentially unchanged or are lower than those from the previous Meeting, the Meeting confirmed its previous recommendations for residues in animal commodities.

RECOMMENDATIONS

On the basis of the data obtained from supervised trials, the Meeting concluded that the residue levels shown in Table 3 below are suitable for establishing maximum residue limits and for IEDI and IESTI assessments.

Definition of the residue for compliance with the MRL and dietary risk assessment for plant commodities: *difenoconazole.*

Definition of the residue for compliance with the MRL and dietary risk assessment for animal commodities: *sum of difenoconazole and 1-[2-chloro-4-(4-chloro-phenoxy)-phenyl]-2-(1,2,4-triazol)-1-yl-ethanol (CGA205375), expressed as difenoconazole*

The residue is fat-soluble.

Table 3 Recommendations for residues of difenoconazole from the 2021 Extra JMPR

CCN	Crop/Commodity	Recommended maximum residue level, mg/kg		STMR or STMR-P, mg/kg	HR or HR-P, mg/kg
		New	Previous		
FT 0336	Guava	0.15	--	0.0335	0.095
FB 0265	Cranberry	0.6	--	0.2	0.26
SO 0691	Cotton seed	0.4	--	0.021	--
DT 1114	Tea, green, black (black, fermented and dried)	20	--	4.85	--
For dietary risk assessment and/or dietary burden calculations					
AB 1203	Cotton seed meal			Median: 0.0019	
AB 0691	Cotton seed hulls			Median: 0.009	
OR 0691	Cotton seed oil, edible			0.0014	
	Tea infusion			0.0072	

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for difenoconazole is 0–0.01 mg/kg bw. The International Estimated Daily Intakes (IEDIs) for difenoconazole were estimated for the 17 GEMS/Food Consumption Cluster Diets using the STMR or STMR-P values estimated by the JMPR. The results are shown in Annex 3 of the 2021 Extra JMPR Report.

The IEDIs ranged from 10–80% of the maximum ADI. The Meeting concluded that long-term dietary exposure to residues of difenoconazole from uses considered by the JMPR is unlikely to present a public health concern.

Acute dietary exposure

The ARfD for difenoconazole is 0.3 mg/kg bw. The International Estimate of Short-Term Intakes (IESTIs) for difenoconazole were calculated for the food commodities and their processed commodities for which HRs/HR-Ps or STMRs/STMR-Ps were estimated by the present Meeting and for which consumption data were available. The results are shown in Annex 4 of the 2021 Extra JMPR Report.

The IESTIs varied from 0–2% of the ARfD for children and 0–2% of the ARfD for the general population. The Meeting concluded that acute dietary exposure to residues of difenoconazole from uses considered by the present Meeting is unlikely to present a public health concern.

REFERENCES

Study Report	Authors	Year	Citation
SYN-018/6-40	Gärtner, C. and Herrchen, M.	2019	Difenoconazole – Metabolism of ^{14}C-Difenoconazole Following Cereal Seed Treatment, Final Report Amendment 1. Syngenta File No. CGA169374_11523

Study Report	Authors	Year	Citation
T009582-07	Findok, D. and Herczog, K.	2010	Difenoconazole – [^{14}C]-Difenoconazole: Uptake and Metabolism of in Confined Rotational Crops. Syngenta File No. CGA169374_10495
TK0208888	Andrews, D. and Fowle, G.	2015	Difenoconazole (CGA169374) – Storage Stability of Residues of Difenoconazole in Crop Matrices Stored Frozen For Up to Two Years. Syngenta File No. CGA169374_10932
TK0211638	McDonald, T. J.	2016	Difenoconazole SC (A13703G) and Difenoconazole EC (A7402T) – Magnitude of the Residues in or on Cotton – USA 2014. Syngenta File No. A13703G_A7402T
PR 10828	Homa, K.	2015	Difenoconazole + Azoxystrobin: Magnitude of the residue on cranberry. Syngenta File No. A13703G_50076
11605	Noegrohati, S.	2016	Difenoconazole + Azoxystrobin: Magnitude of the Residue on Guava. Syngenta File No. A13703G_11491
T010388-05-REG	Jones, S.	2008	Difenoconazole (CGA169374) – Residue Study on Tea in Taiwan in 2006. Syngenta File No. CGA169374_3336
JP2016C029	Takahashi, Y.	2018	Crop Residue Study on Green Tea with Difenoconazole (SCORE) Water Dispersible Granule Syngenta File No. A8885J_10064
A7491J_10043	Kuroda, M. and Higuchi, T.	1993	Crop Residue Analysis Report (tea; open field)
A7491J_10059	Komatsu, K and Yabusaki, T.	1993	Crop Residue Analysis Report (tea; shaded)

ETHION (034)

First draft prepared by Dr M Lee, Andong National University, Republic of Korea

EXPLANATION

Ethion is an organophosphate insecticide and acaricide with non-systemic and contact action. It was first evaluated by the JMPR in 1968, and most recently for toxicology in 1990 and residues in 1994. The 1990 JMPR established an ADI of 0–0.002 mg/kg and the 1994 JMPR defined the residue as *ethion* (*fat-soluble).*

The Thirty-sixth CCPR (2004) agreed to delete the CXL for citrus fruits, as the use of ethion would no longer be supported. In the Thirty-seventh CCPR (2005), for pesticides including ethion, it was decided to apply an approach of use of monitoring data to establish MRLs for spices as no GAP is available.

Thus, currently, ethion is included in the CAC list of pesticides whose MRLs (CXLs) or GLs have been deleted by the CAC and for which no MRLs have been proposed. The CXLs for ethion exist only for spices based on monitoring data.

Ethion was scheduled at the Fifty-first Session of the CCPR for evaluation of additional uses by the 2020 JMPR, which was postponed to the 2021 Extra JMPR. The Meeting received information on GAP and residue trials on chili pepper.

RESIDUE ANALYSIS

Analytical methods

The green chili samples were homogenized at 3,000 rpm for 2 minutes. Representative 15 g homogenized samples were subjected to QuEChERS sample preparation method: add acetonitrile (containing 1% acetic acid) and salts (magnesium sulphate, sodium acetate), shake, centrifuge, and then clean-up of the organic layer by dispersive solid-phase extraction. Determination of ethion was conducted by GC-FPD or GC-ECD. Matrix-matched standard solutions at five different concentrations, showing linearity at $r^2 \geq 0.99$, were used. Recovery tests were performed at three fortification levels (n=3 at 0.05, 0.25 and 0.5 mg/kg). Mean recovery value at each fortification level/test ranged from 74–111% (RSDs, ≤6%). The LOQ values for ethion were 0.05 mg/kg. Table 1 shows the results of recovery test.

Table 1 Recovery test results for ethion in chili pepper, green

Trial location	Fortification level, mg/kg	Individual values, %	Mean value, %	RSD, %	Instrument used
Anand	0.05	85.0, 89.7, 82.4	86	4	GC-FPD
	0.25	98.1, 89.6, 99.6	96	6	
	0.5	97.3, 108, 99.0	102	6	
Hisar	0.05	84.5, 88.5, 84.2	86	3	GC-FPD
	0.25	87.9, 90.7, 87.8	89	2	
	0.5	95.3, 101, 99.4	99	3	
New Delhi	0.05	74.8, 73.4, 74.4	74	1	GC-ECD
	0.25	83.6, 84.0, 83.1	84	1	
	0.5	91.5, 90.8, 92.1	92	1	
Bangalore	0.05	95.6, 93.3, 92.8	94	2	GC-ECD
	0.25	96.1, 97.3, 98.5	97	1	
	0.5	97.3, 99.4, 99.7	99	1	

Trial location	Fortification level, mg/kg	Individual values, %	Mean value, %	RSD, %	Instrument used
Ludhiana	0.05	86.8, 85.1, 87.4	86	1	GC-ECD
	0.25	93.6, 92.8, 91.9	93	1	
	0.5	89.5, 87.8, 88.7	89	1	
Hyderabad	0.05	110, 110, 114	111	2	GC-ECD
	0.25	107, 102, 99.6	103	4	
	0.5	105, 102, 105	104	2	

All LOQ values, <0.05 mg/kg

USE PATTERN

The Meeting received the GAP information on chili pepper from India. The information is summarised in Table 2.

Table 2 Registered use of ethion on chili pepper in India

Crop	Formulation	Application			PHI (days)
		Method	kg ai/ha	Dilution in water, L/ha	
Chili pepper	50 EC (54.5% w/w)	Foliar spray	0.75-1.0	500-1,000	5

Number of sprays and an application interval: not specified

RESULTS OF SUPERVISED RESIDUE TRIALS ON CROPS

The Meeting received residue trials on chili pepper conducted in India. The detailed information are summarised in Table 3 as below.

Fruiting vegetables, other than Cucurbits

Chili pepper

Supervised field trials (six trials) on chili pepper were conducted during 2014–2016 in India under All India Network Project on Pesticide Residues [Sharma, K.K., 2019]. Each trial comprised three plots per treatment and one control plot. Ethion (50 EC form.) was applied with foliar spray (500 L/ha) at a rate of 0.50 kg ai/ha. Two applications were made with an interval of 10 days at fruiting stage. Green chili pepper samples (1 kg) were taken at 0, 1, 3, 5, 7, 10, 15, 20 and (30) days after the last application. Samples, separately collected from each plot, were extracted immediately (within 24 hours) after sampling and analysed. In all control samples, ethion was not detected.

Table 3 Residue concentration of ethion from residue trials on chili pepper, green in India (replicate plots)

Location, Year (Variety)	Application			DALA	Ethion, mg/kg			
	n	kg ai/ha	Int. days		Individual value			Mean
GAP: India		0.75-1.0		PHI, 5 days				
Anand, 2016 (GVC-11)	2	0.50	10	0	5.9	6.6	6.9	6.5
				1	5.0	5.5	4.8	5.1
				3	2.3	1.7	2.3	2.1

Location, Year (Variety)	Application			DALA	Ethion, mg/kg			
	n	kg ai/ha	Int. days		Individual value			Mean
				5	1.8	1.5	1.5	1.6
				7	1.0	1.3	0.97	1.1
				10	1.2	1.2	1.4	1.3
				15	0.82	0.88	0.82	0.84
				20	<0.05	<0.05	<0.05	<0.05
Hisar, 2014 (Kanshi Anmol)	2	0.50	10	0	8.0	7.3	7.7	7.7
				1	5.0	4.2	3.7	4.3
				3	3.5	3.3	3.4	3.4
				5	2.3	2.4	3.0	2.6
				7	2.0	2.3	3.0	2.4
				10	2.0	2.2	2.2	2.1
				15	0.80	1.0	1.2	1.0
				20	<0.05	<0.05	<0.05	<0.05
New Delhi, 2015 (Pusa Sadabahar)	2	0.50	10	0	3.4	3.1	3.2	3.2
				1	3.0	2.9	2. 8	2.9
				3	1.6	1.6	1.4	1.5
				5	1.0	1.1	1.1	1.1
				7	0.44	0.49	0.51	0.48
				10	0.14	0.15	0.07	0.12
				15	<0.05	<0.05	<0.05	<0.05
				20	<0.05	<0.05	<0.05	<0.05
Bangalore, 2014 (Arka suphal)	2	0.50	10	0	1.5	1.5	1.5	1.5
				1	1.5	1.2	1.3	1.3
				3	1.1	1.2	1.1	1.1
				5	0.93	0.96	0.98	0.96
				7	0.39	0.82	1.11	0.77
				10	0.87	0.38	0.67	0.64
				15	0.58	0.35	0.67	0.53
				20	0.42	0.40	0.38	0.40
				30	<0.05	<0.05	<0.05	<0.05
Ludhiana, 2014 (CH-3)	2	0.50	10	0	2.3	2.4	2.0	2.3
				1	1.5	1.5	1.6	1.5
				3	1.3	1.2	1.2	1.2
				5	0.92	0.97	0.99	0.96
				7	0.80	0.87	0.71	0.79
				10	0.56	0.48	0.61	0.55
				15	0.36	0.31	0.35	0.34
				20	0.27	0.27	0.26	0.27
				30	<0.05	<0.05	<0.05	<0.05
Hyderabad, 2014 (Aspire Green)	2	0.50	10	0	4.2	4.3	4.1	4.2
				1	3.6	3. 8	3.9	3.7
				3	2.3	2.3	2.1	2.2
				5	1.9	1.9	1.9	1.9

Location, Year (Variety)	Application			DALA	Ethion, mg/kg			
	n	kg ai/ha	Int. days		Individual value			Mean
				7	1.9	1. 9	1.5	1.8
				10	0.70	0.81	0.74	0.75
				15	0.58	0.63	0.64	0.62
				20	0.22	0.31	0.31	0.28
				30	<0.05	<0.05	<0.05	<0.05

50 EC formulation was applied.

APPRAISAL

Ethion is an organophosphate insecticide and acaricide with non-systemic and contact action. It was first evaluated by the JMPR in 1968, and most recently for toxicology in 1990 and residues in 1994. The 1990 JMPR established an ADI of 0–0.002 mg/kg bw and the 1994 JMPR defined the residue as ethion (fat-soluble).

Currently, ethion is included in the Codex Alimentarius Commission (CAC) list of pesticides whose MRLs (CXLs) or Guideline levels (GLs) have been deleted by the CAC and for which no MRLs have been proposed. The CXLs for ethion exist only for spices based on monitoring data.

Ethion was scheduled at the Fifty-first Session of the CCPR for evaluation of additional uses by the 2020 JMPR, which was postponed to the 2021 Extra JMPR. The Meeting received information on GAP and residue trials on chili pepper.

Methods of analysis

Analysis of ethion in chili peppers was conducted by a new method using QuEChERS sample preparation and GC-FPD or GC-ECD. Recovery test results showed recoveries of 74–111%. The LOQ value was 0.05 mg/kg. The analytical method used in the chili pepper residue trials was considered sufficiently validated.

Stability of residues in stored analytical samples

Residue analysis was performed within 24 hours after sample collection.

Results of supervised residue trials on crops

Fruiting vegetables, other than Cucurbits

Chili pepper

The critical GAP for the use of ethion on chili pepper in India involves foliar sprays at 1.0 kg ai/ha and a 5-day PHI (the maximum number of sprays and minimum re-treatment intervals are not specified).

Six residue trials (decline) on chili pepper were conducted in India during 2014–2016, at a rate of 0.50 kg ai/ha with 2 applications at a 10-day interval and a PHI of 5 days. The residue concentrations of ethion in chili pepper were (n = 6): 0.96, 0.96, 1.1, 1.6, 1.9 and 2.6 mg/kg (highest value of 3.0 mg/kg from replicate plots). The trials did not match the application rate of the critical GAP.

Communication by the sponsor indicated that the local agricultural practice involves re-treatment intervals of 2–4 days. Residues declined with a median half-life of 4.1 days (1st order) and the modelled residue at a spray interval of 2 days and 2 applications differed by more than 25% compared to the supervised field trials. The Meeting concluded that the supervised field trials were conducted at a significantly longer re-treatment interval in combination with lower application rates and cannot be used for the estimation of a maximum residue level

REFERENCES

Author	Year	Study title, Institute
K.K. Sharma	2019	Data/Information for Fixation of MRL of Ethion on Green Chili. All India Network Project on Pesticide Residues, ICAR-Indian Agricultural Research Institute, New Delhi-110012, India

ETHIPROLE (304)

First draft prepared by Dr Yukiko Yamada, International Food Safety Consultant and Adjunct Professor, Azabu University, Japan

EXPLANATION

Ethiprole is a non-systemic phenylpyrazole insecticide. It was first evaluated by the 2018 JMPR for toxicology and residues. The 2018 JMPR established an ADI of 0–0.005 mg/kg bw and an ARfD of 0.005 mg/kg bw.

The 2018 JMPR reviewed information on identity, physical and chemical properties, plant metabolism and environmental fate, residue analysis and storage stability, use pattern, supervised trials on many crops, processing, and animal feeding; and recommended the following residue definitions:

Definition of the residue for compliance with the MRL for plant commodities: *Ethiprole*

Definition of the residue for dietary risk assessment for plant commodities: *Sum of ethiprole, 5-amino-1-[2,6-dichloro-4-(trifluoromethyl)phenyl]-4-(ethylsulfinyl)-1H-pyrazole-3-carboxamide (ethiprole amide) and 5-amino-1-(2,6-dichloro-4-(trifluoromethylphenyl)-4-ethylsulfonylpyrazole-3-carbonitrile (ethiprole-sulfone), expressed as parent equivalents.*

Definition of the residue for compliance with the MRL and dietary risk assessment for animal commodities: *Sum of ethiprole and 5-amino-1-(2,6-dichloro-4-(trifluoromethylphenyl)–4-ethylsulfonylpyrazole-3-carbonitrile (ethiprole-sulfone), expressed as parent equivalents.*

The residue is fat-soluble.

On the basis of the above residue definitions, the 2018 JMPR estimated maximum residue levels for rice, coffee and related commodities as well as commodities of animal origin.

The Forty-third Codex Alimentarius Commission (2020) approved the new work proposals including the priority list of pesticides for evaluation by the current Meeting. The priority list included ethiprole for evaluation of an additional use on soya beans. The present Meeting received new validation information on an analytical method, supervised residue trials and processing studies on soya beans.

RESIDUE ANALYSIS

Analytical methods

The 2018 JMPR reviewed three LC-MS/MS methods, including Method 01128, used for data generation, and their validation data. Method 01128 was used in the supervised trials on soya beans submitted to the current Meeting. In Method 01128, ethiprole, ethiprole-sulfone and ethiprole-amide were extracted with acetonitrile/water (9:1, v/v) with homogenization; the extract was centrifuged; and an aliquot of supernatant was diluted with acetonitrile/water (9:1, v/v) for analysis by LC-MS/MS (Santiago, 2013, M-455162-01-2). The 2018 JMPR concluded that Method 01128 was sufficiently validated for the determination of ethiprole, ethiprole-sulfone and ethiprole-amide in soya beans at fortification levels of 0.01 and 1.0 mg/kg for each analyte with the LOQ at 0.01 mg/kg.

The current Meeting received new information on the validation and procedural recovery data on Method 01128 generated in the supervised trial studies and processing studies on soya beans. The data on validation are summarized in Table 1 below as well as the results of independent validation on soya beans

reviewed by the 2018 JMPR. In the new validation studies, at the fortified levels of 0.002–2.0 mg/kg in soya beans, the recoveries and RSD values were within the acceptable range. The validated LOQ was 0.002 mg/kg for the three analytes in soya beans.

The results of concurrent recoveries on soya beans and processed soya bean commodities are summarized in Table 2. At all fortification levels in soya beans and processed commodities, the recoveries and RSD values were within the acceptable range. However, for aspirated grain fractions, recoveries were tested at only one fortification level of 100 mg/kg for each analyte, which is much higher than the concentrations of these analytes reported in the processing study.

Table 1 Summary of validation of Method 01128 (LC-MS/MS) for the determination of ethiprole residues in soya beans (validation data evaluated by the 2018 JMPR and those in supervised trial studies evaluated by the current Meeting)

Matrix	Fortification level (mg/kg)	n	Recovery (%)			Recovery (%)		
			Range	Mean	RSD	Range	Mean	RSD
			Quantitation			Confirmation		
Ethiprole			m/z 395 → 250			m/z 395 → 330		
Soya beans [a]	0.01	6	100-110	106	3.5	93-108	101	6.3
	1.0	5	89-104	96	6.6	88-105	97	6.6
			m/z 395 → 330			m/z 395 → 250		
Soya beans [b]	0.002	5	107-118	114	4.6	106-120	114	4.8
	0.2	5	86-107	98	8.8	90-112	98	9.1
	2.0	5	82-118	97	14.9	76-113	96	16.3
Soya beans [c]	0.002	5	83-110	96	11	92-118	102	9.9
Ethiprole-sulfone			m/z 411 → 375			m/z 411 → 282		
Soya beans [a]	0.01	7	97-120	112	6.9	108-116	112	3.2
	1.0	5	81-100	91	9.2	82-101	90	9.1
Soya beans [b]	0.002	5	113-120	116	2.5	112-119	116	2.2
	0.2	5	89-110	97	8.8	88-112	97	9.5
Soya beans [c]	0.002	5	88-113	100	9.1	80-105	97	10
Ethiprole-amide			m/z 415 → 398			m/z 415 → 255		
Soya beans [a]	0.01	5	94-108	103	5.5	96-99	98	1.6
	1.0	5	92-104	97	4.7	88-101	94	5.4
Soya beans [b]	0.002	5	90-101	96	4.1	87-98	94	4.6
	0.2	5	95-107	98	5.4	89-100	93	5.0
Soya beans [c]	0.002	5	89-103	96	6.1	78-103	90	11

[a] Santiago, 2012, M-455162-01-2 (reviewed by the 2018 JMPR)

[b] Abdal, 2019, M-674212-01-1

[c] Alves, 2018, M-636713-01-1

Table 2 Summary of concurrent recovery data of Method 01128 (LC-MS/MS) for the determination of ethiprole residues in soya beans in supervised trial studies and processed commodities in processing studies

Matrix	Fortification level (mg/kg)	n	Recovery (%)			Reference
			Range	Mean	RSD	
Ethiprole			m/z 395 → 330			
Soya beans	0.002	5	71-99	84	13.2	Alves, 2018, M-636713-01-1
	1.0	5	94-109	101	5.5	
Soya beans	0.002	3	91-111	98	11.8	Abdal, 2019, M-674212-01-1
	0.2	3	78-101	87	13.9	
	2.0	2	82-104	93	-	
Soya beans	0.002	7	75-100	89	10.9	Lenz and Fischer, 2016, M-565737-01-1
	0.10	3	88-98	94	6.0	
Aspirated grain fractions	100	3	96-117	103	11.7	
Flour	0.002	3	81-92	85	7.2	
	0.004	3	85-97	92	6.3	
	0.10	3	72-78	76	3.8	
Hulls	0.002	3	101-111	107	4.8	
	0.10	3	85-86	86	0.6	
	0.80	3	80-85	82	3.2	
Soya milk	0.002	3	85-89	86	3.0	
	0.10	3	83-86	85	1.6	
Refined oil (solvent extraction)	0.002	3	87-97	93	5.5	
	0.10	3	85-94	89	5.1	
Ethiprole-sulfone			m/z 411 → 375			
Soya beans	0.002	7	72-78	75	4.0	Alves, 2018, M-636713-01-1
	1.0	5	97-106	100	3.8	
Soya beans	0.002	2	74	74	-	Abdal, 2019, M-674212-01-1
	0.2	2	73-76	75	-	
Soya beans	0.002	7	84-92	88	3.2	Lenz and Fischer, 2016, M-565737-01-1
	0.10	3	95-104	101	5.2	
Aspirated grain fractions	100	3	96-106	99	5.4	
Flour	0.002	3	103-108	104	2.6	
	0.004	3	91-99	95	4.0	
	0.10	3	88-93	90	3.0	
Hulls	0.002	3	99-103	101	2.4	
	0.10	3	104-107	105	1.8	
	0.80	3	84-90	87	3.1	
Soya milk	0.002	3	102-109	104	3.5	
	0.10	3	98-102	100	2.2	
Refined oil (solvent extraction)	0.002	3	83-87	85	2.2	
	0.10	3	76-80	79	3.1	
Ethiprole-amide			m/z 415 → 398			

Matrix	Fortification level (mg/kg)	n	Recovery (%)			Reference
			Range	Mean	RSD	
Soya beans	0.002 1.0	5 5	89-115 92-111	102 102	2022222 229.5 7.3	Alves, 2018, M-636713-01-1
Soya beans	0.002 0.2	1 1	87 116	- -	- -	Abdal, 2019, M-674212-01-1
Soya beans	0.002 0.10	7 3	90-111 92-95	100 93	6.8 1.6	Lenz and Fischer, 2016, M-565737-01-1
Aspirated grain fractions	100	3	98-101	100	1.6	
Flour	0.002 0.004 0.10	3 3 3	97-107 94-97 106-107	101 96 106	5.2 1.5 0.5	
Hulls	0.002 0.10 0.80	3 3 3	97-105 113-117 98-100	100 115 99	4.0 1.6 0.9	
Soya milk	0.002 0.10	3 3	85-101 93-95	93 94	8.7 0.8	
Refined oil (solvent extracted)	0.002 0.10	3 3	99-101 94-102	100 97	0.8 4.5	

The 2018 JMPR evaluated storage stability data including data on soya beans. The 2018 JMPR concluded that residues of ethiprole, ethiprole-sulfone and ethiprole-amide were stable in all of the five key categories of commodities (including soya beans) for at least 24 months under frozen storage conditions (at about -18 °C).

USE PATTERN

Ethiprole has been registered in several countries for use on crops including soya beans, rice, cotton, sugar cane and coffee. The current Meeting received GAP information on a registered use of ethiprole on soya bean in Brazil, as summarized below.

Table 3 Registered use of ethiprole on soya beans in Brazil

Crop	Country	Conc. g ai/L Form	Application					Min. PHI days
			Method	Rate kg ai/ha	Max No.	RTI days	Water L/ha	
Soya bean	BR	200 SC	Boom sprayer, Backpack sprayer	0.15-0.2	2 [a]	7	150-200	14

[a] A maximum of 2 applications per crop cycle on a 7-day interval.

Note: On seed crops, apply when 1 big stink bug per sampling is found. The highest dose should be used under conditions of increased infestation or where there is a history of pest presence.

RESULTS OF SUPERVISED RESIDUE TRIALS ON CROPS

The current Meeting received information on supervised trials using foliar sprays of ethiprole on soya beans. The results of these supervised trials are summarized in Table 4.

Group/Sub-group	Commodity	Country	Table No.
Pulses			
Dry beans	Soya bean (dry)	Brazil	Table 4

In addition to the descriptions and details of the field trials, each study report includes a summary of the analytical methods, together with the corresponding procedural recoveries, LOQ, LOD, and information on storage of samples. Durations of freezer storage between sampling and analysis were reported for all trials and were covered by the demonstrated storage stability in the storage stability studies.

In the trials, where multiple analyses were conducted on a single sample, the mean value is reported. Where multiple samples were taken from a single plot, the individual and mean values are reported. Where results from separate plots with distinguishing characteristics such as different varieties or treatment schedules were reported, results are listed for each plot.

When residues were not quantifiable, they are shown as below the LOQ of the relevant analytical method (e.g., < 0.002 mg/kg). Residues and application rates have generally been rounded to two significant figures or, for residues near the LOQ, to one significant figure.

Although control plots were included in the trials, control data are not reported in the following tables unless residues in control samples exceeded the LOQ. Results have not been corrected for concurrent method recoveries.

Residue values from the trials conducted according to the critical GAP that were used for the estimation of maximum residue levels, STMR and HR, are underlined in the tables.

For the calculation of the sum of ethiprole, ethiprole-sulfone and ethiprole-amide, expressed as parent equivalents (total residues), the Meeting followed the same approach as used by the 2018 JMPR: i.e., when the concentration of any analyte expressed as ethiprole was below the LOQ, it was regarded to be at the LOQ; and when the concentrations of all three analytes were below the LOQ, the total residue is expressed as below the sum of the LOQ values (<0.006 mg/kg).

Pulses

Soya bean (dry)

A total of eight field trials were conducted on soya beans in 2018–2019 in Brazil. Ethiprole (200 SC) was applied twice as foliar sprays with a retreatment interval of 6–7 days at a nominal application rate of 0.2 kg ai/ha.

In each trial, samples of mature soya bean seeds were collected 7, 14, 21 and 28 days after the second (last) application (DALA). At the time of the sample collection, soya bean seeds were at BBCH 89–97 growth stage, corresponding to fully ripe, dry and hard.

The residues of ethiprole, ethiprole-sulfone and ethiprole-amide were determined with Method 01128 (LC-MS/MS). The LOQs were 0.002 mg/kg for all these analytes. The concurrent recoveries for these analytes were within the acceptable range as shown in Table 2.

Soya bean seed samples were stored frozen from the day of sample collection for a maximum of 169 days (ca 5.6 months) in the studies. The storage stability studies indicated that these three analytes were stable at least 24 months under the frozen storage in soya beans.

Residue concentrations in Table 4 are expressed in ethiprole equivalents.

Table 4 Residues of ethiprole, ethiprole-sulfone and ethiprole-amide in soya bean (dry) from supervised trials in Brazil after foliar application of ethiprole (200 SC formulation)

Trial No, Location in Brazil, Year (Variety)	Application		DALA days	Residues, mg-ethiprole/kg (mean)				Reference Study/code
	kg ai/ha	No.		Ethiprole	E-sulfone	E-amide	Total	
GAP in Brazil	0.2	2	14					
I17-018-01, Jaboticabal, São Paulo 2018 (BMX Potência RR)	0.191 0.200	2	7 14 21 28	0.005 <0.002 <0.002 <0.002	<0.002 <0.002 <0.002 <0.002	<0.002 <0.002 <0.002 <0.002	0.009 <0.006 <0.006 <0.006	I17-018 / M-636713-01-1
I17-018-02, Trindade, Goiás 2018 (M 8210 IPRO RR)	0.190 0.206	2	7 14 21 28	<0.002 <0.002 <0.002 <0.002	<0.002 <0.002 <0.002 <0.002	<0.002 <0.002 <0.002 <0.002	<0.006 <0.006 <0.006 <0.006	I17-018 / M-636713-01-1
I17-018-03, Ponta Grossa, Paraná 2018 (BMX Potência RR)	0.190 0.202	2	7 14 21 28	0.032 0.028 0.012 0.009	0.005 0.008 0.003 <0.002	<0.002 <0.002 <0.002 <0.002	0.039 0.038 0.017 0.013	I17-018 / M-636713-01-1
I17-018-05, Paulínia, São Paulo 2018 (BMX Potência RR)	0.202 0.203	2	7 14 21 28	0.019 <0.002 <0.002 <0.002	0.002 <0.002 <0.002 <0.002	<0.002 <0.002 <0.002 <0.002	0.023 <0.006 <0.006 <0.006	I17-018 / M-636713-01-1
Ri18b-12/01/03, Restinga Seca, Rio Grande do Sul 2019 (NS 5445 IPRO)	0.207 0.209	2	7 14 21 28	0.11; 0.079 (0.095) 2x <0.002 (<0.002) 2x <0.002 (<0.002) 2x <0.002 (<0.002)	0.005; 0.004 (0.004) 2x <0.002 (<0.002) 2x <0.002 (<0.002) 2x <0.002 (<0.002)	2x <0.002 (<0.002) 2x <0.002 (<0.002) 2x <0.002 (<0.002) 2x <0.002 (<0.002)	0.10 <0.006 <0.006 <0.006	RAEH0068 / M-674212-01-1
Ri18b-12/01/04, São Pedro do Sul, Rio Grande do Sul 2019 (BMX GARRA IPRO)	0.209 0.209	2	7 14 21 28	0.0032; 0.002 (0.003) 0.0025; 0.0036 (0.003) 0.0030; 0.0043 (0.004) 0.0027; 0.0042 (0.003)	2x <0.002 (<0.002) 2x <0.002 (<0.002) 2x <0.002 (<0.002) 2x <0.002 (<0.002)	2x <0.002 (<0.002) 2x <0.002 (<0.002) 2x <0.002 (<0.002) 2x <0.002 (<0.002)	0.007 0.007 0.007, 0.008 (0.008) 0.007, 0.008 (0.007)	RAEH0068 / M-674212-01-1

Trial No, Location in Brazil, Year (Variety)	Application		DALA days	Residues, mg-ethiprole/kg (mean)				Reference Study/code
	kg ai/ha	No.		Ethiprole	E-sulfone	E-amide	Total	
GAP in Brazil	0.2	2	14					
Ri18b-12/01/05, Tamarana, Paraná 2019 (7063 (Intacta))	0.202 0.209	2	7 14 21 28	2x <0.002 (<0.002) 0.0024; 0.0028 (0.003) 0.0024; 0.0020 (0.002) 0.0023; 0.0024 (0.002)	2x <0.002 (<0.002) 2x <0.002 (<0.002) 2x <0.002 (<0.002) 2x <0.002 (<0.002)	0.0028; <0.002 (0.002) 2x <0.002 (<0.002) 2x <0.002 (<0.002) 2x <0.002 (<0.002)	<0.006 0.006, 0.007 (0.007) 0.006 0.006	RAEH0068 / M-674212-01-1
Ri18b-12/01/06, Luis Eduardo, Magalhães, Bahia 2019 (SYN 1687 IPRO)	0.199 0.215	2	7 14 21 28	0.13; 0.67 (0.40) <0.002; 0.012 (0.007) <0.002; 0.005 (0.004) 2x <0.002 (<0.002)	0.014; 0.084 (0.049) 0.002; 0.0031 (0.003) 2x <0.002 (<0.002) 2x <0.002 (<0.002)	2x <0.002 (<0.002) 2x <0.002 (<0.002) 2x <0.002 (<0.002) 2x <0.002 (<0.002)	0.45 0.006, 0.017 (0.012) 0.008 <0.006	RAEH0068 / M-674212-01-1

FATE OF RESIDUES IN STORAGE AND IN PROCESSING

Information and Data from Residues in Processed Commodities

The effects of processing on the concentrations of ethiprole residues were evaluated by the 2018 JMPR for rice and coffee beans.

The current Meeting received information on the processing of soya beans, relevant to the current evaluation. (Lenz and Fischer, 2016, M-565737-01-1)

Two field trials were performed in the USA in 2013 for obtaining the soya bean samples for a processing study. Two foliar applications were made at rates around 0.5 kg ai/ha (2.5 times the maximum GAP rate) with RTI of 7 days. The second application was conducted at the growth stage BBCH 80–81 (beginning of ripening), at 20–23 days before harvest.

Soya bean seeds were collected and dried in an oven until a moisture content of 13% was achieved. Seeds were aspirated using sieves. The material which passed through the 2360 µm sieve was collected as the aspirated grain fraction. For the production of hulls, meal, flour and solvent-extracted oil, whole soya beans were fed into a roller mill for hulling before the material was aspirated to separate the hull and kernel material. The kernel material was then heated to 71–79 °C and flaked in a flaking roll. A portion of the flakes were removed for direct solvent extraction and the remaining flakes were extruded, where they turned into collets by steam injection and compression. After extrusion, the collets were ground in a disc mill and dried.

Ground collets and flakes were extracted with hexane in batches at 49–60 °C. The crude oil and hexane extracts were combined. The solvent was removed from the extracted flakes with warm air before being ground to produce flour. Extracted collets were then toasted using steam injection and cooled before screening. Material passing through the screen (meal) was collected. The crude oil/hexane extracts were separated into fractions and the crude oil was neutralized and centrifuged. The refined oil was then filtered and bleached using activated bleaching earth (1.0% by weight of oil) under vacuum. The bleached oil was finally steam bathed under vacuum at 220–230 °C before the addition of 0.5% citric acid to generate the refined solvent-extracted oil and deodorizer distillates.

For the cold-pressed oil, cleaned soya bean samples were moisture-adjusted to 16% before pressing to separate the crude oil from the press cake (meal). The crude oil was processed into refined cold-pressed oil using the same procedure as described for refined solvent-extracted oil.

Whole cleaned soybeans used for soya milk production were washed and soaked in water for at least 12 hours before the soaked beans were ground and filtered to separate the milk from solids. The liquid fraction was heated to 91–97 °C for 9–11 minutes before the milk fraction was collected.

Samples of soya bean seeds, aspirated grain fractions, flour, hulls, meal, soya milk and refined oil (cold-pressed and solvent-extracted) were analyzed for residues of ethiprole, ethiprole-sulfone and ethiprole-amide by Method 01128. Samples were analyzed within 208 days (ca. 7 months) from sampling.

Residues of ethiprole, ethiprole-sulfone and ethiprole-amide in soya beans and the processed commodities are shown in Table 5. Processing factors were calculated from these results for ethiprole and the total residues and presented in Table 5.

Table 5 Residues of ethiprole in soya beans and their processed commodities and calculated processing factors

Trial No., Location, Year	Sample	Residues as ethiprole (mg/kg) [a]				Processing factor	
		Ethiprole	Ethiprole-sulfone	Ethiprole-amide	Total	Ethiprole	Total
EH010-13A Stewardson, IL, USA 2011	Seed (RAC)	0.047	0.006	<0.002	0.055	-	-
	Aspirated grain fractions [b]	35.5	6.54	0.054	42.1	755	765
	Flour	0.012	0.002	<0.002	0.016	0.26	0.29
	Hulls	0.331	0.049	<0.002	0.382	7.0	6.9
	Meal	0.010	<0.002	<0.002	0.014	0.21	0.25
	Soya milk	<0.002	<0.002	<0.002	<0.006	<0.04	<0.11
	Refined cold-pressed oil	<0.002	0.009	<0.002	0.013	<0.04	0.24
	Refined solvent-extracted oil	<0.002	0.002	<0.002	0.006	<0.04	0.11
EH011-13PA Atlantic, IA, USA 2013	Seed (RAC)	0.004	<0.002	<0.002	0.008	-	-
	Aspirated grain fractions [b]	1.6	1.0	0.006	2.57	400	323
	Flour	0.002	<0.002	<0.002	0.006	0.50	0.75
	Hulls	0.018	0.004	<0.002	0.025	4.5	3.1
	Meal	0.002	<0.002	<0.002	0.006	0.50	0.75
	Soya milk	<0.002	<0.002	<0.002	<0.006	<0.50	<0.75
	Refined cold-pressed oil each	<0.002	<0.002	<0.002	<0.006	<0.50	<0.75
	Refined solvent-extracted oil	<0.002	<0.002	<0.002	<0.006	<0.50	<0.75

[a] Mean value of triplicate analysis.

[b] Recoveries of each of these analytes were tested only at the fortification level of 100 mg/kg. (Recoveries of each analyte were tested at the fortification levels up to 2.0 mg/kg in soya beans.)

Table 6 Best estimates of processing factors of ethiprole and total ethiprole residues (soya beans to their processed products)

Commodity	Ethiprole		Total ethiprole residues	
	Individual processing factors	Best estimate [a]	Individual processing factors	Best estimate [a]
Seed (RAC)	(0.047, 0.004 mg/kg)	-	(0.055, 0.008 mg/kg)	-
Aspirated grain fractions	755, 400	755	765, 323	765
Flour	0.26, 0.50	0.26	0.29, 0.75	0.29
Hulls	7.0, 4.5	7.0	6.9, 3.1	6.9
Meal	0.21, 0.50	0.21	0.25, 0.75	0.25
Soya milk	<0.04, 0.50	<0.04	<0.11, <0.75	<0.11
Refined cold-pressed oil	<0.04, 0.50	<0.04	0.24, <0.75	0.24
Refined solvent-extracted oil	<0.04, 0.50	<0.04	0.11. <0.75	0.11

[a] Since the residue concentration in the second trial (EH011-13PA) was very low at 0.004 mg/kg (ethiprole), the Meeting selected processing factors calculated using the analytical results in the first trial (EH010-13A) as best estimates.

APPRAISAL

Ethiprole is a non-systemic phenylpyrazole insecticide. It was first evaluated by the 2018 JMPR for toxicology and residues. The 2018 JMPR established an ADI of 0–0.005 mg/kg bw, an ARfD of 0.005 mg/kg bw and recommended the following residue definitions:

Definition of the residue for compliance with the MRL for plant commodities: Ethiprole

Definition of the residue for dietary risk assessment for plant commodities: Sum of ethiprole, 5-amino-1-[2,6-dichloro-4-(trifluoromethyl)phenyl]-4-(ethylsulfinyl)-1H-pyrazole-3-carboxamide (ethiprole amide) and 5-amino-1-(2,6-dichloro-4-(trifluoromethylphenyl)-4-ethylsulfonylpyrazole-3-carbonitrile (ethiprole-sulfone), expressed as parent equivalents.

Definition of the residue for compliance with the MRL and dietary risk assessment for animal commodities: Sum of ethiprole and 5-amino-1-(2,6-dichloro-4-(trifluoromethylphenyl)-4-ethylsulfonylpyrazole-3-carbonitrile (ethiprole-sulfone), expressed as parent equivalents.

The residue is fat-soluble.

On the basis of the above residue definitions, the 2018 JMPR estimated maximum residue levels for rice, coffee and related commodities as well as commodities of animal origin.

The Forty-third Codex Alimentarius Commission (2020) approved the new work proposals including the priority list of pesticides for evaluation by the current Meeting. The priority list included ethiprole for evaluation of an additional use on soya beans. The present Meeting received new validation information on an analytical method, supervised residue trials and processing studies on soya beans.

Methods of analysis

The 2018 JMPR evaluated three LC-MS/MS methods for the determination of ethiprole, ethiprole-sulfone and ethiprole-amide in supervised trials, including Method 01128. The current Meeting received new information on the validation of Method 01128 used in the supervised trials on soya beans. The Method was validated with a LOQ of 0.002 mg/kg for the three analytes in soya beans and their processed commodities except aspirated grain fractions. In aspirated grain fractions, recoveries were only tested at a fortification level of 100 mg/kg for each analyte with the resulting procedural recoveries and relative standard deviations within the acceptable range.

Results of supervised residue trials on crops

The current Meeting received information on supervised trials using foliar spray of ethiprole conducted in support of estimating maximum residue level for soya bean (dry).

For the calculation of the sum of ethiprole, ethiprole-sulfone and ethiprole-amide, expressed as parent equivalents (total residues), the Meeting used the approach agreed at the 2018 JMPR. When the concentration of any analyte was below the LOQ, it was regarded to be at the LOQ; and when the concentrations of all three analytes were below the LOQ, the total residue is expressed as below the sum of the LOQ values (< 0.006 mg/kg).

Pulses

Soya bean (dry)

Critical GAP in Brazil for soya bean allows two foliar applications at a maximum rate of 0.2 kg ai/ha with a re-treatment interval of 7 days, and PHI of 14 days.

Eight field trials were conducted on soya beans in Brazil in 2018–2019. In the trials conducted in 2019, duplicate samples were taken at each DALA point.

Ethiprole residues from the trials matching the critical GAP were in rank order (n = 8): < 0.002 (4), 0.003, 0.004, 0.007 and 0.028 mg/kg.

The Meeting estimated a maximum residue level of 0.05 mg/kg for soya bean (dry).

Corresponding total residues from the trials were in rank order (n = 8): < 0.006 (4), 0.007, 0.008, 0.012 and 0.038 mg/kg.

The Meeting estimated an STMR of 0.0065 mg/kg for soya bean (dry).

Fate of residues during processing

Processing

The effects of processing on the concentrations of ethiprole residues were evaluated by the 2018 JMPR for rice and coffee beans.

The current Meeting received information on the processing of soya beans to processed commodities, relevant to the current evaluation.

The calculated processing factors for these commodities together with calculated STMR-Ps are summarized below. Since the residue concentrations in the second trial were close to the LOQ or lower than LOQ, the Meeting selected processing factors calculated using the first trial (first value for commodity) as best estimates.

Table 1 Processing Factors and STMR-P/HR-P

Commodity	Ethiprole		Total ethiprole residues		
	Individual processing factors	Best estimate	Individual processing factors	Best estimate	STMR/STMR-P
Seed (RAC)	(0.047, 0.004 mg/kg)	-	(0.055, 0.008 mg/kg)	-	0.0065
Flour	0.26, 0.50	0.26	0.29, 0.75	0.29	0.002
Soya milk	< 0.04, 0.50	< 0.04	< 0.11, < 0.75	< 0.11	0.0007
Refined oil		< 0.04		0.24	0.002
Cold-pressed	< 0.04, 0.50	< 0.04	0.24, < 0.75	0.24	0.002
Solvent-extracted	< 0.04, 0.50	< 0.04	0.11. < 0.75	0.11	0.0007
Meal	0.21, 0.50	0.21	0.25, 0.75	0.25	0.002
Aspirated grain fractions [a]	755, 400	755	765, 323	765	4.97
Hulls	7.0, 4.5	7.0	6.9, 3.1	6.9	0.045

[a] Recoveries were only validated for the three analytes at a fortification level of 100 mg/kg in aspirated grain fractions, while the concentrations of the analytes were 0.054–35.5 mg eq/kg.

Based on the maximum residue level of 0.05 mg/kg for soya bean (dry) and the processing factor of 7.0 for ethiprole in soya bean hulls, the Meeting estimated a maximum residue level of 0.4 mg/kg for soya bean hulls.

Residues in animal commodities

The 2018 JMPR calculated the maximum and mean dietary burdens using the STMR/STMR-Ps estimated for rice bran, rice grain and rice hulls on the basis of the OECD Animal Feeding Table. The current Meeting evaluated the supervised residue trials and processing studies on soya beans. Among those commodities for which data were available, soya beans, soya aspirated grain fractions, soya bean meal, soya bean hulls may be fed to livestock. The newly calculated dietary burdens showed minor increase of 0.01 ppm which corresponded to a less than 1% increase compared to the previously calculated highest dietary burdens for cattle and poultry. The current Meeting concluded that it was unnecessary to review the recommendations for animal commodities made by the 2018 JMPR.

RECOMMENDATIONS

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

Definition of the residue for compliance with the MRL for plant commodities: *Ethiprole*

Definition of the residue for dietary risk assessment for plant commodities: *Sum of ethiprole, 5-amino-1-[2,6-dichloro-4-(trifluoromethyl)phenyl]-4-(ethylsulfinyl)-1H-pyrazole-3-carboxamide (ethiprole-amide) and 5-amino-1-(2,6-dichloro-4-(trifluoromethylphenyl)-4-ethylsulfonylpyrazole-3-carbonitrile (ethiprole-sulfone), expressed as parent equivalents.*

Definition of the residue for compliance with the MRL and dietary risk assessment for animal commodities: *Sum of ethiprole and 5-amino-1-(2,6-dichloro-4-(trifluoromethylphenyl)-4-ethylsulfonylpyrazole-3-carbonitrile (ethiprole-sulfone), expressed as parent equivalents.*

The residue is fat-soluble.

Table 2 Recommendations for residues of ethiprole from the 2021 Extra JMPR

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)	0.05	-	0.0065	-
AB 0541	Soya bean hulls	0.4		Median: 0.045	-
OR 0541	Soya bean oil, refined			0.002	-
	Soya bean flour			0.002	
	Soya milk			0.0007	-
AB 1265	Soya bean meal			Median: 0.002	-
	Aspirated grain fractions			Median: 4.97	-

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for ethiprole is 0–0.005 mg/kg bw. The International Daily Intakes (IEDIs) for ethiprole were estimated for the 17 GEMS/Food Consumption Cluster Diets using the STMR and STMR-P values estimated by JMPR. The results are shown in Annex 3 of the 2021 Extra JMPR Report.

The IEDIs ranged from 1–6% of the maximum ADI. The Meeting concluded that long-term dietary exposure to residues of ethiprole from uses considered by JMPR is unlikely to present a public health concern.

Acute dietary exposure

The ARfD for ethiprole is 0.005 mg/kg bw. The international Estimate of Short-Term Intakes (IESTIs) for ethiprole were calculated for soya bean (dry) and the processed commodities for which STMR/STMR-Ps were estimated by the present Meeting and for which consumption data were available. The results are shown in Annex 4 of the 2021 Extra JMPR report.

The IESTIs varied from 0–2% and 0–1% of the ARfD for children and general population, respectively. The Meeting concluded that acute dietary exposure to residues of ethiprole from uses considered by the present Meeting is unlikely to present a public health concern.

REFERENCES

Reference No.	Author(s)	Year	Title, Date, etc.
-	FAO/WHO	2019	Pesticide residues in food 2018 EVALUATIONS 2018 PART 1 - RESIDUES Joint FAO/WHO Meeting on Pesticide Residues (FAO PLANT PRODUCTION AND PROTECTION PAPER 235), pp. 139-251
M-455162-01-2	Santiago, L	2013	Validation Study of the Analysis Methodology for Residues of Ethiprole and its metabolites RPA 097973 and RPA 112916 in various matrices; Report No.: VM12-007 5 April 2013 GLP, unpublished

Reference No.	Author(s)	Year	Title, Date, etc.
M-565737-01-1	Lenz C. and Fischer D. R.	2016	Ethiprole + Imidacloprid SC 200 (100+100 g/L) – Magnitude of the Residue in/on Soya bean Processed Commodities; Report No.: RAEHL016 18 August 2016 GLP, unpublished
M-636713-01-1	Alves F. M.	2018	Determination of the residues of ethiprole in/on Soya bean (seed) after Spraying of ETHIPROLE SC 200A G in the Field in Brazil; Report No.: I17-018 28 September 2018 GLP, unpublished
M-674212-01-1	Abdal M. F.	2019	Magnitude of the residues of Ethiprole in/on soya bean crop (seeds) after treatment with Curbix 200 SC in Brazil; Report No.: RAEH0068 19 November 2019 GLP, unpublished

FENBUCONAZOLE (197)

First draft by Dr Hidetaka Kobayashi Ministry of Agriculture, Forestry and Fisheries, Japan

EXPLANATION

Fenbuconazole, whose IUPAC name is 4-(4-chlorophenyl)-2-phenyl- 2-(1*H*-1,2,4-triazol-1-ylmethyl) butyronitrile, is a triazole fungicide intended for agricultural and horticultural use for the control of a variety of fungal infections of crops.

Fenbuconazole was first evaluated by the 1997 JMPR and an ADI of 0–0.03 mg/kg bw was established. The residue definition is *fenbuconazole*, both for compliance with the MRL and dietary risk assessment for plant and animal commodities.

The residue is not fat-soluble.

The 2012 JMPR established an ARfD of 0.2 mg/kg bw. The compound was also evaluated by the JMPR in 2009 and 2013 for additional uses.

At the Fifty-first Session of CCPR (2019), fenbuconazole was scheduled for evaluation of an additional use on tea by the 2020 JMPR. The current Meeting received new information on use patterns, an additional analytical method, supervised field trials and a processing study on tea.

RESIDUE ANALYSIS

Analytical methods

The Meeting received information on new methods of analysis for fenbuconazole in tea (leaves and infusion: filtrate of soaking tea leaves in boiling water) as used in the supervised field trials as well as for enforcement.

Methods for supervised trials

LC-MS/MS Method (JP2016C059, JP2017C340)

Extract of tea leaves (dry) was prepared as follows. Five grams of tea leaves were pulverized using a blender and soaked in 20 mL of water (room temperature). It was allowed to stand still for 15 minutes, to which 50 mL of acetonitrile was added and homogenized for 3 minutes. It was filtered using Celite as filtration aid. Filter cake was washed three times with acetonitrile and all filtrates were combined.

Tea infusion was prepared by soaking 9 g of tea leaves in 540 mL of boiling water, standing still for 5 minutes and filtered through a cotton plug.

The extract of tea leaves or tea infusion was cleaned up by using a C_{18} mini-column and a graphite carbon/ethylene-diamine-N-propyl silylated silica gel (GC/PSA) mini-column.

The analyte was quantified by LC-MS/MS with the following conditions: column was C_{18} reversed phase (diameter 2 mm x 75 mm, particle size 3 µm); mobile phase was acetonitrile-water (3:7 -> 9:1, v/v) containing 0.1% (v/v) of formic acid; ionization method was ESI positive; and monitoring ions were m/z 337.1 → 125.0. The reported LOD and LOQ were 0.005 mg/kg and 0.01 mg/kg, respectively. The mean recoveries of fenbuconazole in tea leaves and tea infusion were in the range of 89–104% (Table 1). The calibration curve was linear (R^2>0.99) between 0.5–10 mg/kg.

Table 1 Recovery of fenbuconazole in tea (LC-MS/MS method, JP2017C340)

Matrix	analyte	Fortification level (mg/kg)	n	Recovery range (%)	Recovery, mean (%)	RSD (%)
Tea leaves	Fenbuconazole	0.01	6	89-98	92	4.6
		0.1	6	88-91	89	1.4
		20	6	90-97	93	3.3
Tea infusion	Fenbuconazole	0.01	6	96-103	102	2.7
		0.1	6	87-100	95	5.4
		5	6	101-108	104	2.4

GC-Method (7P-5-97)

Pulverized tea leaf sample (5 g) was extracted with 20 mL of water (room temperature) for 2 hours and then 100 mL of methanol was added. The flask was vigorously shaken for 30 minutes and filtered.

Tea infusion was prepared by soaking 5 g of tea leaves in 300 mL of boiling water, standing still for 5 minutes and filtered through a cotton plug.

The filtrate from tea leaf extract or filtered tea infusion was extracted with dichloromethane, cleaned up using silica gel column chromatography and Florisil column chromatography. The solution was analysed by GC with nitrogen-phosphorus detector (NPD) using DB-17 column. The reported LOD was 0.02 mg/kg (LOQ was not reported). The recovery was tested only at the fortification level of 0.8 mg/kg of fenbuconazole in tea leaves and the mean recovery was 96% (Table 2). The calibration curve of standard solution (0.01–0.4 mg/kg) was linear (R^2>0.99).

No validation study was available for this method on tea leaves.

Table 2 Recovery of fenbuconazole in tea (GC-Method, 7P-5-97)

Matrix	analyte	Fortification level (mg/kg)	n	Recovery range (%)	Recovery, mean (%)	RSD (%)
Tea leaves	Fenbuconazole	0.8	2	95, 97	96	-

Method for enforcement (DAS150556)

A QuEChERS method was available for enforcement. Samples were extracted by agitation with acetonitrile after addition of water (acetonitrile:water=2:1, v/v) in presence of magnesium sulphate, sodium chloride, sodium citrate dibasic sesquihydrate and sodium citrate tribasic dihydrate. After centrifugation, an aliquot of the acetonitrile layer was purified with a mixture of primary/secondary amine (PSA), silylated-C_{18} and magnesium sulphate. After centrifugation, an aliquot was diluted in water and analysed with LC-MS/MS (m/z 337.0 → 70.1 for quantification and m/z 337.5 → 125.0 for confirmation). The mean recoveries of fenbuconazole in apple (high water content), orange (high acid content), olive (high oil content) and wheat grain (dry/high starch content) were in the range of 88–102% (Table 3). The LOD and LOQ were reported to be 0.003 mg/kg and 0.01 mg/kg, respectively. The accepted calibration range (R^2>0.99) was 0.0025–0.5 mg/kg.

Table 3 Recovery of fenbuconazole analyzed with the method for enforcement

Matrix	Fortification level (mg/kg)	n	Quantification (m/z 337.0 → 70.1)		Confirmation (m/z 337.5 → 125.0)	
			Recovery range (mean) (%)	RSD (%)	Recovery range (mean) (%)	RSD (%)
Apple	0.01	5	86-90 (88)	1.7	88-90 (89)	1.3
(high water content)	0.1	5	98-103 (101)	2.0	98-105 (102)	2.5
Orange	0.01	5	84-91 (89)	3.3	84-96 (90)	5.2
(high acid content)	0.1	5	87-94 (90)	3.2	88-95 (91)	2.8
Olive	0.01	5	96-106 (98)	2.7	88-95 (91)	3.1
(high oil content)	0.1	5	97-106 (102)	3.5	95-100 (98)	2.6
Wheat grain	0.01	5	94-100 (97)	2.4	95-102 (98)	2.6
(dry/high starch content)	0.1	5	91-99 (95)	3.4	91-99 (95)	4.2

Stability of pesticide residues in stored analytical samples

In one study, different samples of pulverized tea leaves were fortified with fenbuconazole at a concentration of 0.5 mg/kg and then stored frozen at ≤-20 °C for intervals up to 98 days. All samples were analysed on the same day (at the end of the storage periods). Samples were analysed using LC-MS/MS method with an LOQ of 0.01 mg/kg.

In the earlier study, pulverized tea leaves were fortified with fenbuconazole at a concentration of 2.0 or 2.5 mg/kg and then stored frozen at ≤-20 °C for 78 and 104 days. Samples were analyzed using the GC method.

Fenbuconazole was stable for at least 3 months.

Table 4 Storage stability of fenbuconazole in tea leaves

Fortification (mg/kg)	Storage period (day)	Residue analytical value (mean) (mg/kg)	Residues remaining (%)	Concurrent recovery (%)	Analytical method
0.5	19	0.45, 0.45 (0.45)	90	86	LC-MS/MS
0.5	35	0.43, 0.44 (0.44)	87		
0.5	47	0.44, 0.46 (0.45)	90		
0.5	83	0.42, 0.43 (0.42)	86		
0.5	91	0.45, 0.47 (0.46)	92		
0.5	98	0.42, 0.44 (0.43)	86		
2	78	1.6, 1.6, 1.7, 1.7, 1.8, 1.9 (1.7)	90	98	GC
2.5	104	1.9, 2.0, 2.0, 2.1, 2.1, 2.2 (2.0)	85	92	

USE PATTERN

The Meeting received a label for tea in Japan as shown in Table 5. The label provided covers a broader spectrum of uses.

Table 5 Representative use pattern of fenbuconazole on tea

Crop	Country	Concentration Formulation	Application					PHI (days)
			Dilution	g ai/hL	Application volume	Type	Max no	
Tea	Japan	220 g ai/L SC	×5000	4.4 g ai/hL	2000-4000 L/ha [a]	Foliar	See footnote [b]	7

[a] Recommended

[b] Maximum 2 applications before first pick and maximum 2 applications between picks (e.g. maximum 2 applications before first pick, up to a total of 4 (2+2) before second pick, 6 (2+2+2) before third pick etc.). Retreatment interval between the two applications is not specified on the label, but an interval of 7 days is common in practice in Japan.

RESULTS OF SUPERVISED RESIDUE TRIALS ON CROPS

Residue levels were reported as measured. When residues were not detected they are shown as below the LOQ, e.g., <0.01 mg/kg. Application rates, concentrations and mean residue results have been rounded to two significant figures.

Laboratory reports included method validation. Dates of analyses or duration of residue sample storage were provided. Field reports provided data on the sprayers used and their calibration, plot size, residue sample size and sampling date.

Tea, green, black (black, fermented and dried)

Six residue trials were conducted on tea in Japan between 1995 and 2017 (7P-5-97, JP2016C059 and JP2017C340). In three trials, tea received two foliar applications of fenbuconazole (220 g/L SC) at 4.4 g ai/hL (dilution rate of ×5000) with an interval of 6–8 days and was harvested 7–21 days after last treatment (DALT). In three other trials, foliar sprays of fenbuconazole (220 g/L SC) at 4.4 g ai/hL (dilution rate of x5000) were applied: twice at 13–15 days and 7 days before the first picking and twice between first and second picks (13–15 days and 7 days before the second picks).

Fresh tea leaves harvested (2.2 kg) were steamed for 35 seconds by using a belt conveyer type steam heating machine and then dried for 120 minutes at 80 °C for preparation of tea leaves (approximately 0.4 kg) to be analyzed in the supervised trials.

The residues of fenbuconazole were analyzed using a GC-method (7P-5-97, trials conducted in 1995) with an LOD of 0.02 mg/kg or LC-MS/MS method (JP2016C059 and JP2017C340, trials conducted in 2016 and 2017) with an LOQ of 0.01 mg/kg. Analyses were completed within 104 days from sampling, except in one trial conducted in Oita in 1995 (109 days). The residues of fenbuconazole in the trials harvested 7 DALT were 2.1–14 mg/kg (Table 6).

Table 6 Residues of fenbuconazole in tea leaves

Tea Location, year (Variety)	Application No/	Concentration (g ai/hL)	Water volume (L)	RTI (days)	pick	DALT (days)	Residue (mg/kg)	Report no.
GAP in Japan	Footnote [a]	4.4	2 x 2000-4000			7		
Kyoto, Japan [b] 1995	2	4.4	 2000	7	1st pick	7 14	2.1 1.7	7P-5-97

Tea Location, year (Variety)	Application No/	Concentration (g ai/hL)	Water volume (L)	RTI (days)	pick	DALT (days)	Residue (mg/kg)	Report no.
(Kyoken No. 129)			2000			21	0.76	
Oita, Japan [b] 1995 (Yabukita)	2	4.4	 2000 2000	7	1st pick	7 13 21	3.5 1.7 1.1	7P-5-97
Kagoshima, Japan 2016 (Yamato-midori)	2	4.4	 3850 3850	7	1st pick	7	2.4	JP2016C059
Ibaraki, Japan 2017 (Yabukita)	2	4.4	 3130 3130	8	1st pick	7	4.2	JP2017C340
	4	4.4	 3130 3130 3110 3110	8 55 8	2nd pick	7	11	
Kochi, Japan 2017 (Yabukita)	2	4.4	 3780 3780	6	1st pick	7	4.0	JP2017C340
	4	4.4	 3780 3780 3800 3800	6 51 8	2nd pick	7	3.5	
Miyazaki, Japan 2017 (Yabukita)	2	4.4	 3410 3410	7	1st pick	7	14	JP2017C340
	4	4.4	 3410 3410 3410 3410	7 56 6	2nd pick	7	4.5	

[a] Two applications before the first pick. For second and following picks, two additional applications between picks are allowed (e.g. 2 applications for the first pick, 4 for the second pick, 6 for the third pick etc.)

[b] Samples were analyzed by a method that was not validated according to the OECD guidance document.

FATE OF RESIDUES IN STORAGE AND PROCESSING

Tea infusion was prepared from tea leaves as follows: 5 g (trials in 1995) or 9 g (other trials) of tea leaves were soaked in 300 mL (trials in 1995) or 540 mL (other trials) of boiling water for 5 minutes and filtered

through a cotton plug. Tea infusion was analyzed with the GC method (trials conducted in 1995; not validated method) or the LC-MS/MS method with a LOQ of 0.01 mg/kg (trials conducted in 2016 and 2017).

The processing factors ranged from 0.0035 to < 0.01 with a best estimate (median) of 0.0044 (Tables 7 and 8).

Table 7 Processing factor of fenbuconazole from dried tea leaves to infusion

Tea	Application	picking	Tea preparation		Residue (mg/kg)		Processing factor	Report no.
Location, year (Variety)	water volume L		Tea leaves (g)	Boiling water (mL)	Tea leaves (mg/kg)	Infusion (mg/L)		
Kyoto, Japan 1995 (Kyoken No. 129)	2000 2000	1st pick	5	300	2.1	< 0.02 [a]	<0.01	7P-5-97
Oita, Japan 1995 (Yabukita)	2000 2000	1st pick	5	300	3.5	< 0.02 [a]	<0.006	7P-5-97
Kagoshima, Japan 2016 (Yamatomidori)	3850 3850	1st pick	9	540	2.4	< 0.01	<0.005	JP2016C059
Ibaraki, Japan 2017 (yabukita)	3130 3130	1st pick	9	540	4.2	0.020	0.0046	JP2017C340
	3130 3130 3110 3110	2nd pick	9	540	11	0.052	0.0046	JP2017C340
Kochi, Japan 2017 (Yabukita)	3780 3780	1st pick	9	540	4.0	0.014	0.0035	JP2017C340
	3780 3780 3800 3800	2nd pick	9	540	3.5	0.013	0.0037	JP2017C340
Miyazaki, Japan 2017 (Yabukita)	3410 3410	1st pick	9	540	14	0.060	0.0044	JP2017C340
	3410 3410 3410 3410	2nd pick	9	540	4.5	0.019	0.0041	JP2017C340

[a] Analyses were conducted with a method that was not validated.

Table 8 Processing factor from tea leaves to tea infusion (Summary)

RAC	Processed commodity	Calculated processing factors [a]	PF (mean or best estimate)
Tea leaves	Tea infusion	0.0035, 0.0037, 0.0041, 0.0044, 0.0046 (2), <0.005	0.0044

[a] Each value represents a separate trial. Data from trials in which the analytical method was not validated was excluded from the table. The factor is the ratio of the residue in processed commodity divided by the residue in the RAC.

APPRAISAL

Fenbuconazole, whose IUPAC name is 4-(4-chlorophenyl)-2-phenyl- 2-(1*H*-1,2,4-triazol-1-ylmethyl) butyronitrile, is a triazole fungicide intended for agricultural and horticultural use for the control of a variety of fungal infections of crops.

Fenbuconazole was first evaluated by the 1997 JMPR and an ADI of 0–0.03 mg/kg bw was established. The residue definition is fenbuconazole, both for compliance with the MRL and dietary risk assessment for plant and animal commodities. The residue is not fat-soluble.

The 2012 JMPR established an ARfD of 0.2 mg/kg bw. The compound was also evaluated by the JMPR in 2009 and 2013 for additional uses.

At the Fifty-first Session of CCPR (2019), fenbuconazole was scheduled for evaluation of an additional use on tea by the 2020 JMPR, which was postponed to the 2021 Extra JMPR. The current Meeting received new information on use patterns, an additional analytical method, supervised field trials and a processing study on tea.

Methods of analysis

The Meeting received information on a new analytical method for fenbuconazole.

In the new method based on LC-MS/MS, fenbuconazole was extracted from dry tea leaves with acetonitrile and cleaned-up before analysis. Tea infusion (filtrate of soaking tea leaves in boiling water) was directly analysed by the same LC-MS/MS method. Recovery data support an LOQ of 0.01 mg/kg. The Meeting agreed that the method is suitable for analysing fenbuconazole in dried tea leaves and tea infusion.

Stability of pesticide residues in stored analytical samples

Stability studies of fenbuconazole in tea leaves (dry) were available. The Meeting concluded that fenbuconazole in tea leaves stored at ≤-20 °C was stable for at least 3 months. All the field trial residue samples were analysed within this period.

Results of supervised residue trials on crops

Tea, green, black (black, fermented and dried)

The critical GAP for tea in Japan is two applications before the first pick and two applications between subsequent picks at 4.4 g ai/hL with a PHI of 7 days. Re-treatment interval between the two applications is

not specified on the label. However, the Meeting noted that an interval of 7 days was common in practice in Japan.

In trials matching the GAP, residues of fenbuconazole in tea leaves from the first pick were (n = 4): 2.4, 4.0, 4.2, and 14 mg/kg.

For the second pick, following two further applications, residues were (n = 3): 3.5, 4.5 and 11 mg/kg after 7 DALA.

The Meeting considered that residues after the first pick and before the second set of sprays would not contribute significantly to the final residue taking into account growth dilution during the interval of at least 50 days between the second treatment (before the first pick) and the third treatment (after the first pick).

The Meeting agreed that the trials were sufficiently independent even though the samples were taken at the first pick and 2nd pick from the same plot and decided to use the residue data from both picks for estimation of maximum residue levels. The combined data set of residues of fenbuconazole in tea leaves (from both first and second picks) were (n = 7): 2.4, 3.5, 4.0, 4.2, 4.5, 11 and 14 mg/kg.

The Meeting estimated a maximum residue level and STMR value of 30 mg/kg and 4.2 mg/kg, respectively, for fenbuconazole in tea, green, black (black, fermented and dried).

Fates of residues during processing

The Meeting received information on the fate of fenbuconazole residues when tea infusion was produced from tea leaves. An estimated processing factor and the derived STMR-P are summarized in the table below.

Table 1 Processing Factors and STMR-P/HR-P

RAC	Processed commodity	Calculated processing factors a	PF (mean or best estimate)	RAC STMR (mg/kg)	STMR-P (mg/kg)
Tea leaves	Tea infusion	0.0035, 0.0037, 0.0041, 0.0044, 0.0046, 0.0046, < 0.005	0.0044	4.2	0.0018

[a] Each value represents a separate trial. The factor is the ratio of the residue in processed commodity divided by the residue in the RAC.

RECOMMENDATIONS

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

Definition of the residue for compliance with the MRL and dietary risk assessment for plant and animal commodities: fenbuconazole.

The residue is not fat-soluble.

Table 2 Recommendations for residues of fenbuconazole from the 2021 Extra JMPR

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
DT1114	Tea, green, black (black, fermented and dried)	30		4.2	
	Tea infusion			0.0018	

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for fenbuconazole is 0–0.03 mg/kg bw. The International Estimated Daily Intakes (IEDIs) for fenbuconazole were estimated for the 17 GEMS/Food Consumption Cluster Diets using the STMR or STMR-P values estimated by the JMPR. The results are shown in Annex 3 of the 2021 Extra JMPR Report.

The IEDIs ranged from 0–3% of the maximum ADI for fenbuconazole. The Meeting concluded that long-term dietary exposure to residues of fenbuconazole from uses considered by the JMPR is unlikely to present a public health concern.

Acute dietary exposure

The ARfD for fenbuconazole is 0.2 mg/kg bw. The International Estimate of Short-Term Intakes (IESTIs) for fenbuconazole were calculated for tea leaves and infusion for which STMRs/STMR-Ps were estimated by the present Meeting and for which consumption data were available. The results are shown in Annex 4 of the 2021 Extra JMPR Report.

The IESTIs from tea varied from 2–3% of the ARfD for children and 0–3% of the ARfD for the general population. The Meeting concluded that acute dietary exposure to residues of fenbuconazole from uses considered by the present Meeting is unlikely to present a public health concern.

REFERENCES

Code	Author(s)	Year	Title
DAS 150556	Dorange, J.B.	2016	Fenbuconazole – Validation of the Analytical Method for the Determination of Fenbuconazole in Different Matrices of Plant, Dow AgroSciences Method 150556 GLP, Unpublished 09 December 2016.
7P-5-97	Kazuhiro Komatsu	1995	Crop Residue Analysis Report Fenbuconazole Residues in Processed Tea Leaves and Tea Infusion Japan Food Research Laboratories Document ID: Crop 7P-5-97 Non-GLP, Unpublished September 25, 1995
JP2016C059	Koji Nakamura	2016	Residue analysis study of fenbuconazole (Indar) SC in tea (General Incorporated Association) Japan Plant Protection Association Study number: JP2016C059 GLP, Unpublished October 31, 2016
JP2017C340	Koji Nakamura	2017	Residue analysis study of fenbuconazole (Indar) SC in tea (General Incorporated Association) Japan Plant Protection Association Study number: JP2017C340 GLP, Unpublished October 20, 2017

FENHEXAMID (215)

The first draft was prepared by Dr Chris Anagnostopoulos, Benaki Phytopathological Institute, Greece

EXPLANATION

Fenhexamid is a hydroxyaniline protective fungicide whose mode of action is by the inhibition of germ tube elongation, mycelial growth and spore germination. Fenhexamid was evaluated for the first time by the 2005 JMPR where an ADI of 0–0.2 mg/kg bw was established and it was concluded that an ARfD was unnecessary. The 2005 JMPR also concluded that the residue definition for plant and animal commodities for compliance with the MRL and dietary risk assessment was parent *fenhexamid*.

Fenhexamid was scheduled by the Fifty-first Session of the CCPR for the evaluation of additional uses by the 2020 JMPR, which was postponed to the 2021 Extra JMPR. The current Meeting received new information on analytical methods, storage stability and on the use patterns for fenhexamid in pear, ginseng, asparagus, spring onion and bulb vegetables supported by supervised field trials.

RESIDUE ANALYSIS

Analytical methods

Analytical methods for the determination of fenhexamid in pear, ginseng, asparagus and onion (green and dry) with an LOQ of 0.02 mg/kg were provided to the current meeting. Validation of the analytical methods used in the field trials was performed as part of the study and a brief description is provided below.

Method:	Method codes were not available.				
Reference:	Cornell Analytical Laboratories method "Residue Analysis of Fenhexamid on Pear using LC with MS Detection"	Cornell Analytical Laboratories method "Residue Analysis of Fenhexamid on Ginseng using LC with MS Detection"	Cornell Analytical Laboratories method "Residue Analysis of Fenhexamid on Asparagus using LC with MS Detection"	Working method for PR 07149: Analysis of fenhexamid in onion.	
Commodity:	Pears	Ginseng	Asparagus	Green onion	Bulb onion
Analyte:	Fenhexamid	Fenhexamid	Fenhexamid	Fenhexamid	Fenhexamid
Determination:	LC-MS	LC-MS	LC-MS	LC-MS	LC-MS
LOQ:	0.02 mg/kg	0.02 mg/kg	0.02 mg/kg	0.02 mg/kg	0.02 mg/kg
Accuracy:	102% (0.02 mg/kg, n=3) 97% (0.5 mg/kg, n=4) 93% (2 mg/kg, n=22) 100% (7 mg/kg, n=3)	84% (0.02 mg/kg, n=13) 53% (0.05 mg/kg, n=12) 67.5% (0.2 mg/kg, n=6)	98 % (0.02 mg/kg, n=6) 95% (0.2 mg/kg, n=4)	77-116% 99 (0.02 mg/kg, n=7) 80 (0.5 mg/kg, n=7) 82 (5 mg/kg, n=6) 92 (20 mg/kg, n=4)	79-113% 93 (0.02 mg/kg, n=8) 84 (0.5 mg/kg, n=8) 89 (5 mg/kg, n=7)

Method:	Method codes were not available.				
Reference:	Cornell Analytical Laboratories method "Residue Analysis of Fenhexamid on Pear using LC with MS Detection"	Cornell Analytical Laboratories method "Residue Analysis of Fenhexamid on Ginseng using LC with MS Detection"	Cornell Analytical Laboratories method "Residue Analysis of Fenhexamid on Asparagus using LC with MS Detection"	Working method for PR 07149: Analysis of fenhexamid in onion.	
Repeatability:	3% (0.02 mg/kg) 6% (0.5 mg/kg) 6% (2 mg/kg) 6% (7 mg/kg)	14% (0.02 mg/kg) 8.5% (0.05 mg/kg) 11.3% (0.2 mg/kg)	4% (0.02 mg/kg) 13% (0.2 mg/kg)	11% (0.02 mg/kg) 4% (0.5 mg/kg) 3% (5 mg/kg) 7% (20 mg/kg)	10% (0.02 mg/kg) 4% (0.5 mg/kg) 4% (5 mg/kg)
Specificity:	3 ions	3 ions	3 ions	3 ions	3 ions
Description:	25 grams of sample is blended with acetone and filtered. After evaporation of the acetone the aqueous residue is cleaned up on a ChemElute column and eluted with cyclohexane/ethyl acetate 85/15. Extract is then evaporated to dryness, taken up in anhydrous methanol and analyzed with LC-MS.				

Stability of residues in stored analytical samples

The current Meeting received residue stability data for pear, asparagus, ginseng and green onions spiked at 0.2 mg/kg, and 2 mg/kg, stored concurrently with the residue trial samples. The stability of fenhexamid residues in stored samples of pears, asparagus and green onions was demonstrated for at least 232, 217 and 532 days. respectively. For ginseng storage stability data up to 133 days were available but recoveries were found to be 60% at 133 days, thus stability was not demonstrated. In addition, residue levels at time 0 were not reported. The value of 0.2 mg/kg is the theoretical spiked concentration and was not confirmed by an analysis. For bulb onions storage stability data were not available.

Table 1 Storage stability of fenhexamid in pear, ginseng, asparagus and green onion stored below -18 °C

Commodity	FL (mg/kg)	Storage interval (month)	% Fresh fortification recovery (mean)	% Remaining residue in stored sample (mean)	Report No. Reference
pear	2	0 230-231 (7.5)	- 90; 90; 85; 100; 95 (91)	- 70; 70; 70; 65; 80; 80 (72.5)	IR-4 Study No. 07402
ginseng	0.2	0 133 (4.3)	- 60; 65 (62.5)	- 60; 60 (60)	PR.No 07846
asparagus	0.2	0 217 (7)	- 90	- 80;90;80 (83,3)	PR.No 08692
green onion	1	0 532 (17.5)	- -	- 73; 76; 70 (73)	IR-4 Study No. 07149

The maximum storage periods for all samples from field trials included in this evaluation are given in Table 2.

Table 2 Maximum sample storage intervals

Study No.	Targets	Sample material	Storage period

			Days	Months
IR-4 Study No. 07402	pears	fruit	245	8
PR.No 07846	ginseng	Root (dried mature)	179	6
PR Study No. 08692	asparagus	spears	228	7.5
IR-4 Study No. 07149	green onion	green onion	487	16
IR-4 Study No. 07149	bulb onion	bulb onion	483	16

USE PATTERN

A fenhexamid label from the USA was made available to the Meeting. Relevant uses are summarized in Table 3.

Table 3 Registered uses of fenhexamid relevant to the evaluation

Crop	Country	Site	Formulation		Application						PHI (days)	Remarks
			Active ingredient content (%)	Type	Method	No.	Rate (g ai/ha)	Timing	Interval (days)	Seasonal max. (g ai/ha)		
Pears	USA	I	50	WG	Low-Volume Packing- line spray	1	0.23 - 0.34 [a]				0	Post-harvest
					High-Volume Packing-line spray	1	0.23 - 0.34[a]				0	
					Fruit dip treatment	1	0.23 - 0.34 [b]				0	
Ginseng	USA	F	50	WG	Groundboom, airblast, high and low pressure handheld, aerial	4	840	not reported	7-14	3.360	0	
Asparagus	USA	F	50	WG	Groundboom, airblast, high and low pressure handheld, aerial	4	840	at fern stage only	7-14	3.360	180	
Green onion	USA	F	50	WG	Groundboom, airblast, high and low pressure handheld, aerial	4	840	not reported	7	3.360	0	

Crop	Country	Site	Formulation		Application						PHI (days)	Remarks
			Active ingredient content (%)	Type	Method	No.	Rate (g ai/ha)	Timing	Interval (days)	Seasonal max. (g ai/ha)		
Subgroup of bulb onions	USA	F	50	WG	Groundboom, airblast, high and low pressure handheld, aerial	4	840	not reported	7	3.360	0	

[a] Post-harvest low volume spray: 0.23–0.34 kg a.i. per 90.7 tn. fruit in 378.5 L of an appropriate water, wax/oil emulsion, or aqueous dilution of a wax/oil emulsion (equiv. to 0.09 kg ai/hL or 3.7 g ai/1 tn. fruit).

[b] Post-harvest low volume spray: 0.23–0.34 kg a.i. per 90.7 tn. fruit in 378.5 L of an appropriate water, wax/oil emulsion, or aqueous dilution of a wax/oil emulsion (equiv. to 0.09 kg ai/hL or 3.7 g ai/1 tn. fruit). Fruit must remain in dip solution for 20 to 30 seconds.

RESULTS OF SUPERVISED RESIDUE TRIALS ON CROPS

Pear

Five (5) post-harvest residue trials with fenhexamid on pears were conducted in the USA during 2000.

In the submitted trials, fenhexamid was applied post-harvest as a WDG formulation once at approximately 0.09 kg ai/hL water (drench or dip) or per 90.7 tn fruit (packing line spray). In two trials (OO-CA51 and 00-WA32) pears were treated on a packing line spray or with a combination of a drench and packing line spray. Fruit wax was added to the solution for the dip and packing line spray applications, as well as the drench application in the OO-NJ30 trial.

Samples were frozen immediately and stored for up to 245 days (-21 °C) before analysis for fenhexamid using Cornell Analytical Laboratories method "*Residue Analysis of Fenhexamid on Pear using LC with MS Detection*" with a LOQ of 0.02 mg/kg. The results are summarized in Tables, 4, 5, 6 and 7.

Table 4 Results of residue trials conducted in the USA with fenhexamid on pears by dip application

Location, year, (variety)	Application				Residues			Reference
	Method	No	kg ai /hL	GS	Portion analyzed	DALT (days)	Fenhexamid (mg/kg)[a]	
Parlier, CA (00-CA51)	Dip	1	0.09	87	fruit	NA	2.8;3 (2.9)	IR-4 Study No. 07402 00-CA51
Wenatchee,WA (00-WA32)	Dip	1	0.09	87	fruit	NA	1.8;1.8 (1.8)	IR-4 Study No. 07402 00-WA32
Kimberly, ID (00-ID02)	Dip	1	0.09	87	fruit	NA	0.82;0.97 (0.9)	IR-4 Study No. 07402 00-ID02
Bridgeton, NJ USA 2014 (00-NJ30)	Dip	1	0.09	87	fruit	NA	1.9;3.3 (2.6)	IR-4 Study No. 07402 00-NJ30

Location, year, (variety)	Application				Residues			Reference
	Method	No	kg ai /hL	GS	Portion analyzed	DALT (days)	Fenhexamid (mg/kg)[a]	
Davis, CA (00-CA50)	Dip	1	0.09	87	fruit	NA	2.8;3.1 (2.9)	IR-4 Study No. 07402 00-CA50

[a] the mean values in mg/kg are included in brackets.

Table 5 Results of residue trials conducted in the USA with fenhexamid on pears by drench application

Location, year, (variety)	Application				Residues			Reference
	Method	No	kg ai/hL	GS	Portion analyzed	DALT (days)	Fenhexamid (mg/kg)	
Parlier, CA (00-CA51)	Drench	1	0.09	87	fruit	NA	2.2;2.2 (2.2)	IR-4 Study No. 07402 00-CA51
Wenatchee, WA (00-WA32)	Drench	1	0.09	87	fruit	NA	1;1.6 (1.3)	IR-4 Study No. 07402 00-WA32
Kimberly, ID (00-ID02)	Drench	1	0.09	87	fruit	NA	0.88;1.4 (1.1)	IR-4 Study No. 07402 00-ID02
Bridgeton, NJ USA 2014 (00-NJ30)	Drench	1	0.072	87	fruit	NA	2;2.1 (2.05)	IR-4 Study No. 07402 00-NJ30
Davis, CA (00-CA50)	Drench	1	0.09	87	fruit	NA	1.6;1.8 (1.7)	IR-4 Study No. 07402 00-CA50

Table 6 Results of residue trials conducted in the USA with fenhexamid on pears by spray application in the packing line

Location, year, (variety)	Application				Residues			Reference
	Method	No	kg ai/hL	GS	Portion analyzed	DALT (days)	Fenhexamid (mg/kg)	
Parlier, CA (00-CA51)	Packing line spray	1	0.09	87	fruit	NA	2.9;3.6 (3.25)	IR-4 Study No. 07402 00-CA51
Wenatchee, WA (00-WA32)	Packing line spray	1	0.12	87	fruit	NA	4.4;4.7 (4.55)	IR-4 Study No. 07402 00-WA32

Table 7 Results of residue trials conducted in the USA with fenhexamid on pears by spray application in the packing line and drench application

Location, year, (variety)	Application				Residues			Reference
	Method	No	kg ai/hL	GS	Portion analyzed	DALT (days)	Fenhexamid (mg/kg)	
Parlier, CA (00-CA51)	Drench + spray	1	0.09 + 0.09	87	fruit	NA	6;6.5 (6.25)	IR-4 Study No. 07402 00-CA51
Wenatchee, WA (00-WA32)	Drench + spray	1	0.09 + 0.1	87	fruit	NA	4.6;6.4 (5.5)	IR-4 Study No. 07402 00-WA32

Ginseng

Five (5) residue trials with fenhexamid on ginseng were conducted in Canada and the USA during 2000. The two trials conducted in Canada (ON01 and ON02) were conducted in the same year (2002) and in the same location, thus cannot be considered as independent.

In the ginseng trials, four broadcast applications of a fenhexamid WDG formulation at 0.73–0.92 kg ai/ha were applied at a 7-day interval. Roots were harvested on the day of the last application (0 days) and dried in a drying facility at approximately 10% moisture (the drying process lasted 2–6 weeks). Samples were frozen immediately and stored for up to **179 days** (-21 °C) before analysis for fenhexamid using Cornell Analytical Laboratories method "*Residue Analysis of Fenhexamid on Ginseng using LC with MS Detection*" with a LOQ of 0.02 mg/kg. Procedural recoveries of fenhexamid in roots were performed at two spiking levels with average procedural recoveries of 58% (at 0.02 mg/kg) and 58.3% (at 0.2 mg/kg). The RSD was ≤13.1%. In the validation experiments the mean recoveries were also low (53% and 67.5% at 0.05 mg/kg and 0.2 mg/kg, respectively).

Table 8 Results of residue trials with fenhexamid on ginseng conducted in Canada and the USA

Location, year, (variety)	Application				Residues			Reference
	Method	No	kg ai/ ha	GS	Portion analysed	DALT (days)	Fenhexamid (mg/kg)	
Kamloops, BC, Canada 2002 (ginseng)	Broadcast	4	0.87 0.85 0.84 0.83	92 to 99	Root (dried mature)	0	0.02; 0.024 (0.022)	IR-4 Study No. 07846 BC01
Edneyville, NC USA 2002 (ginseng)	Broadcast	4	0.82 0.80 0.84 0.83	92 to 99	Root (dried mature)	0	0,033; 0,026 (0.03)	IR-4 Study No. 07846 NC23

Location, year, (variety)	Application				Residues			Reference
	Method	No	kg ai/ ha	GS	Portion analysed	DALT (days)	Fenhexamid (mg/kg)	
Ontario, ONN0E 2A0 Canada 2002 (North America ginseng)	Broadcast	4	0.74 0.87 0.82 0.86	92 to 99	Root (dried mature)	0	0.03; 0.04 (0.035)	IR-4 Study No. 07846 ON01 Trials ON01 and ON02 are not independent.
Ontario, ONN0E 2A0 Canada 2002 (North America ginseng)	Broadcast	4	0.87 0.85 0.83 0.82	92 to 99	Root (dried mature)	0	0.024; <0.02 (0.022)	IR-4 Study No. 07846 ON02 Trials ON01 and ON02 are not independent.
Wisconsin USA 2020 (ginseng)	Broadcast	4	0.78 0.92	92 to 99	Root (dried mature)	0	0.076; 0.14 (0.11)	PR Study No. 07846 WI41

Asparagus

Two (2) residue trials with fenhexamid on asparagus were conducted in the USA during 2003.

In the asparagus trials, four broadcast applications of fenhexamid (WDG formulation) were applied at 4.19–4.26 kg ai/ha with a 7-day interval. Spears were harvested 92–100 days after the last treatment.

Samples were frozen immediately and stored for up to 228 days (-21 °C) before analysis for fenhexamid using Cornell Analytical Laboratories method "*Residue Analysis of Fenhexamid on Asparagus using LC with MS Detection. Version #8*" with a LOQ of 0.02 mg/kg. Procedural recoveries for fenhexamid in spears were performed at two spiking levels (0.02 and 0.2 mg/kg). The average recoveries were 96.7% at 0.02 mg/kg and 90% at 0.2 mg/kg. The RSD was ≤6%.

Table 9 Results of residue trials with fenhexamid on asparagus conducted in the USA

Location, year, (variety)	Application				Residues			Reference
	Method	No	kg ai/ ha	GS	Portion analyzed	DALT (days)	Fenhexamid (mg/kg)	
Riverside, CA USA 92003 (UC 157)	Foliar broadcast	4	4.25 4.24 4.26 4.20	92 to 95	spears	92	<0.02; <0.02 (<0.02	PR Study No. 08692 CA92

Location, year, (variety)	Application				Residues			Reference
	Method	No	kg ai/ ha	GS	Portion analyzed	DALT (days)	Fenhexamid (mg/kg)	
San Ardo USA 2003 (UC 157)	Foliar broadcast	4	4.19 4.21 4.26 4.26	88 to 92	spears	100	<0.02; <0.02 (<0.02	PR Study No. 08692 CA100

Spring onion

Three (3) residue trials with fenhexamid on green onions were conducted in the USA during 2007 and 2008.

In the trials four broadcast applications of fenhexamid (WDG formulation) at 0.81–0.93 kg ai/ha were applied at a 6–8 day interval. Samples were harvested after the last application.

Samples were frozen immediately and stored for up to 487 days (-21 °C) before analysis for fenhexamid using the method "Working method for PR 07149: Analysis of fenhexamid in onion" with a LOQ of 0.02 mg/kg. Procedural recoveries for fenhexamid in green onion were performed at spiking levels of 0.02, 0.04, 0.5, 1 and 5 mg/kg with the average recoveries being 101%, 116% (n=1), 79.3%, 88% (n=1) and 81.7%, respectively. The RSD was ≤10.8%.

Table 10 Results of residue trials with fenhexamid on green onions conducted in the USA

Location, year, (variety)	Application				Residues			Reference
	Method	No	kg ai/ha	GS	Portion analyzed	DALT (days)	Fenhexamid (mg/kg)	
Salinas, CA 2007 (Oasis)	directed	4	0.86 0.83 0.74 0.86	15 to 40	Green onion	0	4.1; 4.6 (4.35)	IR-4 Study No. 07149 CA27
Tifton, GA 2008 (Pegasus)	directed	4	0.84 0.85 0.85 0.85	15 to 40	Green onion	0	11.1; 12.3 (11.7)	IR-4 Study No. 07149 GA10
Charleston, SC 2007 (Onion parade seed)	directed	4	0.92 0.93 0.93 0.92	15 to 40	Green onion	0	2.9; 2.2 (2.55)	IR-4 Study No. 07149 SC08

Bulb onion, garlic, shallots

Eight (8) residue trials with fenhexamid on bulb onions were conducted in the USA during 2007 and 2008.

In the trials four broadcast applications of fenhexamid (WDG formulation) at 0.72–0.83 lb ai/A (0.83–0.93 kg ai/ha) were applied at a 6–8 day interval. Samples were harvested the last application.

Samples were frozen immediately and stored for up to **483 days** (-21 °C) before analysis for fenhexamid using the method "Working method for PR 07149: Analysis of fenhexamid in onion" with an LOQ of 0.02 mg/kg. Procedural recoveries for fenhexamid in bulbs were performed at three spiking level (0.02, 0.5 and 5 mg/kg). The average recoveries were 96.2, 84 and 88.5% respectively. The RSD was ≤12.3%.

Table1 Results of residue trials with fenhexamid on bulb onions conducted in the USA

Location, year, (variety)	Application				Residues			Reference
	Method	No	kg ai/ha	GS	Portion analysed	DALT (days)	Fenhexamid (mg/kg)	
Hotville, CA 2008 (Serengeti)	directed	4	0,83 0,86 0,85 0,84	15 to 48	Dry bulb	0	1.3; 1.4 (1.35)	CA26
Salinas, CA 93908 2007 (Olympic)	directed	4	0,84 0,85 0,85 0,83	15 to 48	Dry bulb	0	0.25; 0.4 (0.33)	CA28
Salinas, CA 939005 2007 (Olympic)	directed	4	0,86 0,81 0,84 0,84	15 to 48	Dry bulb	0	0.068; 0.041 (0.055)	CA29
Weld Co, CO 2007 (Tioga)	directed	4	0,84 0,86 0,87 0,84	15 to 48	Dry bulb	0	0.032; 0.036 (0.034)	CO08
Freeville, NY 2007 (BGS 233)	directed	4	0,85 0,84 0,83 0,84	15 to 48	Dry bulb	0	<0.02; <0.02 (<0.02)	NY21
Weslaco, TX 2008 (Texas grano 1015Y)	directed	4	0,83 0,84 0,84 0,84	15 to 48	Dry bulb	0 3 7 13	0,055;0,046 (0.051) 0.036; 0.044 0.024; 0.021 <0.021; <0.02	TX15
Weslaco, TX 2008 (XON210W)	broadcast directed	4	0,84 0,84 0,85 0,84	15 to 48	Dry bulb	0	0.081; 0.056 (0.069)	TX16
Moxee, WA 2007 (Tamara F1)	directed	4	0,84 0,84 0,85 0,86	15 to 48	Dry bulb	0	<0.02; <0.02 (<0.02)	WA07

APPRAISAL

Fenhexamid is a hydroxyaniline protective fungicide whose mode of action is by the inhibition of germ tube elongation, mycelial growth and spore germination. Fenhexamid was evaluated for the first time by the 2005 JMPR where an ADI of 0–0.2 mg/kg bw was established and it was concluded that an ARfD was unnecessary. The 2005 JMPR also concluded that the residue definition for plant and animal commodities for compliance with the MRL and dietary risk assessment was parent fenhexamid.

Fenhexamid was scheduled by the Fifty-first Session of the CCPR for the evaluation of additional uses by the 2020 JMPR, which was postponed to the 2021 Extra JMPR. The current Meeting received new information on analytical methods, storage stability and on the use patterns for fenhexamid in pear, ginseng, asparagus, spring onion and bulb vegetables supported by supervised field trials.

Methods of analysis

The Meeting received descriptions and validation data for the analytical methods (specific method codes were not available) in plant matrices. Plant matrices (high water content) are extracted with acetone and cleaned up by solid phase extraction. The residues are detected with LC-MS and generally achieved a LOQ of 0.02 mg/kg for pear, asparagus and onions. The recoveries were in the range of 77–102% with repeatability below 14%.

For ginseng, the Meeting noted that the performance of the analytical method was poor (53–84%) and the mean procedural recoveries were not acceptable (58–58.3%).

Stability of residues in stored analytical samples

Information provided to the 2005 JMPR demonstrated that fenhexamid residues were stable for 12 months in high-water commodities (peaches, cherries, plums, tomatoes). The current Meeting received additional residue stability data for pear, ginseng, asparagus and green onions from samples stored concurrently with residue trial samples. Stability was demonstrated for at least 7.7, 7.2 and 17.7 months for pear, asparagus and green onion, respectively, covering the storage period of samples in the residue trials.

In ginseng, 60% of the residue was found after 4.4 months, however the procedural recoveries were not acceptable (mean recovery of the fresh fortification samples was 62.5%) thus the meeting could not conclude on an acceptable storage stability period for ginseng. **Error! Reference source not found.**

Results of supervised residue trials on crops

The Meeting received supervised residue trial data for foliar applications of fenhexamid on pears, ginseng, asparagus, spring and bulb onion.

Pear

Fenhexamid is registered in the USA for use as a post-harvest dip or drench application on pears at a maximum rate of 0.34 kg ai in 378.5 L of water to 90.7 tn. of fruit (equivalent to 0.09 kg ai/hL or 3.7 g ai/1 tn. fruit). The GAP does not allow a combination of a post-harvest and pre-harvest treatment.

In five trials matching the GAP for post-harvest dip, residues of fenhexamid were (n = 5): 0.9, 1.8, 2.6, 2.9 and 2.9 mg/kg. In five additional trials matching the GAP for post-harvest drench, residues of fenhexamid were (n = 5): 1.1, 1.3, 1.7, 2.05 and 2.2 mg/kg.

Two further trials were carried out with one post-harvest spray application of 0.34 kg ai in 378.5 L of water in the packing line, however only one of the trials matched the GAP for post-harvest use with residues of fenhexamid at 3.25 mg/kg.

The combined data were (n = 11): 0.9, 1.1, 1.3, 1.7, 1.8, 2.05, 2.2, 2.6, 2.9, 2.9 and 3.25 mg/kg.

The Meeting estimated a maximum residue level of 6 mg/kg (based on the mean + 4×SD) and a STMR of 2.05 mg/kg for pears based on the combined dataset.

Ginseng

The critical GAP for fenhexamid on ginseng in the USA is for 4 applications up to BBCH 99 at 840 g ai/ha (interval 7–14 days) and 0 days PHI. The meeting received five trials according to the critical GAP from which 4 were considered independent. The Meeting noted that the performance of the analytical method was poor (53–84%) and the mean procedural recoveries were not acceptable (58–58.3%), thus the meeting was unable to recommend a maximum residue level for ginseng roots.

Asparagus

The critical GAP for fenhexamid on asparagus in the USA is for 4 applications to the ferns up to BBCH 95 at 840 g ai/ha (interval 7–14 days) and 180 days PHI. The meeting received 2 independent, overdosed trials (at 4190–4260 g ai/ha). The residues in spears were (n = 2): < 0.02 and < 0.02 mg/kg. Since in both trials residues were below the LOQ and the applications were made to ferns prior to senescence, about 6 months before spear emergence, the limited number of trials was considered acceptable.

The Meeting estimated a maximum residue level of 0.02 mg/kg and a STMR of 0.02 mg/kg for asparagus.

Subgroup of Bulb Onions

The critical GAP for fenhexamid on bulb onion (Onion, bulb, subgroup 3-07A) in the USA is for 4 applications up to BBCH 48 at 840 g ai/ha (7-day intervals up to the day of harvest) and 0 days PHI. The meeting received seven independent trials matching the critical GAP. The residues in bulb onions were (n = 7): < 0.02(2), 0.034, 0.055, 0.069, 0.33 and 1.4 mg/kg.

The Meeting estimated a maximum residue level of 3 mg/kg and a STMR of 0.055 mg/kg for the Subgroup of Bulb Onions.

Subgroup of Green Onions

The critical GAP for fenhexamid on spring onion (Onion, green, subgroup 3-07B) in the USA is for 4 applications up to BBCH 40 at 840 g ai/ha (7-day intervals up to day of harvest) and a 0-day PHI. The meeting received 3 independent trials according to the critical GAP. The residues in green onions were (n = 3): 2.55, 4.35 and 11.7 mg/kg.

The Meeting concluded that the data, were not sufficient to give a recommendation for Green onions as a minor crop.

RECOMMENDATIONS

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

The residue definition for compliance with the MRL and dietary risk assessment for plant and animal commodities is *fenhexamid*.

The residue is fat-soluble

Table 1 Recommendations for residues of fenhexamid from the 2021 Extra JMPR

CCN	Commodity name	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg
		New	Previous	
VS 0621	Asparagus	0.02		0.02
VA 2031	Bulb onions, Subgroup of	3		0.055
FP 0230	Pear	6 Po		2.05

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for fenhexamid is 0–0.2 mg/kg bw. The International Estimated Daily Intakes (IEDIs) for fenhexamid were estimated for the 17 GEMS/Food Consumption Cluster Diets using the STMR or STMR-P values estimated by the JMPR. The results are shown in Annex 3 of the 2021 Extra JMPR Report.

The IEDIs ranged from 0–5% of the maximum ADI. The Meeting concluded that long-term dietary exposure to residues of fenhexamid from uses considered by the JMPR is unlikely to present a public health concern.

Acute dietary exposure

The 2005 JMPR decided that an ARfD for fenhexamid was unnecessary. The Meeting therefore concluded that the acute dietary exposure to residues of fenhexamid from the uses considered is unlikely to present a public health concern.

Table 6REFERENCES

Report number	Author(s)	Year	Title, Source, Company name, Report No., Date, GLP status published or not
07402	Thompson, D.	2003	Fenhexamid: magnitude of the residue on pear following post-harvest treatment. IR-4 Study No. 07402 GLP Unpublished
07846	Larsson-Kovach, P.	2003	Fenhexamid: magnitude of the residue on ginseng following post-harvest treatment PR.No 07846 GLP Unpublished

Report number	Author(s)	Year	Title, Source, Company name, Report No., Date, GLP status published or not
08692	Corley, J.	2006	Fenhexamid: magnitude of the residue on asparagus following post-harvest treatment PR.No 08692 GLP Unpublished
07149	Corley, J.	2011	Fenhexamid: magnitude of the residue on onion. IR-4 Study No. 07149 GLP Unpublished

FENPICOXAMID (305)

The first draft was prepared by Dr Chris Anagnostopoulos, Benaki Phytopathological Institute, Greece

EXPLANATION

Fenpicoxamid is a picolinamide fungicide. The mode of action is by the inhibition of mitochondrial complex III to disrupt spore germination and germ tube elongation. Fenpicoxamid was evaluated for the first time by the 2018 JMPR where an ADI of 0–0.05 mg/kg bw was established and it was concluded that an ARfD was unnecessary.

The residue definition for plant commodities for compliance with the MRL and dietary risk assessment was *parent fenpicoxamid*. The 2018 JMPR did not recommend residue definitions for animal commodities (compliance with the MRL and dietary risk assessment) but concluded that if future uses of fenpicoxamid resulted in an increase in the dietary exposure to the animal metabolite X12326349 and the hydrolysis products X12314005, X12019520, X12335723 and X12264475, reconsideration of the residue definitions may become necessary.

Fenpicoxamid was scheduled at the Fifty-first Session of the CCPR for the evaluation of additional uses by the 2020 JMPR. The current Meeting received new information on use patterns for fenpicoxamid in wheat, similar grains and pseudo cereals without husks supported by additional analytical methods and recovery data, storage stability studies, supervised field trials, feeding studies and studies simulating typical processing conditions.

METABOLISM AND ENVIRONMENTAL FATE

The metabolism of fenpicoxamid was investigated by the 2018 JMPR. No additional data were provided to the current Meeting.

RESIDUE ANALYSIS

Analytical methods

An analytical method (120615) for the determination of fenpicoxamid and its plant metabolite X642188 in wheat grain, straw and whole plant with an LOQ of 0.01 mg/kg for each analyte was evaluated by the 2018 JMPR. This method was used in the following new studies provided to the current Meeting: S14-01568; S14-01569; S15-02628; S15-02629, S12-02427 and S12-01369. For the additional new processing, feeding and storage stability studies, a modification of the previously validated method 120615 was employed and a brief description is provided below.

Method:	No code	No code
Reference:	S14-02186 (processing study)	CEMS-6149 (feeding study) CEMS-6104 (storage stability study)
Commodity:	Wheat grain, wheat bran, refined flour, wholemeal bread	Bovine whole milk, skim milk, cream, muscle, liver, kidney, fat
Analyte:	Fenpicoxamid X642188 X12335723 X12019520 X12314005 X12264475	Fenpicoxamid X642188 X12326349
Determination:	LC-MS/MS	LC-MS/MS
LOQ:	0.01 mg/kg	0.01 mg/kg

Method:	No code	No code
Accuracy:	The mean recovery in wheat grain, wheat bran, wheat refined flour and wheat wholemeal bread was between 70–105%, 87–102%, 88–109% and 81–111 % at each fortification level.	The mean recovery in whole milk, skim milk, cream, muscle, liver, kidney and fat was between 88–100%, 84–97%, 91–100%, 79–93%, 81–101% and 99–111% at each fortification level.
Repeatability:	The RSDs of all analyte recoveries at each fortification level and overall, during method validation were all below 19.6%	The RSDs of all analyte recoveries at each fortification level and overall, during method validation were all below 12.6%
Specificity:	No interfering peaks were detected in control samples above the LOD.	No interfering peaks were detected in control samples above the LOD.
Description:	Extraction and clean-up: Residues were extracted from a 5.0 g portion of sample by the addition of acetonitrile/water/phosphoric acid (90/10/0.1, v/v/v). Samples were homogenised, shaken and centrifuged. For the analysis of the polar analytes X12335723 and X12264475, 2.0 mL aliquots are pH adjusted and liquid-liquid extracted with water and ethyl acetate. 1.0 mL aliquots of aqueous layer are taken for analysis. For analysis of fenpicoxamid, X642188, X12019520 and X12314005, 1.0 mL aliquots of samples were diluted with 1 mL acetonitrile/water/phosphoric acid (90/10/0.1, v/v/v) prior to analysis.	Extraction and clean-up: Residues were extracted from a 1.0 g portion of sample by the addition of acetonitrile/water/phosphoric acid (75/25/0.1, v/v/v). Following the addition of lysing matrix, muscle, liver, kidney and fat samples were blended in a FastPrep homogenizer for 60 seconds. All samples were shaken and centrifuged. A 250 µL aliquot of the extract was mixed with 1750 µL of acetonitrile/water/formic acid (50/50/0.1, v/v/v).

Stability of residues in stored analytical samples

Additional studies were provided investigating the stability of residues of fenpicoxamid and metabolite X642188 in wheat grain, straw, forage and processed commodities and the stability of fenpicoxamid and metabolites X642188 and X12326349 in animal commodities.

Plant commodities

A study (S12-02427) investigating the stability of residues of fenpicoxamid and metabolite X642188 in wheat grain, straw, forage and processed commodities was provided.

In this study, wheat grain, wheat straw, wheat forage bran, germ, white bread and white flour samples were fortified separately with fenpicoxamid or X642188 at a fortification level of 1.0 mg/kg and stored at < -18 °C.

At each storage time point (initial day=T0, 1, 3, 6, 9 and 24 months storage), analysis consisted of the following samples: 1 unfortified control sample, 2 freshly fortified control samples at 1 mg/kg with both fenpicoxamid and X642188 for procedural recovery calculations, 3 stored samples fortified with fenpicoxamid and 3 stored samples fortified with X642188.

Residue analysis was performed according to the method 120615. This method has been validated in various crop matrices, including wheat grain, straw, bran, flour and bread, with an LOQ of 0.01 mg/kg for each analyte, by the 2018 JMPR.

The recoveries from the stored fortified and the concurrent recoveries from the freshly fortified samples are presented in Table 1 and Table 2 for fenpicoxamid and X642188 respectively.

Both parent fenpicoxamid and metabolite X642188 are stable in wheat raw commodities and processed fractions under freezer conditions for up to *ca.* 2 years (741 days for wheat grain, straw and forage and 745 days for processed commodities).

Table 1 Stability of fenpicoxamid residues in crops following storage at ≤ 18 °C

Commodity	Level (mg/kg)	Storage interval (days)	Concurrent recovery (mean, %)	Residues after storage (mg/kg)		% remaining
				Individual values	Mean	% Nominal spiking level
Wheat grain	1.0	0	104 (102, 106)	1.0702, 1.1027, 1.0957	1.0895	109
		33	103 (103, 103)	0.9440, 0.9560, 1.0240	0.9747	97
		98	99 (98, 100)	0.8914, 0.8930, 0.9001	0.8948	89
		180	99.5 (97, 102)	0.9266, 0.8975, 0.9031	0.9091	91
		273	110.5 (107, 114)	0.9999, 1.0200, 0.9859	1.0019	100
		741	106 (104, 108)	0.9881, 0.9778, 0.9557	0.9739	97
Wheat straw	1.0	0	105 (104, 106)	1.0415, 1.0153, 1.0164	1.0244	102
		33	104.5 (104, 105)	1.0477, 1.0997, 1.1157	1.0877	109
		98	100.5 (98, 103)	0.9313, 0.9631, 0.9169	0.9371	94
		180	101.5 (100, 103)	0.9241, 0.9277, 0.8887	0.9135	91
		273	106.5 (106, 107)	0.9321, 0.9609, 0.9463	0.9464	95
		741	103 (103, 103)	0.9193, 0.9155, 0.9416	0.9255	93
Wheat forage	1.0	0	95 (95, 95)	0.9072, 0.8752, 0.9552	0.9125	91
		33	101.5 (99, 104)	0.8352, 0.8992, 0.8632	0.8659	87
		98	103 (98, 108)	0.8684, 0.8312, 0.8494	0.8479	85
		180	93 (92, 24)	0.8305, 0.8978, 0.8332	0.8538	85
		273	96 (96, 96)	0.7897, 0.7773, 0.7681	0.7784	78
		741	98.5 (94, 103)	0.8991, 0.9450, 0.9188	0.9210	92
Wheat bran	1.0	0	92 (94, 90)	0.9515, 0.9555, 0.9555	0.9542	95
		31	100 (9, 101)	0.9184, 0.8946, 0.9228	0.9119	91
		97	104.5 (104, 105)	0.9535, 0.9467, 0.9273	0.9425	94
		182	96 (99, 93)	0.8541, 0.8507, 0.8245	0.8431	84
		276	103 (104, 102)	0.7854, 0.7823, 0.8067	0.7915	79

Commodity	Level (mg/kg)	Storage interval (days)	Concurrent recovery (mean, %)	Residues after storage (mg/kg)		% remaining
				Individual values	Mean	% Nominal spiking level
		745	101.5 (101, 102)	0.7399, 0.7418, 0.9067	0.7295	73
Wheat germ	1.0	0	102.5 (97, 108)	1.0280, 1.1120, 1.0680	1.0693	107
		31	96 (97, 95)	0.7624, 0.8140, 0.8312	0.8025	80
		97	107.5 (106, 109)	0.8653, 0.8427, 0.8439	0.8506	85
		182	94 (94, 94)	0.7702, 0.8005, 0.7786	0.7831	78
		276	98.5 (98, 99)	0.7104, 0.7281, 0.6763	0.7049	70
		745	89.5 (85, 94)	0.7749, 0.7706, 0.8387	0.7947	79
Wheat flour	1.0	0	106 (101, 111)	1.1142, 1.1022, 1.0542	1.0902	109
		31	94 (95, 93)	0.8858, 0.9490, 0.8698	0.9015	90
		97	106.5 (107,106)	0.8841, 0.9459, 0.9153	0.9151	92
		182	97.5 (98, 97)	0.8837, 0.8368, 0.8731	0.8645	86
		276	96.5 (94, 99)	0.8846, 0.8565, 0.8388	0.8600	86
		745	103.5 (104, 103)	0.7666, 0.8227, 0.7827	0.7907	79
Wheat bread	1.0	0	99.5 (102, 97)	1.0220, 1.0044, 0.9883	1.0049	100
		31	90.5 (91, 90)	0.8215, 0.8847, 0.8815	0.8626	86
		97	103.5 (104, 103)	0.9736, 0.9375, 0.9388	0.9500	95
		182	94.5 (94, 94)	0.8620, 0.8497, 0.8308	0.8475	85
		276	94 (92, 96)	0.8613, 0.8146, 0.8632	0.8464	85
		745	89 (89, 89)	0.8792, 0.8515, 0.8656	0.8654	87

Table 2 Stability of X642188 residues in crops following storage at ≤ 18 °C

Commodity	Level (mg/kg)	Storage interval (days)	Fresh recovery (mean, %)	Residues after storage (mg/kg)		Recovery
				Individual values	Mean	% Nominal spiking level
Wheat grain	1.0	0	97.5 (97, 98)	0.9722, 0.9236, 0.9550	0.9503	95
		33	102.5 (101, 104)	1.0080, 1.0160, 1.0120	1.0120	101

Commodity	Level (mg/kg)	Storage interval (days)	Fresh recovery (mean, %)	Residues after storage (mg/kg)		Recovery
				Individual values	Mean	% Nominal spiking level
		98	93.5 (94, 93)	1.0227, 1.0876, 1.0450	1.0518	105
		180	97.5 (101, 94)	0.9129, 0.9416, 0.9165	0.9237	92
		273	88 (87, 89)	0.8198, 0.7948, 0.8113	0.8086	81
		741	104 (108, 104)	1.0603, 1.0527, 1.0516	1.0549	105
Wheat straw	1.0	0	95.5 (98, 93)	0.9295, 0.9335, 0.9538	0.9389	94
		33	107.5 (107, 108)	0.9600, 1.0840, 1.0880	1.044	104
		98	103 (106, 100)	1.1633, 1.1027, 1.1879	1.1513	115
		180	96 (96, 96)	0.9041, 0.8982, 0.9247	0.9090	91
		273	92.5 (93, 92)	0.7673, 0.7889, 0.7715	0.7759	78
		741	90 (91, 89)	0.6428, 0.7228, 0.7285	0.6980	70
Wheat forage	1.0	0	91.5 (92, 91)	0.9560, 0.9600, 0.9920	0.9693	97
		33	101.5 (103, 100)	0.9160, 0.9120, 0.9040	0.9107	91
		98	98 (101, 95)	1.0050, 1.0483, 1.0794	1.0442	104
		180	89 (89, 89)	0.8099, 0.8283, 0.8240	0.8207	82
		273	86 (86, 86)	0.7216, 0.6963, 0.7537	0.7239	72
		741	96 (98, 94)	0.9210, 0.9596, 0.9008	0.9271	93
Wheat bran	1.0	0	103 (102, 104)	1.0638, 1.0518, 1.0478	1.0545	105
		31	96 (96, 96)	0.9262, 0.9140, 0.9236	0.9213	92
		97	104.5 (108, 101)	1.0332, 1.0266, 1.1008	1.0535	105
		182	108.5 (110, 107)	0.9940, 0.9926, 0.9650	0.9839	98
		276	94.5 (95, 94)	0.8304, 0.7990, 0.8113	0.8136	81
		745	102.5 (103, 102)	0.7994, 0.8696, 0.8661	0.8450	85
Wheat germ	1.0	0	97 (98, 96)	1.0080, 0.9640, 0.9840	0.9853	99
		31	95 (98, 92)	0.8448, 0.8143, 0.7929	0.8173	82
		97	110 (110, 110)	1.0741, 1.1137, 1.1231	1.1036	110
		182	106.5 (106, 107)	1.0016, 0.9771, 0.9571	0.9786	98
		276	92 (92, 92)	0.7359, 0.7297, 0.7138	0.7265	73

Commodity	Level (mg/kg)	Storage interval (days)	Fresh recovery (mean, %)	Residues after storage (mg/kg)		Recovery
				Individual values	Mean	% Nominal spiking level
		745	111.5 (107, 116)	1.0150, 1.0358, 1.0215	1.0241	102
Wheat flour	1.0	0	88 (82, 94)	0.9320, 1.0560, 1.0120	1.0000	100
		31	92 (94, 90)	0.8952, 0.9011, 0.8400	0.8788	88
		97	101 (101, 101)	1.1367, 1.0397, 1.0978	1.0914	109
		182	105.5 (106, 105)	0.9294, 0.9813, 1.0133	0.9747	97
		276	88.5 (88, 89)	0.8136, 0.8138, 0.7751	0.8008	80
		745	107.5 (114, 101)	0.9346, 0.9281, 0.9516	0.9381	94
Wheat bread	1.0	0	107 (107, 107)	1.0714, 1.0513, 1.0732	1.0653	107
		31	92 (92, 92)	0.8694, 0.8433, 0.8859	0.8662	87
		97	98.5 (98, 99)	1.1547, 1.0946, 1.1057	1.1193	112
		182	105 (109, 101)	0.9516, 0.9060, 0.9015	0.9197	92
		276	86.5 (85, 88)	0.7399, 0.7985, 0.8226	0.7870	79
		745	100.5 (101, 100)	0.9976, 0.9706, 0.9384	0.9689	97

Animal commodities

The study was carried out on bovine whole milk, bovine muscle, bovine liver and bovine fat. The samples were fortified separately with fenpicoxamid, X642188 or X12326349 at a fortification level of 0.10 mg/kg for each compound. Additional samples used as unfortified controls, procedural recovery samples and the stored fortified samples were stored in a freezer set to maintain a temperature of < -18 °C.

At each storage time point (initial day=T0, 1, 3, 6 and 9 months storage), analysis consisted of the following samples and a reagent blank: 1 unfortified control sample, 2 freshly fortified control samples with fenpicoxamid, X642188 and X12326349 for procedural recovery calculations, 3 stored samples fortified with fenpicoxamid, 3 stored samples fortified with X642188 and 3 stored samples fortified with X12326349.

Residues of fenpicoxamid and metabolites X642188 and X12326349 were determined by the analytical method validated in this study. The method was validated with an LOQ of 0.01 mg/kg for each compound in all matrices. The performance of the method was verified by obtaining mean procedural recoveries in the acceptable range of 70–120% at fortification level of 0.10 mg/kg. The recoveries from the stored fortified and the concurrent recoveries from the freshly fortified samples are presented in Tables 3, 4 and 5 for fenpicoxamid, X642188 and X12326349 respectively.

Significant decrease is observed in bovine milk for X12326349 after 3 months and for X642188 after 6.5 months whereas fenpicoxamid is stable for 9 months. Similar decrease is observed in bovine muscle, however significant residues of fenpicoxamid were lost after approximately 7 months. In bovine

liver, residues are stable only up to 3 months for the parent compound and decrease significantly after 6–7 months for X642188 and X12326349. In bovine fat, fenpicoxamid and X642188 remain stable for 9 months; however significant residues of X123263489 were lost after 3 months.

In conclusion, fenpicoxamid, X642188 and X12326349 are stable in animal commodities under freezer conditions for up to 3 months.

Table 3 Stability of fenpicoxamid residues in animal matrices following storage at ≤ 18 °C

Commodity	Level (mg/kg)	Storage interval (days)	Fresh recovery (mean, %)	Residues after storage (mg/kg)		Recovery
				Individual values	Mean	% Nominal spiking level
Bovine whole milk	0.1	0	93.5	0.0967, 0.0958, 0.0920	0.0948	95
		28	100.5	0.1043, 0.0908, 0.0960	0.0970	97
		78–85	107.5	0.0851, 0.0918, 0.0918	0.0896	90
		196	89	0.0910, 0.0958, 0.0962	0.0943	94
		273	88.5	0.0844, 0.0915, 0.0843	0.0867	87
Bovine muscle	0.1	0	97	0.1073, 0.1112, 0.1004	0.1063	106
		28	97	0.0758, 0.0819, 0.0862	0.0813	81
		78–85	101	0.0887, 0.0821, 0.0794	0.0834	83
		206	95	0.0769, 0.0766, 0.0792	0.0776	78
		276	92	0.0660, 0.0685, 0.0648	0.0664	66
Bovine liver	0.1	0	93.5	0.0842, 0.0903, 0.0938	0.0894	89
		28	110.5	0.0774, 0.0834, 0.0933	0.0847	85
		78–85	118.5	0.0994, 0.0700, 0.0754	0.0816	82
		206–208	94.5	0.0241, 0.0529, 0.0589	0.0453	45
		276	93.5	0.0230, 0.0631, 0.0095	0.0319	32
Bovine fat	0.1	0	96	0.0969, 0.0967, 0.0997	0.0978	98
		28	89	0.1058, 0.1026, 0.0971	0.1018	102
		78–85	119.5	0.1142, 0.1100, 0.1116	0.1119	112
		207	84	0.0889, 0.0875, 0.0902	0.0889	89
		277	101	0.0892, 0.0950, 0.0990	0.0944	94

Table 4 Stability of X642188 residues in animal commodities following storage at ≤ 18 °C

Commodity	Level (mg/kg)	Storage interval (days)	Fresh recovery (mean, %)	Residues after storage (mg/kg)		Recovery
				Individual values	Mean	% Nominal spiking level
Bovine whole milk	0.1	0	113	0.1068, 0.1108, 0.1036	0.1071	107
		28	91.5	0.0917, 0.0998, 0.0874	0.0930	93
		78–85	95.5	0.0808, 0.0832, 0.0834	0.0825	83
		196	97.5	0.0920, 0.1009, 0.0923	0.0951	95
		273	78	0.0674, 0.0684, 0.0732	0.0697	70
Bovine muscle	0.1	0	93	0.0966, 0.0909, 0.0925	0.0933	93
		28	90	0.0912, 0.0854, 0.0848	0.0871	87
		78–85	100.5	0.0775, 0.0771, 0.0673	0.0740	74
		206	95	0.0683, 0.0749, 0.0838	0.0757	76
		276	81.5	0.0554, 0.0588, 0.0507	0.0550	55
Bovine liver	0.1	0	81.5	0.0898, 0.0896, 0.0951	0.0915	92
		28	99	0.0863, 0.0804, 0.0938	0.0868	87
		78–85	108	0.0705, 0.0775, 0.0776	0.0752	75
		206–208	94.5	0.0743, 0.0750, 0.0786	0.0760	76
		276	85.5	0.0498, 0.0532, 0.0554	0.0528	53
Bovine fat	0.1	0	101	0.0969, 0.1070, 0.1071	0.1037	104
		28	93	0.0871, 0.0757, 0.0831	0.0820	82
		78–85	94	0.0646, 0.0946, 0.0809	0.0800	80
		207	85.5	0.0742, 0.0765, 0.0800	0.0769	77
		277	86	0.0778, 0.0678, 0.0733	0.0730	73

Table 5 Stability of X12326349 residues in animal commodities following storage at ≤ 18 °C

Commodity	Level (mg/kg)	Storage interval (days)	Fresh recovery (mean, %)	Residues after storage (mg/kg)		Recovery
				Individual values	Mean	% Nominal spiking level
Bovine whole milk	0.1	0	99.5	0.1025, 0.1006, 0.1052	0.1028	103
		28	93	0.0867, 0.0825, 0.0968	0.0887	89
		78–85	89	0.0727, 0.0885, 0.0740	0.0784	78
		196	93	0.0685, 0.0689, 0.0634	0.0660	66

Commodity	Level (mg/kg)	Storage interval (days)	Fresh recovery (mean, %)	Residues after storage (mg/kg)		Recovery
				Individual values	Mean	% Nominal spiking level
		273	85.5	0.0640, 0.0663, 0.0786	0.0696	70 (69.6)
Bovine muscle	0.1	0	102.5	0.0943, 0.1048, 0.1054	0.1015	102
		28	103.5	0.0606, 0.0754, 0.0752	0.0704	70
		78–85	88	0.0698, 0.0730, 0.0673	0.0740	74
		206	92	0.0683, 0.0749, 0.0838	0.0757	76
		276	73.5	0.0554, 0.0588, 0.0507	0.0550	55
Bovine liver	0.1	0	97	0.1001, 0.0969, 0.1034	0.1001	100
		28	101	0.1015, 0.0969, 0.0870	0.0951	95
		78–85	104.5	0.0934, 0.0895, 0.0936	0.0922	92
		206–208	85	0.0739, 0.0706, 0.0718	0.0721	72
		276	78.5	0.0621, 0.0650, 0.0684	0.0652	65
Bovine fat	0.1	0	115	0.0859, 0.1198, 0.1094	0.1050	105
		28	82.5	0.0713, 0.0762, 0.0998	0.0824	92
		78–85	116.5	0.0838, 0.0900, 0.0991	0.0910	91
		207	92.5	0.0604, 0.0581, 0.0679	0.0621	62
		277	82.5	0.0546, 0.0658, 0.0578	0.0594	59

USE PATTERN

A copy of the fenpicoxamid label from New Zealand and Europe was made available to the Meeting. These registered uses are summarised in Table 6.

Table 6 Registered uses of fenpicoxamid relevant to the evaluation

Crop	Country	Form	Application			PHI, days
			Method	Rate g ai/ha	Number	
Wheat	New Zealand	50 g/L, EC	Foliar	100	2	Apply BBCH 30–69 Grain and straw: n/a For fodder and green feed: 28
Wheat, durum wheat, rye, triticale and spelt wheat	Europe (CZ: BE, CH, CZ, DE, IE, LU, HU, NL, AT, PL, RO, SI, SK, UK, NEU: EE, FI, LV, LT, NO, SE SEU: BG, EL, ES, HR, IT)	50 g/L, EC	Foliar	100	2	Apply up to BBCH 69

Crop	Country	Form	Application			PHI, days
			Method	Rate g ai/ha	Number	

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

Cereals

Thirty-six (36) residue trials with fenpicoxamid on wheat were conducted in Europe during 2014–2015 according to the critical GAP, from which 18 were considered independent.

Two foliar spray applications of an EC or SC formulation were made at a rate of 92–121 g ai/ha, with re-treatment intervals of 11–19 days and with the final application at growth stage BBCH 69. Each of the field trial sites normally consisted of an untreated control plot and treated plots.

Samples of at least 1 kg were harvested at the normal commercial harvest (NCH). Decline data was also collected on **whole plants (forage)** at -0, 0, 6–8, 14, 26–30 days and **grain and straw** samples were taken 6/7 days before NCH, at NCH, and 6–8 and 12–14 days after NCH. Samples were frozen immediately after sampling. The maximum period of sample storage at -20 °C was less than 247 days. This period is covered by the available storage stability data.

Samples were analysed for residues of fenpicoxamid and its metabolite X642188 using the validated analytical method 120615 (previously evaluated by the 2018 JMPR). The LOQ was 0.01 mg/kg for each analyte. The performance of the method was verified by obtaining mean procedural recoveries of fenpicoxamid and X642188 in the acceptable range of 84–109.4% and 94–110% for fenpicoxamid and X642188, respectively, at fortification levels 0.01–20 mg/kg. Laboratory reports included method validation with procedural recoveries, RSD and calibration curves from spiking at residue levels similar to those occurring in samples from the supervised trials. Residue data are not corrected for percentage recovery. The results from the supervised trials conducted in Europe with products GF-3307, GF-3309, GF-3310, and GF-3312 (EC formulations) and GF-2925 (SC formulation) on wheat (winter and spring) after foliar application are summarized in Table 7.

Table 7 Results of fenpicoxamid residue trials in wheat grain

Location, year, (variety	Application			Residues			Reference
	No	kg/ha (a.i.)	GS	Portion analysed	DALT (days)	Fenpicoxamid (mg/kg)	
45300, Loiret, Rouvres-Saint-Jean France NEU 2014	2	99.9 98.4	55 69	Grain Grain Grain Grain	43 50 57 64	<0.01 <0.01 <0.01 <0.01	S14-01568-01

Location, year, (variety	Application			Residues			Reference
	No	kg/ha (a.i.)	GS	Portion analysed	DALT (days)	Fenpicoxamid (mg/kg)	
(Winter wheat / Arezzo)	2	101.1 101.6	BBCH 55 BBCH 69	Grain Grain Grain Grain	43 50 57 64	<0.01 <0.01 <0.01 <0.01	
CV9 3DT, Atherstone, Warwickshire UK NEU 2014 (Spring wheat / Mulika)	2	98.3 104.4	BBCH 61 BBCH 69	Grain Grain Grain Grain	28 35 42 49	0.024 0.010 <0.01 (0.008) <0.01 (0.006)	S14-01568-02
	2	102.8 104.4	BBCH 61 BBCH 69	Grain Grain Grain Grain	28 35 42 49	0.021 <0.01 (0.006) 0.012 <0.01 (0.009)	
DE73 7FW, Twyford, Derbyshire, UK NEU 2014 (Winter wheat / KWS Santiago)	2	101.6 99.2	BBCH 55 BBCH 69	Grain	45	<0.01 (0.007)	S14-01568-03
	2	100.8 100.0	BBCH 55 BBCH 69	Grain	45	<0.01 (0.006)	
71691, Freiberg, Baden-Württemberg, Germany NEU GLP 2014 (Spring wheat / Triso	2	100.0 95.8	BBCH 61 BBCH 69	Grain	42	0.012	S14-01568-04
	2	100.0 95.8	BBCH 61 BBCH 69	Grain	42	<0.01 (0.009)	
40054, Prunaro Di Budrio, Province of Bologna, Italy SEU 2014	2	100.7 99.3	BBCH 53 BBCH 69	Grain Grain Grain Grain	49 55 62 67	0.020 0.017 0.011 ND	S14-01568-05

Location, year, (variety	Application			Residues			Reference
	No	kg/ha (a.i.)	GS	Portion analysed	DALT (days)	Fenpicoxamid (mg/kg)	
(Winter wheat / Masarrio)	2	97.4 103.3	BBCH 53 BBCH 69	Grain Grain Grain Grain	49 55 62 62	0.018 0.035 0.010 ND	
44492 Fonfría, Teruel, Spain SEU GLP 2014 (Spring wheat / Marius)	2	95.6 98.9	BBCH 39-45 BBCH 69	Grain Grain Grain Grain	53 60 67 74	0.017 0.021 0.027 0.014	S14-01568-06
	2	96.1 101.9	BBCH 39-45 BBCH 69	Grain Grain Grain Grain	53 60 67 74	0.012 0.017 0.016 0.015	
66750, Saint-Cyprien, Pyrénées Orientales, France SEU 2014 (Winter wheat / Babylone)	2	94.2 100.0	BBCH 61 BBCH 69	Grain	36	0.066	S14-01568-07
	2	95.0 104.2	BBCH 61 BBCH 69	Grain	36	0.051	
44492, Lagueruela, Aragon, Spain SEU 2014 (Spring wheat / Marius)	2	105.6 103.9	BBCH 39-45 BBCH 69	Grain	41	0.020	S14-01568-08
	2	102.8 101.7	BBCH 39-45 BBCH 69	Grain	41	0.022	
45300, Loiret, Rouvres-Saint-Jean France NEU 2014 (Winter wheat / Arezzo)	2	102.2 98.8	BBCH 51 BBCH 69	Grain Grain Grain Grain	43 50 57 64	<0.01 (0.003) <0.01 (0.003) <0.01 (0.004) <0.01 (ND)	S14-01569-01
	2	97.1 99.4	BBCH 51 BBCH 69	Grain Grain Grain Grain	43 50 57 64	0.011 0.010 <0.01 (0.009) 0.011	

Location, year, (variety	Application			Residues			Reference
	No	kg/ha (a.i.)	GS	Portion analysed	DALT (days)	Fenpicoxamid (mg/kg)	
CV9 3DT, Atherstone, Warwickshire UK NEU 2014 (Spring wheat / Mulika)	2	99.4 105.6	BBCH 61 BBCH 69	Grain Grain Grain Grain	28 35 42 49	0.028 (0.006) (0.007) (0.003)	S14-01569-02
	2	98.3 99.4	BBCH 61 BBCH 69	Grain Grain Grain Grain	28 35 42 49	0.032 0.024 0.030 0.018	
DE73 7FW, Twyford, Derbyshire, UK NEU 2014 (Winter wheat / KWS Santiago)	2	101.7 120.8	BBCH 55 BBCH 69	Grain	45	<0.01 (ND)	S14-01569-03
	2	103.3 115.0	BBCH 55 BBCH 69	Grain	45	0.012	
71691, Freiberg, Baden-Württemberg, Germany NEU 2014 (Spring wheat / Triso)	2	97.5 103.3	BBCH 61 BBCH 69	Grain	42	<0.01 (ND)	S14-01569-04
	2	96.7 105.8	BBCH 61 BBCH 69	Grain	42	0.024	
40054, Prunaro Di Budrio, Province of Bologna, Italy SEU 2014 (Winter wheat / Masarrio)	2	96.7 105.9	BBCH 53 BBCH 69	Grain Grain Grain Grain	49 55 62 67	<0.01 (0.008) 0.038 0.014 <0.01 (ND)	S14-01569-05
	2	103.0 97.0	BBCH 53 BBCH 69	Grain Grain Grain Grain	49 55 62 67	0.027 0.040 0.012 <0.01 (ND)	

Location, year, (variety	Application			Residues			Reference
	No	kg/ha (a.i.)	GS	Portion analysed	DALT (days)	Fenpicoxamid (mg/kg)	
44492 Fonfría, Teruel, Spain SEU 2014 (Spring wheat / Marius)	2	95.0 101.4	BBCH 39-45 BBCH 69	Grain Grain Grain Grain	53 60 67 74	0.013 <0.01 (0.009) <0.01 (0.009) 0.011	S14-01569-06
	2	99.4 105.3	BBCH 39-45 BBCH 69	Grain Grain Grain Grain	53 60 67 74	0.017 0.023 0.036 0.021	
66750, Saint-Cyprien, Pyrénées Orientales, France SEU 2014 (Winter wheat / Babylone)	2	94.2 105.0	BBCH 61 BBCH 69	Grain	36	0.050	S14-01569-07
	2	98.3 105.0	BBCH 61 BBCH 69	Grain	36	0.095	
44492, Lagueruela, Aragon, Spain SEU 2014	2	107.2 105.0	BBCH 39-45 BBCH 69	Grain	41	0.013	S14-01569-08
	2	106.1 97.2	BBCH 39-45 BBCH 69	Grain	41	0.040	
HU17 8JF, Bishop Burton, East Yorkshire, UK NEU 2015 (Winter wheat / Reflection)	2	102.1 97.6	BBCH 61 BBCH 69	Grain Grain Grain Grain	45 51 59 65	<0.01 (0.008) 0.012 0.014 <0.01 (0.009)	S15-02628-01
L39 6SX, Lydiate, Lancashire, UK NEU 2015 (Winter wheat / KWS Kielder)	2	110.0 106.7	BBCH 65-67 BBCH 69	Grain	42	0.034	S15-02628-02

Location, year, (variety	Application			Residues			Reference
	No	kg/ha (a.i.)	GS	Portion analysed	DALT (days)	Fenpicoxamid (mg/kg)	
67140 Saint-Pierre, Alsace, Bas-Rhin, France NEU 2015 (Winter wheat / Pakilo)	2	95.8 100.4	BBCH 59 BBCH 69	Grain	42	0.011	S15-02628-03
86120 Morton, Vienne, France SEU 2015 (Winter wheat / Cellule)	2	100.0 103.2	BBCH 56 BBCH 69	Grain Grain Grain Grain	39 45 51 59	0.025 0.010 0.023 <0.01 (0.009)	S15-02628-04
71290 Simandre, Saône-et-Loire, France SEU 2015 (Spring wheat / Sensus)	2	108.7 93.2	BBCH 59 BBCH 69	Grain	26	0.059	S15-02628-05
50367 Retascón, Spain SEU 2015 (Winter wheat / Garcia)	2	108.3 92.9	BBCH 47 BBCH 67-69	Grain	41	0.010	S15-02628-06
44492 Fonfría, Teruel, Spain SEU 2015 (Winter wheat / Marius)	2	91.9 95.6	BBCH 57-61 BBCH 69	Grain	50 50	0.010	S15-02628-07
NG34 8PE, Silk Willoughby, Lincolnshire, UK NEU 2015 (Spring wheat / Mulika)	2	105.4 108.3	BBCH 39 BBCH 69	Grain Grain Grain Grain	44 51 58 65	0.020 0.011 <0.01 (0.008) <0.01 (0.007)	S15-02628-08

Location, year, (variety	Application			Residues			Reference
	No	kg/ha (a.i.)	GS	Portion analysed	DALT (days)	Fenpicoxamid (mg/kg)	
LE12 5RQ, West Leake, Leicestershire, UK NEU 2015 (Spring wheat / Mulika)	2	112.5 95.0	BBCH 61 BBCH 69	Grain	46	<0.01 (0.008)	S15-02628-09
40057, Granarolo, Emilia-Romagna, Italy SEU 2015 (Spring wheat / Aquilante)	2	105.1 108.4	BBCH 51 BBCH 69	Grain Grain Grain Grain	33 40 47 54	0.013 <0.01 (0.007) <0.01 (0.004) <0.01 (0.006)	S15-02628-10
HU17 8JF, Bishop Burton, East Yorkshire, UK NEU 2015	2	99.0 98.3	BBCH 61 BBCH 69	Grain Grain Grain Grain	45 51 59 65	0.011 0.014 0.012 <0.01 (0.006)	S15-02629-01
L39 6SX, Lydiate, Lancashire, UK NEU 2015 (Winter wheat / KWS Kielder)	2	108.3 110.0	BBCH 65-67 BBCH 69	Grain	42	0.023	S15-02629-02
67140 Saint-Pierre, Alsace, Bas-Rhin, France NEU 2015 (Winter wheat / Pakilo)	2	98.8 102.7	BBCH 59 BBCH 69	Grain	42	0.016	S15-02629-03
86120 Morton, Vienne, France SEU 2015 (Winter wheat / Cellule)	2	94.7 105.0	BBCH 56 BBCH 69	Grain Grain Grain Grain	39 45 51 59	0.028 0.011 0.020 <0.01 (0.009)	S15-02629-04

Location, year, (variety	Application			Residues			Reference
	No	kg/ha (a.i.)	GS	Portion analysed	DALT (days)	Fenpicoxamid (mg/kg)	
71290 Simandre, Saône-et-Loire, France SEU 2015 (Spring wheat / Sensus)	2	105.3 93.0	BBCH 59 BBCH 69	Grain	26	0.069	S15-02629-05
50367 Retascón, Spain SEU 2015 (Winter wheat / Garcia)	2	95.2 92.9	BBCH 47 BBCH 67-69	Grain	41	<0.01 (0.003)	S15-02629-06
44492 Fonfría, Teruel, Spain SEU 2015 (Winter wheat / Marius)	2	108.1 95.6	BBCH 57-61 BBCH 69	Grain	50	<0.01 (0.007)	S15-02629-07
NG34 8PE, Silk Willoughby, Lincolnshire, UK 2015 (Spring wheat / Mulika)	2	108.3 107.5	BBCH 39 BBCH 69	Grain Grain Grain Grain	44 51 58 65	0.022 0.012 0.011 <0.01 (0.008)	S15-02629-08
LE12 5RQ, West Leake, Leicestershire, UK NEU 2015	2	115.0 96.7	BBCH 61 BBCH 69	Grain	46	0.012	S15-02629-09
40057, Granarolo, Emilia-Romagna, Italy SEU 2015 (Spring wheat / Aquilante)	2	109.1 108.9	BBCH 51 BBCH 69	Grain Grain Grain Grain	33 40 47 54	0.031 0.010 <0.01 (0.008) 0.020	S15-02629-10

Table 8 Results of fenpicoxamid residue trials in wheat straw

Location, year, (variety	Application			Residues			Reference
	No	kg/ha (ai)	GS	Portion analysed	DALT (days)	Fenpicoxamid (mg/kg)	
45300, Loiret, Rouvres-Saint-Jean France NEU 2014 (Winter wheat / Arezzo)	2	99.9 98.4	55 69	Straw Straw Straw Straw	43 50 57 64	1.221 1.251 1.105 1.586	S14-01568-01
	2	101.1 101.6	BBCH 55 BBCH 69	Straw Straw Straw Straw	43 50 57 64	1.040 1.250 1.142 1.102	
CV9 3DT, Atherstone, Warwickshire UK NEU 2014 (Spring wheat / Mulika)	2	98.3 104.4	BBCH 61 BBCH 69	Straw Straw Straw Straw	28 35 42 49	1.585 1.531 1.414 1.652	S14-01568-02
	2	102.8 104.4	BBCH 61 BBCH 69	Straw Straw Straw Straw	28 35 42 49	2.094 1.729 1.457 1.312	
DE73 7FW, Twyford, Derbyshire, UK NEU 2014 (Winter wheat / KWS Santiago)	2	101.6 99.2	BBCH 55 BBCH 69	Straw	45	1.912	S14-01568-03
	2	100.8 100.0	BBCH 55 BBCH 69	Straw	45	2.038	
71691, Freiberg, Baden-Württemberg, Germany NEU GLP 2014 (Spring wheat / Triso	2	100.0 95.8	BBCH 61 BBCH 69	Straw	42	5.465	S14-01568-04
	2	100.0 95.8	BBCH 61 BBCH 69	Straw	42	5.200	
40054, Prunaro Di Budrio, Province of Bologna, Italy SEU	2	100.7 99.3	BBCH 53 BBCH 69	Straw Straw Straw Straw	49 55 62 67	1.342 1.391 0.405, <0.01 (ND) 1.319, <0.01 (0.005)	S14-01568-05

Location, year, (variety	Application			Residues			Reference
	No	kg/ha (ai)	GS	Portion analysed	DALT (days)	Fenpicoxamid (mg/kg)	
2014 (Winter wheat / Masarrio)	2	97.4 103.3	BBCH 53 BBCH 69	Straw Straw Straw Straw	49 55 62 67	1.815 1.683 0.711, <0.01 (0.005) 0.056, <0.01 (ND)	
44492 Fonfría, Teruel, Spain SEU GLP 2014 (Spring wheat / Marius)	2	95.6 98.9	BBCH 39-45 BBCH 69	Straw Straw Straw Straw	53 60 67 74	5.147 5.692 4.077 5.089	S14-01568-06
	2	96.1 101.9	BBCH 39-45 BBCH 69	Straw Straw Straw Straw	53 60 67 74	3.874 4.903 4.493 3.267	
66750, Saint-Cyprien, Pyrénées Orientales, France SEU 2014 (Winter wheat / Babylone)	2	94.2 100.0	BBCH 61 BBCH 69	Straw	36	11.967	S14-01568-07
	2	95.0 104.2	BBCH 61 BBCH 69	Straw	36	8.308	
44492, Lagueruela, Aragon, Spain SEU 2014 (Spring wheat / Marius)	2	105.6 103.9	BBCH 39-45 BBCH 69	Straw	41	7.095	S14-01568-08
	2	102.8 101.7	BBCH 39-45 BBCH 69	Straw	41	5.811	
45300, Loiret, Rouvres-Saint-Jean France NEU 2014 (Winter wheat / Arezzo)	2	102.2 98.8	BBCH 51 BBCH 69	Straw Straw Straw Straw	43 50 57 64	0.729 0.879 1.160 0.972	S14-01569-01
	2	97.1 99.4	BBCH 51 BBCH 69	Straw Straw Straw Straw	43 50 57 64	2.675 3.110 3.455 3.125	
CV9 3DT, Atherstone, Warwickshire UK NEU 2014 (Spring wheat / Mulika)	2	99.4 105.6	BBCH 61 BBCH 69	Straw Straw Straw Straw	28 35 42 49	0.999 1.029 1.145 1.182	S14-01569-02
	2	98.3 99.4	BBCH 61 BBCH 69	Straw Straw Straw Straw	28 35 42 49	6.312 5.600 6.800 6.200	
	2	101.7 120.8	BBCH 55 BBCH 69	Straw	45	0.594	S14-01569-03

Location, year, (variety	Application			Residues			Reference
	No	kg/ha (ai)	GS	Portion analysed	DALT (days)	Fenpicoxamid (mg/kg)	
DE73 7FW, Twyford, Derbyshire, UK NEU 2014 (Winter wheat / KWS Santiago)	2	103.3 115.0	BBCH 55 BBCH 69	Straw	45	5.337	
71691, Freiberg, Baden-Württemberg, Germany NEU 2014 (Spring wheat / Triso)	2	97.5 103.3	BBCH 61 BBCH 69	Straw	42	1.486	S14-01569-04
	2	96.7 105.8	BBCH 61 BBCH 69	Straw	42	9.912	
40054, Prunaro Di Budrio, Province of Bologna, Italy SEU 2014 (Winter wheat / Masarrio)	2	96.7 105.9	BBCH 53 BBCH 69	Straw Straw Straw Straw	49 55 62 67	1.421 1.768 0.023 0.030	S14-01569-05
	2	103.0 97.0	BBCH 53 BBCH 69	Straw Straw Straw Straw	49 55 62 67	2.563 1.409 0.425 0.143	
44492 Fonfría, Teruel, Spain SEU 2014 (Spring wheat / Marius)	2	95.0 101.4	BBCH 39-45 BBCH 69	Straw Straw Straw Straw	53 60 67 74	2.935 3.224 2.596 2.328	S14-01569-06
	2	99.4 105.3	BBCH 39-45 BBCH 69	Straw Straw Straw Straw	53 60 67 74	017 8.349 8.698 7.495 7.185	
66750, Saint-Cyprien, Pyrénées Orientales, France SEU 2014 (Winter wheat / Babylone)	2	94.2 105.0	BBCH 61 BBCH 69	Straw	36	4.669	S14-01569-07
	2	98.3 105.0	BBCH 61 BBCH 69	Straw	36	9.590	
44492, Lagueruela, Aragon, Spain	2	107.2 105.0	BBCH 39-45 BBCH 69	Straw	41	3.583	S14-01569-08

Location, year, (variety	Application			Residues			Reference
	No	kg/ha (ai)	GS	Portion analysed	DALT (days)	Fenpicoxamid (mg/kg)	
SEU 2014	2	106.1 97.2	BBCH 39-45 BBCH 69	Straw	41	10.560	
HU17 8JF, Bishop Burton, East Yorkshire, UK NEU 2015 (Winter wheat / Reflection)	2	102.1 97.6	BBCH 61 BBCH 69	Straw Straw Straw Straw	45 51 59 65	1.155 0.457 0.712 0.839	S15-02628-01
L39 6SX, Lydiate, Lancashire, UK NEU 2015 (Winter wheat / KWS Kielder)	2	110.0 106.7	BBCH 65-67 BBCH 69	Straw	42	2.380	S15-02628-02
67140 Saint-Pierre, Alsace, Bas-Rhin, France NEU 2015 (Winter wheat / Pakilo)	2	95.8 100.4	BBCH 59 BBCH 69	Straw	42	2.690	S15-02628-03
86120 Morton, Vienne, France SEU 2015 (Winter wheat / Cellule)	2	100.0 103.2	BBCH 56 BBCH 69	Straw Straw Straw Straw	39 45 51 59	2.655 2.316 3.698 3.612	S15-02628-04
71290 Simandre, Saône-et-Loire, France SEU 2015 (Spring wheat / Sensus)	2	108.7 93.2	BBCH 59 BBCH 69	Straw	26	2.488	S15-02628-05
50367 Retascón, Spain SEU 2015 (Winter wheat / Garcia)	2	108.3 92.9	BBCH 47 BBCH 67-69	Straw	41	6.260	S15-02628-06

Location, year, (variety	Application			Residues			Reference
	No	kg/ha (ai)	GS	Portion analysed	DALT (days)	Fenpicoxamid (mg/kg)	
44492 Fonfría, Teruel, Spain SEU 2015 (Winter wheat / Marius)	2	91.9 95.6	BBCH 57-61 BBCH 69	Straw	50	2.454	S15-02628-07
NG34 8PE, Silk Willoughby, Lincolnshire, UK NEU 2015 (Spring wheat / Mulika)	2	105.4 108.3	BBCH 39 BBCH 69	Straw Straw Straw Straw	44 51 58 65	2.671 3.828 4.557 5.174	S15-02628-08
LE12 5RQ, West Leake, Leicestershire, UK NEU 2015 (Spring wheat / Mulika)	2	112.5 95.0	BBCH 61 BBCH 69	Straw	46	0.762	S15-02628-09
40057, Granarolo, Emilia-Romagna, Italy SEU 2015 (Spring wheat / Aquilante)	2	105.1 108.4	BBCH 51 BBCH 69	Straw Straw Straw Straw	33 40 47 54	0.892 1.193 0.722 1.265	S15-02628-10
HU17 8JF, Bishop Burton, East Yorkshire, UK NEU 2015	2	99.0 98.3	BBCH 61 BBCH 69	Straw Straw Straw Straw	45 51 59 65	0.713 0.487 0.767 0.198	S15-02629-01
L39 6SX, Lydiate, Lancashire, UK NEU 2015 (Winter wheat / KWS Kielder)	2	108.3 110.0	BBCH 65-67 BBCH 69	Straw	42	1.465	S15-02629-02
67140 Saint-Pierre, Alsace, Bas-Rhin, France NEU 2015 (Winter wheat / Pakilo)	2	98.8 102.7	BBCH 59 BBCH 69	Straw	42	2.580	S15-02629-03

Location, year, (variety	Application			Residues			Reference
	No	kg/ha (ai)	GS	Portion analysed	DALT (days)	Fenpicoxamid (mg/kg)	
86120 Morton, Vienne, France SEU 2015 (Winter wheat / Cellule)	2	94.7 105.0	BBCH 56 BBCH 69	Straw Straw Straw Straw	39 45 51 59	3.571 2.693 4.168 4.742	S15-02629-04
71290 Simandre, Saône-et-Loire, France SEU 2015 (Spring wheat / Sensus)	2	105.3 93.0	BBCH 59 BBCH 69	Straw	26	2.742	S15-02629-05
50367 Retascón, Spain SEU 2015 (Winter wheat / Garcia)	2	95.2 92.9	BBCH 47 BBCH 67-69	Straw	41	2.124	S15-02629-06
44492 Fonfría, Teruel, Spain SEU 2015 (Winter wheat / Marius)	2	108.1 95.6	BBCH 57-61 BBCH 69	Straw	50	2.048	S15-02629-07
NG34 8PE, Silk Willoughby, Lincolnshire, UK 2015 (Spring wheat / Mulika)	2	108.3 107.5	BBCH 39 BBCH 69	Straw Straw Straw Straw	44 51 58 65	3.134 3.589 4.702 6.263	S15-02629-08
LE12 5RQ, West Leake, Leicestershire, UK NEU 2015	2	115.0 96.7	BBCH 61 BBCH 69	Straw	46	1.148	S15-02629-09
40057, Granarolo, Emilia-Romagna, Italy SEU 2015 (Spring wheat /Aquilante)	2	109.1 108.9	BBCH 51 BBCH 69	Straw Straw Straw Straw	33 40 47 54	0.979 1.579 1.750 4.728	S15-02629-10

Table 9 Results of fenpicoxamid residue trials in forage

Location, year, (variety	Application			Residues			Reference
	No	kg/ha (ai)	GS	Portion analysed	DALT (days)	Fenpicoxamid (mg/kg)	
45300, Loiret, Rouvres-Saint-Jean France NEU 2014 (Winter wheat / Arezzo)	2	99.9 98.4	55 69	Whole plant Whole plant Whole plant Whole plant Whole plant	-0 0 8 14 28	0.36 1.6 0.37 0.37 0.35	S14-01568-01
	2	101.1 101.6	BBCH 55 BBCH 69	Whole plant Whole plant Whole plant Whole plant Whole plant	-0 0 8 14 28	0.2 1.6 0.36 0.4 0.30	
CV9 3DT, Atherstone, Warwickshire UK NEU 2014 (Spring wheat / Mulika)	2	98.3 104.4	BBCH 61 BBCH 69	Whole plant Whole plant Whole plant Whole plant Whole plant	-0 0 7 14 28	0.25 1.3 0.5 0.6 0.4	S14-01568-02
	2	102.8 104.4	BBCH 61 BBCH 69	Whole plant Whole plant Whole plant Whole plant Whole plant	-0 0 7 14 28	0.152 1.171 0.73 0.45 0.42	
DE73 7FW, Twyford, Derbyshire, UK NEU 2014 (Winter wheat / KWS Santiago)	2	101.6 99.2	BBCH 55 BBCH 69	Whole plant	-	Not analysed	S14-01568-03
	2	100.8 100.0	BBCH 55 BBCH 69	Whole plant	-	Not analysed	
71691, Freiberg, Baden-Württemberg, Germany NEU GLP 2014 (Spring wheat / Triso	2	100.0 95.8	BBCH 61 BBCH 69	Whole plant	-	Not analysed	S14-01568-04
	2	100.0 95.8	BBCH 61 BBCH 69	Whole plant	-	Not analysed	

Location, year, (variety	Application			Residues			Reference
	No	kg/ha (ai)	GS	Portion analysed	DALT (days)	Fenpicoxamid (mg/kg)	
40054, Prunaro Di Budrio, Province of Bologna, Italy SEU 2014 (Winter wheat / Masarrio)	2	100.7 99.3	BBCH 53 BBCH 69	Whole plant Whole plant Whole plant Whole plant Whole plant	-0 0 6 14 28	0.61 1.6 0.97 0.65 0.71	S14-01568-05
	2	97.4 103.3	BBCH 53 BBCH 69	Whole plant Whole plant Whole plant Whole plant Whole plant	-0 0 6 14 28	0.5 1.9 0.87 0.52 0.50	
44492 Fonfría, Teruel, Spain SEU GLP 2014 (Spring wheat / Marius)	2	95.6 98.9	BBCH 39-45 BBCH 69	Whole plant Whole plant Whole plant Whole plant Whole plant	-0 0 6 14 26	0.82 2.8 2.5 1.9 1	S14-01568-06
	2	96.1 101.9	BBCH 39–45 BBCH 69	Whole plant Whole plant Whole plant Whole plant Whole plant	-0 0 6 14 26	0.87 2.4 2.3 1.6 0.77	
66750, Saint-Cyprien, Pyrénées-Orientales, France SEU 2014 (Winter wheat / Babylone)	2	94.2 100.0	BBCH 61 BBCH 69	Whole plant	-	Not analysed	S14-01568-07
	2	95.0 104.2	BBCH 61 BBCH 69	Whole plant	-	Not analysed	
44492, Lagueruela, Aragon, Spain SEU 2014 (Spring wheat / Marius)	2	105.6 103.9	BBCH 39–45 BBCH 69	Whole plant	-	Not analysed	S14-01568-08
	2	102.8 101.7	BBCH 39–45 BBCH 69	Whole plant	-	Not analysed	
45300, Loiret, Rouvres-Saint-Jean France NEU 2014	2	102.2 98.8	BBCH 51 BBCH 69	Whole plant Whole plant Whole plant Whole plant Whole plant	-0 0 8 14 28	0.35 1.48 0.40 0.28 0.22	S14-01569-01

Location, year, (variety	Application			Residues			Reference
	No	kg/ha (ai)	GS	Portion analysed	DALT (days)	Fenpicoxamid (mg/kg)	
(Winter wheat / Arezzo)	2	97.1 99.4	BBCH 51 BBCH 69	Whole plant Whole plant Whole plant Whole plant Whole plant	-0 0 8 14 28	0.65 1.7 0.88 0.57 0.66	
CV9 3DT, Atherstone, Warwickshire UK NEU 2014 (Spring wheat / Mulika)	2	99.4 105.6	BBCH 61 BBCH 69	Whole plant Whole plant Whole plant Whole plant Whole plant	-0 0 7 14 28	0.16 1 0.52 0.26 0.32	S14-01569-02
	2	98.3 99.4	BBCH 61 BBCH 69	Whole plant Whole plant Whole plant Whole plant Whole plant	-0 0 7 14 28	1.1 1.5 1.5 1.4 2.2	
DE73 7FW, Twyford, Derbyshire, UK NEU 2014 (Winter wheat / KWS Santiago)	2	101.7 120.8	BBCH 55 BBCH 69	Whole plant	-	Not analysed	S14-01569-03
	2	103.3 115.0	BBCH 55 BBCH 69	Whole plant	-	Not analysed	
71691, Freiberg, Baden-Württemberg, Germany NEU 2014 (Spring wheat / Triso)	2	97.5 103.3	BBCH 61 BBCH 69	Whole plant	-	Not analysed	S14-01569-04
	2	96.7 105.8	BBCH 61 BBCH 69	Whole plant	-	Not analysed	
40054, Prunaro Di Budrio, Province of Bologna, Italy SEU 2014 (Winter wheat / Masarrio)	2	96.7 105.9	BBCH 53 BBCH 69	Whole plant Whole plant Whole plant Whole plant Whole plant	-0 0 6 14 28	0.51 2.4 1.2 0.5 0.4	S14-01569-05
	2	103.0 97.0	BBCH 53 BBCH 69	Whole plant Whole plant Whole plant Whole plant Whole plant	-0 0 6 14 28	1.0 2.0 1.2 1.0 0.77	

Location, year, (variety	Application			Residues			Reference
	No	kg/ha (ai)	GS	Portion analysed	DALT (days)	Fenpicoxamid (mg/kg)	
44492 Fonfría, Teruel, Spain SEU 2014 (Spring wheat / Marius)	2	95.0 101.4	BBCH 39–45 BBCH 69	Whole plant Whole plant Whole plant Whole plant Whole plant	-0 0 6 14 26	0.6 3.1 1.8 1.0 0.58	S14-01569-06
	2	99.4 105.3	BBCH 39–45 BBCH 69	Whole plant Whole plant Whole plant Whole plant Whole plant	-0 0 6 14 26	0.9 2.3 2.4 1.6 1.3	
66750, Saint-Cyprien, Pyrénées-Orientales, France SEU 2014 (Winter wheat / Babylone)	2	94.2 105.0	BBCH 61 BBCH 69	Whole plant	-	Not analysed	S14-01569-07
	2	98.3 105.0	BBCH 61 BBCH 69	Whole plant	-	Not analysed	
44492, Lagueruela, Aragon, Spain SEU 2014	2	107.2 105.0	BBCH 39-45 BBCH 69	Whole plant	-	Not analysed	S14-01569-08
	2	106.1 97.2	BBCH 39–45 BBCH 69	Whole plant	-	Not analysed	
HU17 8JF, Bishop Burton, East Yorkshire, UK NEU 2015 (Winter wheat / Reflection)	2	102.1 97.6	BBCH 61 BBCH 69	Whole plant Whole plant Whole plant Whole plant Whole plant	-0 0 7 14 28	0.28 1.12 0.48 0.31 0.32	S15-02628-01
L39 6SX, Lydiate, Lancashire, UK NEU 2015 (Winter wheat / KWS Kielder)	2	110.0 106.7	BBCH 65–67 BBCH 69	Whole plant	-	Not analysed	S15-02628-02
67140 Saint-Pierre, Alsace, Bas-Rhin, France NEU 2015 (Winter wheat / Pakilo)	2	95.8 100.4	BBCH 59 BBCH 69	Whole plant	-	Not analysed	S15-02628-03

Location, year, (variety	Application			Residues			Reference
	No	kg/ha (ai)	GS	Portion analysed	DALT (days)	Fenpicoxamid (mg/kg)	
86120 Morton, Vienne, France SEU 2015 (Winter wheat / Cellule)	2	100.0 103.2	BBCH 56 BBCH 69	Whole plant Whole plant Whole plant Whole plant Whole plant	-0 0 7 14 30	0.67 2.35 1.51 0.53 0.81	S15-02628-04
71290 Simandre, Saône-et-Loire, France SEU 2015 (Spring wheat / Sensus)	2	108.7 93.2	BBCH 59 BBCH 69	Whole plant	-	Not analysed	S15-02628-05
50367 Retascón, Spain SEU 2015 (Winter wheat / Garcia)	2	108.3 92.9	BBCH 47 BBCH 67–69	Whole plant	-	Not analysed	S15-02628-06
44492 Fonfría, Teruel, Spain SEU 2015 (Winter wheat / Marius)	2	91.9 95.6	BBCH 57–61 BBCH 69	Whole plant	-	Not analysed	S15-02628-07
NG34 8PE, Silk Willoughby, Lincolnshire, UK NEU 2015 (Spring wheat / Mulika)	2	105.4 108.3	BBCH 39 BBCH 69	Whole plant Whole plant Whole plant Whole plant	0 7 14 30	1.1 0.8 0.41 0.62	S15-02628-08
LE12 5RQ, West Leake, Leicestershire, UK NEU 2015 (Spring wheat / Mulika)	2	112.5 95.0	BBCH 61 BBCH 69	Whole plant	-	Not analysed	S15-02628-09

Location, year, (variety	Application			Residues			Reference
	No	kg/ha (ai)	GS	Portion analysed	DALT (days)	Fenpicoxamid (mg/kg)	
40057, Granarolo, Emilia-Romagna, Italy SEU 2015 (Spring wheat / Aquilante)	2	105.1 108.4	BBCH 51 BBCH 69	Whole plant Whole plant Whole plant Whole plant Whole plant	-0 0 7 14 28	0.33 1.36 0.24 0.19 0.17	S15-02628-10
HU17 8JF, Bishop Burton, East Yorkshire, UK NEU 2015	2	99.0 98.3	BBCH 61 BBCH 69	Whole plant Whole plant Whole plant Whole plant Whole plant	-0 0 7 14 28	0.29 1.66 0.42 0.27 0.22	S15-02629-01
L39 6SX, Lydiate, Lancashire, UK NEU 2015 (Winter wheat / KWS Kielder)	2	108.3 110.0	BBCH 65–67 BBCH 69	Whole plant	-	Not analysed	S15-02629-02
67140 Saint-Pierre, Alsace, Bas-Rhin, France NEU 2015 (Winter wheat / Pakilo)	2	98.8 102.7	BBCH 59 BBCH 69	Whole plant	-	Not analysed	S15-02629-03
86120 Morton, Vienne, France SEU 2015 (Winter wheat / Cellule)	2	94.7 105.0	BBCH 56 BBCH 69	Whole plant Whole plant Whole plant Whole plant Whole plant	-0 0 7 14 30	0.67 2.8 1.57 0.8 1.6	S15-02629-04
71290 Simandre, Saône-et-Loire, France SEU 2015 (Spring wheat / Sensus)	2	105.3 93.0	BBCH 59 BBCH 69	Whole plant	-	Not analysed	S15-02629-05

Location, year, (variety	Application			Residues			Reference
	No	kg/ha (ai)	GS	Portion analysed	DALT (days)	Fenpicoxamid (mg/kg)	
50367 Retascón, Spain SEU 2015 (Winter wheat / Garcia)	2	95.2 92.9	BBCH 47 BBCH 67–69	Whole plant	-	Not analysed	S15-02629-06
44492 Fonfría, Teruel, Spain SEU 2015 (Winter wheat / Marius)	2	108.1 95.6	BBCH 57–61 BBCH 69	Whole plant	-	Not analysed	S15-02629-07
NG34 8PE, Silk Willoughby, Lincolnshire, UK 2015 (Spring wheat / Mulika)	2	108.3 107.5	BBCH 39 BBCH 69	Whole plant Whole plant Whole plant Whole plant	0 7 14 30	1.65 1.6 0.83 0.83	S15-02629-08
LE12 5RQ, West Leake, Leicestershire, UK NEU 2015	2	115.0 96.7	BBCH 61 BBCH 69	Whole plant	-	Not analysed	S15-02629-09
40057, Granarolo, Emilia-Romagna, Italy SEU 2015 (Spring wheat / Aquilante)	2	109.1 108.9	BBCH 51 BBCH 69	Whole plant Whole plant Whole plant Whole plant Whole plant	-0 0 7 14 28	0.87 2.5 0.45 0.68 0.31	S15-02629-10

FATE OF RESIDUES IN STORAGE AND PROCESSING

In Storage

Further data were not provided.

In Processing

Eight processing trials with fenpicoxamid on wheat were conducted in Europe during 2012 and 2014. In treated plots GF-2925, a suspension concentrate (SC) formulation containing fenpicoxamid (130 g ai/L), was applied in 2 post-emergent foliar applications with a 14-day interval between applications and with the last application targeted at BBCH 69. Fenpicoxamid was applied at a nominal rate of 130 g ai/ha or 390 g ai/ha in each of the two applications (1.3–3.9 times the maximum label rate of 2 × 100 g ai/ha).

Wheat grain balk samples (approximately 100 kg) were taken at the NCH (corresponding to BBCH 89). All samples were stored at ambient temperature prior to processing (max. 6–15 days). Processed products/fractions produced in this study included shorts, coarse bran, fine bran, total bran, middlings, refined flour (Type 550), white bread, wholemeal flour, wholemeal bread, dried gluten, dried starch, gluten feed meal and wheat germs. "Total bran", is a 1:1 mixture of fine bran and coarse bran and is representative of the bran fraction normally produced commercially.

Processed samples were stored frozen for a maximum of 744 days (*ca.* 24 months) prior to analysis. Storage stability in wheat and processed fractions was demonstrated for 741 days (RAC) to 745 days (processed commodities). At 741 days in grain, straw and forage generally high recoveries were reported (92–105%), demonstrating no decline in stability. A recovery of 70% for X642188 in straw was observed, but taking into account the procedural recovery of 90%, the corrected stored recovery is 78%. Therefore, an additional 3-days storage is not likely to have an impact on the stability of fenpicoxamid or X642188.

In the 4 trials conducted in 2012 (S12-01369), residue analysis was performed according to the method validated in study 120615. This method has been validated in various crop matrices, including wheat grain, straw, bran, flour and bread, with an LOQ of 0.01 mg/kg for each analyte, by the 2018 JMPR. The performance of the method was verified by obtaining mean procedural recoveries of 81.1–107.5% and 87.3–110.3% for fenpicoxamid and X642188 respectively at fortification levels of 0.01–0.10 mg/kg.

In the 4 trials conducted in 2014 (S14-02186), residue analysis of fenpicoxamid and X642188, X12335723, X12019520, X12314005 and X12264475 was performed according to the method validated within the study. This method has been validated in with an LOQ of 0.01 mg/kg for each analyte. The performance of the method was verified by obtaining mean procedural recoveries of 91.3–102.5% for parent, 90.6–100.5% for X642188, 81–96.9% for X12335723, 89.9–101.3% for X12019520, 91.6–99.3% for X12314005 and 77.3–93.3 % for X12264475 at fortification levels of 0.01–0.10 mg/kg.

The data demonstrate that residues of parent do not concentrate in processed fractions. Processing factors have been calculated for parent in Table 10 and 11. All processing factors are <1, apart from coarse bran (PF ~1.3), but it is noted that the representative commodity is "total bran" for which the processing factor is <1.

Table 10 Fenpicoxamid and X642188 residues in wheat and its processed commodities from supervised trials in 2012 in Europe (S12-01369)

Winter wheat country, year (variety)	Application				PHI (days)	Mean residues (mg/kg)				Reference
	Formulation	No	g/ha (ai)	BBCH at last application		Portion analysed	Fenpicoxamid	X642188	Pf [a]	
Germany. 2012 (Tabasco)	SC	2	130 129	69	54	grain, not cleaned (from processor) [RAC]	0.077	<0.01		S12-01369 01 Plot 2
						cleaned grain	0.024	<0.01		
						shorts	0.027	<0.01		
						fine bran	0.022	<0.01		
						coarse bran	0.069	<0.01		
						total bran	0.044	<0.01	0.57	
						middlings	0.021	<0.01		

Winter wheat country, year (variety)	Application				PHI (days)	Mean residues (mg/kg)				Reference
	Formulation	No	g/ha (ai)	BBCH at last application		Portion analysed	Fenpicoxamid	X642188	Pf [a]	
						refined flour (type 550)	0.020	<0.01	0.26	
						white bread	<0.01	<0.01	<0.13	
						wholemeal flour	0.028	<0.01	0.36	
						wholemeal bread	0.011	<0.01	0.14	
						dried starch	<0.01	<0.01	<0.13	
						dried gluten	0.025	<0.01	0.32	
						gluten feed meal	0.012	<0.01		
						wheat germ	<0.01	0.031	<0.13	
Germany. 2012 (Tabasco)	SC	2	393 391	69	54	grain, not cleaned (from processor) [RAC]	0.253	<0.01		S12-0136901 Plot 3
						cleaned grain	0.143	<0.01		
						shorts	0.120	<0.01		
						fine bran	0.051	<0.01		
						coarse bran	0.355	0.022		
						total bran	0.218	0.015	0.86	
						middlings	0.053	<0.01		
						refined flour (type 550)	0.088	<0.01	0.35	
						white bread	0.032	<0.01	0.13	
						wholemeal flour	0.111	0.012	0.44	
						wholemeal bread	0.046	0.012	0.18	
						dried starch	<0.01	<0.01	<0.04	
						dried gluten	0.132	0.065	0.52	
						gluten feed meal	0.073	0.028		
						wheat germ	0.020	0.048	0.08	
Germany. 2012 (Tabasco)	GF-2925, SC	2	129 134	69	60	grain, not cleaned (from processor) [RAC]	0.061	<0.01		S12-0136902 Plot 2
						cleaned grain	0.026	<0.01		
						shorts	0.060	<0.01		
						fine bran	0.018	<0.01		
						coarse bran	0.051	<0.01		
						total bran	0.034	<0.01	0.56	

Winter wheat country, year (variety)	Application				PHI (days)	Mean residues (mg/kg)				Reference
	Formulation	No	g/ha (ai)	BBCH at last application		Portion analysed	Fenpicoxamid	X642188	Pf [a]	
						middlings	0.016	<0.01		
						refined flour (type 550)	0.027	<0.01	0.44	
						white bread	0.010	<0.01	0.16	
						wholemeal flour	0.023	<0.01	0.38	
						wholemeal bread	<0.01	<0.01	<0.16	
						dried starch	<0.01	<0.01	<0.16	
						dried gluten	<0.01	0.014	<0.16	
						gluten feed meal	<0.01	<0.01		
						wheat germ	<0.01	0.017	<0.16	
UK, 2012 (Glasgow)	SC	2	405 386	69	60	grain, not cleaned (from processor) [RAC]	0.316	0.017		S12-0136902 Plot 3
						cleaned grain	0.139	0.010		
						shorts	0.158	<0.01		
						fine bran	0.080	<0.01		
						coarse bran	0.265	0.020		
						total bran	0.194	0.017	0.61	
						middlings	0.071	<0.01		
						refined flour (type 550)	0.097	<0.01	0.31	
						white bread	0.036	<0.01	0.11	
						wholemeal flour	0.120	0.012	0.38	
						wholemeal bread	0.062	<0.01	0.2	
						dried starch	0.014	<0.01	0.04	
						dried gluten	0.048	0.036	0.15	
						gluten feed meal	0.034	0.019		
						wheat germ	0.050	0.087	0.16	
Spain, 2012 (Botticelli)	SC	2	128 131	69	39	grain, not cleaned (from processor) [RAC]	0.183	<0.01		S12-0136903 Plot 2
						cleaned grain	0.085	<0.01		
						shorts	0.072	<0.01		
						fine bran	0.058	<0.01		
						coarse bran	0.258	<0.01		

Winter wheat country, year (variety)	Application				PHI (days)	Mean residues (mg/kg)				Reference
	Formulation	No	g/ha (ai)	BBCH at last application		Portion analysed	Fenpicoxamid	X642188	Pf [a]	
						total bran	0.170	<0.01	0.93	
						middlings	0.080	<0.01		
						refined flour (type 550)	0.040	<0.01	0.22	
						white bread	0.017	<0.01	0.09	
						wholemeal flour	0.074	<0.01	0.4	
						wholemeal bread	0.036	<0.01	0.2	
						dried starch	<0.01	<0.01	<0.05	
						dried gluten	0.033	0.014	0.18	
						gluten feed meal	0.021	<0.01		
						wheat germ	0.021	0.051	0.11	
Spain, 2012 (Botticelli)	SC	2	386 393	69	39	grain, not cleaned (from processor) [RAC]	0.296	<0.01		S12-01369 03 Plot 3
						cleaned grain	0.180	<0.01		
						shorts	0.092	<0.01		
						fine bran	0.161	<0.01		
						coarse bran	0.547	0.022		
						total bran	0.368	0.015	1.24	
						middlings	0.188	<0.01		
						refined flour (type 550)	0.083	<0.01	0.28	
						white bread	0.038	<0.01	0.13	
						wholemeal flour	0.170	<0.01	0.57	
						wholemeal bread	0.069	<0.01	0.23	
						dried starch	<0.01	<0.01	<0.03	
						dried gluten	0.091	0.038	0.31	
						gluten feed meal	0.058	0.013		
						wheat germ	0.077	0.113	0.26	
Spain, 2012 (Botticelli)	SC	2	123 123	69	40	grain, not cleaned (from processor) [RAC]	0.056	<0.01		S12-01369 04 Plot 2
						cleaned grain	0.030	<0.01		
						shorts	0.021	<0.01		
						fine bran	0.015	<0.01		

Winter wheat country, year (variety)	Application				PHI (days)	Mean residues (mg/kg)				Reference
	Formulation	No	g/ha (ai)	BBCH at last application		Portion analysed	Fenpicoxamid	X642188	Pf [a]	
						coarse bran	0.102	<0.01		
						total bran	0.070	<0.01	1.25	
						middlings	0.022	<0.01		
						refined flour (type 550)	0.014	<0.01	0.25	
						white bread	<0.01	<0.01	<0.18	
						wholemeal flour	0.023	<0.01	0.41	
						wholemeal bread	0.010	<0.01	0.18	
						dried starch	<0.01	<0.01	<0.18	
						dried gluten	0.016	<0.01	0.29	
						gluten feed meal	<0.01	<0.01		
						wheat germ	<0.01	0.028	<0.18	
Spain, 2012 (Botticelli	SC	2	382 376	69	40	grain, not cleaned (from processor) [RAC]	0.306	<0.01		S12-0136904 Plot 3
						cleaned grain	0.112	<0.01		
						shorts	0.047	<0.01		
						fine bran	0.070	<0.01		
						coarse bran	0.359	0.027		
						total bran	0.240	0.015	0.78	
						middlings	0.088	<0.01		
						refined flour (type 550)	0.046	<0.01	0.15	
						white bread	0.022	<0.01	0.07	
						wholemeal flour	0.084	<0.01	0.27	
						wholemeal bread	0.039	<0.01	0.13	
						dried starch	<0.01	<0.01	<0.03	
						dried gluten	0.052	0.023	0.17	
						gluten feed meal	0.035	<0.01		
						wheat germ	0.045	0.105	0.15	

[a] the Processing Factor values are listed with a less than sign (<) if the residue value for the processed product used in the calculation was below the LOQ of 0.01. In cases when the processed product residue was below the LOQ (<0.01 mg/kg), the value of 0.01 mg/kg was used in the calculation or if the residue value was below the LOQ.

Table 11 Fenpicoxamid and metabolite residues in wheat and its processed commodities from supervised trials in 2014 in Europe (S14-02186)

Winter wheat country, year (variety)	Application				PHI (days)	Mean residues (mg/kg)								Reference
	Formulation	No	g/ha (ai)	BBCH at last application		Portion analysed	Fenpicoxamid	X642188	X12335723	X12019520	X12314005	X12264475	Pf [a]	
Germany. 2014 (Asano)	SC	2	137 133	69	57	grain, not cleaned (from processor) [RAC]	0.034	<0.01	<0.01	<0.01	<0.01	<0.01		S14-02186 01 Plot 2
						cleaned grain	0.021	<0.01	<0.01	<0.01	<0.01	<0.01		
						shorts	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01		
						fine bran	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01		
						coarse bran	0.061	<0.01	<0.01	<0.01	<0.01	<0.01		
						total bran	0.031	<0.01	<0.01	<0.01	<0.01	<0.01	0.91	
						middlings	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01		
						refined flour (type 550)	0.010	<0.01	<0.01	<0.01	<0.01	<0.01	0.29	
						white bread	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.29	
						wholemeal flour	0.017	<0.01	<0.01	<0.01	<0.01	<0.01	0.5	
						wholemeal bread	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.29	
						dried starch	ND	<0.01	<0.01	<0.01	<0.01	<0.01	<0.29	
						dried gluten	ND	<0.01	<0.01	<0.01	<0.01	<0.01	<0.29	
						gluten feed meal	ND	<0.01	<0.01	<0.01	<0.01	<0.01		
						wheat germ	ND	<0.01	<0.01	<0.01	<0.01	<0.01	<0.29	
UK, 2014 (Solstice)	SC	2	122 132	69	77	grain, not cleaned (from processor) [RAC]	0.050	<0.01	<0.01	<0.01	<0.01	<0.01		S14-02186 02 Plot 2
						cleaned grain	0.020	<0.01	<0.01	<0.01	<0.01	<0.01		
						shorts	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01		
						fine bran	0.011	<0.01	<0.01	<0.01	<0.01	<0.01		

Winter wheat country, year (variety)	Application				PHI (days)	Mean residues (mg/kg)								Reference
	Formulation	No	g/ha (ai)	BBCH at last application		Portion analysed	Fenpicoxamid	X642188	X12335723	X12019520	X12314005	X12264475	Pf [a]	
						coarse bran	0.043	<0.01	<0.01	<0.01	<0.01	<0.01		
						total bran	0.025	<0.01	<0.01	<0.01	<0.01	<0.01	0.5	
						middlings	0.010	<0.01	<0.01	<0.01	<0.01	<0.01		
						refined flour (type 550)	0.010	<0.01	<0.01	<0.01	<0.01	<0.01	0.2	
						white bread	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.2	
						wholemeal flour	0.013	<0.01	<0.01	<0.01	<0.01	<0.01	0.26	
						wholemeal bread	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.2	
						dried starch	ND	<0.01	<0.01	<0.01	<0.01	<0.01	<0.2	
						dried gluten	ND	<0.01	<0.01	<0.01	<0.01	0.012	<0.2	
						gluten feed meal	ND	<0.01	<0.01	<0.01	<0.01	<0.01		
						wheat germ	ND	<0.01	<0.01	<0.01	<0.01	<0.01	<0.2	
Spain, 2014 (Marius)	SC	2	129 128	69	55	grain, not cleaned (from processor) [RAC]	0.042	<0.01	<0.01	<0.01	<0.01	<0.01		S14-02186 03 Plot 2
						cleaned grain	0.028	<0.01	<0.01	<0.01	<0.01	<0.01		
						shorts	0.011	<0.01	<0.01	<0.01	<0.01	<0.01		
						fine bran	0.016	<0.01	<0.01	<0.01	<0.01	<0.01		
						coarse bran	0.049	<0.01	<0.01	<0.01	<0.01	<0.01		
						total bran	0.037	<0.01	<0.01	<0.01	<0.01	<0.01	0.88	
						middlings	0.014	<0.01	<0.01	<0.01	<0.01	<0.01		
						refined flour (type 550)	0.015	<0.01	<0.01	<0.01	<0.01	<0.01	0.36	
						white bread	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.24	

Winter wheat country, year (variety)	Application				PHI (days)	Mean residues (mg/kg)								Reference
	Formulation	No	g/ha (ai)	BBCH at last application		Portion analysed	Fenpicoxamid	X642188	X12335723	X12019520	X12314005	X12264475	Pf [a]	
						wholemeal flour	0.025	<0.01	<0.01	<0.01	<0.01	<0.01	0.6	
						wholemeal bread	0.011	<0.01	<0.01	<0.01	<0.01	<0.01	0.26	
						dried starch	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.24	
						dried gluten	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.24	
						gluten feed meal	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01		
						wheat germ	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.24	
France, 2014 (Ingenio)	SC	2	121 130	69	50	grain, not cleaned (from processor) [RAC]	0.035	<0.01	<0.01	<0.01	<0.01	<0.01		S14-02186 04 Plot 2
						cleaned grain	0.023	<0.01	<0.01	<0.01	<0.01	<0.01		
						shorts	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01		
						fine bran	0.021	<0.01	<0.01	<0.01	<0.01	<0.01		
						coarse bran	0.095	<0.01	<0.01	<0.01	<0.01	<0.01		
						total bran	0.061	<0.01	<0.01	<0.01	<0.01	<0.01	1.74	
						middlings	0.018	<0.01	<0.01	<0.01	<0.01	<0.01		
						refined flour (type 550)	0.012	<0.01	<0.01	<0.01	<0.01	<0.01	0.34	
						white bread	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.29	
						wholemeal flour	0.025	<0.01	<0.01	<0.01	<0.01	<0.01	0.71	
						wholemeal bread	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.29	
						dried starch	ND	<0.01	<0.01	<0.01	<0.01	<0.01	<0.29	
						dried gluten	ND	<0.01	<0.01	<0.01	<0.01	<0.01	<0.29	
						gluten feed meal	ND	<0.01	<0.01	<0.01	<0.01	<0.01		
						wheat germ	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.29	

[b] the Processing Factor values are listed with a less than sign (<) if the residue value for the processed product used in the calculation was below the LOQ of 0.01. In cases when the processed product residue was below the LOQ (<0.01 mg/kg), the value of 0.01 mg/kg was used in the calculation or if the residue value was below the LOQ.

Flowcharts illustrating the (a) wheat cleaning process (b) refined flour (Type 550) process (c) white bread process (d) wholemeal flour (e) wholemeal bread process and (f) gluten, starch and gluten feed meal are presented in Figures 1–6, respectively.

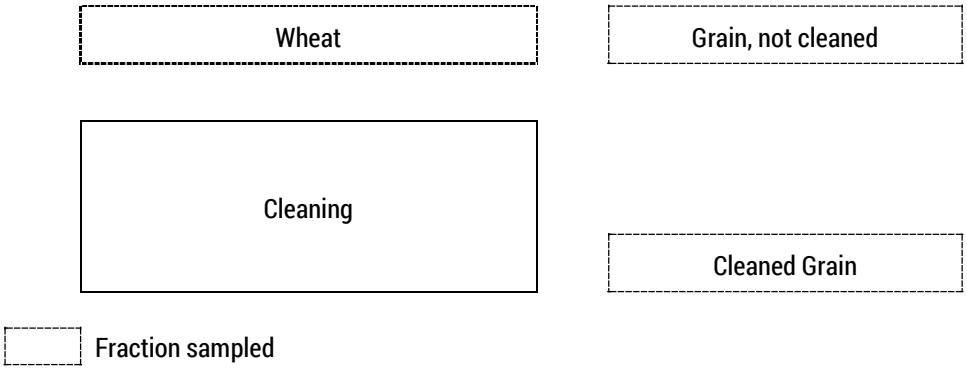
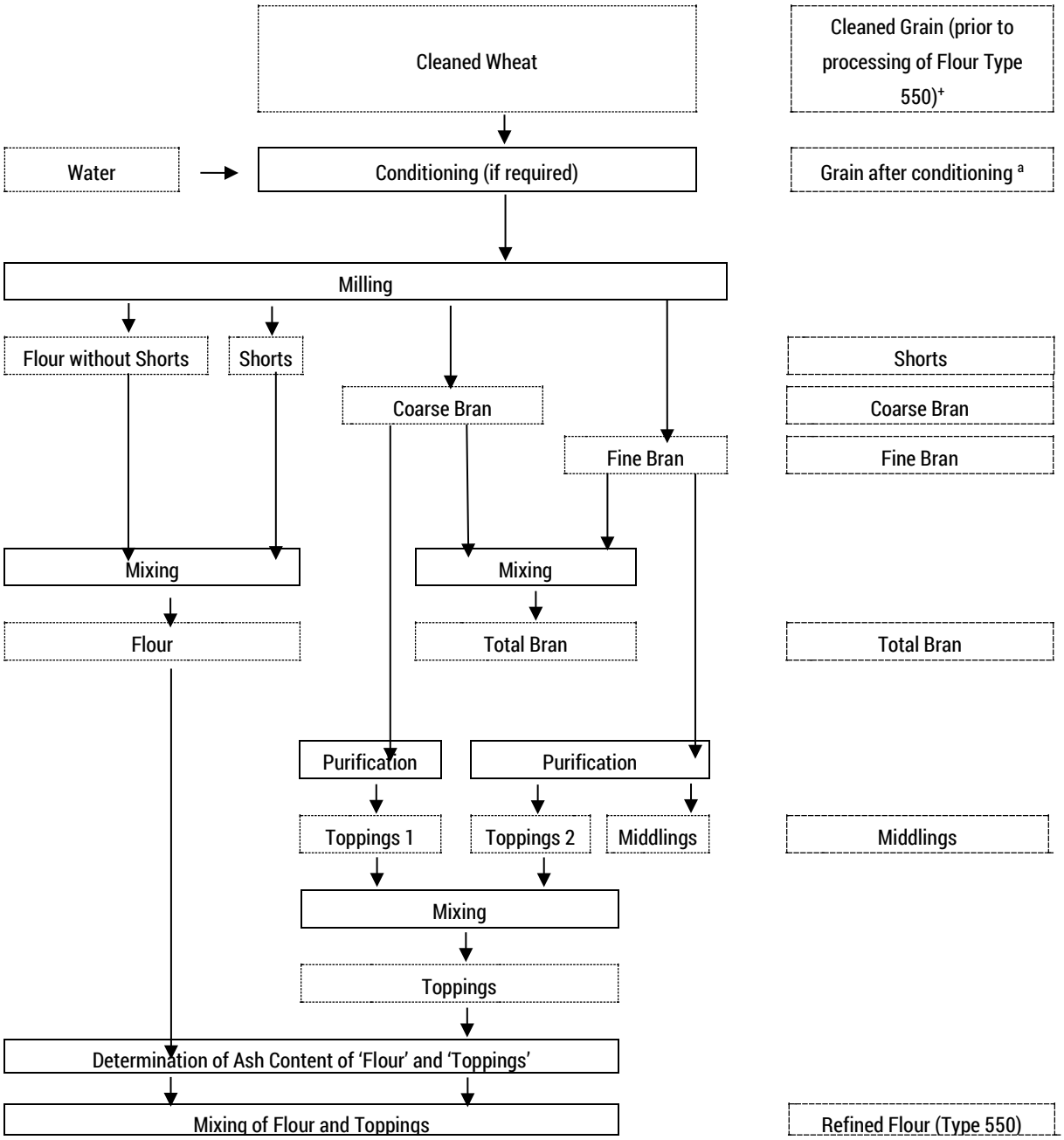


Figure 1 Wheat cleaning processing flowchart.



Fraction sampled

+ If grain was not conditioned, only sample "Cleaned grain (prior to Flour Type 550)" had been taken

a If grain was conditioned, only sample "Grain (after conditioning)" had been taken

Figure 2 Figure 6.5.3/2-2. Wheat milling – refined flour (Type 550) processing flowchart

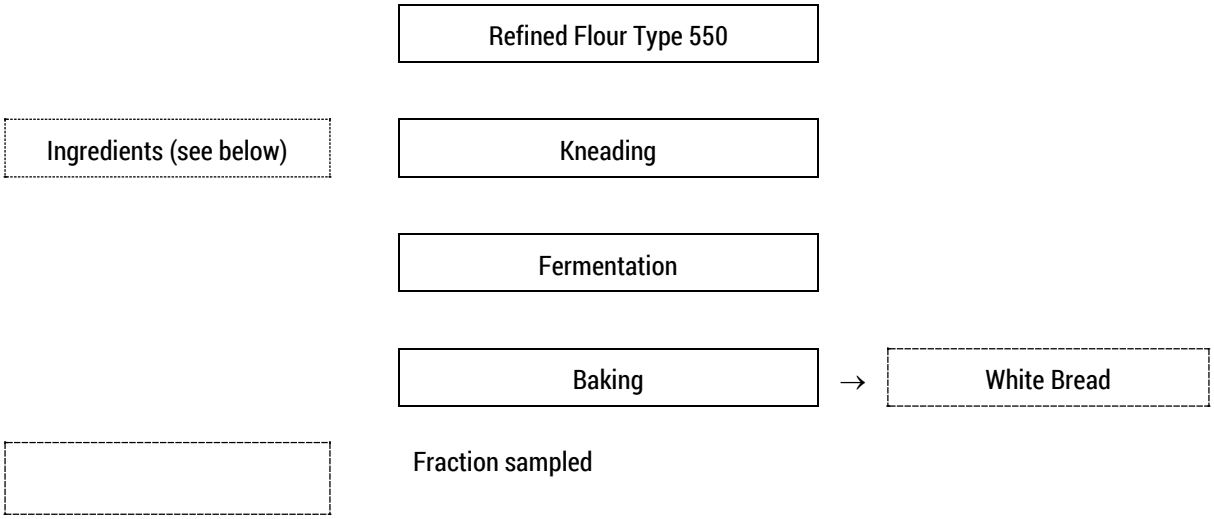


Figure 3 White bread processing flowchart

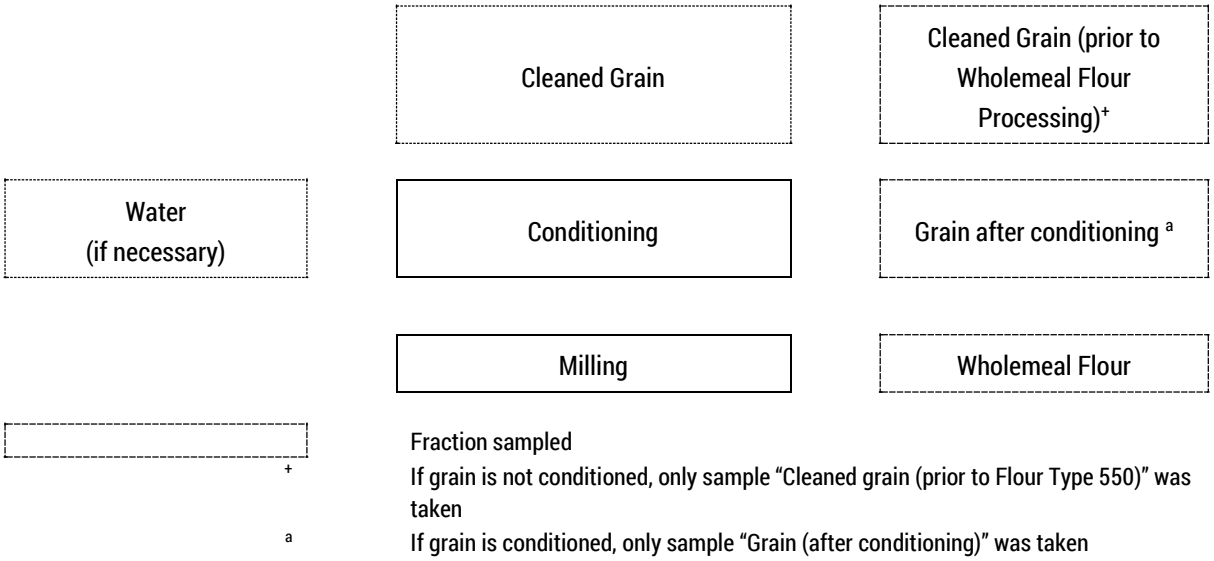


Figure 4 Wholemeal flour processing flowchart

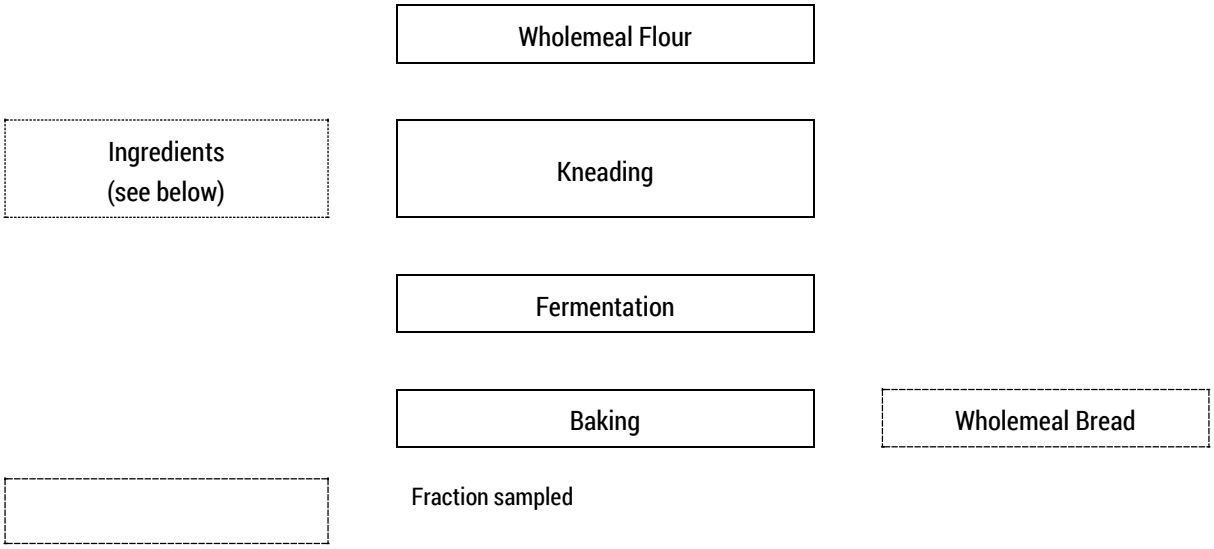


Figure 5 Wholemeal bread processing flowchart

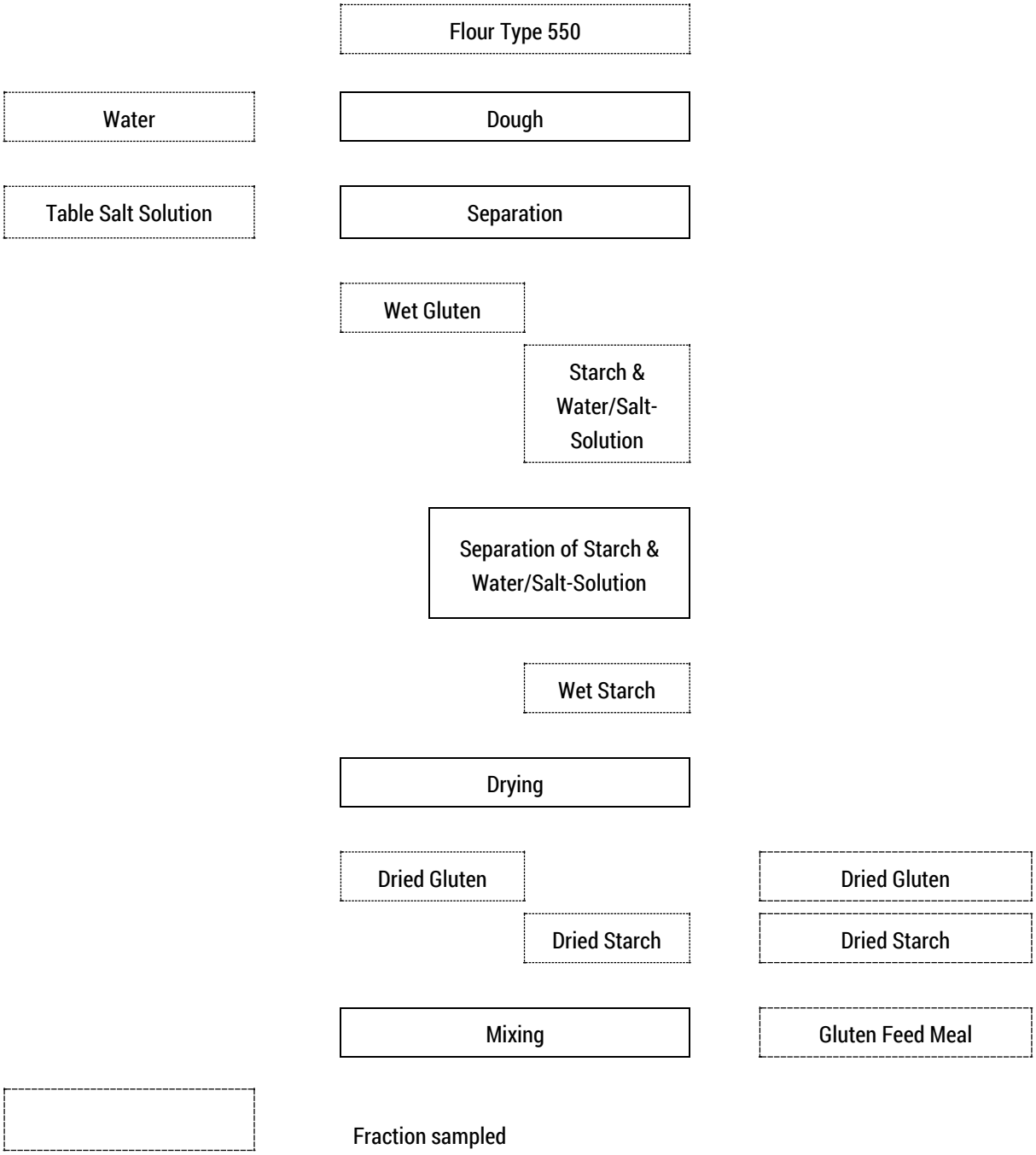


Figure 6 Gluten, starch and gluten feed meal processing flowchart

RESIDUES IN ANIMAL COMMODITIES

Ruminants

A feeding study was conducted for fenpicoxamid in lactating dairy cows (Rawle, N.W., 2013). Twenty-seven lactating Friesian/Holstein dairy cows (divided in 4 groups) were selected for the study, Group A (three cows) were maintained as control and received no test compound. Group B (four cows) received doses of fenpicoxamid equivalent to 4.5 ppm diet (0.142 mg/kg bw), Group C (four cows) were dosed at 13.44 ppm diet (0.408 mg/kg bw), and Group D (sixteen cows) were dosed at 67.31 ppm diet (2.151 mg/kg bw). All

cows, at Groups A, B and D were sacrificed after 28 days or 29 days in Group C of dosing, within 6 hours of receiving the final dose. Four depuration cows were sacrificed following withdrawal of 3, 7, 17 and 21 days on days 31, 35, 42 and 49, respectively.

Milk samples were collected twice daily and composites prepared from the p.m. milkings of one day and the a.m. milkings of the following day. On days 22 and 26, milk was separated into cream and skim milk fractions. Samples of muscle, mesenteric fat, subcutaneous fat, perirenal fat, liver and kidney were collected following slaughter. All samples were frozen at -18 °C prior to analysis. All samples were analysed within 38 days of sampling. This period is covered by the available storage stability study on animal commodities (up to 3 months).

Residues of fenpicoxamid and metabolites X642188 and X12326349 were determined by an analytical method validated under the current study and presented above. The method was validated with an LOQ of 0.01 mg/kg for each compound in all matrices. The performance of the method was verified by obtaining mean procedural recoveries in the acceptable range of 85–120%, 84–120% and 89–107% for fenpicoxamid, X642188 and X12326349 respectively at fortification levels of 0.01–0.10 mg/kg.

Fenpicoxamid, X642188 and X12326349 residues in whole milk during the dosing and depuration periods were below the limit of quantification (<0.01 mg/kg), and in most cases were not detected (<0.003 mg/kg). Samples of milk on days 22 and 26 were mechanically separated into skim milk and cream. Residues of fenpicoxamid, X642188 and X12326349 were below the limit of quantification (<0.01 mg/kg) in all samples from all dose groups and in most cases were not detected (<0.003 mg/kg).

A summary of the residues found in tissues are presented in Table 12, 13 and 14. Residues of fenpicoxamid were not detected (<0.003 mg/kg) in all samples from all dose groups. Residues of X642188 were below the limit of quantification (<0.01 mg/kg) in all samples from all dose groups, and in most cases was not detected (<0.003 mg/kg). Residues of X12326349 were below the limit of quantification (<0.01 mg/kg) in all samples in Group B (4.5 ppm diet) and in muscle, kidney and fat samples in Group C (13.44 ppm diet). Low residues were detected in liver in Group C at 0.011 mg/kg. At the highest dose rate in Group D (67.31 ppm diet) residues of X12326349 were below the limit of quantification (<0.01 mg/kg) at sacrifice apart from in liver (0.047 mg/kg), kidney (0.061 mg/kg and perirenal fat (0.012 mg/kg). In all matrices residues declined to <0.003 mg/kg after 3 days of withdrawal.

Table 12 Mean residues of fenpicoxamid in tissues following 28–29 days oral administration of fenpicoxamid to dairy cows

Matrix	DAY sampled	Mean concentration of fenpicoxamid (mg/kg)		
		Dose level (mg/kg in feed/day)		
		4.5 ppm (Group B)	13.44 ppm (Group C)	67.31 ppm (Group D)
Muscle	28-29	<0.003	<0.003	<0.003
	31	-	-	<0.003
	35	-	-	<0.003
	42	-	-	<0.003
	49	-	-	<0.003
Liver	28-29	<0.003	<0.003	<0.003
	31	-	-	<0.003
	35	-	-	<0.003
	42	-	-	<0.003
	49	-	-	<0.003

Matrix	DAY sampled	Mean concentration of fenpicoxamid (mg/kg)		
		Dose level (mg/kg in feed/day)		
		4.5 ppm (Group B)	13.44 ppm (Group C)	67.31 ppm (Group D)
Kidney	28-29	<0.003	<0.003	<0.003
	31	-	-	<0.003
	35	-	-	<0.003
	42	-	-	<0.003
	49	-	-	<0.003
Subcutaneous fat	28-29	<0.003	<0.003	<0.003
	31	-	-	<0.003
	35	-	-	<0.003
	42	-	-	<0.003
	49	-	-	<0.003
Mesenteric fat	28-29	<0.003	<0.003	<0.003
	31	-	-	<0.003
	35	-	-	<0.003
	42	-	-	<0.003
	49	-	-	<0.003
Perirenal fat	28-29	<0.003	<0.003	<0.003
	31	-	-	<0.003
	35	-	-	<0.003
	42	-	-	<0.003
	49	-	-	<0.003

Table 13 Mean residues of X642188 in tissues following 28-29 days oral administration of fenpicoxamid to dairy cows

Matrix	DAY sampled	Mean concentration of X642188 (mg/kg) [a]		
		Dose level (mg/kg in feed/day)		
		4.5 ppm (Group B)	13.44 ppm (Group C)	67.31 ppm (Group D)
Muscle	28-29	<0.003	<0.003	<0.003
	31	-	-	<0.003
	35	-	-	<0.003
	42	-	-	<0.003
	49	-	-	<0.003
Liver	28-29	<0.003	<0.003	<0.003
	31	-	-	<0.003
	35	-	-	<0.003
	42	-	-	<0.003
	49	-	-	<0.003
Kidney	28-29	<0.003	<0.003	<0.003
	31	-	-	<0.003
	35	-	-	<0.003

Matrix	DAY sampled	Mean concentration of X642188 (mg/kg) [a]		
		Dose level (mg/kg in feed/day)		
		4.5 ppm (Group B)	13.44 ppm (Group C)	67.31 ppm (Group D)
	42	-	-	<0.003
	49	-	-	<0.003
Subcutaneous fat	28-29	<0.003	<0.003	<0.003
	31	-	-	<0.003
	35	-	-	<0.003
	42	-	-	<0.003
	49	-	-	<0.003
Mesenteric fat	28-29	<0.003	<0.003	<0.01
	31	-	-	<0.003
	35	-	-	<0.003
	42	-	-	<0.003
	49	-	-	<0.003
Perirenal fat	28-29	<0.003	<0.003	<0.01 (0.008)
	31	-	-	<0.003
	35	-	-	<0.003
	42	-	-	<0.003
	49	-	-	<0.003

[a] In case of residues > 0.003, the highest residues from the individual results (4 replicates) is included in brackets.

Table 14 Mean residues of X12326349 in tissues following 28–29 days oral administration of fenpicoxamid to dairy cows

Matrix	DAY sampled	Mean concentration of X12326349 (mg/kg)[a]		
		Dose level (mg/kg in feed/day)		
		4.5 ppm (Group B)	13.44 ppm (Group C)	67.31 ppm (Group D)
Muscle	28-29	<0.003	<0.003	<0.003
	31	-	-	<0.003
	35	-	-	<0.003
	42	-	-	<0.003
	49	-	-	<0.003
Liver	28-29	<0.01(0.007)	0.011 (0.012)	0.047 (0.066)
	31	-	-	<0.003
	35	-	-	<0.003
	42	-	-	<0.003
	49	-	-	<0.003
Kidney	28-29	<0.01 (0.007)	<0.01 (0.009)	0.061 (0.082)
	31	-	-	<0.003
	35	-	-	<0.003
	42	-	-	<0.003
	49	-	-	<0.003

Matrix	DAY sampled	Mean concentration of X12326349 (mg/kg)[a]		
		Dose level (mg/kg in feed/day)		
		4.5 ppm (Group B)	13.44 ppm (Group C)	67.31 ppm (Group D)
Subcutaneous fat	28-29	<0.003	<0.003	<0.01 (0.016)
	31	-	-	<0.003
	35	-	-	<0.003
	42	-	-	<0.003
	49	-	-	<0.003
Mesenteric fat	28-29	<0.003	<0.003	<0.01 (0.011)
	31	-	-	<0.003
	35	-	-	<0.003
	42	-	-	<0.003
	49	-	-	<0.003
Perirenal fat	28-29	<0.003	<0.003	0.012 (0.019)
	31	-	-	<0.003
	35	-	-	<0.003
	42	-	-	<0.003
	49	-	-	<0.003

[a] In case of residues > 0.003, the highest residues from the individual results (4 replicates) is included in brackets.

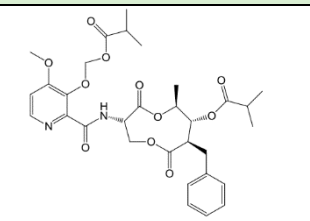
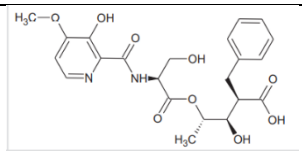
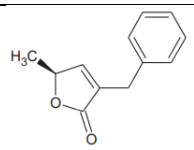
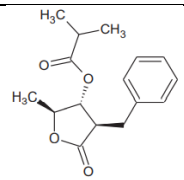
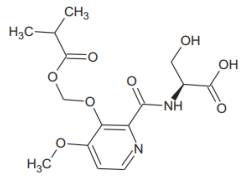
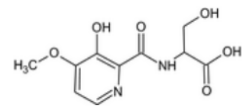
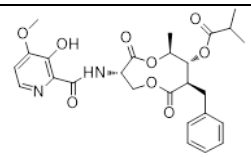
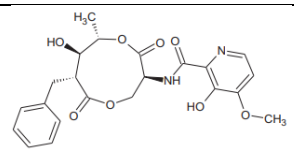
APPRAISAL

Fenpicoxamid is a picolinamide fungicide. The mode of action is by the inhibition of mitochondrial complex III to disrupt spore germination and germ tube elongation. Fenpicoxamid was evaluated for the first time by the 2018 JMPR where an ADI of 0–0.05 mg/kg bw was established and it was concluded that an ARfD was unnecessary. The residue definition for plant commodities for compliance with the MRL and dietary risk assessment was parent fenpicoxamid. The 2018 JMPR did not recommend residue definitions for animal commodities (compliance with the MRL and dietary risk assessment) but concluded that if future uses of fenpicoxamid resulted in an increase in the dietary exposure to the animal metabolite X12326349 and the hydrolysis products X12314005, X12019520, X12335723 and X12264475, reconsideration of the residue definitions may become necessary.

It was scheduled at the Fifty-first Session of the CCPR for the evaluation of additional uses by the 2020 JMPR, which was postponed to the 2021 Extra JMPR. The current Meeting received new information on use patterns for fenpicoxamid in wheat, similar grains and pseudo cereals without husks supported by additional analytical methods and recovery data, storage stability studies, supervised field trials, feeding studies and studies simulating typical processing conditions.

The abbreviations and structures used for the major metabolites discussed in this appraisal are shown in Table 1.

Table 1 List of identified metabolites for fenpicoxamid discussed by the current Meeting

Name	Chemical name	Structure
fenpicoxamid	(3*S*,6*S*,7*R*,8*R*)-8-benzyl-3-{3-[(isobutyryloxy) methoxy]-4-methoxypyridine-2-carboxamido}-6-methyl-4,9-dioxo-1,5-dioxonan-7-yl isobutyrate	
X12326349	2-benzyl-2,5-dideoxy-4-O-[*N*-(3-hydroxy-O-4-methoxypyridine-2-carbonyl)-L-seryl]- L -arabinonic acid	
X12019520	(5S)-3-benzyl-5-methylfuran-2(*5H*)-one	
X12314005	(2*S*,3*R*,4*R*)-4-benzyl-2-methyl-5-oxotetrahydrofuran-3-yl 2-methylpropanoate	
X12335723	*N*-(4-methoxy-3-{[(2-methylpropanoyl) oxy]methoxy}pyridine-2-carbonyl)-L-serine	
X12264475	*N*-(3-hydroxy-4-methoxypyridine-2-carbonyl)-DL-serine	
X642188	(3S,6S,7R,8R)-8-benzyl-3-{[(3-hydroxy-4-methoxypyridin-2-yl)carbonyl]amino}-6-methyl-4,9-dioxo-1,5-dioxonan-7-yl 2-methylpropanoate	
X696872	N-[(3S,7R,8R,9S)-7-benzyl-8-hydroxy-9-methyl-2,6-dioxo-1,5-dioxonan-3-yl]-3-hydroxy-4-methoxypyridine-2-carboxamide	

Methods of analysis

A LC-MS/MS based analytical method (120615) for the determination of fenpicoxamid and its plant metabolite X642188 in wheat grain, straw and whole plant with an LOQ or 0.01 mg/kg for each analyte was evaluated by the 2018 JMPR. The current Meeting received new procedural recovery data for a similar method involving the same principles acetonitrile/water/phosphoric acid extraction and LC-MS/MS determination of fenpicoxamid and its metabolites (X642188, X12335723, X12019520, X12314005,

X12264475) in wheat grain, wheat bran, refined flour and wholemeal bread supporting the LOQ of 0.01 mg/kg.

A similar method was evaluated by the 2018 JMPR for the analysis of fenpicoxamid and its animal metabolites X642188 and X12326349 in animal commodities with a LOQ of 0.01 mg/kg for each analyte.

The Meeting concluded that both methods were demonstrated to have adequate performance for recovery of fenpicoxamid and its metabolites, with an LOQ of 0.01 mg/kg (for each compound).

Stability of residues in stored analytical samples

The storage stability of fenpicoxamid and its metabolite X642188 was investigated by the 2018 JMPR. Additional storage stability studies for dry and animal commodities were submitted to the current meeting. The Meeting concluded that fenpicoxamid and X642188 are stable for at least 24 months in wheat grain, straw, forage, bran, flour, germ and bread when stored frozen at -18 °C. All samples of animal commodities were analysed within 38 days of sampling. This period is covered by the available storage stability study on animal commodities.

Definition of the residue

The 2018 JMPR established the current residue definition for fenpicoxamid in plant commodities.

For plant commodities, the 2018 JMPR concluded that fenpicoxamid is a suitable marker for enforcement of maximum residue levels and for dietary risk assessment for plant commodities. However, it was also recognised that in processed commodities, the degradates X12314005, X12016520, X12335723 and X12264475 may become relevant for commodities subject to high-temperature conditions. The new uses evaluated by the current Meeting relate to cereal grains, which are mostly subject to thermal treatment before consumption. In four processing trials involving field treatment according to the critical GAP, all of these degradates were analysed in raw and processed wheat commodities. Quantifiable residues above the LOQ of 0.01 mg/kg were only found for X12264475 in dried gluten. In absence of specific toxicological data, X12264475 was characterized as a Cramer Class III compound by the 2018 Meeting. Based on the very low X12264475 residue levels in dried gluten (up to 0.014 mg/kg) and the negligible relevance of the commodity in the daily diet, the current Meeting concluded that the dietary exposure will not be significant or reach the TTC for a Cramer Class III compound.

The Meeting confirmed its previous residue definition for fenpicoxamid in plant matrices. Should the Meeting evaluate future uses involving commodities subject to high-temperature conditions, these compounds may need to be re-considered for assessing dietary risk.

For animal commodities, the 2018 JMPR did not recommend a residue definition. The compounds X12326349, 13495S-3S, X12264475 were found as major metabolites in ***ruminants*** while the co-eluting metabolites X119634422/MW208 and the metabolites X696872, X12264475 and X129300 represented the predominant residue in ***poultry***. Since total radioactive residues in both species were very low, the 2018 Meeting concluded that dietary burdens up to 20 ppm for ruminants and 10 ppm for laying hens will not lead to residues above 0.01 mg/kg in animal commodities.

The current Meeting received a new farm animal feeding study on dairy cows with dose rates of 4.5, 13.4 and 67.3 ppm. While residues of parent fenpicoxamid and X642188 remained below the LOQ in all matrices and dose groups, X12326349 was detected (LOD 0.003 mg/kg) in the 4.5 ppm dose group and quantified at concentrations of 0.011 mg/kg and 0.047 mg/kg in tissues in the 13.4 ppm and 67.3 ppm dose groups, respectively. In the highest dose group quantifiable residues were also found in kidney (0.061 mg/kg) and perirenal fat (0.012 mg/kg). In view of the maximum dietary burden for beef cattle of 13.6 ppm arising from the new use on cereals, residues near or above 0.01 mg/kg have to be expected in

ruminant liver and kidney. The current Meeting reviewed the available data and recognized that urinary levels of X12326349 represented 30–40% of the absorbed dose. Accordingly, it can be considered as having been addressed by studies with the parent and as such HBGVs for fenpicoxamid also apply to X12326349.

Residues of X12326349 were primarily found in liver and kidney. Only in the highest dose group were residues quantified in perirenal fat (0.012 mg/kg), while residues in muscle remained below the LOQ (0.003 mg/kg). However, the data does not indicate a strong tendency to partition into fatty compartments.

Since X12326349 was the only residue found in livestock animal feed studies and an analytical method capable of measuring the analyte at LOQs of 0.01 mg/kg is available, the current Meeting concluded that it represents a suitable residue definition both for setting of maximum residue levels and for the estimation of the dietary exposure.

The definition of the residue for compliance with the MRL and dietary risk assessment for milk and tissues of mammals (other than marine mammals): *2-benzyl-2,5-dideoxy-4-O-{N- [(3-hydroxy-4-methoxypyridin- 2-yl)carbonyl]-L-seryl}-L-arabinoic acid (X12326349), expressed as fenpicoxamid.*

The residue is not fat-soluble.

The current Meeting confirms its previous decision not to recommend a residue definition for poultry commodities (muscle, fat, offals and eggs). If future uses of fenpicoxamid result in an increase in the dietary exposure to the animal metabolite X12326349 and the hydrolysis products X12314005, X12019520, X12335723 and X12264475 in poultry, reconsideration of the residue definitions may become necessary.

Results of supervised residue trials on crops

The Meeting received new GAP information and supervised residue trial data for the foliar application of fenpicoxamid on wheat and related cereal grains.

Rye, triticale and wheat

The critical GAP for fenpicoxamid on rye, triticale and wheat (including durum and spelt) is similar in several European countries. As a representative, the GAP in Belgium is for 2 foliar applications up to BBCH 69 (end of flowering) at 100 g ai/ha with a 14 day re-treatment interval. The PHI is limited by the growth stage at the last application. The meeting received 36 supervised field trials matching the critical GAP, from which 18 were considered independent.

In wheat grain, residues of fenpicoxamid were (n = 18): 0.010 (2), 0.011, 0.012 (3), 0.014, 0.016, 0.020, 0.023, 0.024, 0.030, 0.034, 0.036, 0.04 (2), 0.069 and 0.095 mg/kg.

The Meeting estimated a maximum residue level of 0.15 mg/kg and a STMR of 0.0215 mg/kg for wheat. Noting that the GAP also applied to rye and triticale the Meeting decided to extrapolate the maximum residue level to rye and triticale.

Residues in animal feeds

Forage of cereal straw

Based on a New Zealand GAP for wheat of 2 foliar applications up to BBCH 69 (end of flowering) at 100 g ai/ha (interval 14 days) with a PHI of 28 days for forage. The Meeting received eight independent trials that reported residues of fenpicoxamid in wheat forage, residues were (n = 8): 0.31, 0.32, 0.66, 0.77, 0.83,1.3, 1.6, 2.2 mg/kg.

The Meeting estimated a median and highest residue of 0.80 mg/kg and 2.2 mg/kg, respectively, for wheat forage.

Fodder and straw

The critical GAP for fenpicoxamid on rye, triticale and wheat (including durum and spelt) is similar in several European countries. As a representative, the GAP in Belgium is for 2 foliar applications up to BBCH 69 (end of flowering) at 100 g ai/ha (interval 14 days). This GAP only allows for feeding of wheat straw and fodder. The PHI is covered by the growth stage at the last application. The meeting received 36 supervised field trials according to the critical GAP, from which 18 were considered as independent.

In wheat straw residues of fenpicoxamid were (n = 18): 0.84, 1.2, 1.8, 2.4, 2.5, 2.7, 2.7, 3.5, 4.7, 4.7, 5.3, 6.3, 6.3, 6.8, 8.7, 9.9, 10.6 and 12 mg/kg.

The Meeting estimated a maximum residue level of 30 mg/kg (dry weight basis) and a median residue of 4.7 mg/kg (as received) and a highest residue of 12 mg/kg (as received) for wheat straw and fodder, dry.

Fate of residues during processing

The Meeting received new processing studies on wheat. Processed commodities were derived using simulated commercial practices. The resulting processing factors and STMR-P estimates for fenpicoxamid are summarized in Table 2 below.

Table 2 Estimation of 1Processing Factors for wheat processed fractions from Field-Incurred Residues from Foliar Treatment with fenpicoxamid

Raw commodity	Processed commodity	fenpicoxamid			
		Individual processing factors	Median or best estimate processing factor	STMR or STMR-P (mg/kg)	Maximum residue level (mg/kg)
Wheat grain (STMR: 0.0215 mg/kg)	Bran	0.5, 0.56, 0.57, 0.61, 0.78, 0.86, 0.88, 0.91, 0.93, 1.24, 1.25, 1.7 4	0.87	0.019	-
	Refined flour (type 550)	0.15, 0.2, 0.22, 0.25, 0.26, 0.28, 0.29, 0.31, 0.34, 0.35, 0.36, 0.44	0.29	0.0062	-
	White bread	0.07, 0.09, 0.11, < 0.13, 0.13, 0.13, 0.16, < 0.18, < 0.2, < 0.24, < 0.29, < 0.29	0.15	0.0032	-
	Wholemeal flour	0.26, 0.27, 0.36, 0.38, 0.38, 0.4, 0.41, 0.44, 0.5, 0.57, 0.6, 0.71	0.41	0.0088	-
	Wholemeal bread	0.13, 0.14, < 0.16, 0.18, 0.18, 0.2, 0.2, < 0.2, 0.23, 0.26, < 0.29, < 0.29	0.20	0.0043	-
	Wheat germ	0.08, 0.11, < 0.13, 0.15, < 0.16, 0.16, < 0.18, < 0.2, < 0.24, 0.26, < 0.29, < 0.29	0.17	0.0036	-

	Wheat starch	< 0.03, < 0.03, < 0.04, 0.04, < 0.05, **< 0.13, < 0.16**, < 0.18, < 0.2, < 0.24, < 0.29, < 0.29	0.15	0.0032	-
	Wheat gluten	0.15, < 0.16, 0.17, 0.18, < 0.2, < 0.24, < 0.29, < 0.29, 0.29, 0.31, 0.32, 0.52	0.27	0.0058	-

Residues in animal commodities

The 2018 JMPR did not recommend residue definitions for animal commodities as they were not necessary for the uses considered by that meeting. Commodities considered by the current meeting can be used as feed items, thus dietary burdens were calculated for beef cattle, dairy cattle, broilers and laying poultry based on the feed items (cereal forage and grain) evaluated by the current meeting. The dietary burdens, estimated using the most recent version of the OECD livestock dietary burden calculator, are presented in Annex 6 of the 2021 Extra JMPR Report and summarised in Table 3 below.

Table 3 Estimated maximum and mean dietary burdens of farm animals

Animal dietary burden, fenpicoxamid residues, ppm of dry matter diet (DM)					
		US-Canada	EU	Australia	Japan
Beef cattle	max	1.4	2.7	12.7[a]	0.009
	mean	0.54	1.1	4.9[b]	0.009
Dairy cattle	max	2.2	2.7	6.3[c]	0
	mean	0.86	1.1	2.4[d]	0
Poultry Broiler	max	0.025	0.018	0.013	0
	mean	0.025	0.018	0.013	0
Poultry Layer	max	0.025	1.4[e]	0.009	0
	mean	0.025	0.54[f]	0.009	0

[a] Suitable for estimation of maximum residue levels in meat.

[b] Suitable for estimation of median residue levels in meat

[c] Suitable for estimation of maximum residue levels in milk

[d] Suitable for estimation of median residue levels in milk

[e] Suitable for estimation of maximum residue levels in poultry meat and eggs

The fenpicoxamid dietary burden reached a maximum level of 12.7 ppm diet in beef cattle, 6.3 ppm diet in dairy cattle and 1.4 mg/kg diet in poultry. The mean dietary burdens were 4.9 mg/kg in beef cattle, 2.4 mg/kg diet in dairy cattle and 0.54 mg/kg in poultry.

Residue in animal commodities

Farm animal feeding studies

Cattle

A new lactating dairy cow feeding study was provided with dosing at three levels equivalent to 4.5, 13.44 and 67.31 ppm in the diet. Residues of fenpicoxamid and X642188 were < 0.01 mg/kg in all matrices.

Residues of X12326349 were < 0.01 mg/kg in milk, subcutaneous/mesenteric fat and muscle, but were quantifiable in liver (0.011–0.047 mg/kg), kidney (0.061 mg/kg) and perirenal fat (0.012 mg/kg).

Table 4 Maximum residue level, STMR and HR in cattle commodities

	Feed level (ppm DM) for milk residues	Residues (mg/kg) in milk [a]	Feed level (ppm DM) for tissue residues	Residues [a] (mg/kg)			
				Muscle	Liver	Kidney	Fat
Maximum residue level and HR in beef or dairy cattle							
Feeding study	4.5	< 0.013	4.5	< 0.013	< 0.013	< 0.013	< 0.013
	13.4	< 0.013	13.4	< 0.013	0.015	< 0.013	< 0.013
MRL estimate [b]	6.3	0.015* [c]	12.7	0.015* [c]	0.02	0.015 [c]	0.015 [c]
STMR in beef or dairy cattle							
Feeding study	4.5	< 0.013	4.5	< 0.013	< 0.013	< 0.013	< 0.013
	13.4	< 0.013	13.4	< 0.013	0.015	< 0.013	< 0.013
Dietary burden and STMR estimate [c]	2.4	0 [b]	4.9	0 [b]	0.013	0.013	0.013

[a] Residues of X12326349 expressed as fenpicoxamid calculated using a molecular weight conversion factor of 1.33. X12326349 in fenpicoxamid equivalents = X12326349 mg/kg × (614.66/462.2)

[b] Residues are below LOQ at an overdosed rate and therefore residues at the dietary burden are essentially zero

[c] MRL estimate based on limit of quantification (i.e. calculated value of < 0.013 mg/kg rounded up to the next MRL class of 0.015 mg/kg)

Since the residue definition is X12326349 expressed as fenpicoxamid, a conversion factor of 1.33 was used based on the molecular weight (X12326349 in fenpicoxamid equivalents = X12326349 mg/kg × (614.66/462.2)).

The Meeting estimated maximum residue levels of 0.02 mg/kg for edible offal, mammalian, 0.015 mg/kg for fat and 0.015(*) mg/kg for milk and meat.

The Meeting estimated STMRs of 0.013 mg/kg for liver, kidney and fat and STMRs of 0 mg/kg for muscle and milk.

Poultry

For poultry, in the 2018 JMPR a metabolism study in laying hens was conducted at a dose level of *ca.* 10 ppm DM which covers the currently calculated dietary burden (1.4 ppm). In this study, TRR were very low in eggs (0.001–0.007 mg/kg) and low in tissues (0.005–0.153 mg/kg). Only liver samples were taken for identification and fenpicoxamid was not detected (< 0.003 mg/kg). Other metabolites were determined to be below the limit of quantification (< 0.01 mg/kg). Therefore, fenpicoxamid and metabolites are expected to be below the LOQ (< 0.01 mg/kg) in all poultry products when fenpicoxamid is applied according to the intended GAP.

The current Meeting confirms its previous decision not to recommend a residue definition for poultry commodities (muscle, fat, offal and eggs). If future uses of fenpicoxamid result in an increase of the dietary exposure of the animal metabolite X12326349 and the products X12314005, X12019520, X12335723 and X12264475 in poultry, reconsideration of the residue definitions may become necessary.

RECOMMENDATIONS

On the basis of the data from supervised trials, the Meeting concluded that the residue levels listed below in Table 5 are suitable for establishing maximum residue limits and for IEDI assessment.

The residue definition for compliance with the MRL and dietary risk assessment for plant commodities is *fenpicoxamid*.

The residue definition for compliance with the MRL and dietary risk assessment for milk and tissues of mammals (other than marine mammals): is *2-benzyl-2,5-dideoxy-4-O-{N- [(3-hydroxy-4-methoxypyridin- 2-yl)carbonyl]-L-seryl}-L-arabinoic acid (X12326349), expressed as fenpicoxamid*.

The residue is not fat-soluble.

Table 5 Recommendations for residues of fenpicoxamid from the 2021 Extra JMPR

CCN	Commodity name	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg	
		New	Previous		
MO 0105	Edible offal (mammalian)	0.02		0.013	
MF 0100	Mammalian fats (except milk fats)	0.015		0.013	
MM 0095	Meat (from mammals other than marine mammals)	0.015(*)		0	
ML 0106	Milks	0.015(*)		0	
GC 0650	Rye	0.15		0.0215	
GC 0653	Triticale	0.15		0.0215	
AS 0654	Wheat straw and fodder, dry	30 (dw)		Median: 4.7 (as)	Highest: 12 (as)
GC 0654	Wheat	0.15		0.0215	
GC 0653	Triticale flour (white and wholemeal)			0.0088[a]	
CF 1210	Wheat germ			0.0036	
CP 1212	Wheat wholemeal bread			0.0043	
CP 1211	Wheat white bread			0.0032	
CF 1211	Wheat white flour			0.0062	
CF 1250	Rye flour (white and wholemeal)			0.0088[a]	
-	Wheat, bulgur			0.0088[a]	
-	Wheat starch			0.0032	
-	Wheat gluten			0.0058	

[a]: PF for wheat wholemeal flour was used.

Table 6 Additional values used in estimating livestock dietary burdens.

CCN	Commodity name	Median residue (-P) mg/kg	highest residue (-P) mg/kg
AF/AS	Wheat forage	0.8	2.2
AF/AS	Wheat straw and fodder, dry	4.7	12

CCN	Commodity name	Median residue (-P) mg/kg	highest residue (-P) mg/kg
GC	Rye grain	0.0215	-
GC	Triticale grain	0.0215	-
GC	Wheat grain	0.0215	-
CM/CF	Wheat milled by products	0.0215	-

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for fenpicoxamid is 0–0.05 mg/kg bw. The International Estimated Daily Intakes (IEDIs) for fenpicoxamid were estimated for the 17 GEMS/Food Consumption Cluster Diets using the STMR or STMR-P values estimated by the JMPR. The results are shown in Annex 3 of the 2021 Extra JMPR Report.

The IEDIs were 0% of the maximum ADI. The Meeting concluded that long-term dietary exposure to residues of fenpicoxamid from uses considered by the JMPR is unlikely to present a public health concern.

Acute dietary exposure

The 2018 JMPR decided that an ARfD for fenpicoxamid was unnecessary. The Meeting therefore concluded that the acute dietary exposure to residues of fenpicoxamid from the uses considered is unlikely to present a public health concern.

REFERENCES

Report number	Author(s)	Year	Title, Source, Company name, Report No., Date, GLP status published or not
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CEMS-6149	Rawle, N.W.	2013	XDE-777 Livestock Feeding Study: Magnitude of Residue in Milk, Muscle, Liver, Kidney and Fat of Lactating Dairy Cattle Study No. CEMS-6149 Dow Agrosciences Study ID 130949 Dow Agrosciences Document No. 2021796 GLP Unpublished

Report number	Author(s)	Year	Title, Source, Company name, Report No., Date, GLP status published or not
CEMS-6104	Betson, S.	2013	Frozen Storage Stability of Residues of XDE-777 and Its Metabolites (X642188 and X12326349) in Animal Matrices – Final report Study No. CEMS-6104 Dow Agrosciences Study ID 130709 Dow Agrosciences Document No. 2020534 GLP Unpublished
S12-02427	Watson, G.	2014	XDE-777 and its Metabolite X642188 –Storage Stability in wheat and wheat processed fractions stored frozen for up to 24 months Study No. S12-02427 Dow Agrosciences Study ID 120749 Dow Agrosciences Document No. 2024197 GLP Unpublished
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S14-01568	Eversfield, S.	2016	Determination of Residue of XDE-777 and Prothioconazole after Two Applications of GF-3307 and after Two Applications of GF-3310 in Winter Wheat and Spring Wheat at 4 sites in Northern Europe and 4 sites in Southern Europe in 2014 Study No. S14-01568 Dow Agrosciences Study ID 140649 Dow Agrosciences Document No. 2036755 GLP Unpublished
S14-01569	Eversfield, S.	2017	Determination of Residues of XDE-777 and Pyraclostrobin after Two Applications of GF-3312 and after Two Applications of GF-2925 in Winter Wheat and Spring Wheat at 4 sites in Northern Europe and 4 sites in Southern Europe in 2014 Study No. S14-01569 Dow Agrosciences Study ID 140648 Dow Agrosciences Document No. 2036756 GLP Unpublished

Report number	Author(s)	Year	Title, Source, Company name, Report No., Date, GLP status published or not
S15-02628	White, T.	2016	Determination of Residues of XDE-777 and Pyraclostrobin after Two Applications of GF-3309 to Spring and Winter Wheat, at 5 sites in Northern Europe and 5 sites in Southern Europe, 2015 Study No. S15-02628 Dow Agrosciences Study ID 150650 Dow Agrosciences Document No. 2036014 GLP Unpublished
S15-02629	White, T.	2016	Determination of Residues of XDE-777 and Prothioconazole after Two Applications of GF-3307 to Spring and Winter Wheat, at 5 sites in Northern Europe and 5 sites in Southern Europe, 2015 Study No. S15-02629 Dow Agrosciences Study ID 150649 Dow Agrosciences Document No. 2036015 GLP Unpublished
S12-01369	Tandy, R.	2014	Determination of Residues of XDE-777 in Grain and Processed Products After Two Applications of GF-2925 in Winter Wheat on 2 Sites in Northern Europe and 2 Sites in Southern Europe in 2012 Study No. S12-01369 Dow Agrosciences Study ID 120435 Dow Agrosciences Document No. 2024328 GLP Unpublished
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FLUOPYRAM (243)

First draft prepared by Dr Yukiko Yamada, International Food Safety Consultant and Adjunct Professor, Azabu University, Japan

EXPLANATION

Fluopyram, a pyridylethylamide broad spectrum fungicide, is registered in many countries for protection against a range of Ascomycete and Deuteromycete diseases in many horticultural and arable crops. Within plants, fluopyram shows translaminar activity and some upwards movement within the xylem. Fluopyram was first evaluated by the JMPR for toxicology and residues in 2010 and subsequently in 2012, 2014, 2015 and 2017 for residues.

The 2010 JMPR established an ADI of 0–0.01 mg/kg bw and an ARfD of 0.5 mg/kg bw.

The 2010 JMPR reviewed information on identity, physical and chemical properties, plant and animal metabolism, environmental fate, residue analysis and storage stability, use pattern, supervised trials on many crops, processing, and animal feeding; and recommended the following residue definitions:

Definition of the residue for compliance with the MRL and dietary risk assessment 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 dietary risk assessment for animal commodities: *Sum of fluopyram, 2-(trifluoromethyl)benzamide and the combined residues of 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 residue is not fat-soluble.

On a basis of the above residue definitions, the Meeting estimated maximum residue levels for many plant commodities (including cereal grains, pulses and oilseeds) and foods of animal origin.

The Forty-thrid Codex Alimentarius Commission (2020) approved the new work proposals including the priority list of pesticides for evaluation by the current Meeting. The priority list included fluopyram for the evaluation of an additional use on coffee plants.

The current Meeting received new information on GAP, validation of analytical methods, supervised residue trials on coffee and processing studies on coffee beans.

In this evaluation, (2-trifluoromethyl)benzamide is expressed as "BZM" as in the previous evaluations.

RESIDUE ANALYSIS

Analytical methods

The 2010 JMPR and subsequent evaluations have reviewed validation data on the LC-MS/MS methods, Method 00984 and Method 00984/M001 (slight modification of Method 00984), in various matrices for which supervised trial data were provided. These methods were used in the supervised trials and processing studies on coffee provided to the current Meeting.

In these methods, fluopyram residues were extracted by homogenization with acetonitrile/water (4:1, v/v) followed by filtration and/or centrifugation. The extract was diluted with the extraction solvent to a known volume. An aliquot of the extract was diluted under basic conditions, and internal standard solutions were added for analysis by reversed phase HPLC equipped with tandem mass spectrometry (MS/MS) with electrospray ionization. Mass transition for quantitation: m/z 397 → 173 for fluopyram; and m/z 190 → 170 (Method 00984) or 190 → 102 (Method 00984/M001) for BZM.

The current Meeting received information on new validation studies on Method 00984 and Method 00984-M001 for coffee beans and their processed commodities. The data on validation for determination of fluopyram and BZM are summarized in Table 1 below. At the fortification levels of 0.01 and 1.0 mg/kg in green coffee beans, roasted coffee beans and instant coffee, the recoveries and RSD values were within the acceptable range and the validated LOQs were 0.01 mg/kg, expressed in fluopyram, in green coffee beans, roasted coffee beans and instant coffee.

Table 1 Summary of validation of the HPLC-MS/MS methods (Method 00984 and Method 00984-M001) for the determination of fluopyram residues in coffee beans and their processed commodities

Matrix	Analyte	Fortification (mg/kg)	n	Recovery (%)			Reference (Report No)
				Range	Mean	RSD	
Method 00984							
Green beans	Fluopyram	0.01	5	85-104	94	7.2	Sarti, A. 2018 M-564093-02-2 (I15-018)
		1.0	5	97-106	101	3.7	
	BZM	0.01	5	79-114	95	15	
		1.0	4	96-102	98	2.8	
Method 00984-M001							
Green beans	Fluopyram	0.01	12	76-101	86	10	Li, Y. 2017 M-597632-01-1 (RAGMN212)
		1.0	8	72-110	91	14	
	BZM	0.01	12	70-107	88	14	
		1.0	8	63-98	81	13	
Roasted Beans	Fluopyram	0.01	7	84-96	90	4.8	Li, Y. 2019 M-608569-02-2 (RAGMN213-01)
		1.0	5	83-103	97	7.7	
	BZM	0.01	7	71-94	84	8.0	
		1.0	5	77-96	91	7.7	
Instant coffee	Fluopyram	0.01	7	87-106	98	5.7	
		1.0	5	68-100	91	13	
	BZM	0.01	7	63-110	84	20	
		1.0	5	64-99	88	14	

Stability of residues in stored analytical samples

The 2010 and 2012 JMPR evaluated storage stability data including data on rapeseed in the "high oil" commodity group. The 2012 JMPR concluded that fluopyram and BZM were stable for at least 36 months under frozen storage conditions in rapeseed (at or below -18 ºC). As coffee beans is also categorized in the "high oil" group, the current Meeting confirmed that fluopyram and BZM would be stable in frozen conditions for at least 36 months.

USE PATTERN

Fluopyram has been registered in many countries for use on many crops. The relevant label in Brazil, Guatemala and Nicaragua were provided to the current Meeting. As the supervised trial data provided to the current Meeting were on coffee, registered uses on coffee are summarized below to support the interpretation of the supervised residue trials on coffee.

Table 2 Registered uses of fluopyram on coffee relevant to the current evaluation of supervised trials on coffee

Crop	Country	Conc. g ai/L Form	Application			Max/season		Min. PHI days	Note
			Method	Max rate kg ai/ha	Water L/ha	No.	kg ai/ha		
Coffee	BR	500 SC	Strip or drench to soil	0.50	200-500	1	0.50	90	[a]
Coffee	GT NI	500 SC	Drench (nursery)	0.075 g ai/ plant	30 mL/ bag	1 [b]	-	-	[b]
			Spray to the ground (Mature plantation)	0.50	500	1 [b]	-	120	

[a] Should be applied in strips or as a drench on both sides of the plant, in the projection of the coffee tree canopy in order to reach most of the roots. The soil should be clean, free of leaves and crop residues and weeds. Perform one application per crop cycle.

[b] Apply once in the nursery and apply once in the field 30 days after the first heavy rain. The rate of 0.5 kg ai/ha corresponds to 0.05 kg ai/plant according to the label.

Notes on the coffee cultivation: The first coffee harvests start 3 to 4 years after planting the nursery plants in the field plantation. Nursery plants are growing in bags.

RESULTS OF SUPERVISED RESIDUE TRIALS ON CROPS

The current Meeting received information on supervised trials using fluopyram on coffee plants. The results of these supervised trials are summarized in the following table:

Group/Sub-group	Commodity	Country	Table No.
Seed for beverages and sweets	Coffee beans	Brazil	Table 3
		Colombia Costa Rica Guatemala Mexico	Table 4

In addition to the descriptions and details of the field trials, each study report included a summary of the analytical methods, together with the corresponding procedural recoveries, LOQ, LOD, and information on storage of samples. The durations of freezer storage between sampling and analysis were reported for all trials and were covered by the conditions of the freezer storage stability studies.

All appropriate trials are summarized and used. In the trials, where multiple analyses were conducted on a single sample, the mean value is reported. Where multiple samples were taken from a single plot, the individual and mean values are reported. Where results from separate plots with distinguishing

characteristics such as different varieties or treatment schedules were reported, results are listed for each plot.

When residues were not quantifiable, they are shown as below the LOQ of the relevant analytical method (e.g., < 0.01 mg/kg). Residues and application rate have generally been rounded to two significant figures or, for residues near the LOQ, to one significant figure.

Although control plots were included in the trials, control data are not reported in the following tables unless residues in control samples exceeded the LOQ. Results have not been corrected for concurrent method recoveries.

Residue values from the trials conducted according to the critical GAP were used for the estimation of maximum residue levels, STMR and HR. Those results included in the tables are underlined.

Seed for beverages and sweets

Coffee beans - Brazil

A total of five field trials were conducted on coffee in 2015 in coffee growing regions of Brazil. Fluopyram (500 SC) was applied once as a drench spray to soil at the base of the plants at a rate equivalent to 0.50 kg ai/ha.

In each trial, duplicate samples of coffee cherries were collected 70, 80, 90, 98–100 and 110 days after treatment in decline trials, or 90 days after treatment in other trials. At the time of the sample collection, coffee cherries were at BBCH 85–89 growth stage, corresponding to "Increase in intensity (variety-specific), yellow or red, fruit colour; fruit not yet ready for picking" to "Overripe; beginning of darkening or drying; fruits stay on the tree or abscission begins".

The collected coffee cherries were processed by drying under ambient conditions and then removing the pulp mechanically to obtain green coffee beans (dry method). The whole process took 2–20 days. The obtained green beans were frozen at <-20 ºC until analysis. The maximum storage duration of treated green coffee beans from collection to analysis was 256 days (about 8.5 months). The storage stability study on rapeseed (high oil matrix) reviewed by the 2010 JMPR indicated that fluopyram was stable for at least 36 months in coffee beans.

Fluopyram in green coffee beans was determined with Method 00984 (LC-MS/MS) with the LOQ of 0.01 mg/kg. The concurrent recoveries and their RSD for fluopyram in green coffee beans were within the acceptable range.

BZM in green coffee beans was also determined using the same method and found to be 0.014 mg eq/kg in one 90-DAT sample in trial I15-018-04 and <0.01 mg eq/kg in all other samples.

Table 3 Residues of fluopyram in green coffee beans from supervised trials conducted using drench spray application of fluopyram (500 SC formulation) in Brazil in 2015

Trial No Location in Brazil (Variety)	Application				DAT days	Fluopyram, mg.kg		Reference Study No
	kg ai/ha	kg ai/hL	Water, L/ha	No		Individual	Mean	
GAP in Brazil	0.50		200-500	1	90			
I15-018-01 Paulínia (Catuai Vermelho)	0.51	0.10	510	1	70 80 90 98 110	<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	M-564093-02-2 I15-018
I15-018-02 Araguari (Mundo Novo)	0.49	0.10	490	1	70 80 90 100 110	<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	
I15-018-03 Ribeirão Preto (Ubata)	0.51	0.10	510	1	70 80 90 100 110	<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	
I15-018-04 Ituverava (Catuai Amarelo)	0.50	0.10	500	1	90	0.014, <0.01	0.012	
I15-018-05 São José da Bela Vista (Catuai Amarelo)	0.50	0.10	500	1	90	<0.01, <0.01	<0.01	

DAT: days after treatment

Coffee beans - Colombia, Costa Rica, Guatemala and Mexico

A total of five field trials were conducted on coffee in 2016 in coffee growing regions of Colombia (2), Costa Rica (1), Guatemala (1) and Mexico (1). Fluopyram (500 SC) was applied once as a drench spray to soil at a rate equivalent to 0.50 kg ai/ha in accordance with GAP in Brazil.

In each trial, duplicate samples of ripe coffee cherries were collected 70, 79–80, 90, 100 and 110 days after treatment in decline trials, or 88–90 days after treatment in other trials. At the time of the sample collection, coffee cherries were at BBCH 87–89 growth stage, corresponding to “Fruit is fully ripe colour and ready for picking” to “Overripe; beginning of darkening or drying; fruits stay on the tree or abscission begins”.

The collected coffee cherries were processed through a wet processing method common in Central America to obtain green coffee beans as follows: removal of the outer hulls on the day of harvest, fermentation in water for several hours for less than 1 day; removal of the mucilage by washing with water; drying for up to 14 days; and removal of parchment. The obtained green coffee beans were frozen at <-

20 °C until analysis. The maximum storage duration of treated green coffee beans from collection to analysis was 197 days (about 6.5 months).

Fluopyram in green coffee beans was determined with Method 00984/M001 (LC-MS/MS) with the LOQ of 0.01 mg/kg. The concurrent recoveries and their RSD for fluopyram in green coffee beans were within the acceptable range.

BZM in green coffee beans was also determined using the same method and found to be <0.01 mg eq/kg in all samples.

Table 4 Residues of fluopyram in green coffee beans from supervised trials conducted using drench spray application of fluopyram (500 SC formulation) in Colombia, Costa Rica, Guatemala and Mexico in 2016

Trial No Location (Variety)	Application				DAT days	Fluopyram, mg.kg		Reference Study No
	kg ai/ha	kg ai/hL	Water, L/ha	No		Individual	Mean	
GAP in Brazil	0.50		200-500	1	90			
GM001-16DA Caldas, Colombia (Castillo)	0.50	0.11	450	1	70 79 90 100 110	<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	M-597632-01-1 RAGMN212
GM002-16DA San Jose, Costa Rica (Catuai)	0.49	0.16	310	1	70 80 90 100 110	<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	
GM003-16DA Departamento El Progreso, Guatemala (Pache)	0.50	0.24	210	1	70 80 90 100 110	<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	
GM004-16HA Risaralda, Colombia (Supremo)	0.50	0.11	440	1	88	<0.01, <0.01	<0.01	
GM005-16HA Veracruz, Mexico (Marsellesa/ Sarchimor)	0.51	0.11	460	1	90	<0.01, <0.01	<0.01	

DAT: days after treatment

FATE OF RESIDUES IN STORAGE AND IN PROCESSING

Information and Data from Residues in Processed Commodities

The current Meeting received information on the processing of green coffee beans to roasted coffee beans and instant coffee. (Li, Y., 2019, M-608569-02-2)

Two field trials were conducted in Brazil and Colombia in each of which fluopyram (500 SC) was applied as a drench spray to the soil at an exaggerated rate (5N of GAP rate in Brazil) of 2.5 kg ai/ha and ripe coffee cherries were harvested at a DAT of 90 days. The harvested coffee cherries were processed to green coffee beans either by the dry method (trial in Brazil) or the wet method (trial in Colombia). The obtained green coffee bean samples were stored frozen until processing to roasted beans and instant coffee.

Before processing, the moisture content in green beans was ensured to be 10–13% or, if necessary, adjusted by drying to 10–13%. The processing method in the study simulated industrial practice as closely as possible. However, beans were processed in batches rather than in continuous flow as in the industrial practice due to the sample size.

Green beans were cleaned, and, if necessary, aspirated and screened. Clean green beans were roasted using a modified table-top roaster at 188–204 ºC and maintained for 2–15 minutes to a level similar to "mild roast". The roasted beans were allowed to cool.

Roasted coffee beans were ground to produce material for instant coffee production. After grinding, the material was sifted with a sifter equipped with a 16- and 36-mesh sieves. Material below the 16-mesh sieve and remaining on the top of the 36-mesh sieve was extracted in a system consisting of two steam stainless steel jacketed vessels, in-line pressure regulator to raise internal pressure above atmospheric conditions, a positive displacement pump with reservoir tank, in-line thermometer, and chilled-water heat exchanger to cool exit product. After filling the jacketed vessels with ground material, water was pumped into bottom vessel. Steam was applied to the vessel and once bottom vessel was heated, pumping of water resumed and steam was applied to the top vessel. Water is pumped through the system until the exit solution became amber in colour. Exit temperature of liquid from top vessel was 129–163 °C. The temperature of extract entering the chilled-water heat exchanger was decreased to below 24 °C. Extracts were screened/filtered twice with a 120-mesh screen upon exiting the heat exchanger. Coffee extract was concentrated in a laboratory vacuum evaporator until the solid content was greater than 25%. Temperature was maintained below 24 °C during the concentration. Extract was filtered with a 120-mesh screen and was frozen. Frozen extract was freeze dried to produce instant coffee.

Sub-samples of green coffee beans, roasted coffee beans and instant coffee were stored frozen at <-23 ºC until analysis. In the trials, the maximum storage duration from collection to analysis was 232 days (ca 8 months) for green coffee beans, 749 days (ca 25 months) for roasted beans and 726 days (ca 24 months) for instant coffee.

Residues of fluopyram and BZM were determined with Method 00984-M001 (LC-MS/MS) with the LOQ of 0.01 mg/kg in each matrix. Concurrent recoveries and their RSD were within the acceptable range.

Residues of fluopyram in green coffee beans and the processed commodities are shown in Table 5. Since the residues of fluopyram in green coffee beans (RAC) was <0.01 mg/kg in the two trials with a 5N exaggerated rate, it was not possible to calculate processing factors for roasted beans and instant coffee.

The analytical result of BZM in instant coffee from the green coffee beans obtained in the trial in Colombia was above the LOQ of 0.01 mg eq/kg, the analytical results of BZM are also included in Table 5 5, even though this metabolite has not been included in the residue definition for plant commodities.

Table 5 Residues of fluopyram in green coffee beans and their processed commodities

(Drench application of fluopyram in 500 SC formulation at a rate of 2.5 kg ai/ha; and coffee cherries were harvested at 90 DAT) (M-608569-02-2, Study No. RAGMN213-1)

Trial No. Year, Location (Variety)	Application				Sample	Fluopyram mg/kg a	BZM mg eq/kg a
	kg ai/ha	kg ai/hL	Water L/ha	No			
GM006-16PA 2016, São Paulo, Brazil (Café Obatã)	2.5	0.50	500	1	Green bean (RAC)	<0.01	<0.01
					Roasted bean	<0.01	<0.01
					Instant coffee	<0.01	<0.01
GM007-16PA 2016, Quindio, Colombia (Caturro)	2.5	0.57	440	1	Green bean (RAC)	<0.01	<0.01
					Roasted bean	<0.01	<0.01
					Instant coffee	<0.01	0.027

[a] For green beans, average of three separate samples; and for the processed commodities, each sample was analysed in triplicate.

APPRAISAL

Fluopyram, a pyridylethylamide broad spectrum fungicide, was first evaluated by the JMPR for toxicology and residues in 2010 and subsequently in 2012, 2014, 2015 and 2017 for residues. The 2010 JMPR established an ADI of 0–0.01 mg/kg bw and an ARfD of 0.5 mg/kg bw.

The 2010 JMPR recommended the following residue definitions:

Definition of the residue for compliance with the MRL and dietary risk assessment 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 dietary risk assessment for animal commodities: *Sum of fluopyram, 2-(trifluoromethyl)benzamide and the combined residues of 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 residue is not fat-soluble.

The Forty-third Codex Alimentarius Commission (2020) approved the new work proposals including the priority list of pesticides for evaluation by the current Meeting. The priority list included fluopyram for the evaluation of an additional use on coffee plants.

The current Meeting received new information on GAP, validation of analytical methods, supervised residue trials on coffee and processing studies on coffee beans.

Methods of analysis

The current Meeting received new information on the validation of Method 00984 and Method 00984-M001 used in supervised trials on coffee. These LC-MS/MS methods were evaluated by the JMPR in 2010 and later for determining fluopyram and its three metabolites in many other matrices.

The methods were validated with LOQs of 0.01 mg/kg for fluopyram and (2-trifluoromethyl)benzamide in green coffee beans, roasted coffee beans and instant coffee. The mean recoveries and RSDs were within the acceptable range in these commodities.

Results of supervised residue trials on crops

The current Meeting received information on supervised trials on coffee plants using drench spray application of fluopyram.

Seed for beverages and sweets

Coffee beans

Critical GAP in Brazil for coffee plants allows one strip or drench application to soil at a maximum rate of 0.5 kg ai/ha per season, and a PHI of 90 days.

GAP in Guatemala and Nicaragua for coffee plants allows one application in nursery plants (0.075 g ai/plant), and one application to the ground in mature plantations at the same rate as in Brazil with a PHI of 120 days. However, since it normally takes more than three years for coffee plants to become harvestable, the Meeting considered that the application in nursery plants would not lead to residues in harvested coffee beans and that the GAP in Brazil with a shorter PHI was more critical than that in Guatemala and Nicaragua. In the trials conducted in Central America, samples were collected at DAT that were shorter than the PHI specified in the GAP in Guatemala and Nicaragua.

A total of ten field trials were conducted on coffee plants in coffee growing regions in Brazil (5) in 2015, and in Colombia (2), Costa Rica (1), Guatemala (1) and Mexico (1) in 2016. Duplicate samples were taken in these trials. The harvested coffee cherries were processed using the dry method in the trials in Brazil, and using the wet method in the trials in Central America.

Fluopyram residues from trials in Brazil according to the GAP in Brazil were in rank order (n = 5): < 0.01 (4) and 0.012 mg/kg.

Fluopyram residues from trials in Central America according to the GAP in Brazil were in rank order (n = 5): < 0.01 (5) mg/kg.

While the green coffee bean samples were prepared by two different methods (dry method and wet method) in Brazil and Central America, the Meeting concluded that since the residue levels were low, the residue populations could be considered together.

After combining the data, fluopyram residues were in rank order (n = 10): < 0.01 (9) and 0.012 mg/kg. The Meeting estimated a maximum residue level of 0.015 mg/kg for coffee beans. The Meeting estimated an STMR of 0.01 mg/kg.

Fate of residues during processing

The current Meeting received information on the processing of green coffee beans to roasted coffee beans and freeze-dried instant coffee. For obtaining the raw material green beans, fluopyram was applied at an

exaggerated rate (5N of the maximum rate specified in GAP in Brazil) in the field. Fluopyram concentrations in all the samples of green beans (RAC) and roasted coffee and instant coffee were < 0.01 mg/kg. Therefore, processing factors could not be calculated.

As fluopyram was detected slightly above the LOQ in one sample of green beans from the trial conducted in accordance with GAP in Brazil and was below the LOQ in all the samples in the processing studies using an exaggerated 5N rate in the field phase, the Meeting agreed to use 0.01 mg/kg as the STMR-P for roasted beans and instant coffee for dietary exposure assessment.

Residues in animal commodities

As coffee beans or their by-products/processed commodities are not included in the OECD Animal Feeding Table, the current Meeting concluded that it was unnecessary to re-calculate animal dietary burdens or review the recommendations for animal commodities made by the 2017 JMPR.

RECOMMENDATIONS

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

Definition of the residue for compliance with the MRL and dietary risk assessment 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 dietary risk assessment for animal commodities: *Sum of fluopyram, 2-(trifluoromethyl)benzamide and the combined residues of 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 residue is not fat-soluble.

Table 1 Recommendations for residues of fluopyram from the 2021 Extra JMPR

CCN	Commodity	Recommended maximum residue level mg/kg		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
SB 0716	Coffee beans	0.015	-	0.01	-
SM 0716	Coffee beans, roasted			0.01	-
	Instant coffee			0.01	

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for fluopyram is 0–0.01 mg/kg bw. The International Daily Intakes (IEDIs) for fluopyram were estimated for the 17 GEMS/Food Consumption Cluster Diets using the STMR and STMR-P values estimated by JMPR. The results are shown in Annex 3 of the 2021 Extra JMPR Report.

The IEDIs ranged from 10–80% of the maximum ADI. The Meeting concluded that long-term dietary exposure to residues of fluopyram from uses considered by the JMPR is unlikely to present a public health concern.

Acute dietary exposure

The ARfD for fluopyram is 0.5 mg/kg bw. The international Estimate of Short-Term Intakes (IESTIs) for fluopyram were calculated for coffee beans and the processed commodities for which STMR/STMR-Ps were estimated by the present Meeting and for which consumption data were available. The results are shown in Annex 4 of the 2021 Extra JMPR report.

The IESTIs were 0% of the ARfD for children and the general population. The Meeting concluded that acute dietary exposure to residues of fluopyram from uses considered by the present Meeting is unlikely to present a public health concern.

REFERENCES

Reference No.	Author(s)	Year	Title, Date, etc.
-	FAO/WHO	2010	Pesticide residues in food 2010 EVALUATIONS 2010 PART 1 - RESIDUES Joint FAO/WHO Meeting on Pesticide Residues (FAO PLANT PRODUCTION AND PROTECTION PAPER 206), pp. 1415-1701
M-564093-02-2	Sarti, A.	2018	Determination of residues of fluopyram and its metabolite in the coffee crop (grains) following the application of fluopyram in trials in Brazil. Report No. I15-018 Addendum No. 01 GLP; Unpublished 2016 (Final report) 2018 (Amendment 1)
M-597632-01-1	Li, Y.	2017	Fluopyram 500 SC - Magnitude of Residues in/on Coffee; Import Tolerances. Report No. RAGMN212 GLP; Unpublished 2011 (Final report) 2017 (Amendment 1)
M-608569-02-2	Li, Y.	2019	Fluopyram 500 SC - Magnitude of Residues in/on Coffee Processed Commodities; Import Tolerances Report No. RAGMN213-01 GLP; Unpublished 2017 (Final report) 2019 (Amendment 1, authored by Harbin. A)

IMAZALIL (110)

First draft prepared by Mr D Lunn, Ministry for Primary Industries, Wellington, New Zealand

EXPLANATION

Imazalil, an imidazole fungicide with protective, curative and anti-sporulant activity, was first evaluated by the JMPR in 1977 and was re-evaluated under the periodic review programme for toxicology and residues by the 2018 JMPR. FAO Specifications for imazalil (technical material and related formulations) were published in 2001.

The 2018 JMPR confirmed the ADI of 0–0.03 mg/kg bw (established by the 1991 JMPR) and the ARfD of 0.05 mg/kg bw (established by the 2005 JMPR).

Residue definitions established by the 2018 JMPR are:

Definition of the residue for compliance with the MRL for plant and animal commodities: *imazalil.*

Definition of the residue for dietary risk assessment for plant commodities: *free and conjugated imazalil.*

Definition of the residue for dietary risk assessment animal commodities: *sum of imazalil and the metabolite R061000 ((RS)-3-[2-(2,4-dichlorophenyl)-2-(2,3- dihydroxypropoxy)ethyl]imidazolidine- 2,4-dione (+)-1-[2-(2,4-dichlorophenyl)-2-[(2,3-dihydroxypropyl)oxy]ethyl]-2,5-imidazolidinedione), expressed as imazalil equivalents.*

The residue is not fat-soluble.

The Fifty-first Session of the CCPR (2019) agreed to advance the proposed draft MRLs for lemons and limes (sub-group) and for sweet and sour oranges (sub-group), as recommended by the 2018 JMPR, and also agreed to retain the CXL for the other citrus fruits under the 4-year rule, awaiting the evaluation by JMPR in 2021.

The Forty-third Codex Alimentarius Commission (2020) approved the new work proposals including the priority list of pesticides for evaluation by the current Meeting. The priority list included imazalil for evaluation of additional uses by the 2021 Extra JMPR.

The current Meeting received new analytical method validation data and supervised residue trials for mandarins and grapefruit, together with confirmation that the citrus GAP information provided to the 2018 JMPR was still valid. The Meeting also considered relevant information submitted to the 2018 JMPR.

RESIDUE ANALYSIS

Analytical methods

The 2018 JMPR reviewed and summarised analytical method descriptions and validation data for imazalil and its alcohol metabolite R014821 (formed by hydroxylation of the alkyl chain) on a wide range of commodities. These included the QuEChERS extraction and LC-MS/MS Method used to measure residues of imazalil and R014821 in the new supervised residue trials on mandarins and grapefruit.

In the method used in the new trials, samples were suspended in 5–10 mL water for at least 5 minutes before extraction with acetonitrile in presence of the content of a QuEChERS Extraction Kit (magnesium sulphate, sodium chloride, sodium hydrogen citrate sesquihydrate and sodium citrate).

Extracts were diluted 10-fold (flesh) or 20-fold (peel) and injected on the LC-MS/MS system. Mass spectrometry detection was carried out using tandem mass spectrometry with positive ionization in electrospray interface. Quantification was carried out by means of matrix matched external calibration curves.

For imazalil, the selected mass transitions were m/z 297→159 (quantification) and m/z 297→69 (confirmation), these differing from those used in the earlier orange and lemon studies (m/z 297→41 for quantification and m/z 297→159 for confirmation).

For R014821, the selected mass transitions were m/z 257→69 (quantification) and m/z 257→125 (confirmation), the same as in the earlier orange and lemon studies.

Validation studies for this method in mandarins and grapefruit were provided to the current meeting.

Table 1 QuEChERS Method validation data for imazalil in mandarins and grapefruit

	Recovery								
	Imazalil		m/z=297→159			m/z=297→69			Reference
Matrix	Fortification (mg/kg)	n	Range (%)	Mean (%)	RSD (%)	Range (%)	Mean (%)	RSD (%)	
Mandarin (flesh)	0.01	5	83–86	85	1.3	82–89	85	3.0	168SRES19R02
	0.1	5	85–91	87	2.6	84–89	87	2.2	
Mandarin (peel)	0.01	5	79–88	85	4.2	78–94	84	7.0	
	0.1	5	76–83	79	3.6	74–83	79	4.1	
Grapefruit (flesh)	0.01	5	82–87	83	2.4	80–89	84	4.3	168SRES19R03
	0.1	5	84–93	86	4.4	82–94	87	5.4	
Grapefruit (peel)	0.01	5	84–93	88	3.5	73–82	77	4.3	
	0.1	5	84–91	87	2.9	87–95	90	3.5	

Table 2 QuEChERS Method validation data for R014821 in mandarins and grapefruit

	Recovery								
	R014821		m/z=257→69			m/z=257→125			
Matrix	Fortification (mg/kg)	n	Range (%)	Mean (%)	RSD (%)	Range (%)	Mean (%)	RSD (%)	Reference
Mandarin (flesh)	0.01	5	89–94	92	1.8	88–89	88	0.7	168SRES19R02
	0.1	5	88–92	90	1.7	85–91	88	2.4	
Mandarin (peel)	0.01	5	83–88	85	2.4	82–86	84	2.4	
	0.1	5	81–88	84	3.0	81–89	84	3.5	
Grapefruit (flesh)	0.01	5	88–96	91	4.1	89–94	91	2.1	168SRES19R03
	0.1	5	88–93	90	2.1	86–95	91	3.8	
Grapefruit (peel)	0.01	5	81–91	87	5.3	84–93	89	4.1	
	0.1	5	86–92	88	2.8	87–93	89	2.7	

USE PATTERNS

Imazalil is registered in many countries, used mostly as a cereal seed treatment and as a post-harvest treatment on fruit (e.g. citrus, banana). For citrus, imazalil treatments include fogging applications of 0.06 kg ai/tonne, dip, drench or spray applications of 0.05–0.5 kg ai/hL, spray wax applications of 0.15–0.4 kg ai/hL and combination drench/spray wax applications.

Citrus post-harvest use patterns reported to the 2018 JMPR and involving total application rates of 0.15 kg ai/hL or more are re-presented in the following table, together with newly submitted US labels confirming the US GAP.

Table 3 Representative authorised uses of imazalil on citrus involving total post-harvest application rates of 0.15 kg ai/hL or more

Country	Form	Application				PHI days
		Method	kg ai/tonne	Concentration (max) kg ai/hL	No.	
Argentina	750 SP	Spray	-	0.2	1	n/a
	400 SC	Drench	-	0.2	1	n/a
		Wax	-	0.4	1	n/a
Australia	50 EC	Wax	-	0.4	1	0
	200SC	Spray	-	0.2	1	0
		Wax	-	0.3	1	0
Central America	750 SG	Drench	-	0.525	1	0
Chile	750 SG	Wax	-	0.15	1	0
	500 EC	Spray	-	0.15	1	0
		Wax	-	0.4	1	0
European countries	75 SL	Spray	-	0.2	1	0
	75 SC	Spray	-	0.15	1	0
	500 EC	Dip, drench	-	0.5	1	0
		Spray	-	0.5	1	0
		Wax	-	0.4	1	0
Israel	500 EC	Wax	-	0.2	1	0
Mexico	500 EC	Dip	-	0.2	1	0
		Wax	-	0.2	1	0
Morocco	200 EC	Wax	-	0.3	1	0
South Africa	500 EC	Dip+ Wax	-	0.05+ 0.3	2	0
		Spray+ Wax	-	0.1+ 0.3	2	0
		Wax	-	0.3	1	0
Turkey	500 EC	Wax	-	0.2	1	0
Uruguay	750 SP	Spray	-	0.2	1	0
	500 EC	Dip, spray	-	0.2	1	0
		Wax	-	0.2	1	0
	200 SC	Spray, wax	-	0.2	1	0
USA	200 SC	Wax		0.2	1	0
		Dip or drench+ Wax		0.075+ 0.2	2	0
		Spray+ Wax		0.1+ 0.2	2	0
	500 EC	Dip or drench	-	0.075	1	0
		Spray		0.1	1	0
		Wax	-	0.2	1	0
		RTU Wax or foam	-	0.4	1	0
		Dip or drench+ Wax		0.075+ 0.2	2	0
		Spray+ Wax		0.1+ 0.2	2	0
Zimbabwe	800 EC	Wax	-	0.32	1	0

For dipping or drenching applications, the recommended dose is mixed with water in a closed circulating system. The fruit is contained in wooden/plastic bins placed on rolling bands directed to the treatment vessel. Fruits are drenched with treating solution inside the vessel. The fruit bins placed on the rolling band are treated in a single application and, after a drying period, bins are further transported into the storage room.

For spraying or waxing, the product is mixed with water or wax and the treatment solution is sprayed on the fruit in a processing line where fruit pass through the sprayer unit on conveyor belts.

In most cases, the fruits are treated once, at the beginning of storage although in the USA, dual treatments are permitted , except for RTU wax or foam treatments). Depending on the intended storage period, treatment may be before storage and again at pack-out. Another scenario would be to treat at pack-out, but to apply both in water (dip, drench, in-line spray) and then shortly thereafter in wax.

RESIDUES RESULTING FROM SUPERVISED TRIALS

The Meeting reviewed new supervised mandarin and grapefruit post-harvest trial information.

The 2018 JMPR also reviewed information from relevent trials on mandarins, grapefruit, oranges and lemons and these are also included below.

Crop Group	Commodity	Region/Country		Table No.
Citrus	Mandarins	Europe, New Zealand	JMPR 2018	4
		Europe	New data	5
	Grapefruit	USA	JMPR 2018	6
		Europe	New data	7
	Oranges		JMPR 2018	8
	Lemons		JMPR 2018	9

The new 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.

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 2018 JMPR.

Results have not been corrected for concurrent method recoveries. 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. 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 included a summary of the method validation, procedural recoveries, and concurrent recoveries in stored frozen samples.

Citrus fruits

The 2018 JMPR evaluated a range of supervised trials involving post-harvest applications of imazalil to citrus. Treatments included fogging applications of 0.06 kg ai/tonne, dip, drench or spray applications of 0.05–0.15 kg ai/hL, spray wax applications of 0.2–0.3 kg ai/hL and combination drench/spray wax applications.

Mandarins

Results from mandarin trials evaluated by the 2018 JMPR and involving combination dip/drench plus wax treatments are re-presented in the following table.

In the 1997 European trial, samples of at least 12 fruit (min 1 kg) were collected 1–3 days after the last treatment, stored frozen for up to 27 days and analysed for imazalil using Method Gent 53/3b (LOQ of 0.05 mg/kg).

In the 2004 European trials, samples of at least 24 fruit (min 3 kg) were collected immediately after the wax applications, stored frozen for up to 58 days and analysed for imazalil using a validated liquid chromatography with diode array detector (LC/DAD) with an LOQ of 0.05 mg/kg.

Table 4 Residues in mandarins following post-harvest imazalil combination dip/drench and wax treatments [See 2018 JMPR Imazalil Evaluation]

Reference No. Location, Year (Variety)	Application			DALA	Portion analysed	Residues (mg/kg)	
	Formulation, Treatment	Rate (kg ai/hL)	No.			Imazalil (mean)	R014821
GAP: USA	dip/drench+ wax spray	0.075+ 0.2	1+ 1	0			
AGR244 Valencia, Spain, 1997 (Satsuma)	75 SL, drench+ 500 EC, wax spray	0.04+ 0.4	1+ 1	0	whole fruit	3.59, 3.59 (3.59)	
AGR244 Valencia, Spain, 1997 (Satsuma)	75 SL, drench+ 500 EC wax spray	0.04+ 0.4	1+ 1	0	whole fruit	2.73, 2.73 (2.73)	
S04S001R La Font d'en Carrós, Valencia, Spain, 2004 (Fortunas)	400 SC, drench+ 400 SC, wax spray	0.05+ 0.3	1+ 1	0	whole fruit	1.96	
S04S002R La Font d'en Carrós, Valencia, Spain, 2004 Ortanique)	400 SC, drench+ 400 SC, wax spray	0.05+ 0.3	1+ 1	0	whole fruit	1.78	

The current Meeting received information on four new European trials on mandarins where imazalil was applied first as a drench application and then (immediately after drying) as a wax spray treatment, applying 2 g ai in 1 litre of wax per tonne of fruit. Samples of at least 24 fruits were separated into peel (min 0.5 kg) and flesh (min 2 kg) and stored frozen for up to 55 days before imazalil and R014821 analysis using a validated QuECHERS, HPLC-MS/MS method. Mean concurrent recoveries in peel were 82% (imazalil) and 83% (R014821) and in flesh were 92% (imazalil) and 91% (R014821). The LOQs were 0.01 mg/kg for each analyte.

Table 5 Residues in mandarins following post-harvest imazalil (500 EC) drench+wax spray treaments [Ref: 168SRES19R02]

Reference No. Location, Year (Variety)	Application			DALA	Portion analysed [a]	Residues (mg/kg)	
	Formulation, Treatment	Rate (kg ai/hL)	No.			Imazalil (mean)	R014821
GAP: USA	dip/drench+ wax spray	0.075+ 0.2	1+ 1	0			
SRES19-608-168FR Valencia, Spain, 2019 (Clemenrubí)	drench+ wax spray	0.075+ 0.1974	1+ 1	0	peel flesh whole fruit	13.7 0.077 2.6	0.255 n.d. 0.048
SRES19-609-168FR Valencia, Spain, 2019 (Okitsu)	drench+ wax spray	0.075+ 0.1992	1+ 1	0	peel flesh whole fruit	14 0.01 3.8	0.139 n.d. 0.038
SRES19-610-168FR Valencia, Spain, 2019 (Cultifort)	drench+ wax spray	0.075+ 0.1973	1+ 1	0	peel flesh whole fruit	15.7 0.041 3.2	0.134 n.d. 0.028
SRES19-611-168FR Valencia, Spain, 2019 (Mioro)	drench+ wax spray	0.075+ 0.201	1+ 1	0	peel flesh whole fruit	14.9 <0.01 2.1	0.165 n.d. 0.024

[a] Whole fruit residues calculated from residues in peel and flesh based on the weight of each fraction

Grapefruit

Results from grapefruit trials evaluated by the 2018 JMPR and involving combination dip/drench plus wax treatments are re-presented in the following table.

In these trials conducted in the USA, samples collected immediately after the last treatment and stored frozen for up to 60 days were analysed for imazalil and R014821 using a validated GC/MS method, with LOQs of 0.05 mg/kg for each analyte.

Table 6 Residues in grapefruit following post-harvest imazalil spray or spray+wax treatments [See 2018 JMPR Imazalil Evaluation]

Reference No. Location, Year (Variety)	Application			DALA	Portion analysed[(1)]	Residues (mg/kg)	
	Formulation, Treatment	Rate (kg ai/hL)	No.			Imazalil (mean)	R014821
GAP: USA	dip/drench+ wax spray	0.075+ 0.2	1+ 1	0			
F833-0/F834-0 Riverside, CA, USA, 1987 (Not specified)	500 EC, spray+ 500 EC, wax spray	0.1+ 0.1	1+ 1	0	peel flesh whole fruit	2.69 0.1 0.85	0.24 <0.05 0.08
F835-0/F836-0 Riverside, CA, USA, 1987 (Not specified)	500 EC, spray+ 500 EC, wax spray	0.1+ 0.1	1+ 1	0	peel flesh whole fruit	2.92 0.1 0.95	0.12 <0.05 0.04
F993-0/F994-0 Riverside, CA, USA 1987 (Not specified)	500 EC, spray+ 500 EC, wax spray	0.1+ 0.1	1+ 1	0	peel flesh whole fruit	2.36 0.1 0.84	0.2 <0.05 0.07

[a] Whole fruit residues calculated from residues in peel and flesh based on the weight of each fraction

The current Meeting received information on four new European trials on grapefruit where imazalil was applied first as a drench application and then (immediately after drying) as a wax spray treatment, applying 2 g ai in 1 litre of wax per tonne of fruit. Samples of at least 12 fruits were separated into peel (min 0.72 kg) and flesh (min 1.8 kg) and stored frozen for up to 50 days before imazalil and R14821 analysis using a validated QuECHERS, HPLC-MS/MS method. Mean concurrent recoveries in peel were 86% (imazalil) and 97% (R014821) and in flesh were 91% (imazalil) and 91% (R014821). The LOQs were 0.01 mg/kg for each analyte.

Table 7 Residues in grapefruit following post-harvest imazalil (500 EC) drench+wax spray treatments [Ref: 168SRES19R03].

Reference No. Location, Year (Variety)	Application			DALA	Portion analysed [a]	Residues (mg/kg)	
	Formulation, Treatment	Rate (kg ai/hL)	No.			Imazalil (mean)	R014821
GAP: USA	dip/drench+ wax spray	0.075+ 0.2	1+ 1	0			
SRES19-612-168FR Spain, 2019 (Duncan)	500 EC, drench+ 500 EC, wax spray	0.075+ 0.2018	1+ 1	0	peel flesh whole fruit	9.85 <0.01 2.6	0.058 n.d. 0.016
SRES19-613-168FR 46210 Picanya, Valencia, Spain, 2019 (Star Ruby)	500 EC, drench+ 500 EC, wax spray	0.075+ 0.1989	1+ 1	0	peel flesh whole fruit	15.1 <0.01 4.7	0.071 n.d. 0.022
SRES19-614-168FR 46210 Picanya, Valencia, Spain, 2019 (Sambar)	500 EC, drench+ 500 EC, wax spray	0.075+ 0.2013	1+ 1	0	peel flesh whole fruit	12.4 <0.01 3.1	0.099 n.d. 0.025
SRES19-615-168FR 46210 Picanya, Valencia, Spain, 2019 (Henderson)	500 EC, drench+ 500 EC, wax spray	0.075+ 0.1772	1+ 1	0	peel flesh whole fruit	12.4 <0.01 3.2	0.103 n.d. 0.027

[a] Whole fruit residues calculated from residues in peel and flesh based on the weight of each fraction

Oranges

Results from orange trials evaluated by the 2018 JMPR and involving combination dip/drench plus wax treatments are re-presented in the following table.

In two post-harvest trials conducted in Spain on oranges during 2004 (Gimeno Martos, 2004, TRC04-9/10/11/12) involving dual treatments of a drench application at 0.05 kg ai/hL followed by a spray wax application at 0.3kg ai/hl, oranges were collected immediately after treatment (0-DAA) and comprised of at least 12 fruit and weighing at least 2 kg. Residues of imazalil were quantified using a validated GC-MS method. The LOQ of the method was 0.05 mg/kg. The storage intervals from collection to extraction did not exceed 122 days.

In post-harvest trials conducted in Greece, Italy and Spain, during 2016 to 2018, oranges were treated with one post-harvest drench application followed by one storage wax line spray application using an emulsifiable concentrate formulation containing 500 g/L of imazalil (Grote, 2017, S16/06757; Grote, 2018, S17-0771). The post-harvest drench application was applied at concentrations of 0.057–0.075 kg

ai/hL. After fruits were dried, one post-harvest storage wax line spray application followed at concentrations of 0.20–0.46 kg ai/hL. Treated whole oranges were collected immediately after the wax application (0- DAA) and comprised of at least 12 fruits and weighing a minimum of 1 kg. Residues of imazalil and the metabolite R014821 were quantified using a validated QuECHERS, HPLC/MS-MS method. The LOQ of the method was 0.01 mg/kg/analyte. The storage intervals from collection to extraction did not exceed 100 days.

Table 8 Residues in orange following post-harvest imazalil spray or spray+wax treatments [See 2018 JMPR Imazalil Evaluation]

Reference No. Location, Year (Variety)	Application			DALA	Portion analysed	Residues (mg/kg)	
	Formulation, Treatment	Rate (kg ai/hL)	No.			Imazalil (mean)	R014821
GAP: USA	dip/drench+ wax spray	0.075+ 0.2	1+ 1	0			
TRC04-10 La Font d'en Carrós, Valencia, Spain, 2004 (Valencia)	400 SC, drench 400SC spray wax	0.05 0.3	1+ 1	0	whole fruit	2.60	
TRC04-10 La Font d'en Carrós, Valencia, Spain, 2004 (Lane Late)	400 SC, drench 400SC spray wax	0.05 0.3	1+ 1	0	whole fruit	2.15	
S16-06757-01 25100 Aigio, Achaia, Greece 2017 (Navel)	500 EC, drench 500 EC, wax	0.075 0.20	1+ 1	0	whole fruit flesh	4.2 0.07	0.02 <0.01
S16-06757-02 50932, Plati, Imathia, Greece 2017 (Navelina)	500 EC, drench 500 EC, wax	0.075 0.20	1+ 1	0	whole fruit flesh	2.7 0.06	0.02 <0.01
S16-06757-03 95032, Belpasso, Sicily, Italy, 2017 (Tarocco)	500 EC, drench 500 EC, wax	0.075 0.20	1+ 1	0	whole fruit flesh	3.0 0.07	<0.01 <0.01
S16-06757-04 95047, Paterno, Sicily, Italy 2017 (Moro)	500 EC, drench 500 EC, wax	0.075 0.20	1+ 1	0	whole fruit flesh	4.4 0.24, 0.28 (0.26)	0.02 <0.01
S16-06757-05 46469, Beniparrell, Valencia, Spain 2016 (Navelina)	500 EC, drench 500 EC, wax	0.075 0.20	1+ 1	0	whole fruit flesh	3.6 0.06	<0.01 <0.01
S16-06757-06 25003, Lleida, Catalunya, Spain 2017 (Navelina)	500 EC, drench 500 EC, wax	0.075 0.20	1+ 1	0	whole fruit flesh	4.8 0.12, 0.13 (0.13)	0.01 <0.01
S17-07771-01 59032, Plati, Imathia, Greece, 2017 (Merlin)	500 EC, drench 500 EC, wax	0.057 0.46	1+ 1	0	whole fruit flesh	3.0 0.17	<0.01 <0.01
S17-07771-02 46469, Beniparrell, Valencia,	500 EC, drench 500 EC, wax	0.063 0.20	1+ 1	0	whole fruit flesh	2.5 0.11	0.01 <0.01

Reference No. Location, Year (Variety)	Application			DALA	Portion analysed	Residues (mg/kg)	
	Formulation, Treatment	Rate (kg ai/hL)	No.			Imazalil (mean)	R014821
Spain, 2017 (Navelina)							
S17-07771-03 46469, Beniparrell, Valencia, Spain, 2018 (Lane Late)	500 EC, drench 500 EC, wax	0.065 0.21	1+ 1	0	whole fruit flesh	3.3, 3.4 (3.4) 0.21, 0.2 (0.21)	0.03 <0.01

Lemons

Results from lemon trials evaluated by the 2018 JMPR and involving combination dip/drench plus wax treatments are re-presented in the following table.

Nine post-harvest trials were conducted in Greece, Italy and Spain, during 2016, where lemons were treated with one post-harvest drench application followed by one storage wax line spray application of an emulsifiable concentrate formulation containing 500 g/L of imazalil (Grote, 2017, S16/06758; Grote, 2017, S17-07772). The post-harvest drench application was applied at a concentration of 0.075 kg ai/hL. After fruits were dried, one post-harvest storage wax line spray application followed at a concentration of 0.20 kg ai/hL. Treated whole lemons were collected immediately after the wax application (0-DAA). and comprised of at least 12 fruit and weighing a minimum of 1 kg. Residues of imazalil and the metabolite R014821 were quantified using a validated QuECHERS, HPLC/MS-MS method. The LOQ of the method was 0.01 mg/kg/analyte. The storage intervals from collection to extraction did not exceed 106 days

Table 9 Residues in lemon following post-harvest imazalil spray or spray+wax treatments [See 2018 JMPR Imazalil Evaluation]

Reference No. Location, Year (Variety)	Application			DALA	Portion analysed	Residues (mg/kg)	
	Formulation, Treatment	Rate (kg ai/hL)	No.			Imazalil (mean)	R014821
GAP: USA	dip/drench+ wax spray	0.075+ 0.2	1+ 1	0			
S16-06758-01 47100, Arta, Arta, Greece, 2017 (Femminello)	500 EC, drench 500 EC, wax	0.075 0.2	1+ 1	0	whole fruit flesh	9.5, **9.9** (9.7) 0.12	0.01 <0.01
S16-06758-02 59032, Plati, Imathia, Greece 2017 (Maglino)	500 EC, drench 500 EC, wax	0.075 0.2	1+ 1	0	whole fruit flesh	7.9, 7.7 (7.8) 0.19, 0.2 (0.2)	0.01 <0.01
S16-06758-03 95032, Belpasso, Sicily, Italy, 2017 (Femminello)	500 EC, drench 500 EC, wax	0.075 0.2	1+ 1	0	whole fruit flesh	3.3 0.05	0.01 <0.01
S16-06758-04 95047, Paterno, Sicily,	500 EC, drench 500 EC, wax	0.075 0.2	1+ 1	0	whole fruit flesh	3.1 0.05	0.02 <0.01

Reference No. Location, Year (Variety)	Application			DALA	Portion analysed	Residues (mg/kg)	
	Formulation, Treatment	Rate (kg ai/hL)	No.			Imazalil (mean)	R014821
Italy 2017 (Monachello)							
S16-06758-05 46469, Beniparrell, Valencia, Spain 2016 (Eureka)	500 EC, drench 500 EC, wax	0.075 0.2	1+ 1	0	whole fruit flesh	3.0 0.11	0.03 <0.01
S16-06758-06 25003, Lleida, Catalunya, Spain 2017 (Eureka)	500 EC, drench 500 EC, wax	0.075 0.2	1+ 1	0	whole fruit flesh	5.0 0.23, 0.22 (0.23)	0.02 <0.01
S17-07772-01 59032, Plati, Imathia, Greece, 2017 (Maglino)	500 EC, drench 500 EC, wax	0.075 0.2	1+ 1	0	whole fruit flesh	6.4 0.27	0.05 <0.01
S17-07772-02 46469, Beniparrell, Valencia, Spain, 2017 (Fino)	500 EC, drench 500 EC, wax	0.075 0.2	1+ 1	0	whole fruit flesh	4.1 0.36	0.01 <0.01
S17-07772-03 46469, Beniparrell, Valencia, Spain, 2018 (Fino)	500 EC, drench 500 EC, wax	0.075 0.2	1+ 1	0	whole fruit flesh	5.8, 5.5 (5.7) 0.18, 0.17 (0.18)	0.02 <0.01

FATE OF RESIDUES IN STORAGE AND IN PROCESSING

Magnitude of the residue in processing

The effect of processing oranges into juice, marmalade, jam, jelly, oil and dry pomace and lemon into juice, oil and dried pulp on the residues of imazalil was evaluated by the JMPR in 2018 and the following processing factors were derived:

Table 10 Processing factors for imazalil in/on citrus processed fractions (2018 JMPR)

Commodity	Calculated Processing Factors (PF) [a]	Best Estimate
Orange		
Orange juice	0.01, < 0.02, 0.03, 0.05, 0.10, 0.11, 0.14, 0.33, 0.35	0.10 (median)
Cold pressed oil	23.8, 33.4	28.6 (mean)
Orange marmalade	0.15, 0.25, 0.25, 0.27, 0.28, 0.56, 0.68	0.27 (median)
Orange jam	0.03, 0.04	0.04 (mean)
Orange jelly	0.02, 0.03	0.03 (mean)
Orange (canned)	< 0.02, 0.04	0.03 (mean)
Dry pomace (dried pulp)	4.0, 4.0, 4.4, 4.5, 4.9, 6.7, 9.6	4.5 (median)
Lemon		
Lemon juice	0.05, 0.04	0.05 (mean)
Cold pressed oil	4.3, 2.6	3.5 (mean)
Dried peel	1.0, 1.0	1.0 (mean)

[a] The processing factor is the ratio of the total residue in the processed item divided by the total residue in the RAC.

APPRAISAL

Imazalil, an imidazole fungicide with protective, curative and anti-sporulant activity, was first evaluated by the JMPR in 1977 and was re-evaluated under the periodic review programme for toxicology and residues by the 2018 JMPR. FAO Specifications for imazalil (technical material and related formulations) were published in 2001.

The 2018 JMPR confirmed the ADI of 0–0.03 mg/kg bw (established by the 1991 JMPR) and the ARfD of 0.05 mg/kg bw (established by the 2005 JMPR).

Residue definitions established by the 2018 JMPR are:

Definition of the residue for compliance with the MRL for plant and animal commodities: *imazalil*

Definition of the residue for dietary risk assessment for plant commodities: *free and conjugated imazalil*

Definition of the residue for dietary risk assessment animal commodities: *sum of imazalil and the metabolite R061000 ((RS)-3-[2-(2,4-dichlorophenyl)-2-(2,3- dihydroxypropoxy)ethyl]imidazolidine- 2,4-dione (+)-1-[2-(2,4-dichlorophenyl)-2-[(2,3-dihydroxypropyl)oxy]ethyl]-2,5-imidazolidinedione), expressed as imazalil equivalents*.

The residue is not fat-soluble.

The 2018 JMPR also concluded that while conjugated imazalil is likely to contribute to the dietary exposure for foliar-treated crops, it is not relevant for post-harvest and seed treatment uses.

The Forty-third Codex Alimentarius Commission (2020) approved the new work proposals including the priority list of pesticides for evaluation by the current Meeting. The priority list included imazalil for evaluation of additional uses by the 2021 Extra JMPR. The Meeting received new US labels (confirming the citrus GAP information reported by the 2018 JMPR) and new supporting residue information for mandarins and grapefruits.

Methods of analysis

The Meeting received additional validation information on the analytical method (based on the QuEChERS extraction and LC-MS/MS Method evaluated by the 2018 JMPR) used for measuring free imazalil and its alcohol metabolite (R014821) in the new supervised residue trials on mandarins and grapefruits.

The Meeting concluded that for mandarins and grapefruit, the method used in the new residue trials was sufficiently validated with a LOQ of 0.01 mg/kg and is suitable to measure imazalil and metabolite R014821 in citrus commodities.

Results of supervised residue trials on crops

New supervised trials from Europe on mandarins and grapefruit were available to supplement those reported by the 2018 JMPR. The demonstrated stability of residues in frozen samples (at least 12 months) covered the storage intervals in the trials considered by the Meeting.

The Meeting also received product labels from the USA that confirmed the GAP reported by the 2018 JMPR.

Citrus fruits

The 2018 JMPR recommended maximum residue levels of 15 mg/kg for the Subgroup of Lemons and Limes and 8 mg/kg for the Subgroup of Oranges, Sweet, Sour but concluded there were insufficient data to recommend maximum residue levels for the sub-group of Mandarins and the sub-group of Pummelo and Grapefruits.

The current Meeting received new supervised residue trials on mandarins and grapefruit (involving combined post-harvest dip plus wax spray treatments) to supplement the residue trial information reviewed by the 2018 JMPR.

The 2018 JMPR identified the critical GAP in the USA for citrus fruits as being the combination of 2 post-harvest applications, dip or drench at 0.075 kg ai/hL followed by a wax application of 0.2 kg ai/hL, for a total application rate of 0.275 kg ai/hL and a minimum post-treatment interval of 0 days.

Mandarins

In four trials matching the critical GAP for citrus fruits, residues of imazalil in mandarins (whole fruit) were: 2.1, 2.6, 3.2 and 3.8 mg/kg and residues in flesh were: < 0.01, 0.01, 0.041 and 0.077 mg/kg.

Grapefruit

In four trials matching the critical GAP for citrus fruits, residues of imazalil in grapefruit (whole fruit) were: 2.6, 3.1, 3.2 and 4.7 mg/kg and residues in flesh were: < 0.01 (4) mg/kg in flesh. The median whole fruit residue is 3.15 mg/kg.

Orange

The Meeting noted that in the eight orange trials evaluated by the 2018 JMPR and matching the US GAP for citrus, residues of imazalil in oranges (whole fruit) were: 2.5, 2.7, 3.0, 3.4, 3.6, 4.2, 4.4 and 4.8 mg/kg and residues in flesh were: 0.06, 0.06, 0.07, 0.07, 0.11, 0.13, 0.21 and 0.26 mg/kg.

Lemon

The Meeting noted that in the nine lemon trials evaluated by the 2018 JMPR and matching the US GAP for citrus, imazalil residues in lemons (whole fruit) were: 3.0, 3.1, 3.3, 4.1, 5.0, 5.7, 6.4, 7.8 and 9.7 mg/kg and residues in flesh were: 0.05, 0.05, 0.11, 0.12, 0.18, 0.2, 0.23, 0.27, and 0.36 mg/kg. The highest single residue value in whole fruit was 9.9 mg/kg.

Since the US GAP is for the citrus fruits group, the median residues of mandarins, grapefruit, oranges and lemons are within a 5-fold difference and a statistical analysis (Kruskal-Wallis) shows that the four data sets are not significantly different, the Meeting agreed to combine the datasets to estimate a maximum residue level for citrus fruit.

The combined dataset of residues in citrus (whole fruit) is (n = 25): 2.1, 2.5, 2.6, 2.6, 2.7, 3.0, 3.0, 3.1, 3.1, 3.2, 3.2, 3.3, <u>3.4</u>, 3.6, 3.8, 4.1, 4.2, 4.4, 4.7, 4.8, 5.0, 5.7, 6.4, 7.8 and 9.7 mg/kg and imazalil residues in flesh are: < 0.01 (5), 0.01, 0.041, 0.05 (2), 0.06 (2), 0.07, <u>0.07</u>, 0.077, 0.11 (2), 0.12, 0.13, 0.18, 0.2, 0.21, 0.23, 0.26, 0.27 and 0.36 mg/kg. The median whole fruit residue is 3.4 mg/kg.

The Meeting estimated a maximum residue level of 15 mg/kg (based on the mean residue + 4SD) for Citrus fruits and withdrew the previous maximum residue level recommendations of 15 mg/kg for the Subgroup of Lemons and Limes and 8 mg/kg for the Subgroup of Oranges, Sweet, Sour.

The Meeting also estimated a STMR of 0.07 mg/kg and a HR of 0.36 mg/kg for Citrus fruits (flesh) except kumquats and a STMR of 3.4 mg/kg and a HR of 9.9 mg/kg (highest individual residue) for kumquats (whole fruit).

Fate of residues during processing

The 2018 JMPR reviewed information on the fate of imazalil during processing and the estimated processing factors for citrus commodities are listed in Table 1.

STMR-Ps and HR-Ps were calculated for mandarin and grapefruit processed commodities using the higher of the orange or lemon processing factors estimated by the 2018 JMPR.

Residues in processed commodities

Table 1 Citrus commodity processing factors (estimated by the 2018 JMPR) and calculated imazalil STMR-Ps and HR-Ps for citrus commodities based on the highest of the orange or lemon processing factors

Processed commodity	Mean or median processing factors		STMR-P (mg/kg) [$STMR_{RAC}$ × PF]	HR-P (mg/kg) [HR_{RAC} × PF]
	Orange	Lemon	Citrus [median: 3.4]	Citrus [Hi Res: 9.9]
Juice	0.1	0.05	0.34	-
Chopped fresh peel	0.39	0.36	1.3	-
Cold pressed oil	28.6	3.5	97	-
Marmalade	0.27		0.92	-
Canned	0.03		0.1	-
Dry pomace (dried pulp)	4.5		15	

The Meeting applied the orange dry pomace processing factor (4.5) to the estimated maximum residue level for citrus fruits (15 mg/kg) to estimate a maximum residue level of 70 mg/kg (dw) for citrus pulp, dry.

The Meeting applied the orange oil processing factor (28.6) to the estimated maximum residue level for citrus fruits (15 mg/kg) to estimate a maximum residue level of 500 mg/kg for citrus oil, edible.

Residues in animal commodities

Farm animal dietary burden

Dietary burden calculations for beef cattle and dairy cattle and poultry were calculated by the 2018 JMPR, using the OECD diets listed in Appendix IX of the 2016 Edition of the FAO Manual. Maximum dietary burdens were 28.9 ppm (beef cattle), 23.4 ppm (dairy cattle) and 2.3 ppm (poultry) and mean dietary burdens were 25.3 ppm (beef cattle), 19.8 ppm (dairy cattle) and 1.1 ppm (poultry).

The Meeting recalculated these dietary burdens to account for the higher residues in dry citrus pulp, and while the dietary burdens based on the Australian, Japanese and US-Canadian (poultry only) diets

increased albeit insignificantly, the highest maximum and mean dietary burdens (based on the EU diet) remained unchanged.

Residue in animal commodities

Since the revised maximum and mean dietary burdens for beef and dairy cattle and for poultry broilers and layers were the same as those calculated by the 2018 JMPR, the Meeting agreed that the maximum residue levels, STMRs and HRs recommended by the 2018 JMPR for animal commodities did not need to be revised.

RECOMMENDATIONS

On the basis of the data obtained from supervised trials, the Meeting concluded that the residue levels listed below in Table 2 are suitable for establishing maximum residue limits and for IEDI and IESTI assessments.

Definition of the residue for compliance with the MRL for plant and animal commodities: *imazalil.*

Definition of the residue for dietary risk assessment for plant commodities: *free and conjugated imazalil.*

Definition of the residue for dietary risk assessment for animal commodities: *the sum of imazalil and the metabolite R061000 ((RS)-3-[2-(2,4-dichlorophenyl)-2-(2,3- dihydroxypropoxy)ethyl] imidazolidine-2,4-dione (+)-1-[2-(2,4-dichlorophenyl)-2-[(2,3-dihydroxypropyl)oxy]ethyl]-2,5-imidazolidinedione), expressed as imazalil equivalents*.

The residue is not fat-soluble.

Table 2 Recommendations for residues of imazalil from the 2021 Extra JMPR

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
FC 0001	Citrus fruits	15 Po		0.07 (except kumquats) 3.4 (kumquats)	0.36 (except kumquats) 9.9 (kumquats)
FC 0002	Lemons and limes, Subgroup of	W	15 Po		
FC 0004	Oranges, sweet, sour, Subgroup of	W	8 Po		
AB 0001	Citrus pulp, dry	70 (dw) Po		15 (dw)	
OR 0001	Citrus oil, edible	500 Po		97	
JF 0001	Citrus juice			0.34	
	Citrus canned			0.1	
	Citrus marmalade			0.92	
	Citrus peel (chopped)			1.3	

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for imazalil is 0–0.03 mg/kg bw. The International Estimated Daily Intakes (IEDIs) for imazalil were estimated for the 17 GEMS/Food Consumption Cluster Diets using the STMR or STMR-P values estimated by the JMPR. The results are shown in Annex 3 of the 2021 Extra JMPR Report.

The IEDIs ranged from 3–40% of the maximum ADI. The Meeting concluded that long-term dietary exposure to residues of imazalil from uses considered by the JMPR is unlikely to present a public health concern.

Acute dietary exposure

The ARfD for imazalil is 0.05 mg/kg bw. The International Estimate of Short Term Intakes (IESTIs) for imazalil were calculated for the food commodities and their processed commodities for which HRs/HR-Ps or STMRs/STMR-Ps were estimated by the present Meeting and for which consumption data were available. The results are shown in Annex 4 of the 2021 Extra JMPR Report.

The IESTIs varied from 0–40% of the ARfD for children and 0–50% of the ARfD for the general population. The Meeting concluded that acute dietary exposure to residues of imazalil from uses considered by the present Meeting is unlikely to present a public health concern.

REFERENCES

Reference	Author(s)	Year	Title
JMPR 2018	FAO/WHO	2018	Pesticide residues in food 2018. Evaluations. Part 1: Residues. Imazalil (110), pp 573-692.
168SRES19R02	Corts V.	2020	Determination of residues of imazalil and its metabolite R014821 in mandarin (C*itrus paradisi*) following a post-harvest double application by drencher and wax spray of Fecundal 500 EC (Imazalil 50% w/v) in Southern Europe 2019. SynTech Research Spain S.L. Report no. 168SRES19R02. Janssen Pharmaceutica N.V. Reference no. AGR 6001 GLP, unpublished. 28 February 2020
168SRES19R03	Corts V.	2020	Determination of residues of imazalil and its metabolite R014821 in grapefruit (C*itrus reticulata*) following a double post-harvest application by drencher and wax spray of Fecundal 500 EC (Imazalil 50% w/v) in Southern Europe 2019. SynTech Research Spain S.L. Report no. 168SRES19R03. Janssen Pharmaceutica N.V. Reference no. AGR 6002 GLP, unpublished. 16 March 2020

ISOPROTHIOLANE (299)

First draft prepared by Mr D Poflotski, Australian Pesticides and Veterinary Medicines Authority, Armidale, Australia

EXPLANATION

Isoprothiolane is a fungicide belonging to the family of dicarboxylic acids, which act by inhibition of phospholipid biosynthesis. It was considered for the first time for toxicology and residues by the 2017 JMPR. An ADI of 0–0.1 mg/kg bw was established and an ARfD was considered to be unnecessary. The 2017 JMPR decided on the following residue definitions based on a metabolism study on paddy rice and confined rotational crops, and recommended maximum residue levels for husked rice, rice straw and for animal commodities.

The residue definition for compliance with the MRL for plant commodities is *isoprothiolane*.

The residue definition for dietary risk assessment for rice is *isoprothiolane*.

The residue definition for dietary risk assessment for plant commodities other than rice is the sum of *isoprothiolane, diisopropyl-4-hydroxy-1,3-dithiolan-2-ylidenemalonate (M-3); free and conjugated, and 1-hydroxypropan-2-yl propan-2-yl 1,3-dithiolan-2-ylidenemalonate (M-5); free and conjugated, expressed as isoprothiolane*.

The residue definition for compliance with the MRL and dietary risk assessment for animal commodities is the sum of *isoprothiolane and 2-(1,3-dithiolan-2-ylidene)-3-oxo-3-(propan-2- yloxy)propanoic acid (M-2), expressed as isoprothiolane*.

The residue is not fat-soluble.

Isoprothiolane was scheduled at the Fifty-first Session of the CCPR for the consideration by the 2020 Meeting of additional MRLs, which was postponed to the 2021 Extra JMPR.

The Meeting received information on metabolism in apples and grapes, physical and chemical properties, analytical methodology, storage stability, a registered use pattern for bananas and supervised residue trials on bananas.

PHYSICAL AND CHEMICAL PROPERTIES

Technical grade Active Ingredient

The meeting received new physical and chemical property information on the technical grade material. The 2017 JMPR evaluation considered information on the pure active ingredient.

Table 1 Physical and chemical properties of isoprothiolane

Property	Results	Test material purity	Reference
Isoprothiolane			
Melting point	Melting point 54.3 – 55.9 ˚C.	98.9 % w/w	Goia 2019b, PC-2075
Description of the physical state and colour, purity of the ai. and of technical grade	Yellow crystalline solid with characteristic odour	98.9 % w/w	Goia 2019e, PC--2079

Property	Results	Test material purity	Reference
Isoprothiolane			
Solubility of purified active substance in water	4.46 ×10^{-2} g/L at 20 °C (pH 6.15)	98.9 % w/w	Gomes da Silva 2019c, PC-2071
Solubility in organic solvents	[g/L at 20 °C ± 0.5 °C] acetone 1107.78 n-hexane 18.13	98.9 % w/w	
n-Octanol/ water partition coefficient	log P_{ow} 2.86 at pH 7.83 (25 °C)	98.9 % w/w	Gomes da Silva 2019b, PC-2070
Dissociation in water of purified active substance	Isoprothiolane does not dissociate	98.9 % w/w	Goia 2019a, PC-2074
Relative density	1.2588 at 20 °C	98.9 % w/w	Goia 2019b, PC-2075
Vapour pressure	8.53 ×10^{-2} Pa at 25 °C	98.9 % w/w	Gomes da Silva 2019a, PC-2069
pH	5.67 at 20 °C	98.9 % w/w	Goia 2019d, PC-2077

METABOLISM

Metabolism studies on isoprothiolane in paddy rice and a lactating goat were provided to and evaluated by the 2017 JMPR. To support the estimation of maximum residue levels for fruits, new metabolism studies involving foliar application to apples and grapes and a metabolism study involving soil application to apples have been provided.

The studies for plant metabolism, in apples and grapes, were conducted with the test material shown below, with the labelled positions indicated in the following structural formulae:

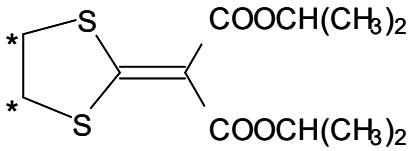
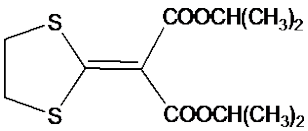
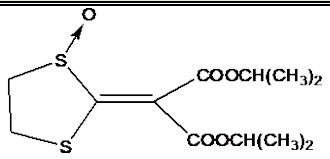
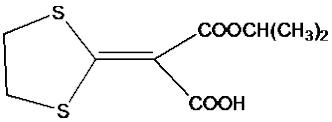
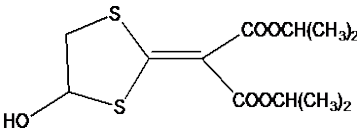
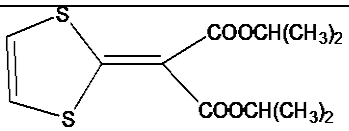
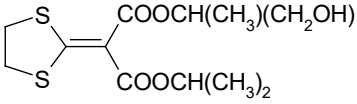
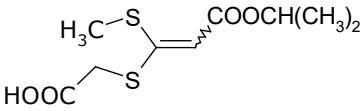
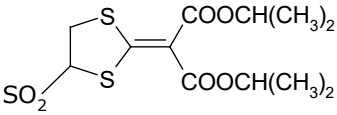
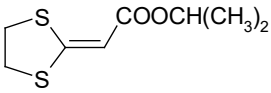
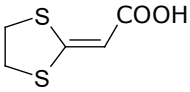
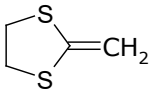


Figure 1 [^{14}C]-isoprothiolane, labelled at the C4 and C5 positions of the dithiolane ring

Table 2 summarizes the names, codes, and structures of the parent and principal metabolites found in plants, lactating goats, rats and rotational crops.

Table 2 Isoprothiolane and metabolites/degradates found in its metabolism in plants and animals

Name	Chemical name	Structure	Compound found in
Isoprothiolane (parent)	Diisopropyl 1,3-dithiolan-2-ylidenemalonate		Rat Mouse Goat fat Rice grain Grape Apple Rot. crops

Name	Chemical name	Structure	Compound found in
Isoprothiolane monosulfoxide (M-1)	Diisopropyl 1- oxo-1,3- dithiolan-2- ylidenemalonate		Mouse (including glucuronide) Rice grain Grape Apple Rot. crops
Isoprothiolane monoester (M-2)	Monoisopropyl 1,3-dithiolan-2- ylidenemalonate		Rat (including glucuronide) Mouse Goat offal [a], muscle, fat Rice grain Rot. crops
4-hydroxy isoprothiolane (M-3)	Diisopropyl 4- hydroxy-1,3-dithiolan-2- ylidenemalonate		Rat Mouse Goat fat Rice grain Grape Apple Rot. crops
Didehydro isoprothiolane (M-4)	Diisopropyl 1,3- dithiol-2-ylidenemalonate		Rat Apple Rot. crops
Hydroxyl-Isopropyl (M-5)	1-Hydroxypropan-2-yl isopropyl 1,3-dithiolan-2-ylidenmalonate		Rot. crops
U9	-		Rat
-	-		Mouse
Isoprothiolane – demonoisopropoxy-carbonyl derivative	-		Mouse
-	-		Mouse (intermediate)
Isoprothiolane- dediisopropoxy-carbonyl derivative	-		Mouse

[a] including glucuronide conjugate (RT-19)

Rot. crops = Rotational crops (lettuce, radish and wheat)

Plant Metabolism

The Meeting received studies for metabolism of isoprothiolane in apples and grapes.

Apple - Foliar treatment

The metabolism of isoprothiolane was also investigated in pot grown dwarf apple trees following foliar treatment (Takahashi, 2007, R-2050). Apple fruit and leaves were directly treated with ^{14}C-isoprothiolane (radiolabelled in the dithiolane ring) as a solution in methanol at 3.14 µg ai/fruit or 1.57 µg ai/leaf. The application rate was equivalent to ca. 0.6 kg ai/ha. The fruits and leaves were collected 7 and 14 days after treatment (DAT).

Samples were surface washed with methanol (MeOH), then homogenised and sequentially extracted with MeOH followed by MeOH/water (4:1 v/v). Obtained extracts were subjected to radioanalysis by LSC. The extracts were profiled by TLC and HPLC against known reference standards.

Most of the radioactive residue in fruit and leaves was readily extractable with MeOH and MeOH/water (92.1–98.8% TRR). From the methanol surface washings, approximately 50% TRR was extracted from the leaf whilst only approximately 20% TRR was extracted from the fruit, suggesting that isoprothiolane is more readily absorbed into the fruit than the leaf. Over time radioactivity concentrations in both fruits and leaves decreased in the methanol washings and extract and increased in the methanol/water extract, suggesting some conversion to more polar metabolites.

The most abundant component of the extractable residue in fruit and leaves was parent isoprothiolane at 49.3% TRR (0.40 mg/kg) and 53.9% TRR (3.24 mg eq/kg), respectively, at 7 DAT. Isoprothiolane residues in fruit and leaves were observed to decrease quickly with a longer DAT. At 14 DAT, residues of isoprothiolane were observed at 26.6% TRR (0.20 mg/kg) in fruit and 40.3% TRR (2.09 mg/kg) in leaves, corresponding with an increase in the amount of polar material (TLC origin). The total components of TLC origin represented 31.9–32.3% TRR in fruit and leaves at 7 DAT and 54.4% TRR in fruit and 42.7% TRR in leaves at 14 DAT. The nature of the metabolites remaining at the TLC origin was investigated, with glucose conjugates of the 4-hydroxy metabolite being characterised as representing approximately 6.9% TRR in fruit and 11.7% TRR in leaves at 7 DAT and 13.1% TRR in fruit and 14.7% TRR in leaves at 14 DAT. The other components, the 4 hydroxy-derivative, monoester derivative, monosulfoxide derivative and didehydro derivative were all observed at levels below 10% TRR.

Table 3 Distribution of residues of ^{14}C-isoprothiolane in apple following surface treatment

	Fruit				Leaves			
	7 DAT		14 DAT		7 DAT		14 DAT	
	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg
MeOH surface wash	19.6	0.16	10.8	0.08	50.3	3.03	42.8	2.22
MeOH extract	72.0	0.58	74.1	0.56	47.3	2.85	53.8	2.79
MeOH/water extract	3.8	0.03	7.1	0.05	1.2	0.07	1.5	0.08
Post-extraction solids	4.7	0.04	7.9	0.06	1.2	0.07	1.9	0.10
TRR	100	0.81	100	0.76	100	6.02	100	5.18

Table 4 Metabolite profile of ^{14}C-isoprothiolane in apple following surface treatment

	Fruit				Leaves			
	7 DAT		14 DAT		7 DAT		14 DAT	
	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg

Total extract profiled[a]	95.3	0.77	92.1	0.70	98.8	5.95	98.1	5.08
Isoprothiolane	49.3	0.40	26.6	0.20	53.9	3.24	40.3	2.09
4-hydroxy-isoprothiolane	2.1	0.02	1.0	<0.01	1.4	0.09	2.6	0.13
Isoprothiolane monoester	ND	ND	ND	ND	ND	ND	0.1	<0.01
Isoprothiolane monosulfoxide	4.2	0.03	4.1	0.03	7.5	0.45	9.0	0.47
Didehydro isoprothiolane	6.6	0.05	4.9	0.04	3.8	0.23	3.4	0.17
TLC-origin[b]	31.9 [6.9]	0.26 [0.06]	54.4 [13.1]	0.41 [0.10]	32.3 [11.7]	1.94 [0.70]	42.7 [14.7]	2.21 [0.76]
Unidentified metabolites	1.2	0.01	1.2	<0.01	ND	ND	ND	ND
Total identified	69.1	0.56	49.7	0.38	78.3	4.71	70.1	3.63

ND; Not detected (LOQ; <0.01 mg eq/kg)

[a] Sum of radioactivity in MeOH wash, MeOH and MeOHCl/H_2O

[b] Values in [] are 4-hydroxy glucose conjugate

Apple - Soil treatment

The metabolism of isoprothiolane was investigated in apples (Takahashi, 2006, R-2011). The soil around pot-grown dwarf apple trees was treated with ^{14}C-isoprothiolane (radiolabelled in the dithiolane ring) as a methanol solution of isoprothiolane of 2.27 mg/mL (equivalent to 3 kg/tree, 12% granule formulation/tree or 360 g ai/tree, based on tree canopy volume). Samples of apple fruits and leaves were collected at 7, 28 and 61 DAT.

Samples were sequentially extracted with MeOH followed by MeOH/water (4:1 v/v). The residues were further extracted with MeOH/1 mol/L HCl (4:1 v/v) and MeOH/1 mol/L NaOH (4:1 v/v). Solvent extracts were subjected to radioanalysis by LSC. The extracts were profiled by TLC and HPLC against known reference standards. Radioactivity remaining at the origin of TLC plates was scraped off the plates and extracted with methanol. The resulting extracts were subjected to β-glucosidase hydrolysis.

In fruit at harvest, residues were very low and most of the radioactive residue (95.0% TRR) was readily extractable with MeOH. As the TRR was <0.01 mg eq/kg, no metabolite identification was performed.

In leaves at 61 DAT, the radioactive residues were extractable with MeOH, MeOH/water (4:1 v/v) and MeOH/1 mol/L HCl (4:1 v/v), with 72.2% TRR being readily solvent extractable. Parent isoprothiolane was detected only at low levels (< 0.01 mg/kg, 0.7% TRR), along with the isoprothiolane monoester and monosulfoxide metabolites. The major metabolite fraction (0.24 mg eq/kg, 65.5% TRR) was at the origin on TLC, part of which was characterised by β-glucosidase treatment as a glucoside conjugate of a monoester metabolite (0.05 mg eq/kg, 13.8% TRR). The remainder of the TLC origin material comprised multiple unidentified components at < 10% TRR each. As the TRR was < 0.01 mg eq/kg in leaves at 7 DAT, no metabolite identification was performed.

Table 5 Distribution of residues of ^{14}C-isoprothiolane in apple following soil application

	Fruit						Leaves					
	7 DAT		28 DAT		61 DAT		7 DAT		28 DAT		61 DAT	
	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg
MeOH	ND	ND	ND	ND	95.0	<0.01	57.7	<0.01	39.0	0.02	48.4	0.18
MeOH/water	ND	ND	ND	ND	ND	ND	42.3	<0.01	26.1	0.01	23.8	0.09

MeOH/1N HCl	-	-	-	-	-	-	-	-	-	-	20.8	0.08
MeOH/1N NaOH	-	-	-	-	-	-	-	-	-	-	3.0	0.01
Post-extraction solids	ND	ND	ND	ND	5.0	<0.01	ND	ND	35.0	0.02	4.0	0.01
TRR	ND	ND	ND	ND	100	<0.01	100	<0.01	100	0.06	100	0.36

ND; Not detected (LOQ; 0.01 mg eq/kg)

Table 6 Metabolite profile of ^{14}C-isoprothiolane in apple following soil application

	Leaves			
	28 DAT		61 DAT	
	%TRR	mg eq./kg	%TRR	mg eq./kg
Total extract profiled [a]	65.0	0.04	72.2	0.26
Isoprothiolane	ND	ND	0.7	<0.01
4-Hydroxy-isoprothiolane	ND	ND	ND	ND
Isoprothiolane monoester	ND	ND	1.2	<0.01
Isoprothiolane monosulfoxide	3.4	<0.01	3.0	0.01
Didehydro isoprothiolane	ND	ND	ND	ND
TLC-origin	61.7	0.03[b]	65.5[c]	0.24[c]
Unidentified metabolites	ND	ND	1.8	<0.01
Total identified	3.4	<0.01	18.7	0.07

ND; Not detected (LOQ; 0.01 mg eq/kg)

[a] Sum of radioactivity in methanol and methanol/water extracts

[b] No single metabolite exceeded 0.01 mg eq/kg

[c] Contained monoester glucoside (13.8% TRR, 0.05 mg eq/kg)

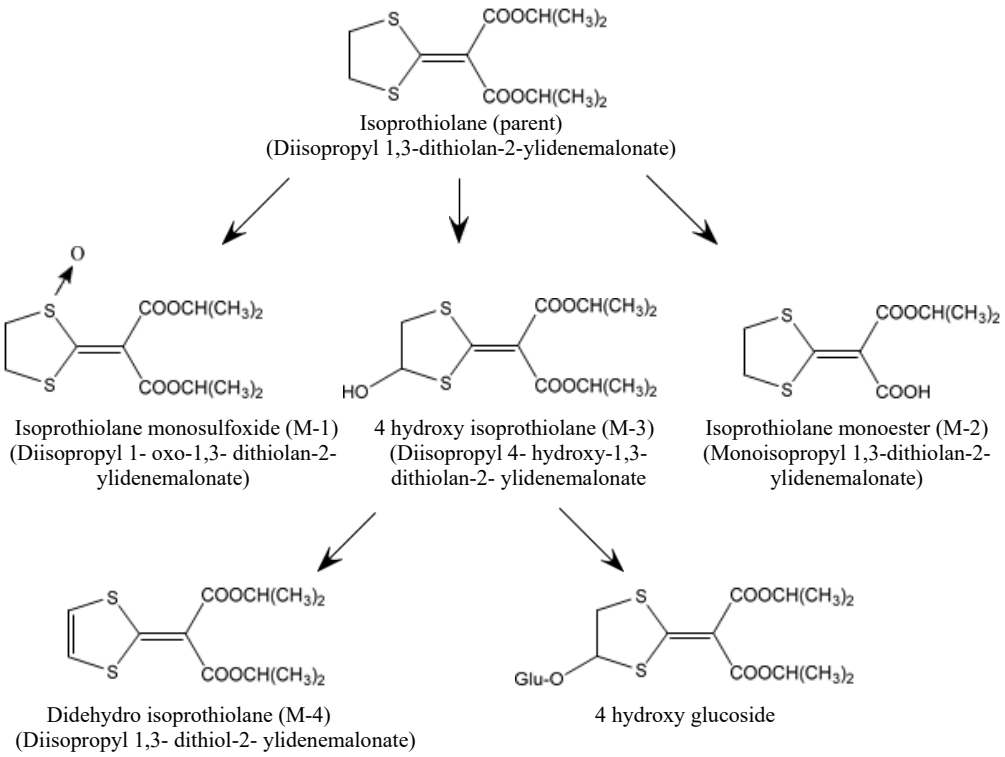


Figure 2 Proposed metabolic pathway of isoprothiolane in apples.

Isoprothiolane was oxidised to isoprothiolane monosulfoxide, dealkylated to form the isoprothiolane monoester and hydroxylated to a 4-hydroxy- isoprothiolane metabolite; the latter subsequently forms a glycoside conjugate and didehydro isoprothiolane.

Grape

The metabolism of isoprothiolane was investigated in grapes (Chang Ahn 2017, R-2151). Isoprothiolane, radiolabelled at positions C4 and C5 of the dithiolane ring was prepared as a 40% emulsifiable concentrate formulation. Isoprothiolane was applied four times to 'Merlot' grapevines (17-year-old vines), grown outdoors in sandy loamy soil, at 70, 42, 21 and 0 days before harvest. The first 3 applications were applied to grapevines at a nominal application rate of 1.46 kg ai/ha. The final application was applied at a nominal rate of 0.4 kg ai/ha. Samples of grapes (fruit) were collected 20 days after 3 applications (20 DAA3, harvest 1) and 0 days after 4 applications (0 DAA4, harvest 2), at a crop growth stage of BBCH 89. Samples were placed into coolers and transported on wet ice to the laboratory on the same day as harvest.

Grape samples were rinsed twice with acetonitrile and stored in a freezer until processing. The acetonitrile rinses were filtered prior to analysis. The total ^{14}C content of the acetonitrile rinses was measured by Liquid Scintillation Counting (LSC). The rinsed grape samples were homogenised in the presence of dry ice and allowed to sublime in the freezer overnight. Samples were extracted twice with acetonitrile/water (ACN/H_2O; 1:1, v/v). An aliquot of each extract was removed and centrifuged followed by further homogenisation. The extraction was performed using mechanical shaking and centrifugation to remove the solid particles from the supernatants. The obtained extracts and post-extraction solids (PES) were combusted and analyzed by LSC. The sum of the radioactivity in all fractions was calculated as the Total Radioactive Residue (TRR), expressed as mg/kg equivalents of the parent compound isoprothiolane (mg eq/kg). Any PES exceeding 10% TRR were further extracted sequentially in mixtures of 0.2 mol/L HCl/ACN (1:1, v/v) and 0.2 mol/L NH_4OH/ACN (1:1, v/v) and if required cellulose treatment, refluxing with 6 mol/L HCl followed by refluxing with 10 mol/L NaOH. The individual acidic and basic extracts were collected separately following centrifugation. The nature of the radioactive residues in the rinses and combined grape extracts were analyzed by both High Pressure Liquid Chromatography (HPLC) and Two-dimensional Thin-Layer Chromatography (2D-TLC).

At 20 DAA3, total radioactive residues were 3.01 mg eq/kg. The majority of radioactive residues (90.6% TRR, 2.73 mg eq.kg) were obtained from the ACN rinse (17.5% TRR, 0.53 mg eq/kg), combined ACH/H_2O (70.9% TRR, 2.13 mg eq/kg) and combined weak acid/base extracts (2.2% TRR, 0.07 mg eq/kg). 9.4% of the TRR (0.28 mg eq/kg) remained in the PES and was not extracted further.

At 0 DAA4, total radioactive residues were 3.43 mg eq/kg. The majority of radioactive residues (89.2% TRR, 3.08 mg eq/kg) were obtained from the ACN rinse (18.0% TRR, 0.62 mg eq/kg), combined ACH/H_2O (69.1% TRR, 2.37 mg eq/kg) and combined weak acid/base extracts (2.7% TRR, 0.09 mg eq/kg). 10.2% of the TRR (0.35 mg eq/kg) remained in the PES. Following further extraction, cellose treatment released 0.7% TRR (0.03 mg eq/kg), acid extraction released 1.5% TRR (0.05 mg eq/kg) and alkaline extraction released 8.0% TRR (0.27 mg eq/kg). No radioactivity remained in the PES after the final alkaline treatment. Following partition of the acid and alkaline extracts with ethyl acetate, the maximum single residual fraction was 2.4% TRR (0.12 mg eq/kg) was the precipitate.

The ACN rinse was analyzed by HPLC. Isoprothiolane, comprising 5.0–8.8% TRR (0.17–0.27 mg/kg), and isoprothiolane monosulfoxide, comprising 7.9–12.5% TRR (0.24–0.43 mg eq/kg), were

determined as the major residues and confirmed by TLC. An unknown residue was present at 0.6–0.7% TRR (0.02 mg eq/kg).

The combined ACN/H_2O extracts, analyzed by HPLC, isoprothiolane represented 41.6–41.7% TRR (1.25–1.43 mg/kg), while isoprothiolane monosulfoxide was a minor residue at 3.3–6.7% TRR (0.10–0.23 mg eq/kg). Isoprothiolane and isoprothiolane monosulfoxide were confirmed by TLC. Three different glycoside conjugates of 4-hydroxy-isoprothiolane were present at 2.3–7.6% TRR (0.07–0.23 mg eq/kg). The presence of the aglycone, 4-hydroxy-isoprothiolane, of the conjugates was confirmed by HPLC and 2D-TLC after hydrolysis with β-glucosidase.

Table 7 Metabolites of isoprothiolane in grapes

DAT	Harvest 1 (20 DAA3)		Harvest 2 (0 DAA4)	
	%TRR	mg eq./kg	%TRR	mg eq./kg
ACN Rinse	17.5	0.53	18.0	0.62
Isoprothiolane	8.8	0.27	5.0	0.17
Isoprothiolane monosulfoxide	7.9	0.24	12.5	0.43
Unassigned other (RT31.8)	0.7	0.02	0.6	0.02
Combined ACN/H_2O Extract	70.9	2.13	69.1	2.37
Isoprothiolane	41.6	1.25	41.7	1.43
Isoprothiolane monosulfoxide	3.3	0.10	6.7	0.23
Glycoside conjugate 3 of 4-Hydroxy-isoprothiolane	3.3	0.10	3.6	0.12
Glycoside conjugate 2 of 4-Hydroxy-isoprothiolane	2.3	0.07	3.8	0.13
Glycoside conjugate 1 of 4-Hydroxy-isoprothiolane	7.6	0.23	6.6	0.23
Ninhydrin-positive protein residues (RT3) [a]	8.6	0.26	6.6	0.23
Unassigned others	4.3	0.13	-	-
Max. other single	2.4	0.07	-	-
0.2N HCl/ACN (1:1, v/v) Extract	1.2	0.04	1.4	0.05
0.2N NH_4OH/ACN (1:1, v/v) Extract	1.0	0.03	1.2	0.04
Combined Weak Acidic/Basic Extracts	2.2	0.07	2.7	0.09
Isoprothiolane	0.3	<0.01	0.3	0.01
Isoprothiolane monosulfoxide	0.2	<0.01	0.3	0.01
Unassigned others	1.7	0.05	2.0	0.07
Max. other single (RT3.4)	0.6	0.02	0.5	0.02
PES	9.4	0.28	10.2	0.35
Cellulase Extract	-	-	0.7	0.03
6N HCl extract at reflux	-	-	1.5	0.05
Acidic aqueous layer	-	-	1.4	0.05
Combined EtOAc layer	-	-	0.1	<0.01
10N NaOH extract at reflux	-	-	8.0	0.27
Basic aqueous layer [A] [b]	-	-	0.4	0.01
Combined EtOAc layer	-	-		
Partition of basic aqueous layer [A] after adjusting to ~pH 3 [c]	-	-	-	-
Acidic aqueous layer [B]	-	-	2.8	0.10
Combined EtOAc layer	-	-	1.3	0.05
Precipitate	-	-	2.4	0.12

[a] Extractable protein/peptide residues, which were visible to purple colour by ninhydrin reaction to free amine groups.

[b] The extract was partitioned with EtOAc under basic conditions.

[c] Aqueous layer [A] obtained from EtOAc partition under basic conditions was acidified and centrifuged to separated biosolids. The acidic supernatant was partitioned.

- Not assayed

20 DAA3 = 20 days after 3 applications (3× 1.46 kg ai/ha)

0 DAA4 = 0 days after 4 applications (3× 1.46 g ai/ha + 1× 0.4 g ai/ha)

The largest component of the radioactivity observed in mature grapes was parent isoprothiolane at 47.0–50.7% TRR (1.53–1.61 mg/kg). The second most abundant component was isoprothiolane monosulfoxide, present at 11.4–19.5% TRR (0.34–0.67 mg eq/kg). Three glycoside conjugates of 4-hydroxy-isoprothiolane were also observed. These were all individually present at < 10% TRR (2.3–7.6% TRR, 0.07–0.23 mg eq/kg), but considering the sum of the conjugates 13.2–14.0% TRR (0.39–0.48 mg eq/kg), these represent a significant portion of radioactivity. Radioactivity was also incorporated into natural products, such as proteins (ninhydrin-positive residues), which were present at 6.6–8.6% TRR (0.23–0.26 mg eq/kg).

Significant proportions of the radioactive residue were identified (75% TRR 20 DAA3; 81% TRR 0 DAA4). Individual unknowns were all < 10% TRR and, in general, were present at low levels (0.01–0.05 mg eq/kg) and were not characterised. The unknown component with the highest concentration was observed at harvest 1 (0.07 mg eq/kg, 2.4% TRR). Residues were shown to be highly extractable (87–88%) in ACN and ACN/water.

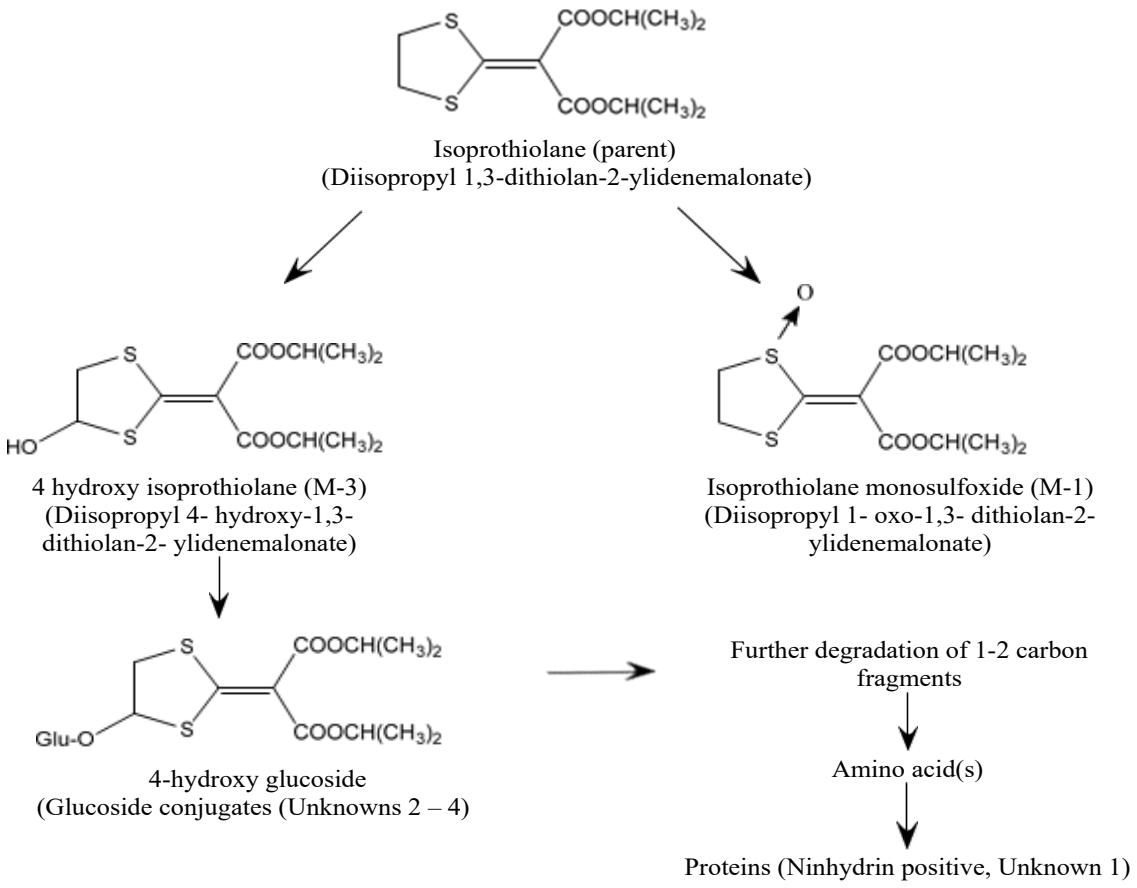


Figure 3 Proposed metabolic pathway of isoprothiolane in grapes

Isoprothiolane was oxidised to isoprothiolane monosulfoxide and hydroxylated to a 4-hydroxy-isoprothiolane metabolite; the latter subsequently forms glycoside conjugates. Further degradation to 1 - 2 carbon units is assumed, which are incorporated into natural products such as proteins/peptides.

Metabolism studies showed that isoprothiolane was the major residue in all plant matrices. Metabolism of the active substance generally produced only low level of minor metabolites except in grapes where isoprothiolane monosulfoxide (M-1) was observed as a significant component (up to 19.5% TRR) and 4-hydroxy-isoprothiolane (M-3) (free and conjugated) was observed at up to 14.0% TRR.

The metabolism in apples, grapes and paddy rice was considered similar. Isoprothiolane was oxidised to isoprothiolane monosulfoxide and hydroxylated to a 4-hydroxy-isoprothiolane metabolite; the latter subsequently forms glycoside conjugates. The apple and rice pathways also proposed that isoprothiolane is dealkylated to form the isoprothiolane monoester.

RESIDUE ANALYSIS

Analytical methods

The Meeting received details of analytical methods including validation data for the determination of isoprothiolane and key metabolites in bananas and these are considered satisfactory. The analytical methods for bananas are summarised below.

Table 8 Summary of the analytical methods submitted for isoprothiolane

Author, Year, Report ID Method ID	Matrix	Analytes	Extraction	Clean-up	Separation / Analysis/LOQ [a)]
Enforcement					
Swaim 2016, A-2031 (Method 82884-M included as an appendix in R-2159)	Banana (whole fruit, peel, and pulp)	Isoprothiolane	Acetonitrile	-	HPLC-MS/MS （internal standard quantification） LOQ: 0.01 mg/kg
Swaim 2019a, - (QuEChERS method 88449)	Banana (whole fruit)	Isoprothiolane	Acetonitrile	-	HPLC-MS/MS （internal standard quantification） LOQ: 0.01 mg/kg
Swaim 2019b, A-2032, (QuEChERS validation for method 88449)					
Keenan 2019, A-2034, (3208W Independent laboratory validation of QuEChERS method)					
Data Generation					
Swaim 2018, A-2033 (Method No. 86846-M)	Banana (whole fruit, peel, and pulp)	Isoprothiolane Isoprothiolane Monosulfoxide 4-HydroxyIsoprothiolane (free and conjugated)	Acetonitrile	SPE clean-up	LC-MS/MS （internal standard quantification） LOQ: 0.01 mg/kg

[a] Defined by the lowest limit of method validation

Method 82884-M (enforcement and data generation): The method for the determination of residues of isoprothiolane in bananas by high performance liquid chromatography with tandem mass spectrometry (HPLC-MS/MS) was reported by Swaim in 2016.

Residues of isoprothiolane were extracted from banana (whole fruit, pulp and peel) samples using a mixture of acetonitrile: water (4:1, v/v). For analysis the extracts were further diluted with acetonitrile and quantified by high performance liquid chromatography with tandem mass spectrometry (HPLC-MS/MS). Quantitation was by external standards monitoring the ion transitions *m/z* 291 → 231 for quantitation and *m/z* 291 → 189 and *m/z* 291→ 145 for confirmation.

Method linearity was validated over the range 0.01 to 1.0 ng/mL for whole fruit and pulp and 0.01 to 2.0 ng/mL for peel (matrix-matched calibration solutions). Correlation coefficients (r) were > 0.99 for both MS/MS transitions.

The accuracy of the method was assessed on the basis of the determined recovery rates. Samples were fortified with isoprothiolane at concentrations of 0.01 and 1.0 mg/kg for whole fruit and pulp and 0.01 and 2.0 mg/kg for peel. Mean recoveries per concentration level were in the range of 99–105%, with acceptable RSD values within the range of 0.9–3.5%.

The limit of quantification (LOQ) for isoprothiolane, defined as the lowest fortification level where acceptable precision and accuracy data were obtained, was demonstrated to be 0.01 mg/kg in banana (whole fruit, pulp and peel).

Table 9 Recoveries for method 86846: isoprothiolane in bananas

Matrix	Analyte	No. of tests	Spiking level [mg/kg]	LC-MS/MS					
				Quantification			Confirmatory		
				Range [%]	Mean	RSD [%]	Range [%]	Mean	RSD [%]
Banana (whole fruit)	Isoprothiolane	-	-	Transition ***m/z*** 291.0 → 231.0			Transition ***m/z*** 291.0 → 189.0		
		5	0.01	103-108	104	2.0	102-104	103	1.0
		5	1.0	98-100	99	0.9	97-101	100	2.0
Banana (pulp)		5	0.01	99-107	105	3.2	101-104	103	1.4
		5	1.0	98-101	100	1.5	98-100	99	0.8
Banana (peel)		5	0.01	95-104	100	3.5	94-100	97	2.9
		5	1.0	95-101	100	2.6	95-101	99	2.5

In the residue trials (R-2159), isoprothiolane (parent only) residues were quantified in bananas (whole fruit, pulp and peel) in accordance with the HPLC-MS/MS analytical method 82884. Concurrent recoveries of isoprothiolane were acceptable in whole fruit, pulp and peel as presented below.

Table 10 Concurrent recoveries of isoprothiolane from the banana residue study R-2159

Matrix	Analyte	No. of tests	Spiking level [mg/kg]	LC-MS/MS			Study ID
				Concurrent recoveries			
				Range [%]	Mean	RSD [%]	
Banana (whole fruit)	Isoprothiolane	-	-	Transition ***m/z*** 291.0 → 231.0			-
		10	0.01	97-113	103	5	Tuinstra and Harbin, 2016, R-2159
		10	1.0	98-111	104	4	
Banana (pulp)		2	0.01	103-104	104	-	
		2	1.0	107-109	108	-	
Banana (peel)		2	0.01	97-104	101	-	
		1	1.0	108	-	-	
		1	2.0	112	-	-	

<u>Method 88449-M (QuEChERS) (enforcement):</u> The QuEChERS method for the determination of residues of isoprothiolane in bananas by high performance liquid chromatography with tandem mass spectrometry (HPLC-MS/MS) was reported by Swaim in 2019a.

Residues of isoprothiolane were extracted from banana (whole fruit) samples using a mixture of acetonitrile: water (1:1, v/v), followed by acetonitrile. Acetonitrile was added to the combined extract, and brought to a known volume with water so that the extract is in 1:1 (v/v) acetonitrile: water. An aliquot of the sample extract was diluted with acetonitrile: water (1:1, v/v). The extracted isoprothiolane was concentrated into the acetonitrile by combining and centrifuging the diluted aliquot with a 1 g QuEChERS salt mixture (800 mg magnesium sulfate + 200 mg sodium chloride).

For analysis, a portion of the acetonitrile extract was further diluted, and the residues were quantified by high performance liquid chromatography with tandem mass spectrometry (HPLC-MS/MS). Quantitation was by external standards monitoring the ion transitions m/z 291 → 231 for quantitation and m/z 291 → 189 for confirmation.

Method linearity was validated over the range 0.0075 to 0.5000 ng/mL (matrix-matched calibration solutions). Correlation coefficients (r) were > 0.99 for both MS/MS transitions.

The accuracy of the method was assessed on the basis of the determined recovery rates. Samples were fortified with isoprothiolane at concentrations of 0.01 and 0.10 mg/kg. Mean recoveries per concentration level were in a range of 90–92%, with acceptable RSD values within the range of 1.2–3.5%.

The limit of quantification (LOQ) for isoprothiolane was demonstrated to be 0.01 mg/kg in banana (whole fruit).

Table 11 Recoveries for method 88449 (QuEChERS; HPLC-MS/MS): isoprothiolane in bananas

Matrix	Analyte	No. of tests	Spiking level [mg/kg]	Quantification			Confirmatory		
				Range [%]	Mean	RSD [%]	Range [%]	Mean	RSD [%]
Banana (whole fruit)	Isoprothiolane	-	-	Transition ***m/z*** 291 → 231			Transition ***m/z*** 291 → 189		
		5	0.01	85-93	90	3.5	87-93	91	2.7
		5	0.10	91-94	92	1.2	91-94	92	1.2

An independent laboratory validation (ILV) was conducted on banana (Keenan 2019, A-2034) for the QuEChERS method 88449. Samples were fortified with isoprothiolane at the nominal concentration levels of 0.01, 0.10 and 1.0 mg/kg and analyzed according to the QuEChERS method 88449-M, as described previously. Mean recoveries per concentration level were in the range of 92–98%, with acceptable RSD values within the range of 1–5%.

Method linearity was validated over the range 0.0075 to 10 ng/mL (matrix-matched calibration solutions). Correlation coefficients (r) were ≥ 0.99 for both MS/MS transitions. The LOQ for isoprothiolane, was confirmed to be 0.01 mg/kg in banana (whole fruit).

Table 12 Recoveries for method 88449 (QuEChERS; HPLC-MS/MS) from ILV study 3208W: isoprothiolane in bananas

Matrix	Analyte	No. of tests	Spiking level [mg/kg]	Quantification			Confirmatory		
				Range [%]	Mean	RSD [%]	Range [%]	Mean	RSD [%]
Banana (whole fruit)	Isoprothiolane	-	-	Transition ***m/z*** 291 → 231			Transition ***m/z*** 291 → 189		
		5	0.01	93-105	98	5	87-100	96	5
		5	0.10	96-101	98	2	97-101	98	2
		5	1.0	91-94	92	1	90-95	92	2

<u>Method 86846-M (data collection):</u> The method for the determination of residues of isoprothiolane and its metabolites (isoprothiolane monosulfoxide and 4-hydroxy isoprothiolane) in banana matrices using liquid chromatography with tandem mass spectrometry (LC-MS/MS) was reported by Swaim in 2018, A-2033.

Residues of isoprothiolane, isoprothiolane monosulfoxide and 4-hydroxy isoprothiolane were extracted from the sample material using a mixture of acetonitrile: water (1:1, v:v), followed by acetonitrile, followed by a mixture of acetonitrile: water (4:1, v:v). The combined extract was brought to a known volume with acetonitrile.

For analysis of isoprothiolane and isoprothiolane monosulfoxide, an aliquot of the sample extract was further diluted with acetonitrile: water (1:1, v:v). The residues were quantified by LC-MS/MS.

For analysis of 4-hydroxy isoprothiolane (free and conjugated), an aliquot of the sample extract was diluted with 0.01% formic acid (aq) and passed through a C-18 solid phase extraction (SPE) cartridge. The residue was eluted from the SPE cartridge with methanol, and an aliquot was concentrated, then reacted with β-glucosidase enzyme and 0.1M sodium acetate buffer solution (pH 5) with a 24-hour incubation period. Post-incubation, sodium chloride was added to the sample, the residue was extracted from the enzymatic solution by partitioning with acetonitrile, and the sample was brought to final volume with water. The residues were quantified by LC-MS/MS. Quantitation was by external standards using the ion transitions presented below.

Table 13 Ion transitions for isoprothiolane and its metabolites for method 86846-M.

Analyte	Ion Transition (*m/z*)	
	Quantification	Confirmation
Isoprothiolane	291 → 231	291 → 189
Monosulfoxide isoprothiolane	307 → 265	307 → 223 307 → 247
4-Hydroxy isoprothiolane (free and conjugated)	307 → 247	307 → 205 307 → 187 307 → 159

Method linearity was validated over the range 0.0075 to 0.50 ng/mL (matrix-matched calibration solutions). Correlation coefficients (r) ≥ 0.99 for all MS/MS transitions.

The accuracy of the method was assessed on the basis of the determined recovery rates. Samples were fortified with isoprothiolane at concentrations of 0.01 and 2.0 mg/kg. Mean recoveries for all analytes, matrices and concentration levels were in the range of 92–109%, with acceptable RSD values within the range of 1.5–11%.

The limit of quantification (LOQ) was demonstrated to be 0.01 mg/kg for isoprothiolane, isoprothiolane monosulfoxide and 4-hydroxy isoprothiolane in banana (whole fruit), banana (pulp) and banana (peel).

Table 14 Recoveries for method 86846-M: isoprothiolane, isoprothiolane monosulfoxide and 4-hydroxy isoprothiolane (free and conjugated) in bananas.

Matrix	Analyte	No. of tests	Spiking level [mg/kg]	LC-MS/MS					
				Quantification			Confirmatory		
				Range [%]	Mean	RSD [%]	Range [%]	Mean	RSD [%]
Banana (whole fruit)	Isoprothiolane	-	-	Transition *m/z* 291.0 → 231.0			Transition *m/z* 291.0 → 189.0		
		5	0.01	96-100	98	1.6	97-104	99	2.8
		5	2.0	92-99	96	3.0	93-99	95	3.2
Banana (pulp)		5	0.01	97-106	101	3.9	96-107	100	4.6
		5	2.0	99-108	104	3.4	101-106	103	2.1
Banana (peel)		5	0.01	98-104	101	2.1	98-103	100	2.3
		5	2.0	96-102	99	2.2	99-104	101	2.1
Banana (whole fruit)	Isoprothiolane-Monosulfoxide	-	-	Transition *m/z* 307.2 → 265.0			Transition *m/z* 307.2 → 223.0		
		5	0.01	93-113	101	8.1	88-98	94	4.4
		5	2.0	94-111	102	6.7	97-101	99	1.5
Banana (pulp)		5	0.01	102-110	106	2.7	99-118	105	7.1
		5	2.0	96-117	104	7.5	101-117	107	6.2
		5	0.01	96-101	98	2.1	99-118	106	6.8

Matrix	Analyte	No. of tests	Spiking level [mg/kg]	LC-MS/MS					
				Quantification			Confirmatory		
				Range [%]	Mean	RSD [%]	Range [%]	Mean	RSD [%]
Banana (peel)		5	2.0	94-105	99	4.0	97-104	100	2.8
Banana (whole fruit)	4-Hydroxy-Isoprothiolane (free and conjugated)	-	-	Transition ***m/z*** 307.1 → 247.2			Transition ***m/z*** 307.1 → 205.1		
		5	0.01	81-112	99	12	99-120	109	8.4
		5	2.0	104-113	110	3.2	95-120	109	8.2
Banana (pulp)		5	0.01	97-113	106	6.7	88-116	100	10
		5	2.0	93-106	98	4.9	92-99	96	3.1
Banana (peel)		5	0.01	81-98	92	7.6	86-117	100	11
		5	2.0	89-97	94	3.3	92-99	96	2.7

Method validation data for 86846-M was reported in Swaim 2019, R-2163

In the residue trials (R-2163), isoprothiolane, isoprothiolane monosulfoxide and 4-hydroxy-isoprothiolane (free and conjugated) residues were quantified in bananas (whole fruit, pulp and peel) in accordance with the HPLC-MS/MS analytical method 82884-M. Concurrent recoveries of isoprothiolane, isoprothiolane monosulfoxide and 4-hydroxy isoprothiolane were acceptable in whole fruit, pulp and peel as presented below.

Table 15 Concurrent recoveries of isoprothiolane, isoprothiolane monosulfoxide and 4-hydroxy isoprothiolane (free and conjugated) from the banana residue study R-2163

Matrix	Analyte	No. of tests	Spiking level [mg/kg]	LC-MS/MS			Study ID
				Concurrent recoveries			
				Range [%]	Mean	RSD [%]	
Banana (whole fruit)	Isoprothiolane	-	-	Transition ***m/z*** 291.0 → 231.0			-
		2	0.01	91-94	93	-	Tuinstra and Harbin, 2019, R-2163
		2	2.0	93-97	95	-	
Banana (pulp)		2	0.01	96-101	99	-	
		2	2.0	98-102	100	-	
Banana (peel)		2	0.01	96-98	97	-	
		2	2.0	100-101	101	-	
Banana (whole fruit)	Isoprothiolane-Monosulfoxide	-	-	Transition ***m/z*** 307.2 → 265.0			
		7	0.01	96-120	106	9	
		7	2.0	91-106	101	5	
Banana (pulp)		1	0.01	110	-	-	
		1	2.0	113	-	-	
Banana (peel)		1	0.01	110	-	-	
		1	2.0	107	-	-	
Banana (whole fruit)	4-Hydroxy-Isoprothiolane (free and conjugated)	-	-	Transition ***m/z*** 307.1 → 247.2			
		7	0.01	82-119	98	12	
		7	2.0	88-104	97	6	
Banana (pulp)		1	0.01	102	-	-	
		1	2.0	100	-	-	
Banana (peel)		1	0.01	92	-	-	
		1	2.0	81	-	-	

In addition, a method comparison (bridging) for concurrent recoveries of isoprothiolane was conducted to verify method performance for the method from original isoprothiolane analysis (Method 82884-M) and the updated method (Method 86846-M). Eight selected representative banana samples (4×

whole fruit, 2× pulp and 2× peel) were also re-analyzed for residues of isoprothiolane using the combined total isoprothiolane method in order to prove equivalency of the extraction scheme. The % difference in isoprothiolane in treated samples via the new and old methods ranged between 4–15% (n = 4) in whole fruit, 14–18% (n = 2) in pulp and 0–7% (n = 2) in peel demonstrating method equivalence.

Stability of pesticide residues in stored analytical samples

Bananas

A study was conducted to determine the freezer storage stability of the residues of isoprothiolane in frozen banana whole fruit, pulp and peel (Swaim 2018, R-2162). Individual control samples of homogenised banana (whole fruit, pulp and peel) were spiked with isoprothiolane at a level of 0.2 mg/kg and stored at -20 °C until analysis.

Fortified control samples were taken out of storage for analysis after about 0, 1, 3 and 6 months of storage to determine isoprothiolane remaining in fortified samples of whole fruit, pulp and peel. An extra fortified control sampling of whole fruit was also analyzed after 14 months of frozen storage. All samples were analyzed according to EAG Laboratories analytical method 82884-M. Residues of isoprothiolane were observed to be stable for at least 14 months in banana whole fruit and 6 months in banana peel and pulp under freezer storage conditions.

Table 16 Stability of isoprothiolane residues in banana whole fruit, pulp and peel following frozen storage at < -20 °C

Matrix	Days of storage	Fortification level (mg/kg)	% remaining (mean)	% concurrent recovery (mean)
Banana whole fruit	0	0.20	102, 103, 103, 105 (103)	-
	30	0.20	95, 96 (96)	98, 96 (97)
	90	0.20	102, 99 (101)	104, 107 (106)
	181	0.20	106, 95 (101)	107, 106 (107)
	419	0.20	99, 97 (98)	100, 100 (100)
Banana pulp	0	0.20	108, 100, 105, 104 (104)	-
	30	0.20	101, 100 (101)	101, 102 (102)
	89	0.20	96, 98 (97)	102, 100 (101)
	180	0.20	105, 98 (102)	102, 103 (103)
Banana peel	0	0.20	103, 97, 98, 101 (100)	-
	29	0.20	94, 92 (93)	92, 96 (94)
	88	0.20	96, 96 (96)	96, 101 (99)
	179	0.20	100, 99 (100)	103, 106 (105)

The frozen storage stability study for isoprothiolane was conducted concurrently with the residue trial study (Tuinstra and Harbin 2018, R-2159).

Another study was conducted to determine the freezer storage stability of residues of 4-hydroxy-isoprothiolane (free and conjugated) and isoprothiolane monosulfoxide in frozen banana whole fruit, pulp and peel (Swaim 2019, R-2164). Individual control samples of homogenised banana (whole fruit, pulp and peel) were spiked with 4-hydroxy-isoprothiolane and isoprothiolane monosulfoxide at a level of 0.1 mg eq/kg and stored at -20 °C until analysis.

Fortified control samples were taken out of storage for analysis after about 1, 3, 6 and 17 months of storage to determine analytes remaining in fortified samples of whole fruit, pulp and peel. All samples were analyzed according to EAG Laboratories analytical method 86846-M. Residues of 4-hydroxy-isoprothiolane (free and conjugated) and isoprothiolane monosulfoxide were stable for at least 17 months

under freezer storage conditions (502 days for whole banana and 511 days for peel and pulp), which covered the longest period of storage for all samples obtained from supervised residue trials.

Table 17 Stability of 4-hydroxy-isoprothiolane residues in banana whole fruit, pulp and peel following storage at < -20 °C

Matrix	Days of storage	Fortification level (mg/kg)	% remaining (mean)	% concurrent recovery (mean)
Banana whole fruit	0	0.10	100, 100, 95, 101 (99)	-
	32	0.10	92, 99 (96)	95, 96 (96)
	91	0.10	98, 104 (101)	95, 98 (97)
	181	0.10	91, 92 (92)	101, 102 (102)
	502	0.10	101, 100 (101)	103, 101 (102)
Banana pulp	0	0.10	111, 111, 101, 110 (108)	-
	31	0.10	96, 96 (96)	99, 102 (101)
	91	0.10	97, 93 (95)	103, 96 (100)
	182	0.10	99, 97 (98)	120, 109 (115)
	511	0.01	104, 99 (102)	104, 104 (104
Banana peel	0	0.10	101, 102, 104, 106 (103)	-
	31	0.10	100, 102 (101)	96, 91 (94)
	91	0.10	97, 89 (93)	97, 102 (100)
	182	0.10	99, 98 (99)	106, 103 (105)
	511	0.10	85, 97 (91)	99, 98 (99)

Table 18 Stability of isoprothiolane-monosulfoxide residues in banana whole fruit, pulp and peel following storage at < -20 °C

Matrix	Days of storage	Fortification level (mg/kg)	% remaining (mean)	% concurrent recovery (mean)
Banana whole fruit	0	0.10	98, 99, 100, 100 (99)	-
	32	0.10	97, 99 (98)	106, 99 (103)
	91	0.10	86, 92 (89)	102, 97 (100)
	181	0.10	89, 88 (89)	111, 107 (109)
	502	0.10	88, 87 (88)	111, 107 (109)
Banana pulp	0	0.10	101, 102, 97, 100 (100)	-
	31	0.10	99, 97 (98)	105, 105 (105)
	91	0.10	101, 101 (101)	105, 110 (108)
	182	0.10	95, 95 (95)	126, 119 (123)
	511	0.10	96, 90 (93)	111, 111 (111)
Banana peel	0	0.10	99, 100, 104, 106 (102)	-
	31	0.10	92, 91 (92)	99, 98 (99)
	91	0.10	97, 95 (96)	106, 113 (110)
	182	0.10	93, 96 (95)	114, 117 (116)
	511	0.10	83, 97 (90)	106, 108 (107)

The frozen storage stability study for 4-hydroxy-isoprothiolane and isoprothiolane monosulfoxide was conducted concurrently with the residue trial study (Tuinstra and Harbin 2019, R-2163).

USE PATTERN

Information on registered uses made available to this Meeting are presented below.

Isoprothiolane is registered for use on bananas in Guatemala. The critical GAP involves up to 12 foliar applications at 400 g ai/ha with a retreatment interval of 21 days in a rotation cycle with other products and a PHI of 0 days.

Table 19 Registered use of isoprothiolane on bananas

Country	Formulation		Application					PHI (days)
	g ai/L	Type	Method	No./year	Rate (kg ai/ ha)	Spray volume (L/ha)	Minimum retreatment interval (RTI, days)	
Guatemala	400	EC	Foliar	12	0.3-0.4	18-28	21	0

RESIDUES RESULTING FROM SUPERVISED TRIALS

The Meeting received information on supervised trials for the use of isoprothiolane on bananas.

All trials were well documented with laboratory and field reports. The studies included method validation including recoveries with spiking at residue levels similar to those occurring in samples from the supervised trials. Dates of analyzes or duration of sample storage were also provided. Samples were collected and stored frozen immediately or soon after sampling. Although trials included control plots, no control data are recorded in the Tables because, unless noted, residues in control samples did not exceed the LOQ. Residues are unadjusted for recoveries.

Bananas

Twelve supervised field trials (5 decline trials and 7 harvest trials) were conducted in South America (Colombia, Costa Rica, Ecuador, Guatemala and Honduras) for both bagged and unbagged Gran Enamo (2), Cavendish (2), William (2), Valery (1), Grand Naine (4), Clon Frances (1) banana varieties during the 2017 and 2018 growing seasons. All trials received 12 applications of an isoprothiolane 40 EC product at a nominal application rate of 400 g ai/ha by ground-based broadcast application at a targeted retreatment interval of 19–21 days. The first application was targeted at 231 weeks before harvest. Individual application rates ranged from 341 to 424 g ai/ha per application. To ensure fruit maturity at sampling, Trials 101-01-15DA and 101-02-15HA received up to three additional applications. Total seasonal rates ranged from 4.8 to 6.1 kg ai/ha. The first applications were made 293 to 209 days prior to harvest. Agricultural spray oil was used at a rate of 5 to 8 L oil/ha in all trials except 101-10-15DA, 101-11-15DA, and 101-14-15DA. For these trials, spray oil was used at 1 L oil/ha. For all trials, a non-ionic surfactant (NIS) was used at label rates (1% of oil) in all tank mixes. The application intervals were 19–21-days, and application spray volumes ranged from 19 to 32 L/ha.

In all trials, control (UTC) and treated (TRT) samples were collected at 0 DALA. In the five decline trials, additional TRT samples were collected at 2–14 DALA. Single UTC samples and duplicate TRT samples were collected, each sample a composite of 24 whole fruits from four hands of at least six plants. Sampling took place between growth stage BBCH 76 (approximately 60% of whole fruit have reached final size and are of commercial size, quality and maturity for export) and BBCH 81 (beginning of ripening, fruit are developing ripe color). In three trials (101-01-15DA, 101-04-15DA and 101-13/15DA), whole fruit samples were collected from unbagged TRT plants, separated into peel and pulp, and evaluated for possible concentration or reduction of isoprothiolane residues.

The maximum duration between sampling and analysis was approximately 14 months for isoprothiolane samples and approximately 17 months for 4-hydroxy-isoprothiolane and isoprothiolane monosulfoxide samples.

Table 20 Summary of residue trials (isoprothiolane, 4-hydroxy-isoprothiolane (free and conjugated) and isoprothiolane-monosulfoxide in bananas whole fruit, peel and pulp)

Trial No., Location, Year (Variety)	Application			Sample	DALA (days)	Residues (mg eq./kg)		
	No. (RTI, days)	Rate (g ai/ha)	Volume (L/ha)			Isoprothiolane (Parent)	4-hydroxy isoprothiolane	Isoprothiolane monosulfoxide
GAP, Guatemala, Banana, Foliar	12 (21)	400	18-28	-	0	-	-	-
101-01-15DA, Sarapiquí, Heredia, Costa Rica, 2016/17, (Gran Enano) (a)	14 (19-21)	341-405	20.5-28.3	Fruit (bagged)	0	<0.01, <0.01	<0.01, <0.01	<0.01, <0.01
					Mean	<0.01	<0.01	<0.01
				Fruit (unbagged)	0	0.08, 0.05	<0.01, <0.01	<0.01, <0.01
					Mean	0.06	<0.01	<0.01
					2	0.05, 0.02	<0.01, <0.01	<0.01, <0.01
					Mean	0.04	<0.01	<0.01
					7	0.02, 0.01	<0.01, <0.01	<0.01, <0.01
					Mean	0.02	<0.01	<0.01
					14	<0.01, 0.01	<0.01, <0.01	<0.01, <0.01
					Mean	0.01	<0.01	<0.01
				Pulp (unbagged)	0	<0.01, <0.01	<0.01, <0.01	<0.01, <0.01
					Mean	<0.01	<0.01	<0.01
				Peel (unbagged)	0	0.09, 0.16	<0.01, <0.01	<0.01, <0.01
					Mean	0.12	<0.01	<0.01
101-02-15HA, Sarapiquí, Heredia, Costa Rica, 2016/17, (Gran Enano) (a)	15 (20-21)	397-418	23.9-31.8	Fruit (bagged)	0	<0.010, <0.010	<0.01, <0.01	<0.01, <0.01
					Mean	<0.010	<0.01	<0.01
				Fruit (unbagged)	0	0.06, 0.05	<0.01, <0.01	<0.01, <0.01
					Mean	0.05	<0.01	<0.01
101-04-15DA, Parroquia, Lorenzo de Garaicoa, Ecuador, 2016/17, (Cavendish)	12 (21)	398-402	26.9-27.1	Fruit (bagged)	0	<0.01, <0.01	<0.01, <0.01	<0.01, <0.01
					Mean	<0.01	<0.01	<0.01
				Fruit (unbagged)	0	0.55, 0.38	0.01, 0.01	<0.01, <0.01
					Mean	0.47	0.01	<0.01
					3	0.42, 0.52	0.01, 0.01	<0.01, <0.01
					Mean	0.47	0.01	<0.01
					7	0.15, 0.11	<0.01, <0.01	<0.01, <0.01
					Mean	0.13	<0.01	<0.01
					14	0.07, 0.07	<0.01, <0.01	<0.01, <0.01
					Mean	0.07	<0.01	<0.01
				Pulp (unbagged)	0	0.02, <0.01	<0.01, <0.01	<0.01, <0.01
					Mean	0.01	<0.01	<0.01
				Peel (unbagged)	0	1.66, 0.69	0.03, 0.02	0.02, <0.01
					Mean	1.17	0.03	0.01
101-05-15HA, Cantón El Triunfo, Ecuador, 2016/17 (William)	12 (21)	399-402	27.9-28.1	Fruit (bagged)	0	<0.01, <0.01	<0.01, <0.01	<0.01, <0.01
					Mean	<0.01	<0.01	<0.01
				Fruit (unbagged)	0	0.20, 0.13	<0.01, <0.01	<0.01, <0.01
					Mean	0.17	<0.01	<0.01
101-06-15HA, Parroquia, Pancho Negro, Ecuador, 2016/17, (Cavendish)	12 (21)	398-401	26.8-27.1	Fruit (bagged)	0	<0.01, <0.01	<0.01, <0.01	<0.01, <0.01
					Mean	<0.01	<0.01	<0.01
				Fruit (unbagged)	0	0.29, 0.26	0.01, <0.01	<0.01, <0.01
					Mean	0.28	0.01	<0.01
101-07-15DA, Apartado Nueva Colonia, Colombia, 2016/17, (Valery)	12 (19)	393-407	26.4-27.3	Fruit (bagged)	0	<0.01, 0.01	<0.01, <0.01	<0.01, <0.01
					Mean	0.01	<0.01	<0.01
				Fruit (unbagged)	0	0.16, 0.10	0.01, <0.01	<0.01, <0.01
					Mean	0.13	0.01	<0.01
					2	0.13, 0.08	0.02, <0.01	<0.01, <0.01

Trial No., Location, Year (Variety)	Application No. (RTI, days)	Rate (g ai/ha)	Volume (L/ha)	Sample	DALA (days)	Residues (mg eq./kg) Isoprothiolane (Parent)	4-hydroxy isoprothiolane	Isoprothiolane monosulfoxide
					Mean	0.11	0.01	<0.01
					7	0.08, 0.05	0.01, 0.01	<0.01, <0.01
					Mean	0.06	0.01	<0.01
					14	0.01, 0.01	<0.01, <0.01	<0.01, <0.01
					Mean	0.01	<0.01	<0.01
101-08-15HA, Chigorodó, Colombia, 2016/17, (William)	12 (19)	374-416	21.8-28.0	Fruit (bagged)	0	0.01, <0.01	<0.01, <0.01	<0.01, <0.01
					Mean	0.01	<0.01	<0.01
				Fruit (unbagged)	0	0.22, 0.09	0.02, 0.01	<0.01, <0.01
					Mean	0.16	0.01	<0.01
101-11-15DA, Bolivia, Santo Domingo Suchitepequez Guatemala, 2016/17, (Clon Frances)	12 (21)	395-404	27.6-28.2	Fruit (bagged)	0	0.02, <0.01	<0.01, <0.01	<0.01, <0.01
					Mean	0.01	<0.01	<0.01
				Fruit (unbagged)	0	0.30, 0.31	0.01, 0.01	<0.01, <0.01
					Mean	0.31	0.01	<0.01
					3	0.15, 0.11	0.01, 0.01	<0.01, <0.01
					Mean	0.13	0.01	<0.01
					5	0.14, 0.13	0.01, 0.01	<0.01, <0.01
					Mean	0.13	0.01	<0.01
					12	0.04, 0.06	0.01, <0.01	<0.01, <0.01
					Mean	0.05	0.01	<0.01
101-10-15HA, La Gomera, Escuintla, Guatemala, 2016/17, (Grand Naine) (b)	12 (20-21)	395-409	18.8-19.4	Fruit (bagged)	0	<0.01, <0.01	<0.01, <0.01	<0.01, <0.01
					Mean	<0.01	<0.01	<0.01
				Fruit (unbagged)	0	0.11, 0.14	<0.01, <0.01	<0.01, <0.01
					Mean	0.13	<0.01	<0.01
101-12-15HA, La Gomera, Escuintla, Guatemala, 2016/17, (Grand Naine) (b)	12 (20-21)	398-406	25.9-26.4	Fruit (bagged)	0	<0.01, 0.01	<0.01, <0.01	<0.01, <0.01
					Mean	0.01	<0.01	<0.01
				Fruit (unbagged)	0	0.52, 0.59	0.01, 0.02	<0.01, <0.01
					Mean	0.55	0.01	<0.01
101-13-15DA, Santiago, Pimienta, Cortes, Honduras, 2016/17, (Grande Naine) (c)	12 (19-21)	394-404	27.6-28.4	Fruit (bagged)	0	<0.01, 0.01	<0.01, <0.01	<0.01, <0.01
					Mean	0.01	<0.01	<0.01
				Fruit (unbagged)	0	0.07, 0.08	<0.01, <0.01	<0.01, <0.01
					Mean	0.07	<0.01	<0.01
					2	0.05, 0.06	<0.01, <0.01	<0.01, <0.01
					Mean	0.05	<0.01	<0.01
					6	0.07, 0.11	0.01, 0.01	<0.01, <0.01
					Mean	0.09	0.01	<0.01
					13	0.07, 0.03	0.01, <0.01	<0.01, <0.01
					Mean	0.05	0.01	<0.01
				Pulp (unbagged)	0	<0.01, <0.01	<0.01, <0.01	<0.010, <0.010
					Mean	<0.01	<0.01	<0.010
				Peel (unbagged)	0	0.14, 0.12	0.02, 0.02	<0.01, <0.01
					Mean	0.13	0.02	<0.01
101-14-15HA, Santiago, Pimienta, Cortes, Honduras, 2016/17, (Grande Naine) (c)	12 (20-22)	395-406	18.8-19.3	Fruit (bagged)	0	0.06*, 0.02*	<0.01, <0.01	<0.01, <0.01
					Mean	0.04	<0.01	<0.01
				Fruit (unbagged)	0	0.02, 0.02	<0.01, <0.01	<0.01, <0.01
					Mean	0.02	<0.01	<0.01

DBH = Days before harvest

LOQ = 0.01 mg/kg for each analyte

* Average of multiple analyses

(a), (b), (c) = non-independent trials sites; therefore, critical (highest) residue endpoints selected from each trial

APPRAISAL

Isoprothiolane is a fungicide belonging to the family of dicarboxylic acids, which act by inhibition of phospholipid biosynthesis. It was considered for the first time for toxicology and residues by the 2017 JMPR. An ADI of 0–0.1 mg/kg bw was established and an ARfD was considered to be unnecessary. The 2017 JMPR decided on the following residue definitions based on a metabolism study on paddy rice and confined rotational crops, and recommended maximum residue levels for husked rice, rice straw and for animal commodities.

The residue definition for compliance with the MRL for plant commodities is *isoprothiolane*.

The residue definition for dietary risk assessment for rice is *isoprothiolane*.

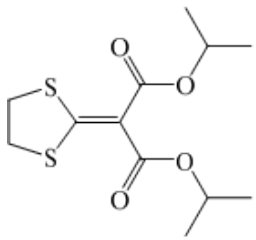
The residue definition for dietary risk assessment for plant commodities other than rice is the sum of *isoprothiolane, diisopropyl-4-hydroxy-1,3-dithiolan-2-ylidenemalonate (M-3); free and conjugated, and 1-hydroxypropan-2-yl propan-2-yl 1,3-dithiolan-2-ylidenemalonate (M-5); free and conjugated, expressed as isoprothiolane*.

The residue definition for compliance with the MRL and dietary risk assessment for animal commodities is the sum of *isoprothiolane and 2-(1,3-dithiolan-2-ylidene)-3-oxo-3-(propan-2- yloxy)propanoic acid (M-2), expressed as isoprothiolane*.

The residue is not fat-soluble.

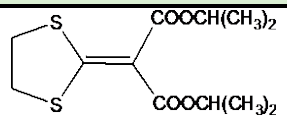
Isoprothiolane was scheduled at the Fifty-first Session of the CCPR for the consideration by the 2020 Meeting of additional MRLs, which was postponed to the 2021 Extra JMPR.

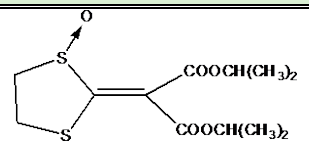
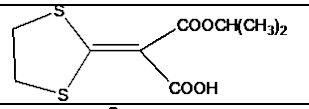
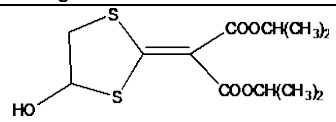
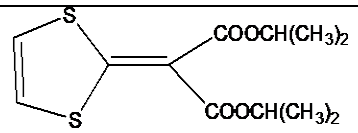
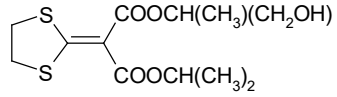
The Meeting received information on metabolism in apples and grapes, physical and chemical properties, analytical methodology, storage stability, a registered use pattern for bananas and supervised residue trials on bananas.



Isoprothiolane (MW 290.4 g/mol)

Table 1 The following abbreviations are used for Isoprothiolane and metabolites/degradates discussed

Name	Chemical name	Structure
Isoprothiolane (parent)	Diisopropyl 1,3-dithiolan-2-ylidenemalonate	

Name	Chemical name	Structure
Isoprothiolane monosulfoxide (M-1)	Diisopropyl 1- oxo-1,3- dithiolan-2-ylidenemalonate	
Isoprothiolane monoester (M-2)	Monoisopropyl 1,3-dithiolan-2- ylidenemalonate	
4-hydroxy isoprothiolane (M-3)	Diisopropyl 4- hydroxy-1,3- dithiolan-2-ylidenemalonate	
Didehydro isoprothiolane (M-4)	Diisopropyl 1,3- dithiol-2-ylidenemalonate	
Hydroxyl-Isopropyl (M-5)	1-Hydroxypropan-2-yl isopropyl 1,3-dithiolan-2-ylidenmalonate	

Plant metabolism

The Meeting received plant metabolism studies on apples and grapes following applications of [^{14}C]-isoprothiolane.

Apples - Direct application to fruit and leaves

In a metabolism study conducted on apples, ^{14}C-isoprothiolane (radiolabelled in dithiolane ring) was applied as a solution in methanol directly to apple fruit and leaves at 3.14 µg ai/fruit or 1.57 µg ai/leaf. The application rate was equivalent to ca. 0.6 kg ai/ha. The fruits and leaves were collected 7 and 14 days after treatment (DAT).

Samples were surface washed with methanol (MeOH), then sequentially extracted with MeOH followed by MeOH/water (4:1 v/v). Extraction efficiencies were 92.1–98.8%.

In fruit, isoprothiolane was the predominant residue, making up 49.3% TRR in the 7DAT samples and 26.6% TRR in the 14DAT samples. The 4-hydroxy-isoprothiolane (M-3) metabolite (up to 2.1% TRR), together with its glucose conjugates, totalled 9% TRR (7DAT) and 14.1% TRR (14DAT). Other characterised metabolites observed in fruit were isoprothiolane-monosulfoxide (M-1) and didehydro-isoprothiolane (M-4), present at 4.1–4.2% TRR and 4.9–6.6% TRR respectively.

In leaves, isoprothiolane was also the predominant residue, making up 53.9% TRR in the 7DAT samples and 40.3% TRR in the 14DAT samples. The 4-hydroxy-isoprothiolane (M-3) metabolite, together with its glucose conjugates, totalled 13.1% TRR (7DAT) and 17.3% TRR (14DAT). Other characterised metabolites observed in leaves were isoprothiolane-monosulfoxide (M-1) and didehydro-isoprothiolane (M-4), present at up to 9% TRR and 3.8% TRR, respectively. The isoprothiolane monoester (M-2) was only detected (0.1% TRR) in the 14DAT leaves.

Apples - Soil application

In a metabolism study conducted on apples, the soil around pot-grown dwarf apple trees was treated once with ^{14}C-isoprothiolane (radiolabelled in dithiolane ring) using a methanol solution of isoprothiolane of 2.27 mg/mL (equivalent to 360 g ai/tree). Samples of apple fruits and leaves were collected at 7, 28 and 61 days

after treatment (DAT). Samples were sequentially extracted with MeOH and MeOH/water (4:1 v/v) with extraction efficiencies of 95% (fruit) and 65–72% (leaves).

In fruit, residues were detected only in the 61 DAT samples and since the TRR was < 0.01 mg eq/kg, no metabolite identification was performed.

In leaves at 61 DAT, isoprothiolane (parent) was detected only at low levels (0.7% TRR), along with the isoprothiolane-monoester (M-2) and isoprothiolane-monosulfoxide (M-1) metabolites, present at 1.2% TRR and 3% TRR, respectively. The major metabolite fraction at 65.5% TRR was at the origin on the thin layer chromatograms (TLC), part of which was characterised by ß-glucosidase treatment as the glucoside of isoprothiolane-monoester (M-2) at 13.8% TRR. The remainder of the TLC origin material comprised multiple unidentified components (each <10% TRR).

Grapes - Foliar application

In the metabolism study conducted on grapes, three foliar applications of [^{14}C]- isoprothiolane were applied to grapevines at 1.46 kg ai/ha followed by a fourth application at 0.4 kg ai/ha. The applications were made at pre harvest intervals (PHIs) of 70, 42, 21 and 0 days. Samples of grapes were collected 20 days after the 3rd application (20DAA3) and 0 days after the 4th application (0 DAA4), at a crop growth stage of BBCH 89. Samples were rinsed twice with acetonitrile and stored in a freezer until processing. The acetonitrile rinses were filtered prior to analysis. The total ^{14}C content of the acetonitrile rinses was measured by Liquid Scintillation Counting (LSC). Sequential extraction with acetonitrile; acetonitrile:water (1:1 v/v); acetonitrile:0.2N HCl (1:1 v/v) and acetonitrile:0.2N NH_4OH (1:1 v/v) was able to extract 89.2–90.6% TRR (2.73–3.43 mg eq/kg).

Isoprothiolane was the major radioactive component identified, representing 47–50.7% TRR in grapes. Isoprothiolane-monosulfoxide (M-1) was also an abundant component representing between 11.4–19.5% TRR. Three different glycoside conjugates of 4-hydroxy-isoprothiolane (M-3) were present, ranging individually from 2.3–7.6% TRR. The total free and conjugated 4-hydroxy-isoprothiolane represented 13.2–14% TRR.

Methods of analysis

Analytical methods have been reported and validated for the analysis of isoprothiolane in bananas.

The data generation method involved a number of extractions with acetonitrile: water (1:1, v/v) and acetonitrile. Conjugates were cleaved with β-glucosidase enzyme and 0.1M sodium acetate buffer solution (pH 5) with a 24-hour incubation period. The residues were quantified by LC-MS/MS. The LOQs for isoprothiolane, isoprothiolane monosulfoxide (M-1) and 4-hydroxy isoprothiolane (M-3) were demonstrated to be 0.01 mg/kg for each analyte in banana (whole fruit), banana (flesh) and banana (peel).

For MRL compliance, a multi-residue method is available for analysis of isoprothiolane on bananas based on QuEChERS extraction/clean-up and HPLC-MS/MS detections. The LOQ for isoprothiolane was determined to be 0.01 mg/kg in banana (whole fruit).

The Meeting concluded that the above methods were sufficiently validated and are suitable for measuring residues of isoprothiolane, and/or its -monosulfoxide and its free and conjugated -4-hydroxy metabolites in bananas.

Stability of residues in stored analytical samples

Residues of isoprothiolane were observed to be stable for at least 14 months in banana whole fruit and for 6 months in banana peel and flesh under freezer storage conditions. Residues of 4-hydroxy-isoprothiolane and isoprothiolane monosulfoxide were observed to be stable for at least 17 months under freezer storage conditions in whole fruit, flesh and peel. These durations covered the longest period of storage for all samples obtained from supervised residue trials.

Definition of the residue

The 2017 JMPR considered a metabolism study conducted on paddy rice, in which isoprothiolane (parent) was the predominant residue, accounting for 16–76% TRR in rice grain, hulls and stems/leaves. The 2017 JMPR also considered a confined rotational study conducted on lettuce, radish and wheat. Isoprothiolane was observed in most matrices at the 30-day plant-back interval, however, the metabolites 4-hydroxy-isoprothiolane (M-3) and hydroxyl-isopropyl (M-5) generally made up the majority of the residue. Isoprothiolane was not observed in wheat grain.

Based on these studies, for plant commodities, the 2017 JMPR established a residue definition of isoprothiolane (parent only) for MRL-compliance and also for dietary risk assessment for rice. For other plant commodities, the established residue definition for dietary risk assessment is the sum of isoprothiolane and metabolites M-3 and M-5 (expressed as parent compound).

Plant commodities

In the grape metabolism study, isoprothiolane (parent) was the major radioactive component identified representing 47–50.7% TRR. Isoprothiolane monosulfoxide (M-1) was also a relatively abundant component representing between 11.4–19.5% TRR. Three different glycoside conjugates of 4-hydroxy-isoprothiolane were present, individually ranging from 2.3–7.6% TRR. The total residues of 4-hydroxy-isoprothiolane (M-3) and its conjugates represented 13.2–14% TRR. No hydroxyl-isopropyl (M-5) was detected in the study.

In the metabolism study on apples involving direct application to leaves and fruit, isoprothiolane (parent) was the most abundant component in fruit representing 26.6–49.3% TRR. In fruit, the 4-hydroxy-isoprothiolane (M-3) and its conjugates accounted for 9–14.1% TRR, with other identified metabolites being the monosulfoxide derivative (M-1), present at about 4.1–4.2% TRR and the didehydro derivative (M-4), accounting for 4.9–6.6% TRR.

In the supervised bananas field trials, isoprothiolane was detected at residue levels of < 0.01–0.59 mg/kg. Residues of 4-hydroxy isoprothiolane (M-3) were detected in whole fruit in 7 of the 9 independent trials at < 0.01–0.02 mg/kg. In the three trials where peel and flesh were analysed, no quantifiable residues of 4-hydroxy isoprothiolane were detected in the flesh. Quantifiable residues of isoprothiolane monosulfoxide (M-1) were not observed in whole fruit and residues of hydroxyl-isopropyl (M-5) were not analysed.

Based on the available metabolism studies, the Meeting noted that isoprothiolane was the predominant residue in fruit matrices. The Meeting confirmed its previous recommendation of isoprothiolane as a suitable residue definition for MRL-compliance in plant matrices.

For the purpose of dietary exposure estimation, parent isoprothiolane and 4-hydroxy isoprothiolane (free and conjugated) represent a significant part of the residue in fruit crops. The only additional major metabolite found in fruit was isoprothiolane-monosulfoxide (M-1) in grapes (up to 19.5% TRR) and in apples (up to 4.2% TRR), but it was not found in the flesh of bananas in the supervised field trials.

The Meeting agreed that the current residue definition for dietary risk assessment also applies to banana but noted that should additional uses be considered in future this residue definition will need to be revisited.

Results of supervised residue trials on crops

The Meeting received GAP information and residue trials on bananas.

Bananas

The cGAP for bananas in Guatemala, is for up to 12 foliar applications at 400 g ai/ha, with a re-treatment interval of 21 days and a PHI of 0 days. Twelve residue trials, matching cGAP, from Colombia, Costa Rica, Ecuador, Guatemala and Honduras were available to the Meeting. The Meeting noted that while all trials matched the cGAP, not all trials were independent. Residues were observed to be higher from the unbagged trials, and these results were used for maximum residue level estimation.

In nine independent trials matching the cGAP the residues in banana (whole fruit) were (n = 9): 0.065, 0.09, 0.13, 0.16, 0.17, 0.28, 0.31, 0.47 and 0.56 mg/kg.

The Meeting estimated a maximum residue level of 1 mg/kg for isoprothiolane in bananas.

Based on the information available the Meeting agreed that the -hydroxyl-isopropyl metabolite (M-5) could be excluded from the STMR calculations in banana as this metabolite has only been reported in rotational crops.

For dietary risk assessment, in the above trials, total residues (i.e., parent plus the free and conjugated M-3 metabolite) in whole fruit were (n = 9): 0.075, 0.1, 0.14, 0.17, 0.18, 0.29, 0.32, 0.48 and 0.57 mg/kg and in three of these trials, total residues in flesh were (n = 3): < 0.02, < 0.02 and 0.02 mg/kg.

In the one trial where quantifiable residues (0.02 mg/kg) were found in the flesh total residues in whole fruit were 0.465 mg/kg (a flesh:whole fruit ratio of 0.043). The Meeting agreed to apply this ratio to the median total residues in whole fruit (0.18 mg/kg) to estimate a STMR of 0.0077 mg/kg for bananas.

Residues in animal commodities

The 2017 JMPR considered residues in animal commodities and recommended maximum residue levels. As bananas are not considered to be a livestock feed item, the livestock dietary burden remains unchanged. No amendments to the previously considered STMRs and maximum residue levels for animal commodities are required.

RECOMMENDATIONS

On the basis of the data obtained from supervised trials, the Meeting concluded that the residue levels listed below in Table 2 are suitable for establishing maximum residue limits and for IEDI assessment.

The residue definition for compliance with the MRL for plant commodities is *isoprothiolane*.

The residue definition for dietary risk assessment for rice is *isoprothiolane*.

The residue definition for dietary risk assessment for plant commodities other than rice is the sum of *isoprothiolane, diisopropyl-4-hydroxy-1,3-dithiolan-2-ylidenemalonate (M-3); free and conjugated, and 1-hydroxypropan-2-yl propan-2-yl 1,3-dithiolan-2-ylidenemalonate (M-5); free and conjugated, expressed as isoprothiolane*.

The residue definition for compliance with the MRL and dietary risk assessment for animal commodities is the sum of *isoprothiolane and 2-(1,3-dithiolan-2-ylidene)-3-oxo-3-(propan-2- yloxy)propanoic acid (M-2), expressed as isoprothiolane*.

The residue is not fat-soluble.

Table 2 Recommendations for residues of isoprothiolane from the 2021 Extra JMPR

CCN	Commodity name	Proposed MRL (mg/kg)	STMR (mg/kg)	HR (mg/kg)
FI 0327	Banana	1	0.0077	

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for isoprothiolane is 0–0.1 mg/kg bw. The International Estimated Daily Intakes (IEDIs) for isoprothiolane were estimated for the 17 GEMS/Food Consumption Cluster Diets using the STMR or STMR-P values estimated by the JMPR. The results are shown in Annex 3 of the 2021 Extra JMPR Report.

The IEDIs ranged from 0–2% of the maximum ADI. The Meeting concluded that long-term dietary exposure to residues of isoprothiolane from uses considered by the JMPR is unlikely to present a public health concern.

Acute dietary exposure

The 2017 JMPR decided that an ARfD is unnecessary. The Meeting concluded that the acute dietary exposure to residues of isoprothiolane from the uses considered is unlikely to present a public health concern.

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PC-2076	Goia, T.G.	2019c	Melting point or range of Isoprothiolane, Study No. 14677.005.027.18, Date: 2019-11-28, unpublished
PC-2077	Goia, T.G.	2019d	Determination of the pH value of an aqueous solution of Isoprothiolane, Study No. 14677.009.100.18, Date: 2019-11-29, unpublished
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ISOXAFLUTOLE (268)

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

EXPLANATION

Isoxaflutole is a herbicide that was first evaluated by the 2013 JMPR, when an ADI was established as 0–0.02 mg/kg bw and an ARfD was considered unnecessary.

The residue definition for isoxaflutole for compliance with the MRL for plant and animal commodities and for dietary risk assessment for plant commodities, including tolerant soya beans, is the *sum of isoxaflutole and isoxaflutole diketonitrile, expressed as isoxaflutole.*

The residue definition for isoxaflutole for dietary risk assessment for animal commodities is the *sum of isoxaflutole, isoxaflutole diketonitrile, RPA 205834 (2-aminomethylene-l-cyclopropyl-3-(2-mesyl-4-trifluoromethylphenyl)-propane-1,3-dione) and RPA 207048 (1-cyclopropyl-2-hydroxymethylene-3- (2-mesyl-4-trifluoromethylphenyl)-propane-1,3-dione), including their conjugates, expressed as isoxaflutole.*

The residue is not fat-soluble.

Isoxaflutole was scheduled at the Fifty-first Session of the CCPR (2019) for evaluation of additional uses by the 2020 JMPR, which was postponed to the 2021 Extra JMPR.

The Meeting received supervised residue trials on soya bean that had been previously evaluated by the 2013 JMPR and new GAP information on isoxaflutole in/on glyphosate/HPPD tolerant soya bean.

USE PATTERNS

Table 1 shows the use patterns of isoxaflutole on soya bean in the USA, for weed control in glyphosate tolerant (GT27) and isoxaflutole-tolerant soya bean.

Table 1 Registered uses of isoxaflutole (SC, 40.5% formulation) for glyphosate tolerant and isoxaflutole-tolerant soya bean in the USA [a, b]

Time of application	Application rate [C] kg ai/ha	Number of application	PHI (days) at least [d]
Early pre-plant, surface applied or incorporated; 8 to 21 days prior to planting	0.045-0.09	2-1	70
Pre-plant, surface applied or incorporated; 0 to 7 days prior to planting or pre-emergence	0.045-0.09	2-1	70
Post-emergence, up to bud not including first bloom growth stage (BBCH 59)	0.045-0.09	2-1	70

[a] Not recommended for use coarse textured soils that have organic matter < 1.5% and a soil pH> 7.5.

[b] Do not graze or feed treated forage or hay from soya beans to livestock.

[c] Max of two applications/year not exceeding 0.09 kg ai/ha in one year.

[d] Do not harvest grain within 70 days of application

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

Soya bean (dry)

A total of 77 residue trials were conducted with isoxaflutole in/on glyphosate/HPPD tolerant soybean in 2009, from which five trials were conducted in Canada and 72 trials in the USA. The trials were also evaluated by the 2013 JMPR and a summary of the results is shown in Table 2.

The residues of isoxaflutole and its metabolite, isoxaflutole-diketonitrile (IFT-DKN) were determined by LC-MS/MS, with a LOQ of 0.01 mg/kg. Samples were held in frozen storage for a maximum of 6.3 months prior to analysis. Storage stability studies in dry soya beans evaluated by the 2013 JMPR showed that isoxaflutole converts to IFT-DKN after 3–6 months of storage at -10 °C, which remain stable for at least 12 months. Hence, total residues (isoxaflutole + IFT-DKN) are stable for up to 12 months.

Table 2 Results of residue trials conducted with isoxaflutole in/on soybean (glyphosate/HPPD tolerant soya bean line, FG72) in Canada and the USA in 2009 using 1 application of a SC formulation

	Application				Residues (mg/kg)			Study Trial No.
Country, location	kg ai/ha	kg ai/hL	BBCH	DALT (days)	Isoxaflutole	Isoxaflutole diketonitrile	Total residues	
Canada, Rockwood, ON Soil: L [b]	0.103	0.079	00	151	<0.01 (2)	<0.01 (2)	<0.02 (2)	RAISP006 IS008-09HA
	0.130	0.066	15	120	<0.01 (2)	0. 012, 0.010	0.022, 0.020	
	0.106	0.068	51	99	<0.01 (2)	<0.01 (2)	<0.02 (2)	
Canada, Rockwood, ON Soil: L [a] + [b]	0.106	0.079	00	90	<0.01 (2)	<0.01 (2)	<0.02 (2)	RAISP010 IS035-09HA
	0.106	0.080	51	90	<0.01 (2)	<0.01 (2)	<0.02 (2)	
USA, Shorter, AL Soil: LSa [b]	0.103	0.067	01	120	<0.01 (2)	<0.01 (2)	<0.02 (2)	RAISP006 IS003-09HA
	0.103	0.068	15	94	<0.01 (2)	<0.01 (2)	<0.02 (2)	
	0.103	0.067	62	82	<0.01 (2)	0.016 (2)	0.026 (2)	
USA, Shorter, AL Soil: SaL [a] + [b]	0.103	0.067	01	72	<0.01 (2)	<0.01 (2)	<0.02 (2)	RAISP010 IS032-09HA
	0.103	0.067	62	72	<0.01 (2)	0.023, 0.031	0.033, 0.041 (0.037)	
USA, Proctor, Arkansas Soil: CL [b]	0.104	0.072	00	111	<0.01 (2)	<0.01 (2)	<0.02 (2)	RAISP006 IS006-09HA
	0.104	0.072	15	87	<0.01 (2)	<0.01 (2)	<0.02 (2)	
	0.104	0.072	60	72	<0.01 (2)	<0.01 (2)	<0.02 (2)	
USA, Proctor, AR Soil: CL [a] + [b]	0.104	0.072	00	62	<0.01 (2)	<0.01 (2)	<0.02 (2)	RAISP010 IS033-09HA
	0.104	0.072	60	62	<0.01 (2)	<0.01 (2)	<0.02 (2)	
USA, Chula, GA Soil: Lsa [b]	0.103	0.055	00	144	<0.01 (2)	<0.01 (2)	<0.02 (2)	RAISP006 IS002-09HA
	0.105	0.046	15	120	<0.01 (2)	<0.01 (2)	<0.02 (2)	
	0.104	0.048	66	98	<0.01 (2)	0.021 (2)	0.031 (2)	
USA, Sheridan, IN Soil: SiL [b]	0.102	0.055	00	133	<0.01 (2)	<0.01 (2)	<0.02 (2)	RAISP006 IS021-09HA
	0.106	0.060	15	104	<0.01 (2)	<0.01 (2)	<0.02 (2)	
	0.103	0.060	60	94	<0.01 (2)	<0.01 (2)	<0.02 (2)	
USA, Carlyle, IL Soil: SiL [b]	0.105	0.053	00	130	<0.01 (2)	<0.01 (2)	<0.02 (2)	RAISP006 IS016-09HA
	0.104	0.043	14	104	<0.01 (2)	0.014, 0.012	0.024, 0.022	
	0.104	0.074	51	97	<0.01 (2)	0.028, 0.025	0.038, 0.035	
USA, Farlin, IA	0.106	0.13	00	77	<0.01 (2)	<0.01 (2)	<0.02 (2)	

	Application				Residues (mg/kg)			Study Trial No.
Country, location	kg ai/ha	kg ai/hL	BBCH	DALT (days)	Isoxaflutole	Isoxaflutole diketonitrile	Total residues	
Soil: CL [a] + [b]	0.104	0.13	60	77	<0.01 (2)	0.014, 0.016	0.024, 0.026 (0.025)	RAISP010 IS034-09HA
USA, Jefferson, IA Soil: SaL [b]	0.107	0.13	00	130	<0.01 (2)	<0.01 (2)	<0.02 (2)	RAISP006 IS007-09DA [c]
	0.104	0.076	13	94	<0.01 (2)	<0.01 (2)	<0.02 (2)	
	0.105	0.062	51	77 79 81 83 85	<0.01 (2) <0.01 (2) <0.01 (2) <0.01 (2) <0.01 (2)	<0.01 (2) <0.01 (2) <0.014 (2) 0.012, <0.01 <0.01, 0.013	<0.02 (2) <0.02 (2) 0.024 (2) 0.022, <0.02 <0.02, 0.023	
USA, Richland, IA Soil: SiCL [b]	0.105	0.0714	00	138	<0.01 (2)	<0.01 (2)	<0.02 (2)	RAISP006 IS015-09HA
	0.098	0.054	13	115	<0.01 (2)	<0.01 (2)	<0.02 (2)	
	0.103	0.0589	60	95	<0.01 (2)	<0.01 (2)	<0.02 (2)	
USA, Gardner, KS Soil: SiL [b]	0.104	0.0727	01	140	<0.01 (2)	<0.01 (2)	<0.02 (2)	RAISP006 IS014-09HA
	0.106	0.0746	13	114	<0.01 (2)	<0.01 (2)	<0.02 (2)	
	0.105	0.0755	60	91	<0.01 (2)	<0.01 (2)	<0.02 (2)	
USA, Gardner, KS Soil: SiL [a] + [b]	0.104	0.075	01	81	<0.01 (2)	<0.01 (2)	<0.02 (2)	RAISP010 IS041-09HA
	0.104	0.073	60	81	<0.01 (2)	<0.01 (2)	<0.02 (2)	
USA, Cheneyville, LA Soil: SiL [b]	0.107	0.079	00	128	<0.01 (2)	<0.01 (2)	<0.02 (2)	RAISP006 IS004-09DA
	0.105	0.067	13	106	<0.01 (2)	<0.01 (2)	<0.02 (2)	
	0.106	0.071	59	87 89 91 93 95	<0.01 (2) <0.01 (2) <0.01 (2) <0.01 (2) <0.01 (2)	<0.01 (2) <0.01 (2) <0.01 (2) <0.01 (2) <0.01 (2)	<0.02 (2) <0.02 (2) <0.02 (2) <0.02 (2) <0.02 (2)	
USA, Campbell, MN Soil: SaCL [b]	0.105	0.0561	00	130	<0.01 (2)	<0.01 (2)	<0.02 (2)	RAISP006 IS012-09HA
	0.104	0.056	15	95	<0.01 (2)	<0.01 (2)	<0.02 (2)	
	0.105	0.056	60	88	<0.01 (2)	<0.01 (2)	<0.02 (2)	
USA, Campbell, MN Soil: SaCL [a] + [b]	0.105	0.056	00	79	<0.01 (2)	<0.01 (2)	<0.02 (2)	RAISP010 IS039-09HA
	0.105	0.0561	60	79	<0.01 (2)	<0.01 (2)	<0.02 (2)	
USA, Geneva, MN Soil: SaCL [b]	0.106	0.069	00	151	<0.01 (2)	<0.01 (2)	<0.02 (2)	RAISP006 IS019-09HA
	0.105	0.062	15	116	<0.01 (2)	<0.01 (2)	<0.02 (2)	
	0.105	0.064	60	92	<0.01 (2)	<0.01 (2)	<0.02 (2)	
	0.105	0.056	60	79	<0.01 (2)	0.010 (2)	0.020 (2)	
USA Greenville, MS Soil: CL [b]	0.104	0.075	00	132	<0.01 (2)	<0.01 (2)	<0.02 (2)	RAISP006 IS005-09HA
	0.105	0.079	15	110	<0.01 (2)	<0.01 (2)	<0.02 (2)	
	0.105	0.082	61	126	<0.01 (2)	<0.01 (2)	<0.02 (2)	
USA, Dudley, MO Soil: SiL [b]	0.106	0.056	05	123	<0.01 (2)	<0.01 (2)	<0.02 (2)	RAISP006 IS010-09HA
	0.106	0.057	15	98	<0.01 (2)	<0.01 (2)	<0.02 (2)	
	0.105	0.056	60	88	<0.01 (2)	0.012 (2)	0.022 (2)	
USA, Dudley, MO	0.106	0.056	05	80	<0.01 (2)	<0.01 (2)	<0.02 (2)	

Country, location	Application			DALT (days)	Residues (mg/kg)			Study Trial No.
	kg ai/ha	kg ai/hL	BBCH		Isoxaflutole	Isoxaflutole diketonitrile	Total residues	
Soil: SiL [a] + [b]	0.106	0.057	60	80	<0.01 (2)	0.019, 0.020	0.029, 0.030 (0.030)	RAISP010 IS037-09HA
USA, Clarence, MO Soil: SiCL [b]	0.106	0.057	00	129	<0.01 (2)	<0.01 (2)	<0.02 (2)	RAISP006 IS011-09HA
	0.110	0.057	14	99	<0.01 (2)	<0.01 (2)	<0.02 (2)	
	0.104	0.059	60	90	<0.01 (2)	<0.01 (2)	<0.02 (2)	
USA, Clarence, MO Soil: SiCL [a] + [b]	0.105	0.057	00	80	<0.01 (2)	<0.01 (2)	<0.02 (2)	RAISP010 IS038-09HA
	0.107	0.055	60	80	<0.01 (2)	<0.01 (2)	<0.02 (2)	
USA, York, NE Soil: SiL [b]	0.107	0.057	00	139	<0.01 (2)	<0.01 (2)	<0.02 (2)	RAISP006 IS013-09HA
	0.104	0.057	15	107	<0.01 (2)	<0.01 (2)	<0.02 (2)	
	0.106	0.056	60	97	<0.01 (2)	<0.01 (2)	<0.02 (2)	
USA, York, NE Soil: SiL [a] + [b]	0.108	0.057	00	89	<0.01 (2)	<0.01 (2)	<0.02 (2)	RAISP010 IS040-09HA
	0.105	0.055	60	89	<0.01 (2)	<0.01 (2)	<0.02 (2)	
USA, Springfield, NE Soil: SiL [b]	0.107	0.083	00	140	<0.01 (2)	<0.01 (2)	<0.02 (2)	RAISP006 IS020-09HA
	0.103	0.08	15	112	<0.01 (2)	<0.01 (2)	<0.02 (2)	
	0.107	0.08	60	87	<0.01 (2)	0.013 (2)	0.023 (2)	
US, ANorthwood, ND Soil: SiL [b]	0.104	0.553	00	147	<0.01 (2)	<0.01 (2)	<0.02 (2)	RAISP006 IS018-09HA
	0.105	0.056	15	110	<0.01 (2)	<0.01 (2)	<0.02 (2)	
	0.107	0.057	60	97	<0.01 (2)	<0.01 (2)	<0.02 (2)	
USA, Marysville, OH Soil: CL [b]	0.103	0.061	00	139	<0.01 (2)	<0.01 (2)	<0.02 (2)	RAISP006 IS009-09HA
	0.103	0.063	15	107	<0.01 (2)	<0.01 (2)	<0.02 (2)	
	0.103	0.062	60	93	<0.01 (2)	<0.01 (2)	<0.02 (2)	
USA, Marysville, OH Soil: CL [a] + [b]	0.103	0.061	00	78	<0.01 (2)	<0.01 (2)	<0.02 (2)	RAISP010 IS036-09HA
	0.105	0.064	60	78	<0.01(2)	0.016, 0.019	0.026, 0.029 (0.028)	
USA, Arkansas, WI Soil: SaL [b]	0.106	0.056	00	144	<0.01 (2)	<0.01 (2)	<0.02 (2)	RAISP006 IS017-09HA
	0.105	0.056	15	110	<0.01 (2)	<0.01 (2)	<0.02 (2)	
	0.106	0.056	60	82	<0.01 (2)	0.010, 0.012	0.020, 0.022 (0.021)	

LSa=loamy sand, SiL = silty loam, CL=clay loam, SaL=sandy loam, L=loam, SiCL=silty clay loam, SaCL=sandy clay loam;

[a] spray mix prepared from SC 480 g/L isoxaflutole and SC 480 g/L of the isopropylammonium salt of glyphosate;

[b] Additionally, adjuvants were used, which included AMS at 2% w/w, a non-ionic surfactant ranging from 0.5% to 1% v/v, a drift control agent, and if necessary anti-foam;

[c] Sample sizes were below the required minimum of 1 kg (FAO manual, 2009).

APPRAISAL

Isoxaflutole is a herbicide that was first evaluated by the 2013 JMPR, when an ADI of 0–0.02 mg/kg bw was established and an ARfD was considered unnecessary.

The residue definition for isoxaflutole for compliance with the MRL for plant and animal commodities and for dietary risk assessment for plant commodities, including tolerant soya beans, is the *sum of isoxaflutole and isoxaflutole diketonitrile, expressed as isoxaflutole.*

The residue definition for isoxaflutole for dietary risk assessment for animal commodities is the *sum of isoxaflutole, isoxaflutole diketonitrile, RPA 205834 (2-aminomethylene-l-cyclopropyl-3-(2-mesyl-4-trifluoromethylphenyl)-propane-1,3-dione) and RPA 207048 (1-cyclopropyl-2-hydroxymethylene-3- (2-mesyl-4-trifluoromethylphenyl)-propane-1,3-dione), including their conjugates, expressed as isoxaflutole.*

The residue is not fat-soluble.

Isoxaflutole was scheduled at the Fifty-first Session of the CCPR (2019) for evaluation of additional uses by the 2020 JMPR, which was postponed to the 2021 Extra JMPR. The Meeting received supervised residue trials on soya bean that had been previously evaluated by the 2013 JMPR and new GAP information on isoxaflutole in/on glyphosate/HPPD tolerant soya bean.

Results of supervised residue trials on crops

Soya bean (dry)

Isoxaflutole is registered for use in the USA as a post-emergence application to glyphosate tolerant and isoxaflutole-tolerant soya bean. The critical GAP is a single application of 0.09 kg ai/ha up to bud not including first bloom growth stage (BBCH 59). The PHI is at least 70 days. Livestock are not allowed to graze or feed forage or hay from treated soya bean.

A total of 77 residue trials were conducted with isoxaflutole in/on glyphosate/HPPD tolerant soya bean in Canada and the USA, where the compound was applied at various BBCH stages and harvested at different days after the application.

The Meeting agreed to consider residue data from all trials approximating the post-emergence (BBCH 59) timing that were conducted with the PHI of at least 70 days.

The residues are (n = 24): < 0.02 (15), 0.02, 0.021, 0.022, 0.023, 0.025, 0.026, 0.028, 0.030 and 0.037 mg/kg.

The Meeting estimated a maximum residue level of 0.04 mg/kg and a STMR of 0.02 mg/kg for isoxaflutole in soya beans.

Fate of residues during processing

Processing factors estimated for soya beans by the 2013 JMPR are 1.2 for meal, 0.79 for hulls, < 0.4 for RBD oil (Refined, bleached and deodorized) and milk, and 6 for aspirated grain fractions.

Based on a STMR of 0.02 mg/kg for soya beans, the Meeting estimated STMR-Ps of 0.008 mg/kg for soya bean refined oil and milk, and median residues of 0.024 mg/kg for soya bean meal, 0.016 mg/kg for soya bean hulls and 0.12 mg/kg for soya bean aspirated grain fractions.

Residues in animal commodities

Including the median residues of soya bean processed commodities and the STMR for soya bean (dry) did not significantly impact the dietary burden of isoxaflutole estimated by the 2013 JMPR.

The Meeting confirmed its previous recommendations for isoxaflutole in animal commodities.

RECOMMENDATIONS

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

Definition of the residue for isoxaflutole for compliance with the MRL for plant and animal commodities and for dietary risk assessment for plant commodities, including tolerant soya beans, is the *sum of isoxaflutole and isoxaflutole diketonitrile, expressed as isoxaflutole.*

The residue definition for isoxaflutole for dietary risk assessment for animal commodities is the *sum of isoxaflutole, isoxaflutole diketonitrile, RPA 205834 (2-aminomethylene-l-cyclopropyl-3-(2-mesyl-4-trifluoromethylphenyl)-propane-1,3-dione) and RPA 207048 (1-cyclopropyl-2-hydroxymethylene-3- (2-mesyl-4-trifluoromethylphenyl)-propane-1,3-dione), including their conjugates, expressed as isoxaflutole.*

The residue is not fat-soluble.

Table 1 Recommendations for residues of isoxaflutole from the 2021 Extra JMPR

Commodity		Maximum residue level, mg/kg	STMR or STMR-P, mg/kg
CCN	Name		
VD 0541	Soya bean (dry)	0.04	0.02
OR 0541	Soya bean oil, refined		0.008
	Soya bean milk		0.008

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for isoxaflutole is 0–0.02 mg/kg bw. The International Estimated Daily Intakes (IEDIs) for isoxaflutole were estimated for the 17 GEMS/Food Consumption Cluster diets using the STMR or STMR-P values estimated by the JMPR. The results are shown in Annex 3 of the 2021 Extra JMPR Report.

The IEDIs accounted for 0% of the maximum ADI. The Meeting concluded that the long-term dietary exposure to residues of isoxaflutole from uses considered by the JMPR is unlikely to present a public health concern

Acute dietary exposure

The 2013 JMPR decided that an ARfD for isoxaflutole is unnecessary. The Meeting therefore concluded that the acute dietary exposure to residues of isoxaflutole from the uses considered is unlikely to present a public health concern.

REFERENCES

Code	Authors	Year	Title, Report reference
M-368661-01-1	Dallstream, K. A.; Fischer, D. R.	2010	Balance® Pro 480 SC - Magnitude of the residue in/on soybeans. Report No. RAISP006. Bayer CropScience AG. Unpublished.
M-368669-01-1	Beedle, E. C.; Dallstream, K. A.	2010	Balance® Pro 480 SC and Glyfos® - Magnitude of the residue in/on soybeans. Report No. RAISP010. Bayer CropScience AG. Unpublished

MANDIPROPAMID (231)

First draft prepared by Dr J Giordano, the Environmental Protection Agency, United States of America

EXPLANATION

Mandipropamid is a fungicide in the mandelamide class used for the control of foliar oomycete pathogens in a range of crops, including *Plasmopara viticola* in grapes, *Phytophthora infestans* in potatoes and tomatoes, and *Pseudoperonospora cubensis* in cucurbits. Mandipropamid was first evaluated by the JMPR in 2008 when an ADI of 0–0.2 mg/kg bw was established, and maximum residue levels were recommended for various crops. An ARfD was considered unnecessary. Additional uses were evaluated by the 2013 and 2018 JMPR. The JMPR agreed on the following residue definition for mandipropamid:

Definition of the residue for compliance with the MRL and dietary risk assessment for plant and animal commodities: *mandipropamid*.

The residue is not fat-soluble.

The Forty-third Codex Alimentarius Commission (2020) approved the new work proposals including the priority list of pesticides for evaluation by the current Meeting. The priority list included mandipropamid for evaluation of an additional use on citrus fruits.

The current Meeting received information on field trials and processing studies to support the estimation of new maximum residue levels in citrus fruit and citrus processed commodities.

RESIDUE ANALYSIS

Analytical methods

Analytical methods for the determination of residues of mandipropamid in food and feedstuffs of plant origin were evaluated by the 2008, 2013, and 2018 JMPRs. The methods previously submitted include RAM 415/01 and RAM 415/02 with LOQs of 0.01 mg/kg validated in high-water, high-acid, high-starch, and high-oil raw agricultural commodities and the processed fractions of tomatoes, grapes, and cocoa beans; WO 9.154, v.3 (based on RAM 415/01) with a LOQ of 0.01 mg/kg validated on green beans; and GRM 001.01A and B with a LOQ of 0.005 mg/kg validated on potatoes and potato processed commodities.

Working Outline WO 9.210 v.1

Method WO 9.210 v.1 was used to quantitate residues of mandipropamid in lemons and grapefruit (whole fruit, flesh, and peel for both commodities). This method is based on the reference method, "Mandipropamid RAM 415/02 Mandipropamid-Residue Method for the Determination of Mandipropamid in Crops," by S. J. Crook, which was evaluated by the 2008 JMPR. Minor modifications are described below. Additional validation data were generated to support the minor modifications.

Residues of mandipropamid were extracted from the sample by homogenizing with acetonitrile:water (80:20, v:v). An aliquot of the extract was subjected to solid phase extraction (SPE) clean-up. Mandipropamid was analysed via LC-MS/MS. Modifications from the reference method include using Agilent LC-MS/MS 6460 (considered equivalent to the instrument used in the reference method), preparing standard in a solution of methanol:water:formic acid (80:20:0.2) instead of methanol:water:acetic acid (50:50:0.2), and using an HLB SPE system instead of C18.

Linearity was demonstrated in the range of 0.011–11 ng/mL with correlation coefficients ≥0.99. Quantitation and confirmation ion transitions are 412→328 and 412→204, respectively; however, only results for the quantitation ion transition were reported. Acceptable mean recoveries were obtained for each matrix at each fortification level. The overall relative standard deviation for each matrix at each fortification level was ≤20%, demonstrating satisfactory analytical precision. A validated LOQ of 0.01 mg/kg has been established for the determination of mandipropamid residues in lemons and grapefruit whole fruit. The Meeting concluded that Working Outline WO 9.210 v.1 and v.2 are sufficiently similar that orange flesh and peel validation at a LOQ of 0.01 mg/kg is adequate to translate to lemon and grapefruit flesh and peel.

Working Outline WO 9.210 v.2

Method WO 9.210 v.2 was used to quantitate residues of mandipropamid in orange whole fruit, flesh, peel, juice, oil, and dried pulp. This method is based on the reference method, "Mandipropamid RAM 415/02 Mandipropamid-Residue Method for the Determination of Mandipropamid in Crops," by S. J. Crook, which was evaluated by the 2008 JMPR. Minor modifications are described below. Additional validation data were generated to support the minor modifications.

Residues of mandipropamid were extracted from the sample by homogenizing with acetonitrile:water (80:20, v:v). An aliquot of the extract was centrifuged, filtered, and diluted. Mandipropamid was analysed via LC-MS/MS. Modifications from the reference method include using a 5g sample size instead of 10g for orange dried pulp, using an incubator/shaker for the oil crop fraction instead of the homogenizer (homogenization was not needed as oil is already homogeneous), not using an SPE clean-up step, and using methanol:water:formic acid (50:50:0.2, v:v:v) instead of methanol:water:acetic acid (50:50:0.2, v:v:v) as the solvent mixture for final dilution.

Linearity was demonstrated in the range of 0.11–11 ng/mL with correlation coefficients ≥0.99. Quantitation and confirmation ion transitions are 412→328 and 412→204, respectively; however, only results for the quantitation ion transition were reported. Acceptable mean recoveries were obtained for each matrix at each fortification level. The overall relative standard deviation for each matrix at each fortification level was ≤20%, demonstrating satisfactory analytical precision. A validated LOQ of 0.01 mg/kg has been established for the determination of mandipropamid residues in oranges and orange processed commodities.

Table 1 Summary of Method Validation (MV) and Concurrent Recovery (CR) of mandipropamid in orange (Raw Agricultural Commodity and Processed Commodities), lemon, and grapefruit

Matrix	Analyte	Fortification level [mg/kg]	Individual recoveries [%]	Range of recoveries [%]	Mean recovery [%]	RSD [%]	Reference
Method WO 9.210 v.1							
Lemon Fruit	Mandipropamid	0.01	MV: 103, 88, 85	85-103	92	10	K, Homa (2017, IR-4 PR No. 11139)
			CR: 77, 70, 70	70-77	72	6	
		0.1	MV: 89, 92, 71	71-92	84	14	
			CR: 96, 87, 86, 93	86-96	91	5	
		1.0	MV: 77, 81, 79	77-81	79	3	
Lemon Peel	Mandipropamid	0.1	CR: 99	NA	NA	NA	
Lemon Flesh	Mandipropamid	0.01	CR: 66, 68, 62, 60	60-68	64	6	

Matrix	Analyte	Fortification level [mg/kg]	Individual recoveries [%]	Range of recoveries [%]	Mean recovery [%]	RSD [%]	Reference
Grapefruit	Mandipropamid	0.01	MV: 93, 96, 94, 94, 94, 93	93-96	94	1	K, Homa (2017, IR-4 PR No. 11140)
			CR: 87, 76, 86	76-87	83	7	
		0.1	MV: 96, 112, 107	96-112	105	8	
			CR: 75, 94, 77, 76, 89, 74, 96	74-96	83	12	
		1.0	MV: 96, 100, 87	87-100	94	7	
Grapefruit Peel	Mandipropamid	0.1	CR: 90, 82	82-90	86	NA	
Grapefruit Flesh	Mandipropamid	0.01	CR: 67	NA	NA	NA	
		0.1	CR: 83	83	83	83	
Method WO 9.210 v.2							
Orange Fruit	Mandipropamid	0.01	MV: 107, 113, 111	107-113	110	3	K, Homa (2017, IR-4 PR No. 11138)
			CR: 83, 96, 101, 77, 82, 102, 90, 78	77-102	89	11	
		0.1	MV: 109, 106, 91	91-109	102	9	
			CR: 115, 93, 90, 98, 93, 87	87-115	96	10	
		1.0	MV: 102, 95, 89	89-102	95	7	
			CR: 107, 86	86-107	97	NA	
Orange Fresh Peel	Mandipropamid	0.01	MV: 96, 88, 88	88-96	91	5	
			CR: 102, 98, 102	98-102	101	2	
		0.1	MV: 91, 94, 102	91-102	96	6	
		1.0	MV: 109, 107, 110	107-110	109	1	
Orange Flesh	Mandipropamid	0.01	MV: 89, 86, 92	86-92	89	3	
			CR: 106, 95, 96	95-106	99	6	
		0.1	MV: 94, 98, 95	94-98	96	2	
		1.0	MV: 99, 96, 100	96-100	98	2	
			CR: 90	NA	NA	NA	
Orange Juice	Mandipropamid	0.01	MV: 98, 100, 101	98-101	100	2	
			CR: 111, 96, 100	96-111	102	8	
		0.1	MV: 101, 103, 103	101-103	102	1	

Matrix	Analyte	Fortification level [mg/kg]	Individual recoveries [%]	Range of recoveries [%]	Mean recovery [%]	RSD [%]	Reference
		1.0	MV: 103, 102, 99	99-103	101	2	
			CR: 97	NA	NA	NA	
Orange Oil	Mandipropamid	0.01	MV: 88, 91, 89	88-91	89	2	
			CR: 106, 111, 113	106-113	110	3	
		0.1	MV: 97, 86, 91	86-97	91	6	
		1.0	MV: 92, 93, 95	92-95	93	2	
			CR: 100	NA	NA	NA	
		10.59	CR: 101, 100, 105	100-105	102	3	
Orange Dried Pulp	Mandipropamid	0.01	MV: 89, 92, 89	89-92	90	2	
			CR: 112, 114, 109	109-114	112	2	
		0.02	CR: 110, 105, 100	100-110	105	5	
		0.1	MV: 97, 99, 98	97-99	98	1	
		1.0	MV: 94, 99, 100	94-100	98	3	
		2.1	CR: 93	NA	NA	NA	

USE PATTERN

Information on GAP for mandipropamid on citrus fruit in the USA was provided to the Meeting and is summarized in Table 2.

Table 2 Registered Use of mandipropamid provided to the 2021 Extra JMPR

Use Site	Country	Conc. (%)	Type	Rate (g/ha/app)	Rate (g/ha/year)	Max No. Apps.	RTI (days)	DALA (days)
Citrus Fruit [a]	USA	23.3	SC	146	291	2	30	0

[a] Label specified all crops within USA's Crop Group 10-10 including: Australian desert lime; Australian finger-lime; Australian round lime; Brown River finger lime; calamondin; citron; citrus hybrids; grapefruit; Japanese summer grapefruit; kumquat; lemon; lime; Mediterranean mandarin; mount white lime; New Guinea wild lime; orange, sour; orange, sweet; pummelo; Russel River lime; satsuma mandarin; sweet lime; tachibana orange; Tahiti lime; tangelo; tangerine (mandarin); tangor; trifoliate orange; uniq fruit; cultivars, varieties, and/or hybrids of these.

* Additional instructions include: Make first foliar application prior to the onset of disease. Apply by ground application in a spray volume sufficient to ensure good coverage of the foliage and fruit.

RESULTS OF SUPERVISED RESIDUE TRIALS ON CROPS

The Meeting received data from supervised residue trials conducted on lemons, mandarins, oranges, and grapefruit.

The field trial reports included method validation and concurrent recovery data at levels reflecting those observed in the field trial samples; dates from critical events during the study, including application,

harvest, storage, and analysis; and 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 method described above.

The field trial study designs included control plots. Measured residues from control plots were <LOQ and are not included in the summary tables in this evaluation.

Individual and mean per-trial residues are presented for all trials. When calculating average residues, values below the LOQ were assumed to be at the LOQ. In the summary tables, residue values used for estimation of maximum residue levels are underlined.

Supervised trials for mandipropamid:

Crop Group	Commodity	Region/Country	Table No.
Citrus fruit	Lemon		Table 3
	Mandarin		Table 4
	Orange		Table 5
	Grapefruit		Table 6

Citrus Fruits

Lemon

A total of five field trials were conducted in the USA during 2014–2015 growing seasons in a study reported by K, Homa (2017, IR-4 PR No. 11139). Treated plots received two foliar-directed applications of an SC formulation, each targeted at 146 g ai/ha (seasonal rate equal to 291 g ai/ha) with a 30-day re-treatment interval (RTI) and harvested 0 days after last application (DALA; equal to USA GAP). Actual application rates ranged from 145.7–150.2 g ai/ha and RTIs ranged from 28–30 days. An adjuvant was added to the spray solution in all trials. At one trial location (14-CA102), residue decline was investigated with additional harvests 1, 4, 7, and 11 DALA. At one trial location (14-CA99), an additional treated sample was harvested 0 DALA to investigate distribution of residues throughout the fruit.

At least 24 fruit (minimum of 2.6 kg) were collected for whole fruit analysis and quartered. At least 12 fruit (minimum of 1.9 kg) were collected for flesh and peel analysis and were left whole. Fruit were placed in frozen storage within 1 hour and 30 minutes of sampling, shipped by ACDS Freezer Truck to the analytical laboratory, and stored frozen prior to extraction for analysis. Fruit designated for flesh and peel analysis were cut in half for separation into ½ fruit (flesh plus peel), flesh only, and peel only samples. All samples were homogenized in the presence of dry ice.

The maximum storage duration was 685 days (23 months). Storage durations and conditions are supported by the storage stability data.

Samples were analysed for residues of mandipropamid using Method WO 9.210 v.1, described above. Concurrent recovery data indicate that the method is suitable.

Table 3 Results of mandipropamid residue trials on lemons conducted in the USA

Trial ID	Variety	Application Rate (g ai/ha)	Application Volume (L ai/ha)	RTI (days)	DALA (days)	Portion	Residues (mg/kg)	Adjuvant
14-CA99 Exeter, CA (4-Nov-2014)	Lisbon	1. 148.0 2. 149.1	1. 1019.6 2. 1015.8	30	0	Fruit	0.20, 0.18 [0.19]	Induce
					0	½ Fruit [a]	0.091, 0.11 [0.10]	
					0	Peel	0.33, 0.30 [0.31]	
					0	Flesh	<0.010, <0.010 [<0.010]	
14-CA102 Exeter, CA (9-Dec-2014)	Lisbon	1. 145.7 2. 148.0	1. 1665.0 2. 1640.7	30	0	Fruit	0.093, 0.077 [0.085]	Induce
					1	Fruit	0.090, 0.075 [0.082]	
					4	Fruit	0.067, 0.073 [0.070]	
					7	Fruit	0.076, 0.067 [0.071]	
					11	Fruit	0.083, 0.054 [0.069]	
15-CA101 Exeter, CA (5-Nov-2015)	Allen Eureka	1. 146.8 2. 148.0	1. 935.4 2. 926.0	28	0	Fruit	0.11, 0.084 [0.098]	Induce
15-CA96 Riverside, CA (5-Feb-2015)	Eureka	1. 148.0 2. 149.1	1. 932.6 2. 940.1	28	0	Fruit	0.24, 0.22 [0.23]	Activator 90
15-CA97 Riverside, CA (6-Feb-2015)	Lisbon	1. 150.2 2. 150.2	1. 1415.3 2. 1406.8	28	0	Fruit	0.094, 0.095 [0.095]	Activator 90

[a] Samples of ½ fruit (flesh plus peel) analysed alongside flesh and peel fractions.

Mandarin

Two field trials were conducted in the USA during the 2015 growing season in a study reported by K, Homa (2017, IR-4 PR No. 11138). The treated plots received two foliar-directed applications of an SC formulation, each targeted at 146 g ai/ha (seasonal rate equal to 291 g ai/ha) with a 28-day RTI and harvested 0 DALA (equal to USA GAP). Actual application rates ranged from 144.6–148.0 g ai/ha and RTIs ranged from 28–29 days. An adjuvant was added to the spray solution for all trials. At one trial location (15-FL158), additional treated samples were harvested 0 DALA to investigate distribution of residues throughout the fruit.

At least 24 fruit (minimum of 1.9 kg) were collected for whole fruit analysis and quartered. At least 12 fruit (minimum of 1.4 kg) were collected for flesh and peel analysis and were left whole. Fruits were placed in frozen storage within 1 hour and 34 minutes of sampling, shipped by ACDS Freezer Truck to the analytical laboratory, and stored frozen prior to extraction for analysis. Fruit designated for flesh and peel

analysis were cut in half for separation into ½ fruit (flesh plus peel), flesh only, and peel only samples. All samples were homogenized in the presence of dry ice.

The maximum storage duration for mandarin whole fruit, flesh, and peel was 763 days (25 months). Since storage stability data do not show dissipation following 24 months of frozen storage in various crop commodities, the extra month of storage is not expected to result in significant residue dissipation.

Samples were analysed for residues of mandipropamid using Method WO 9.210 v.2, described above. Concurrent recovery data indicate that the method is suitable.

Table 4 Results of mandipropamid residue trials on mandarins conducted in the USA.

Trial Number	Variety	Application Rate (g ai/ha)	Application Volume (L/ha)	RTI (days)	Portion	DALA	Residue; Unscaled (mg/kg)	Adjuvant
14-CA100 Exeter, CA (4-Feb-2015)	W. Murcott (mandarin)	1. 148.0 2. 145.7	1. 1,920.4 2. 1,930.7	28	Fruit	0	0.18, 0.17 [0.17]	Induce
					½ Fruit [a]	0	0.16, 0.16 [0.16]	
					Peel	0	0.57, 0.57 [0.57]	
					Flesh	0	<0.010, <0.010 [<0.010]	
15-FL158 Citra, FL (12-Oct-2015)	Satsuma	1. 144.6 2. 144.6	1. 2,793.1 2. 2,802.4	29	Fruit	0	0.11, 0.082 [0.096]	Induce

[a] Samples of ½ fruit (flesh plus peel) analysed alongside flesh and peel fractions.

Orange

A total of ten field trials were conducted in the USA during the 2014–2015 growing seasons in a study reported by K, Homa (2017, IR-4 PR No. 11138). Treated plots received two foliar-directed applications of an SC formulation, each targeted at 146 g ai/ha (seasonal rate equal to 291 g ai/ha) with a 30-day RTI and harvested 0 DALA (equal to USA GAP). Actual application rates ranged from 145.7–149.1 g ai/ha (with two exceptions discussed below) and RTIs ranged from 28–31 days. An adjuvant was added to the spray solution at all trials except one (15-FL404). At two trial locations (14-CA132 and 15-TX422), residue decline was investigated with additional harvests 1, 2–4, 7, and 9–11 DALA. At two trial locations (14-FL170 and 15-FL3), additional treated samples were harvested 0 DALA to investigate distribution of residues throughout the fruit.

At two field trial locations (14-FL170 and 15-FL3), both applications were applied at 57–58 g ai/ha, for a total application rate equal to 114–117 g ai/ha (~40% of the GAP) due to a misinterpretation of the protocol.

At least 24 fruit (minimum of 2.0 kg) were collected for whole fruit analysis and quartered. At least 12 fruit (minimum of 2.2 kg) were collected for flesh and peel analysis and were left whole. Fruits were placed in frozen storage within 3 hours and 20 minutes of sampling, shipped by ACDS Freezer Truck to the analytical laboratory, and stored frozen prior to extraction for analysis. Fruit designated for flesh and peel

analysis were cut in half for separation into ½ fruit (flesh plus peel), flesh only, and peel only samples. All samples were homogenized in the presence of dry ice.

The maximum storage duration for orange whole fruit, flesh, and peel was 763 days (25 months). Since storage stability data do not show dissipation following 24 months of frozen storage in various crop commodities, the extra month of storage is not expected to result in significant residue dissipation.

Samples were analysed for residues of mandipropamid using Method WO 9.210 v.2, described above. Concurrent recovery data indicate that the method is suitable.

Table 5 Results of mandipropamid residue trials on oranges conducted in the USA

Trial Number	Variety	Application Rate (g ai/ha)	Application Volume (L/ha)	RTI (days)	Portion	DALA	Residue; Unscaled (mg/kg)	Adjuvant
14-CA132 Exeter, CA (10-Dec-2014)	Washing-ton Navel	1. 148.0 2. 146.8	1. 1,459.2 2. 1434.9	29	Fruit	0	0.054, 0.059 [0.057]	Induce
					Fruit	1	0.076, 0.10 [0.088]	
					Fruit	4	0.051, 0.063 [0.057]	
					Fruit	7	0.046, 0.045 [0.046]	
					Fruit	11	0.054, 0.061 [0.058]	
14-CA98 Exeter, FL (13-Jun-2014)	Olinda Valencia	1. 145.7 2. 149.1	1. 1,854.9 2. 1,949.4	31	Fruit	0	0.054, 0.045 [0.050]	Latron B-1956
14-FL170 Citra, FL (3-Nov-2014)	Hamlin	1. 58.3 2. 58.3	1. 1132.8 2. 1131.8	30	Fruit	0	0.044, 0.049 [0.047]	De-Fac 820
					½ Fruit[a]	0	0.047, 0.047 [0.047]	
					Peel	0	0.18, 0.17 [0.18]	
					Flesh	0	<0.010, <0.010 [<0.010]	
15-FL157 Citra, FL (17-Nov-2015)	Naval	1. 145.7 2. 145.7	1. 2,824.9 2. 2,824.0	29	Fruit	0	0.082, 0.079 [0.081]	Induce
15-FL159[b] Winter Garden, FL (3-Feb-2015)	Valencia	1. 145.7 2. 148.0	1. 926.0 2. 941.0	28	Fruit	0	0.19, 0.19 [0.19]	D-W Surfactant
15-FL160[b] Winter Garden, FL (3-Feb-2015)	Valencia	1. 146.8 2. 145.7	1. 1,876.4 2. 1,856.8	28	Fruit	0	0.074, 0.073 [0.074]	D-W Surfactant
15-FL3 Citra, FL	Honeybell (tangelo)	1. 57.2 2. 57.2	1. 1,115.9 2. 1,114.1	29	Fruit	0	0.033, 0.034 [0.034]	De-Fac 820

Trial Number	Variety	Application Rate (g ai/ha)	Application Volume (L/ha)	RTI (days)	Portion	DALA	Residue; Unscaled (mg/kg)	Adjuvant
(4-Nov-2014)								
					½ Fruit[a]	0	0.031, 0.027 [0.029]	
					Peel	0	0.12, 0.12 [0.12]	
					Flesh	0	<0.010, <0.010 [<0.010]	
15-FL403[c] St. Lucie County, FL (11-Feb-2015)	Valencia on Swingle	1. 146.8 2. 149.1	1. 934.5 2. 913.9	30	Fruit	0	0.14, 0.16 [0.15]	Activator 90
15-FL404[c] St. Lucie County, FL (11-Feb-2015)	Valencia on Swingle	1. 146.8 2. 146.8	1. 1,329.2 2. 1,302.1	30	Fruit	0	0.070, 0.072 [0.071]	None
15-TX422 Welasco, TX (27-Jan-2015)	Valencia	1. 145.7 2. 145.7	1. 2,204.7 2. 2,288.9	29	Fruit	0	0.079, 0.084 [0.082]	R-11
						1	0.089, 0.091 [0.090]	
						2	0.11, 0.11 [0.11]	
						7	0.086, 0.084 [0.085]	
						9	0.088, 0.091 [0.090]	

[a] Samples of ½ fruit (flesh plus peel) analysed alongside flesh and peel fractions.

[b] Trials 15-FL159 and 15-FL160 were conducted at the same location, pesticide was applied on the same day, and the same variety of fruit was harvested. Therefore, these two trials are not considered independent.

[c] Trials 15-FL403 and 15-FL404 were conducted at the same location, pesticide was applied on the same day, and the same variety of fruit was harvested. Therefore, these two trials are not considered independent.

Grapefruit

A total of six field trials were conducted in the USA during 2014–2015 growing seasons in a study reported by K, Homa (2017, IR-4 PR No. 11140). The treated plots received two foliar-directed applications of an SC formulation, each targeted at 146 g ai/ha (seasonal rate equal to 291 g ai/ha) with a 30-day RTI and harvested 0 DALA (equal to USA GAP). Actual application rates ranged from 145.7–150.2 g ai/ha (with an exception discussed below) and RTIs ranged from 28–32 days. An adjuvant was added to the spray solution at all trials. At one trial location (14-FL171), residue decline was investigated with additional harvests 1, 3, 7, and 10 DALA. At two trial locations (14-FL171 and 15-TX434), an additional treated sample was harvested 0 DALA to investigate distribution of residues throughout the fruit.

At one field trial location (14-FL171), both applications were made at 58 g ai/ha for a total application rate of 115 g ai/ha (~40% of the USA GAP) due to a misinterpretation of the protocol.

At least 24 fruit (minimum of 2.3 kg) were collected for whole fruit analysis quartered. At least 12 fruit (minimum of 3.6 kg) were collected for flesh and peel analysis and were left whole. Where reported, fruits were placed in frozen storage within 1 hour and 45 minutes of sampling; it is noted that the time between sampling and freezing was not reported at one trial for grapefruit used for flesh and peel analysis (15-TX434). Samples were shipped by ACDS Freezer Truck to the analytical laboratory and stored frozen prior to extraction for analysis. Fruit designated for flesh and peel analysis were cut in half for separation into ½ fruit (flesh plus peel), flesh only, and peel only samples. All samples were homogenized in the presence of dry ice.

The maximum storage duration was 623 days (20 months). Storage durations and conditions are supported by the storage stability data.

Samples were analysed for residues of mandipropamid using Method WO 9.210 v.1, described above. Concurrent recovery data indicate that the method is suitable.

Table 6 Results of mandipropamid residue trials on grapefruit conducted in the USA

Trial ID	Variety	Application Rate (g ai/ha)	Application Volume (L ai/ha)	RTI (days)	DALA (days)	Portion	Residues; Unscaled (mg/kg)	Adjuvant
14-CA101 Exeter, CA (18-Nov-2014)	Rio Red	1. 149.1 2. 150.2	1. 1484.5 2. 1513.5	28	0	Fruit	0.060, 0.049 [0.055]	Induce
14-FL171 Citra, FL (6-Nov-2014)	Ruby Red	1. 58.3 2. 58.3	1. 1138.4 2. 1132.8	32	0	Fruit	0.026, 0.034 [0.030]	De-Fac 820
					1	Fruit	0.024, 0.043 [0.034]	
					3	Fruit	0.030, 0.027 [0.029]	
					7	Fruit	0.011, 0.013 [0.012]	
					10	Fruit	0.019, 0.017 [0.018]	
					0	½ Fruit [a]	0.033, 0.028 [0.031]	
					0	Peel	0.087, 0.081 [0.084]	
					0	Flesh	<0.010, <0.010 [<0.010]	
15-CA99 Riverside, CA (3-Mar-2015)	Marsh	1. 149.1 2. 148.0	1. 945.7 2. 938.2	30	0	Fruit	0.069, 0.089 [0.079]	Activator 90
15-FL173 St. Lucie County, FL (11-Feb-2015)	Ruby Red on X-639 Root Stock	1. 146.8 2. 145.7	1. 724.0 2. 707.2	30	0	Fruit	0.064, 0.049 [0.057]	Activator 90

Trial ID	Variety	Application Rate (g ai/ha)	Application Volume (L ai/ha)	RTI (days)	DALA (days)	Portion	Residues; Unscaled (mg/kg)	Adjuvant
15-FL174 Citrus Grove, FL (10-Nov-2015)	Ruby Red	1. 146.8 2. 146.8	1. 2824.9 2. 2833.3	28	0	Fruit	0.049, 0.037 [0.043]	Induce
15-TX434 Welasco, TX (27-Jan-2015)	Rio Red	1. 145.7 2. 145.7	1. 2204.7 2. 2288.9	30	0	Fruit	0.054, 0.045 [0.050]	R-11
					0	½ Fruit [a]	0.086, 0.058 [0.072]	
					0	Peel	0.20, 0.17 [0.19]	
					0	Flesh	<0.010, <0.010 [<0.010]	

[a] Samples of ½ fruit (flesh plus peel) analysed alongside flesh and peel fractions.

FATE OF RESIUDES IN STORAGE AND DURING PROCESSING

In Processing

Oranges

A study was submitted investigating the effect of orange processing on mandipropamid residues (K, Homa; 2017, IR-4 PR No. 11138).

An additional treatment pattern from orange field trial location 14-CA98 (described above) received two foliar applications, each at 442.7–450.6 g ai/ha, on a 30-day RTI (seasonal rate equal to 893 g ai/ha; ~3× greater than the USA GAP application rate). Application spray volumes were 1868.9 and 1966.2 L ai/ha. An adjuvant was added to the spray solution. Samples were harvested 0 DALA.

A total of 117 kg of treated oranges were harvested. Samples were placed in a refrigerated ACDS Freezer Truck within 1 hour and 10 minutes of harvest and shipped to the processing facility. Samples were refrigerated upon arrival and processed into dried pulp, orange juice, and orange oil within 3 hours and 18 minutes of arrival. Processing procedures simulated commercial operations as closely as possible to generate the required fractions. For orange oil processing, whole oranges were scarified to collect flavedo and oil:water emulsion. The oil:water emulsion was centrifuged for separation to orange oil. For juice processing, scarified oranges were halved, and juice was mechanically extracted. Juice was filtered to separate out seed and peel fragments. For dried pulp processing, waste products from oil and juice production (peel, flavedo, and seed) were shredded, hydraulic pressed, and air dried.

Processed fractions were shipped by ACDS Freezer Truck to the analytical laboratory and stored frozen prior to extraction for analysis. All samples were homogenized in the presence of dry ice.

The maximum storage duration was 773 days for oranges, orange dried pulp, and orange oil and 769 days for orange juice (25 months). Since storage stability data do not show dissipation following 24 months of frozen storage in various crop commodities, the extra month of storage is not expected to result in significant residue dissipation.

Samples were analysed for residues of mandipropamid using Method WO 9.210 v.2, described above. Concurrent recovery data indicate that the method is suitable.

Table 7 Residues of mandipropamid in orange processed commodities

Trial ID	Commodity	Residue (mg/kg)	Processing factor
14-CA98	Orange (pre-processing)	0.23	--
	Dried Pulp	0.66	2.9x
	Juice	<0.01	<0.043
	Oil	10.2	45x

NC = Not Calculated.

APPRAISAL

Mandipropamid is a fungicide in the mandelamide class used for the control of foliar oomycete pathogens in a range of crops, including *Plasmopara viticola* in grapes, *Phytophthora infestans* in potatoes and tomatoes, and *Pseudoperonospora cubensis* in cucurbits. Mandipropamid was first evaluated by the JMPR in 2008 when an ADI of 0–0.2 mg/kg bw was established, and maximum residue levels were recommended for various crops. An ARfD was considered unnecessary. Additional uses were evaluated by the 2013 and 2018 JMPR. The JMPR agreed on the following residue definition for mandipropamid:

Definition of the residue for compliance with the MRL and dietary risk assessment for plant and animal commodities: *mandipropamid*.

The residue is not fat-soluble.

The Forty-third Codex Alimentarius Commission (2020) approved the new work proposals including the priority list of pesticides for evaluation by the current Meeting. The priority list included mandipropamid for evaluation of an additional use on citrus fruits.

The current Meeting received information on field trials and processing studies to support the estimation of new maximum residue levels in citrus fruit and citrus processed commodities.

Methods of residue analysis

The current Meeting received method validation and concurrent recovery data for use of Method WO 9.210 v.1 and v.2, both based on the validated reference method RAM 415/02. Both methods were demonstrated to have adequate performance for recovery of mandipropamid in citrus fruits and citrus processed commodities. A LOQ of 0.01 mg/kg was established for both methods for all citrus raw agricultural commodities and processed fractions.

The 2008 JMPR concluded that the multi-residue method DFG S19 is suitable for monitoring residues of mandipropamid in plant commodities, with a LOQ of 0.01 mg/kg.

Stability of pesticide residues in stored analytical samples

Storage stability data were previously provided for mandipropamid in various commodities. The 2018 JMPR concluded that mandipropamid is stable at -20 °C for at least 24 months in tomatoes (fruit and paste),

grapes (fruit and juice), potatoes (tubers and granules/flakes), lettuce, cucumbers, wheat (forage, grain, and straw), and soya beans (beans, hulls, meal, and oil); and for at least 12 months in green beans (beans, pods with seeds and beans, whole plants with pods). The storage stability data are adequate to support the supervised field trials and processing study currently under evaluation.

Results of supervised residue trials on crops

The Meeting received GAP information for mandipropamid on citrus fruits in the USA and supervised residue trials for mandipropamid on lemons, mandarins, oranges, and grapefruit.

Citrus Fruit

For all fruit, samples for whole fruit analysis were quartered in the field and samples for peel and pulp residue distribution were left whole. Comparison of whole fruit residues between quartered samples and samples left whole indicate that quartering in the field does not affect the stability of mandipropamid residue levels in citrus fruit.

The registered GAP in the USA for citrus fruits allows two foliar applications at 146 g ai/ha (seasonal application up to 291 g ai/ha) with a minimum re-treatment interval (RTI) of 30 days and a 0-day pre-harvest interval (PHI).

Lemon

Five field trials on lemons were conducted in the USA matching the USA GAP. Residues of mandipropamid were (n = 5): 0.085, 0.095, 0.098, 0.19, and 0.23 mg/kg.

Mandarin

Two field trials were conducted in the USA matching the USA GAP. Residues of mandipropamid were (n = 2): 0.096 and 0.17 mg/kg.

Orange

Eight independent field trials on oranges were conducted in the USA. Six trials matched the USA GAP. At two trials, both applications were made at ~40% of the GAP application rate (57–58 g ai/ha per application). Residues following applications matching the USA GAP were (n = 6): 0.050, 0.081, 0.088, 0.11, 0.15, and 0.19 mg/kg. Residues following application at ~40% of the GAP application rate were (n = 2): 0.034 and 0.047 mg/kg.

Residues scaled to the USA GAP (scaling factors of 0.98–2.5x) were (n = 8): 0.049, 0.081, 0.085, 0.087, 0.11, 0.12, 0.15, and 0.19 mg/kg.

Grapefruit

Six field trials on grapefruit were conducted in the USA. Five trials matched the USA GAP. At one trial, both applications were made at ~40% of the GAP application rate (58 g ai/ha per application). Residues following applications matching the USA GAP were (n = 5): 0.043, 0.050, 0.055, 0.057, and 0.079 mg/kg. The residue following application at ~40% of the GAP application rate was 0.034 mg/kg.

Residues scaled to the USA GAP (scaling factors of 0.97–2.5) were (n = 6): 0.043, 0.050, 0.053, 0.057, 0.077, and 0.085 mg/kg.

The median whole fruit residues for oranges, lemons, and grapefruits were within 5x of one another. However, the Kruskal-Wallis test showed that the residue values were from different populations. The Meeting noted that the USA GAP covers all citrus fruit and estimated the following maximum residue levels and median values for citrus subgroups:

Subgroup of Lemons and Limes: Maximum residue level of 0.5 mg/kg. Median value of 0.098 mg/kg.

Subgroup of Oranges, Sweet, Sour: Maximum residue level of 0.4 mg/kg. Median value of 0.099 mg/kg.

Subgroup of Pummelo and Grapefruits: Maximum residue level of 0.2 mg/kg. Median value of 0.055 mg/kg.

Subgroup of Mandarins: An insufficient number of trials on mandarin were available to estimate a STMR or maximum residue level. However, the Meeting noted that residues in lemons were shown to be similar to or greater than residues in mandarins. The Meeting decided to extrapolate the residues from lemon and estimated a maximum residue level of 0.5 mg/kg and a median value of 0.098 mg/kg.

At one lemon trial, one mandarin trial, two orange trials, and two grapefruit trials, additional fruit samples were separated into peel and flesh fractions. For all citrus fruit commodities (n = 6), residues in the flesh were below the LOQ (< 0.01 mg/kg). The Meeting concluded that the STMR for the flesh of all citrus fruit is 0.01 mg/kg.

Fate of residues during processing

The Meeting received an orange processing study. The processed fractions included orange dried pulp, orange juice, and orange oil. The Meeting decided to extrapolate processing factors from orange to all citrus commodities. Processing factors and residue estimates are summarized below.

Table 1 Residues of mandipropamid in citrus processed commodities

Processed Commodity	Processing Factor	Residue (RAC)		Residue (Processed Commodity)	
		Maximum Residue Level	Median	Max-P (mg/kg)	STMR-P[a] (mg/kg)
Orange Juice	< 0.043	0.4	0.099	--	< 0.0043
Orange Oil	45			--	4.5
Orange Dried Pulp	2.9			--	0.29
Lemon/Mandarin Juice[a]	< 0.043	0.5	0.098	--	< 0.0042
Lemon/Mandarin Oil[b]	45			22.5	--
Lemon/Mandarin Dried[c] Pulp	2.9			1.45	--
Grapefruit Juice[a]	< 0.043	0.2	0.055	--	< 0.0024
Grapefruit Oil[b]	45			--	--
Grapefruit Dried Pulp[c]	2.9			--	--

[a] Lemon/mandarin and grapefruit juice processing factor extrapolated from orange processing study

[b] Lemon/mandarin and grapefruit oil processing factor extrapolated from orange processing study

[c] Lemon/mandarin and grapefruit dried pulp processing factor extrapolated from orange processing study

Citrus dried pulp: Based on the maximum residue level of 0.5 mg/kg for lemons and median value of 0.099 mg/kg for oranges, the Meeting estimated a maximum residue level of 1.5 mg/kg and a median value of 0.29 mg/kg.

Citrus oil: Based on the maximum residue level of 0.5 mg/kg for lemons and median value of 0.099 mg/kg for oranges, the Meeting estimated a maximum residue level of 30 mg/kg and a STMR of 4.5 mg/kg.

Residues in animal commodities

Farm animal dietary burdens

Dietary burdens were calculated for beef cattle, dairy cattle, broilers, and laying poultry based on feed items evaluated by the JMPR. The dietary burdens were estimated using the most recent version of the OECD livestock dietary burden calculator. Dietary burdens for mandipropamid are presented in Annex 6 and summarised in Table 2, below. In addition to citrus dried pulp, kale leaves were added to the previous dietary burdens as Codex has an established MRL for the leafy vegetable Crop Group.

The Australian beef cattle maximum and mean dietary burdens of 17.51 and 9.07 ppm, respectively, and dairy cattle maximum and mean dietary burdens of 49.21 and 23.77 ppm, respectively, were refined to exclude bean forage and kale. Mandipropamid is not registered for use on beans in Australia and Australia does not import forages. The labelled use for kale in Australia prohibits feeding to livestock, and Australia does not import kale as a feed item. The refined Australian beef cattle maximum and mean dietary burdens are 1.62 and 1.61 ppm, respectively, and the refined Australian dairy cattle maximum and mean dietary burdens are 0 ppm.

Table 2 Livestock dietary burdens for mandipropamid (ppm)

	USA-Canada		EU		Australia		Japan	
	Max	Mean	Max	Mean	Max	Mean	Max	Mean
Beef cattle	0.21	0.13	15.58	7.70	1.62	1.61	--	--
Dairy cattle	0.070	0.043	20.84[a]	10.15[b]	--	--	--	--
Poultry – broiler	--	--	0.041	0.014	--	--	--	--
Poultry - layer	--	--	1.97[c]	1.20[d]	--	--	--	--

[a] Highest maximum beef or dairy cattle dietary burden used for mammalian milk and tissue maximum residue level calculations.

[b] Highest mean beef or dairy cattle dietary burden used for mammalian milk and tissue STMR estimates.

[c] Highest maximum poultry dietary burden used for poultry tissue and egg maximum residue level calculations.

[d] Highest mean poultry dietary burden used for poultry tissue and egg STMR estimates.

Animal commodity maximum residue levels

The highest maximum and mean estimated dietary burdens for cattle are 20.84 and 10.15 ppm, respectively. No animal feeding studies on ruminants are available, therefore the lactating goat metabolism study submitted to the 2008 JMPR was used to estimate residues of mandipropamid in mammalian commodities.

Lactating goats were fed for seven days with [^{14}C] mandipropamid equivalent to 30 ppm in the diet for the methoxyphenyl label and 27 or 45 ppm in the diet for the chlorophenyl label. The methoxyphenyl label produced higher residues of mandipropamid in milk and all tissues and therefore was used as the

basis for anticipated residue calculations. Anticipated residues of mandipropamid in tissues are presented in Table 3.

Table 3 Anticipated Residues in Ruminant Milk and Tissues

	Feed level (ppm)	Estimated Residue (mg/kg)				
		Milk	Muscle	Liver	Kidney	Fat
Metabolism study	30	0.0008	ND	0.0065	ND	0.019
Maximum dietary burden and estimated residues	20.84	< 0.001[a]	NA	0.0045	NA	0.013
Mean dietary burden and estimated residue	10.15	< 0.001[a]	NA	0.0022	NA	0.0064

ND = Not detected. NA = Not applicable.

[a] The STMR anticipated residue of < 0.001 mg/kg in milk is considered negligible and a value of 0 was used in the dietary assessment.

Applying the maximum cattle dietary burden, the Meeting estimated a maximum residue level of 0.02 mg/kg for mandipropamid in mammalian fat (excluding milk fats) to replace its previous recommendation of 0.01(*) mg/kg. The Meeting confirmed its previous maximum residue level estimations of 0.01(*) mg/kg for all other mammalian commodities.

Applying the mean cattle dietary burden, the Meeting concluded that the previous STMR of 0 mg/kg for mammalian meat and milk are appropriate. The Meeting estimated new STMRs of 0.0064 mg/kg for mammalian fat (excluding milk fats) and 0.0022 mg/kg for mammalian edible offal.

The maximum and mean estimated dietary burdens for poultry are unchanged from those estimated by the 2018 JMPR. Therefore, the Meeting confirmed its previous recommendations for poultry commodities.

RECOMMENDATIONS

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

Definition of the residue for compliance with the MRL and dietary risk assessment for plant and animal commodities: *mandipropamid.*

The residue is not fat-soluble.

Table 4 Recommendations for residues of mandipropamid from the 2021 Extra JMPR

CCN	Crop/Commodity	MRL, mg/kg		STMR or STMR-P (mg/kg)
		New	Previous	
OR 0001	Citrus oil, edible	30		4.5
AB 0001	Citrus pulp, dry	1.5		Median: 0.29
MO 0105	Edible offal (mammalian)	0.01(*)	0.01(*)	0.0022
FC 0002	Lemons and Limes, Subgroup of	0.5		0.01
MF 0100	Mammalian fats (except milk fats)	0.02	0.01(*)	0.0064
FC 0003	Mandarins, Subgroup of	0.5		0.01

CCN	Crop/Commodity	MRL, mg/kg		STMR or STMR-P (mg/kg)
		New	Previous	
FC 0004	Oranges, Sweet, Sour (including Orange-like hybrids), Subgroup of	0.4		0.01
FC 0005	Pummelo and Grapefruits (including Shaddock-like hybrids, among other Grapefruit), Subgroup of	0.2		0.01
For dietary risk assessment and/or dietary burden calculations				
	Citrus flesh (excluding kumquat commodities)	--	--	0.01
FC 0303	Kumquat, raw (including juice)	--	--	0.098
--	Lemon/lime/mandarin juice	--	--	0.0042
JF 0004	Orange juice	--	--	0.0043
JF 0203	Grapefruit juice	--	--	0.0024

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for mandipropamid is 0–0.2 mg/kg bw. The International Estimated Daily Intakes (IEDIs) for mandipropamid were estimated for the 17 GEMS/Food Consumption Cluster Diets using the STMR or STMR-P values estimated by the JMPR. The results are shown in Annex 3 of the 2021 Extra JMPR Report.

The IEDIs ranged from 0–6% of the maximum ADI. The Meeting concluded that long-term dietary exposure to residues of mandipropamid from uses considered by the JMPR is unlikely to present a public health concern.

Acute dietary exposure

The 2008 JMPR decided that an ARfD for mandipropamid was unnecessary. Therefore, the Meeting concluded that the acute dietary exposure to residues of mandipropamid from the uses considered is unlikely to present a public health concern.

REFERENCES

Code	Author	Year	Title
IR-4 PR No. 11138	Kathryn Homa	2017	Mandipropamid: magnitude of the residue on orange
IR-4 PR No. 11139	Kathryn Homa	2017	Mandipropamid: magnitude of the residue on lemon
IR-4 PR No. 11140	Kathryn Homa	2017	Mandipropamid: magnitude of the residue on grapefruit

METHOPRENE (147)

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

EXPLANATION

Methoprene is an insect growth regulator that was first evaluated by the JMPR in 1984, it received a periodic review in 2001 for toxicology and in 2005 for residues and was evaluated for additional uses in 2016 and 2019 (extra Meeting).

S-methoprene is the biologically active enantiomer in the racemic methoprene and constitutes 50% of methoprene. An ADI was established at 0–0.09 mg/kg bw for racemic methoprene and at 0–0.05 mg/kg bw for S-methoprene; an ARfD was considered unnecessary.

The residue definition for compliance with the MRL and dietary risk assessment for plant and animal commodities is *methoprene*. *The residue is fat-soluble*.

Methoprene was scheduled at the Fifty-first Session of the CCPR (2019) for evaluation by the 2020 JMPR, but was postponed to the 2021 Extra JMPR Meeting. The Meeting received post-harvest supervised residue trials and GAP information on soya beans

USE PATTERNS

Registered uses of S-methoprene as an insect growth regulator (IGR) are shown in Table 1.

Table 1 Registered uses of S-methoprene in stored soya beans and other commodities in the USA

Country	IGR formulation concentration	Rate	Number of applications	WHP [a]
USA	288 g/L	2.4 g ai/tonne	ns	ns

[a] WHP - withholding period

ns - not stated

RESULTS OF SUPERVISED RESIDUE TRIALS ON CROPS

Soya beans

A total of five post-harvest trials were conducted in 2019 in the USA with a single treatment of S-methoprene. In a cement mixer, soya beans were sprayed at 2.4 g ai/tonne and the seeds were mixed. Within 24 hours of treatment, soya beans were then frozen and samples analysed within a month by HPLC-DAD (Met. CAP 427.05), with concurrent recovery ranging from 70 to 120% (LOQ = 0.02 mg/kg). The results are shown in Table 2.

Table 2 Results of residue trials conducted with S-methoprene using 1 application in storage at 2.4 mg ai/kg of EC formulation (Study 5862).

Location	Variety	Residues (determined as methoprene), mg/kg
Arkansas	-	2.34, 2.78 (2.56)
Mississippi	Pioneer	2.06, 2.08 (2.07)

Location	Variety	Residues (determined as methoprene), mg/kg
North Dakota	-	2.46, 2.34 (2.40)
Illinois	AG 36x6	2.49, 2.34 (2.41)
North Dakota	-	2.41, 2.52 (2.46)

APPRAISAL

Methoprene is an insect growth regulator that was first evaluated by the JMPR in 1984, it received a periodic review in 2001 for toxicology and in 2005 for residues and was evaluated for additional uses in 2016 and 2019 (extra Meeting).

S-methoprene is the biologically active enantiomer in the racemic methoprene and constitutes 50% of methoprene.

An ADI was established at 0–0.09 mg/kg bw for racemic methoprene and at 0–0.05 mg/kg bw for S-methoprene; an ARfD was considered unnecessary.

The residue definition for compliance with the MRL and dietary risk assessment for plant and animal commodities is Methoprene. The residue is fat-soluble.

S-Methoprene was scheduled at the Fifty-first Session of the CCPR (2019) for evaluation by the 2020 JMPR, which was postponed to the 2021 Extra JMPR.

The Meeting received post-harvest supervised residue trials on soya beans for S-methoprene and GAP information.

Results of supervised residue trials on crops

Soya bean (dry) - post harvest use

The critical GAP for S-methoprene in the USA for protection of stored food, grain, animal feed and seeds used for oil is 2.4 g ai/tonne with no withholding period specified. Five trials were conducted in the USA with a single application at 2.4 g ai/tonne and samples collected after treatment gave methoprene residues in rank order (n = 5): 2.1, 2.4, 2.4, 2.5 and 2.6 mg/kg.

As in the trials, where S-methoprene was applied separately to different lots simulating

commercial application practice, the results reflected a high recovery of applied methoprene (88 to 108% of the 2.4 g ai/tonne applied in all the trials), the Meeting decided that the application rate determined the level of residue expected at the zero-day withholding period of the GAP and that residues of up to about 2.4 mg/kg can be anticipated.

The Meeting estimated a maximum residue level of 3 (Po) mg/kg and an STMR of 2.4 mg/kg for soya bean (dry).

Residues in animal commodities

Soya bean seed can be fed to livestock. The 2019 Extra JMPR evaluated residues of methoprene in cereal grains, oilseeds and peanuts. Estimation by the present Meeting, now including soya bean (dry), does not increase the previously estimated (2019) maximum dietary burdens of 13.46 ppm in the diet of cattle and 10.62 ppm for poultry. The Meeting confirmed its previous conclusions for animal commodities.

RECOMMENDATIONS

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

Definition of the residue for compliance with the MRL and dietary risk assessment for plant commodities: methoprene

Definition of the residue for compliance with the MRL and dietary risk assessment for animal commodities: methoprene

The residue is fat-soluble.

Table 1 Recommendations for residues of S-methoprene from the 2021 Extra JMPR

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)	3 Po	-	2.4	-

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for S-methoprene is 0–0.05 mg/kg bw. The International Estimated Daily Intakes (IEDIs) for methoprene were estimated for the 17 GEMS/Food Consumption Cluster Diets using the STMR or STMR-P values estimated by the JMPR. The results are shown in Annex 3 of the 2021 Extra JMPR Report.

Assuming the residues are S-methoprene, the IEDIs ranged from 10-60% of the maximum ADI. The Meeting concluded that long-term dietary exposure to residues of methoprene from uses considered by the JMPR is unlikely to present a public health concern.

Acute dietary exposure

The 2001 JMPR decided that an ARfD for methoprene was unnecessary. The Meeting therefore concluded that the acute dietary exposure to residues of methoprene from the uses considered is unlikely to present a public health concern.

REFERENCE

Reference Number	Author(s)	Year	Study Title
	Witte, J., and Kindel, J.	2019	(S)-Methoprene: Residues on Soybeans. Wellmark International or Central Life Sciences. Study No. 5862.

METHOXYFENOZIDE (209)

First draft prepared by Dr M Lee, Andong National University, Republic of Korea

EXPLANATION

Methoxyfenozide belongs to the diacylhydrazine class of insecticides. It mimics the action of the moulting hormone of Lepidopterous (moths, butterflies) larvae. The JMPR evaluated it first in 2003 for toxicology and residues and in 2006, 2009 and 2012 for additional uses. The 2003 JMPR established an ADI of 0–0.1 mg/kg bw and an ARfD of 0.9 mg/kg bw.

The residue definition for compliance with the MRL and dietary risk assessment for plant and animal commodities is *methoxyfenozide*. The residue is fat-soluble in its distribution between meat muscle and fat, but not in its distribution in milk.

The Forty-third Codex Alimentarius Commission (2020) approved the new work proposals including the priority list of pesticides for evaluation by the current Meeting. The priority list included methoxyfenozide for evaluation of additional uses. The Meeting received information on residue trials on basil, coffee bean, rice, sugar cane and tea.

RESIDUE ANALYSIS

Analytical methods

Analytical methods for methoxyfenozide residues in basil (fresh and dry) and rice (grain, hulls, bran and polished rice) involved an extraction with methanol:0.1 N hydrochloric acid solution (90:10, v/v), clean-up by SPE cartridge or partition with dichloromethane (basil) and determination by LC-MS/MS (monitoring ion, 369.2>313.0 in basil or 369.2/149.1 in rice). The methods used were based on the method 02.25, considered as validated by the 2012 JMPR. The LOQ values were 0.01 mg/kg in fresh basil and 0.05 mg/kg in dried basil and rice matrices.

Method recovery results are shown in Table 1. Mean recoveries of methoxyfenozide in fresh and dried basil ranged from 78–91% (RSDs, ≤12 %) and 90–100% (RSDs, ≤10%) at fortification levels of 0.01–75 mg/kg and 0.05–300 mg/kg, respectively (5 levels; n = 1–6). In rice matrices fortified with methoxyfenozide at levels of 0.05–50 mg/kg (5 levels; n = 3–6) in grain, 0.05–25 mg/kg (4 levels; n = 4–6) in straw, 0.05–50 mg/kg (4 levels; n = 3–6) in bran, 0.05–200 mg/kg (4 levels; n = 3–6) in hulls and 0.05–5 mg/kg (3 levels; n = 3–6) in polished rice, mean recoveries were 98–104 (RSDs, ≤15%), 93–104% (RSDs, ≤10%), 94–103% (RSDs, ≤5%), 103–107 (RSDs, ≤2%) and 89–105% (RSDs, ≤3%), respectively.

For coffee bean (green bean, roasted bean and soluble coffee) and sugar cane (stalk, brown sugar and molasses), analytical methods for methoxyfenozide residues involved an extraction with methanol:water (90:10, v/v), a clean-up by using SPE cartridge or omission of the clean-up step (coffee) and determination by LC-MS/MS (monitoring ion, m/z 369.2/149.1). The methods used were based on the method 02.24, considered as validated by the 2012 JMPR. The LOQ values in coffee and sugar cane matrices were 0.01 mg/kg.

Mean recoveries of methoxyfenozide in coffee beans (green) ranged from 83–110% (RSDs, ≤11%) at fortification levels of 0.01–0.5/1.0 mg/kg (2 levels; n = 6 or 8). In roasted bean and soluble coffee, mean recoveries were 89–95% (RSDs, ≤10%) and 87–92% (RSDs, ≤8%), respectively, at fortification levels of 0.01–0.5 mg/kg (2 levels; n = 6). For sugar cane, mean recoveries were 83–93%.

Table 1 Recovery test results for methoxyfenozide

Matrix	Fortification level, mg/kg	Individual values, %	Mean value, %	RSD, %	LOQ, mg/kg
Basil, fresh	0.01	69, 71, 74, 75, 84, 93	78	12	0.01
	0.3	87, 87, 88, 89, 93	89	3	
	1	86			
	10	86, 86, 99	90	8	
	75	90, 91, 93	91	2	
Basil, dried	0.05	81, 101, 104, 105, 105, 106	100	10	0.05
	2	87, 88, 91, 92, 84	90	3	
	1	88			
	50	94, 95, 96	95	1	
	300	85, 94, 96	92	6	
Coffee beans (2016)	0.01	76, 78, 80, 82, 83, 83, 89, 91	83	6	0.01
	1.0	95, 96, 97, 97, 99, 100	97	2	
Coffee beans (2017)	0.01	87, 110, 112, 115, 117, 118	110	11	0.01
	0.5	96, 96, 97,99, 100, 102	98	3	
Coffee beans, roasted (2017)	0.01	80, 80, 83, 92, 99, 100	89	10	0.01
	0.5	92, 92, 94, 95, 97, 102	95	4	
Coffee, soluble (2017)	0.01	73, 86, 88, 88, 92, 93	87	8	0.01
	0.5	89, 91, 91, 91, 93, 95	92	2	
Rice, grain	0.05	98, 99, 100, 101, 104, 105	101	3	0.05
	0.5	98, 98, 100, 102	100	2	
	5	101, 102, 103, 103, 107, 109	104	3	
	20	80, 108, 111, 111	103	15	
	50	98, 98, 99	98	1	
Rice, straw	0.05	73, 94, 96, 97, 98, 98,	93	10	0.05
	0.5	99, 100, 101, 105	101	3	
	5	102, 102, 106, 107	104	3	
	25	94, 95, 99, 99, 100	97	3	
Rice, bran	0.05	90, 93, 93, 95, 97, 99	95	3	0.05
	0.5	97, 98, 102, 102, 103	100	3	
	5	97, 104, 107	103	5	
	50	91, 95, 97	94	3	
Rice, hulls	0.05	98, 103, 103, 103, 103, 106	103	3	0.05
	0.5	100, 101, 104, 105, 105	103	2	
	5	106, 107, 108	107	1	
	200	101, 104, 105	103	2	
Rice, polished	0.05	85, 87, 88, 90, 91, 92	89	3	0.05
	0.5	100, 101, 101, 101, 106	102	2	
	5	104, 105, 107	105	2	

Matrix	Fortification level, mg/kg	Individual values, %	Mean value, %	RSD, %	LOQ, mg/kg
Sugar cane (2016)	0.01	72, 80, 81, 87, 89, 94	84	9	0.01
	1.0	90, 91, 92, 93, 95, 97	93	3	
Sugar cane, stalks (2017)	0.01	71, 83, 84, 85, 87, 88	83	7	0.01
	0.5	85, 85, 87, 87, 88, 93	88	4	
Sugar cane, molasses (2017)	0.01	82, 85, 86, 87, 89, 93	87	4	0.01
	0.5	87, 88, 89, 91, 92, 93	90	3	
Sugar cane, brown sugar (2017)	0.01	84, 86, 88, 91, 91, 98	90	5	0.01
	0.5	88, 90, 91, 94, 92, 95	92	3	
Tea, dried (1998)	0.8	92, 94, 94, 94	94	1	not rep.
	8	82, 83, 86, 93	86	6	
Tea, infusion (1998)	1.0	96, 98, 101, 104	100	4	not rep.
Tea, dried leaves (2016)	0.01	91, 93, 93, 96, 96	94	2	0.01
	0.1	96, 98, 98, 99, 99	98	1	
	40	87, 88, 89, 89, 90	89	1	
Tea, infusion (2016)	0.01	105, 105, 106, 107, 109	106	2	0.01
	0.1	106, 107, 107, 109, 110	108	2	
	25	92, 93, 95, 97, 98	95	3	
Tea, dried leaves (2017)	0.01	83, 86, 87, 88, 88, 96	88	5	0.01
	0.1	85, 86, 87, 89, 93, 93	89	4	
	40	82, 83, 84, 88, 91, 93	87	5	
Tea, infusion (2017)	0.01	95, 95, 96, 97, 98, 101	97	2	0.01
	0.1	94, 96, 96, 97, 98, 99	97	2	
	25	98, 99, 103, 103, 104, 104	102	3	
Tea, dried leaves (2018)	0.01	72, 77, 81, 90, 91, 96	85	11	0.01
	0.1	78, 80, 81, 84, 84, 85	82	3	
	40	90, 90, 90, 91, 92, 96	92	3	
Tea, infusion (2018)	0.01	106, 110, 112, 113, 114, 118	112	4	0.01
	0.1	94, 94, 95, 96, 97, 98	96	2	
	10	96, 96, 98, 99, 99, 101	98	2	

In coffee and sugar cane, all are concurrent recoveries.

In basil and rice, the underlined values are concurrent recoveries.

(RSDs, ≤9%) in stalks, 87–90% (RSDs, ≤4%) in molasses and 90–92% (RSDs, ≤5%) in brown sugar, at fortification levels of 0.01–0.5/1.0 mg/kg (2 levels; n = 6).

In tea trials conducted in 1998, dried leaves (5 g) were extracted with acetone and the extract was cleaned up by column chromatography methods successively made using C_{18} silica, anion exchange, active charcoal, silica gel, C_{18} silica and florisil. For determination, HPLC-UV was used. Tea infusion sample (equivalent to 5 g dried leaves) was extracted with ethyl acetate/n-hexane, subjected to coagulation treatment and again extracted with ethyl acetate/n-hexane. The extract was cleaned up by using a florisil

column and HPLC-UV was used for determination. The limit of detection was reported as 0.02 mg/kg in both tea matrices (LOQ values, not reported). Dried leaves were fortified with methoxyfenozide at levels of 0.8 mg/kg (n = 4) and 8 mg/kg (n = 4), and for tea infusion, at a level of 1 mg/kg (n = 4). Mean recovery values in dried leaves were 86–94% (RSDs, ≤6%) and in tea infusion, 100% (RSDs, ≤4%).

In tea trials conducted in 2016–2018, water was added to dried tea leaves (pulverized sample of 5 g) and left to stand for 15 min prior to extraction with acetonitrile. The extract was cleaned up by using graphite carbon/ethylene diamine-N-propyl silyl silica gel layered column. Tea infusion sample was cleaned up (omitting the extraction procedure) by using octadecyl silyl silica gel column and successively graphite carbon/ethylene diamine-N-propyl silyl silica gel layered column. LC-MS/MS (m/z 369.2>148.8) was used and the LOQ values were 0.01 mg/kg in both matrices. Mean recoveries in dried leaves ranged from 82–94% (RSDs, ≤11%) at fortification levels of 0.01–40 mg/kg (3 levels; n = 5–6) and in tea infusion, 95–112% (RSDs, ≤4%) at 0.01–10/25 mg/kg, (3 levels; n = 5–6).

Stability of residues in stored analytical samples

The stability of methoxyfenozide residues on frozen storage was studied in basil, rice and tea leaves with results shown in Table 2.

In basil samples stored frozen at below -20 ℃, methoxyfenozide residues were stable for the tested periods of 760 days in fresh basil and 758 days in dried basil (more than 90% of actual storage time 806 days in fresh basil and 805 days in dried basil).

Methoxyfenozide residues in rice samples stored frozen at below -20 ℃ were stable for the test periods of 247 days in hulls and bran and 253 days in polished rice, covering the actual storage days (104 days).

Table 2 Storage stability results of methoxyfenozide in basil, rice and tea

Matrix	Fortification, mg/kg	Recovery, %	Concurrent recovery, %	Storage test days	Actual max. storage days*
Basil, fresh	1	92, 92, 92	86	760	806
Basil, dried	1	83, 83, 84	88	758	805
Rice, hulls	0.1	109, 109, 111	106, 107, 110	247	104
Rice, bran	0.1	105, 109, 112	104, 105, 107	247	104
Rice, polished	0.1	105, 105, 109	100, 102, 106	253	104
Tea, dried leaves (1998)	2	70, 80, 89, 90	94 at 0.8 mg/kg	190, 201	187, 199
Tea, dried leaves (2016)	0.5	89, 90	94 at 0.1 mg/kg	46	20
Tea, dried leaves (2017)	0.5	80, 81, 84, 86, 86, 87	96 at 0.1 mg/kg	39, 49, 62	38, 41, 50
Tea, dried leaves (2018)	0.5	101, 104, 114, 114	85 at 0.1 mg/kg	34, 58	21, 45

* In tea, including storage days of dried leaves used for infusion

In dried tea leaves stored frozen at below -20 ℃, methoxyfenozide residues were stable for the tested periods covering the actual storage days.

USE PATTERN

The Meeting received the GAP information on basil, coffee, rice, sugar cane and tea. The information is summarized in Table 3.

Table 3 Registered use of methoxyfenozide

Crop	Country	Form.	Application					PHI, days
			Method	Volume, L/ha	Rate. kg ai/ha	No.	Int. days	
Basil (fresh and dried) [a]	US	SC 240 g ai/L	Foliar spray A, G	A, min. 90 G, min. 90-180	0.067-0.28 (1.1 kg ai/ha/year)	(4)	10	1
Coffee [b]	Brazil	SC 300 g ai/L	Foliar spray A, G	A, 20-40 G, 400	0.06-0.09	2	30	7
Rice [c]	US	SC 240 g ai/L	Foliar spray A, G		0.13-0.18 (0.36 kg ai/ha/year)	2	10	14
Sugar cane [b]	Brazil	SC 300 g ai/L	Foliar spray A, G	A, 20-40 G, 200	0.03-0.06	2	*	60
Tea	Japan	SC 200 g ai/L	Foliar spray	2,000-4,000, dilution rate, 4,000-8,000 times	0.05-0.2 [d]	2	**	7

A, aerial application; G, ground application

[a] Registered in herbs and not registered for use in New York. Herbs includes angelica, annual marjoram, balm, basil, borage, burnet, chamomile, catnip, chervil (dried), chive, coriander (leaf), costmary, cilantro (leaf), curry (leaf), dill weed, horehound, hyssop, lavender, lemongrass, Lovage (leaf), marigold, marjoram, nasturtium, oregano, parsley (dried), pennyroyal, pot marjoram, rosemary, rue, sage, summer savoury, sweet bay, sweet marjoram, tansy, tarragon, thyme, wild marjoram, wintergreen, winter savoury, woodruff, wormwood.

[b] SC formulation, composed of methoxyfenozide (300 g ai/L) and spinetoram (60 g ai/L); for coffee, add adjuvant organosilicone to 0.05% v/v

[c] Registered in specific counties of California (Buttle, Colusa, Glenn, Merced, Placer, Sacramento, San Joaquin, Sutter, Yolo and Yuba), expires on October 4, 2021

[d] The rate of application, calculated with dilution rates of 4,000-8,000 times and spray volume of 2,000-4,000 L/ha

* Determined by reinfestation

** Not specified; 7-day interval in local agricultural practice

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

The Meeting received residue trials on basil, coffee, rice, sugar cane and tea. The detailed information are summarized in Tables 4 to 8.

Crop Group	Commodity	Table No.
Group 020 Cereal grains	Subgroup 020 C Rice cereals GC 0649 Rice	Table 4
Group 021 Grasses for sugar or syrup production	GS 0659 Sugar cane	Table 5
Group 024 Seeds for beverages and sweets	SB 0716 Coffee bean	Table 6
Group 027 Herbs		
Subgroup 027 A Herbs (herbaceous plants)	HH 0722 Basil, leaves DH 0170 Dried herbs DH 0722 Basil, dried	Table 7
Group 066A Teas	DT 1114 Tea, Green, Black (black, fermented and dried)	Table 8

Cereal grains

Rice

Eight field trials on paddy rice (flooded) were conducted in California, USA in 2017 (four trials) and 2018 (four trials) [Samoil, K., 2019; IR-4 PR No. 11979]. The SC formulation (240 g/L) was foliar sprayed with two applications 8–11 days apart at rates of 0.21–0.22 kg ai/ha (0.42–0.44 kg ai/ha/year), using spray volumes of 140–290 L/ha. In the trials of CA80, CA82 and CA46-CA47, an adjuvant (non-ionic surfactant) was used but not in the CA81, CA83, CA45 and CA48. Samples were harvested 13–14 days after the last application (DALA). At one trial (CA80), samples of rice were collected additionally at 1, 7, 21, and 28 DALA for a decline study. For a processing study, the trial CA81included an additional plot treated at rates of 1.1 kg ai/ha (2.2 kg ai/ha/year).

Each field trial consisted of one untreated control plot and one treated plot. At each plot, duplicate samples were collected. All samples were transported to freezers in coolers and then remained frozen (<-20 °C) until analysis. In residue analysis, concurrent recoveries were satisfactory. In all control samples, methoxyfenozide was not detected (<0.05 mg/kg).

Table 4 Residue concentrations of methoxyfenozide from residue trials on rice in the USA

Location, Year Trial ID (Variety)	Application			Portion analysed	DALA	Methoxyfenozide, mg/kg		
	Rate, kg ai/ha	No.	Int. days			Individual value		Mean
GAP: USA	0.13-0.18 (0.36 kg ai/ha/year)	2	10		PHI, 14 days			
Biggs, CA, 2017 CA80 (M206)[a]	0.21-0.22	2	11	Grain	1	5.6	6.0	5.8
					7	6.1	6.6	6.4
					14	7.5	7.3	7.4
					21	8.4	8.2	8.3
					28	4.8	5.4	5.1
Biggs, CA, 2017 CA81 (M206)[a]	0.22	2	11	Grain	13	7.7	8.9	8.3
	1.1*	2	11	Grain	13	35		
				Bran	13	36		
				Hulls	13	159		
				Polished rice	13	0.46		
Davis, CA, 2017 CA82 (M206)[b]	0.21-0.22	2	8	Grain	13	7.7	5.9	6.8
Davis, CA, 2017 CA83 (M206)[b]	0.21-0.22	2	8	Grain	13	8.2	10	9.1
Davis, CA, 2018 CA45 (M206)[c]	0.21	2	9	Grain	14	6.2	10	8.1
Davis, CA, 2018 CA46 (M206)[c]	0.21-0.22	2	9	Grain	14	9.7	14	12
Davis, CA, 2018 CA47 (L207)[c]	0.21	2	9	Grain	14	8.7	9.0	8.9

Davis, CA, 2018 CA48 (L207)[c]	0.21-0.22	2	9	Grain	14	6.9	7.7	7.3

[a] Not independent trials, conducted on the same place and application dates

[b] Not independent trials, conducted on the same place and application dates

[c] Not independent trials, conducted on the same place and application dates

[*] For a processing study

Grasses for sugar or syrup production

Sugar cane

Eight trials were carried out in Brazil in 2016 (four trials) and 2017 (four trials) [Araujo, N. J. De, 2018; Report No. Ri16b-05-02]. The SC formulation (methoxyfenozide 300 g ai/L plus spinetoram 60 g ai/L) was foliar sprayed with two applications 60–126 days apart at rates of 0.060 kg ai/ha, using spray volumes of 197–213 L/ha. Whole sugar cane plants were harvested 60 DALA in trial ID MG2, MG3, SP2 and SP3, and 30, 50, 60 and 70 DALA in trial ID PR1, MG1, SP1 and PR2. After sampling, sugar cane stalks (min. 2 kg) with about 20 cm obtained from all cane segments (higher, median and lower) were separated. Some samples from the SP2 and SP3 trials were used for processing studies (molasses and brown sugar).

Each field trial consisted of one untreated control plot and one treated plot. All samples were kept under frozen conditions (<-20 °C) until delivery to the processing laboratory, the analysis laboratory and analysis. The maximum storage interval for samples was 200 days. In residue analysis, concurrent recoveries were satisfactory. In all control samples, methoxyfenozide was not detected (<0.01 mg/kg).

Table 5 Residue concentrations of methoxyfenozide from residue trials on sugar cane in Brazil

Location, Year Trial ID (Variety)	Application				Portion Analysed	DALA	Methoxyfenozide, mg/kg
	Rate, kg ai/ha	No.	Int. days	BBCH			
GAP: Brazil	0.030-0.060	2	det. by reinfestation			PHI, 60 days	
Paranavaí, 2016 PR1 (RB 867515)	0.060	2	73	45, 48	Stalk	30	0.022
						50	0.017
						60	<0.01
						70	<0.01
Uberlândia, 2016 MG1 (RB 867515)	0.060	2	68	39, 47	Stalk	30	<0.01
						50	<0.01
						60	<0.01
						70	<0.01
Araguari, 2016 MG2 (RB 867515)	0.060	2	68	38, 48	Stalk	60	<0.01
Conchal, 2016 SP1 (SP81-3250)	0.060	2	60	47, 49	Stalk	30	<0.01

Location, Year Trial ID (Variety)	Application				Portion Analysed	DALA	Methoxyfenozide, mg/kg
	Rate, kg ai/ha	No.	Int. days	BBCH			
						50	<0.01
						60	<0.01
						70	<0.01
Paranavaí, 2017 PR2 (RB 7515)	0.060	2	126	39, 48	Stalk	31	<0.01
						49	<0.01
						60	<0.01
						70	<0.01
Indianópolis , 2017 MG3(RD16726518)	0.060	2	120	22, 48	Stalk	60	<0.01
Conchal, 2017 SP2 (SP80-3280)	0.060	2	121	33, 47	Stalk	60	0.01
					Molasses		0.07
					Brown sugar		0.06
Engenheiro Coelho, 2017 SP3 (SP80-3280)	0.060	2	119	33, 49	Stalk	60	<0.01
					Molasses		0.02
					Brown sugar		<0.01

Seeds for beverages and sweets

Coffee bean

Eight trials were carried out in Brazil in 2016 (four trials) and 2017 (four trials) [Araujo, N. J. De, 2018; Report No. Ri16b-05-01]. The SC formulation (methoxyfenozide 300 g ai/L plus spinetoram 60 g ai/L) was foliar sprayed with two applications 30–31 days apart at rates of 0.086–0.097 kg ai/ha, using spray volumes of 194–216 L/ha containing an adjuvant. Sampling of fruits (*ca.* 1 kg after drying and peeling) were manually made at 7, 14, 21 and 28 DALA in trials of SP1, SP2, MG3 and MG4, at 7, 14 DALA in MG1 and MG2 and at 14 DALA in SP3 and SP4. At harvest, the collected samples were cherry coffee (MG2, MG3, MG4, SP1, SP3) or coquinhos coffee (berries dried on the plant) (MG1, SP2, SP4). Samples from trials of SP3 and SP4 were used for processing studies (roasted bean and soluble coffee).

Each field trial consisted of one untreated control plot and one treated plot. Samples were dried at ambient field temperature through exposure to the sun to reach ca. 10% moisture content, typical of the industrial coffee drying process. The dried fruits were peeled using a coffee processor or manual coffee peeler. The peeled samples (green bean) were kept under frozen conditions (<-20 °C) during transportation to the processing laboratory and analysis laboratory and until analysis. The maximum storage interval for samples was 268 days. In residue analysis, concurrent recoveries were satisfactory. In all control samples, methoxyfenozide was not detected (<0.01 mg/kg).

Table 6 Residue concentrations of methoxyfenozide from residue trials on coffee bean (green) in Brazil

Location, Year Trial ID (Variety)	Application				Sample Analysed	DALA	Methoxyfenozide, mg/kg
	Rate, kg ai/ha	No.	Int. days	BBCH			
GAP: Brazil	0.060-0.090	2	30			PHI, 7 days	
Dois Córregos, 2016 SP1 (Coffee Ubatã)	0.091-0.094	2	31	87, 89	Green bean	7	0.035
						14	0.034
						21	0.021
						28	0.012
Itirapuã, 2016 SP2 (Catuaí 62 Vermelho)	0.092-0.095	2	31	89, 89	Green bean	7	0.045
						14	0.028
						21	0.025
						28	0.014
Serra do Salitre, 2016 MG1 (Catuaí 62)	0.091-0.097	2	30	87,89	Green bean	7	0.011
						14	0.061
Jacutinga, 2016 MG2 (Catuaí 62)	0.092-0.093	2	30	81, 85	Green bean	7	<0.01
						14	<0.01
Monte Carmelo, 2017 MG3 (Mundo Novo)	0.086-0.093	2	30	83, 87	Green bean	7	<0.01
						14	0.01
						21	<0.01
						28	<0.01
Indianópolis, 2017 MG4 (Catuaí Vermelho)	0.086-0.095	2	30	85, 88	Green bean	7	0.02
						14	0.01
						21	<0.01
						28	0.01
Dois Córregos, 2017 SP3 (Rubi MG 1192)	0.088-0.093	2	30	79, 83	Green bean	14	<0.01
					Roasted bean		<0.01
					Soluble coffee		<0.01
Espírito Santo do Pinhal, 2017 SP4 (Catuaí Amarelo)	0.090-0.092	2	30	81, 84	Green bean	14	<0.01
					Roasted bean		<0.01
					Soluble coffee		<0.01

Herbs

Basil

Five field trials were conducted in the USA in 2009 [Switek, T., 2012; IR-4 PR No. 07241]. The SC formulation (240 g/L) was foliar sprayed with four applications 9–11 days apart at rates of 0.28 kg ai/ha (1.1 kg ai/ha/year), using spray volumes of 196–402 L/ha containing a non-ionic surfactant. Samples were

collected at 1 DALA. For dried sample, fresh basil were placed in drying oven or forced air dryer at 32–60 ℃ until dry (1–7 days). At one trial (09-NC04), samples of fresh basil were collected additionally at 3, 6, and 12 DALA for a decline study.

Each field trial consisted of one untreated control plot and one treated plot. At each plot, duplicate samples were collected. All samples were transported to freezers in coolers and then remained frozen (≤-20 ˚C) until analysis. In residue analysis, concurrent recoveries were satisfactory. In all control samples, methoxyfenozide was not detected (fresh basil, <0.01 mg/kg; dried basil, <0.05 mg/kg).

Table 7 Residue concentrations of methoxyfenozide from residue trials on basil in the USA

Location, Year Trial ID (Variety)	Application			Sample Analysed*	DALA	Methoxyfenozide, mg/kg		
	Rate, kg ai/ha	No.	Int. days			Individual value		Mean
GAP: USA	0.067-0.28 (1.1 kg ai/ha/year)	(4)	10		PHI, 1 days			
Citra, FL, 2009	0.28	4	10-11	Fresh basil	1	15	15	15
09-FL46 (Genova)				Dried basil	1	86	98	92
Las Cruces, NM, 2009	0.28	4	9-11	Fresh basil	1	20	21	21
09-NM05 (Genovese)				Dried basil	1	95	109	100
Clinton, NC, 2009	0.28	4	9-10	Fresh basil	1	40	47	43
09-NC04 (Genovese)					3	14	15	14.
					6	11	12	11
					12	0.79	0.86	0.82
				Dried basil	1	181	194	190
Arlington, WI, 2009	0.28	4	9	Fresh basil	1	9.3	11	9.9
09-WI02 (Genova)				Dried basil	1	56	65	60
Agassiz, BC, 2009	0.28	4	9-11	Fresh basil	1	18	20	19
09-BC05 (Di Genova)				Dried basil	1	102	116	110

* Leaves and stems

Teas

Tea, Green, Black (black, fermented and dried)

Residue trials on tea (green) were conducted in Japan. In two trials conducted in 1998, SC formulation (200 g/L) was applied at a dilution rate of 4,000 times in spray volumes of 2,000 L/ha (equivalent to 0.10 kg ai/ha) with two foliar applications 7 days apart. Tea samples were collected at 7, 14 and 21 DALA on the same harvest date. In a total of six trials conducted in 2016 (one trial), 2017 (three trials) and 2018 (two trials), the SC formulation was applied at a dilution rate of 4,000 times in spray volumes of 3,330–3,830 L/ha (equivalent to 0.17–0.19 kg ai/ha) with two foliar applications 7–8 days apart. Tea samples were collected at 7, 14 and 21 DALA in 2017 (on the same harvest date) and at 7 DALA in 2016 and 2018.

In all trials (1998, 2016–2018), one untreated control plot was included. Tea samples from 2016–2018 trials were steamed (50 or 60 sec at 100 ℃), dried (80 or 120 or 140 min at 80 ℃) and packed into a Fres-co bag filled with nitrogen gas or zipped plastic bag with silica gel. For the 1998 trials, the drying method was not reported.

Tea infusion was done on the day of residue analysis in all trials.

Samples of dried green tea were maintained under frozen conditions (≤20 °C) until analysis. All residue analysis was done in duplicate. The analytical method used in 1998 and 2016–2018 was different (see analytical section). Concurrent recoveries (2016–2018), fortified at 0.1 mg/kg (n = 1), were satisfactory (85, 97, 101% for dried leaves and 101, 104, 104% in tea infusion). In 1998, concurrent recovery was not reported. In control samples, methoxyfenozide was detected at <0.02 mg/kg (limit of detection) in the 1998 trials and at <0.01 mg/kg (LOQ) in the 2016–2018 trials.

Table 8 Residue concentrations of methoxyfenozide from residue trials on tea (green) in Japan

Location, Year Trial ID (Variety)	Application				Sample Analysed	DALA	Methoxyfenozide, mg/kg*
	Rate, kg ai/ha	No.	Int. days	Growth stage at last treatment			
GAP: Japan	0.050-0.20	2	Not spec.			PHI, 7 days	
Kanagawa, 1998 (Yabukita)	0.10	2	7	not rep.	Dried leaves	7	7.6
				not rep.		14	4.0
				not rep.		21	1.9
					Tea infusion**	7	0.96
						14	0.28
						21	0.29
Kyoto, 1998 (Kyoken)	0.10	2	7	not rep.	Dried leaves	7	14
				not rep.		14	5.0
				not rep.		21	0.56
					Tea infusion	7	2.5
						14	0.78
						21	0.090
Saitama, 2016 JP2016C058A(Fukumidori)	0.17	2	7	3 leaf stage	Dried leaves	7	29
					Tea infusion	7	10/0.17
Kochi, 2017 JP2017C164B (Yabukita)	0.17	2	7	3 leaf stage	Dried leaves	7	28
				2 leaf stage		14	0.82
				1 leaf stage		21	0.82
					Tea infusion	7	7.8/0.13
						14	0.18
						21	0.18
Miyazaki, 2017 JP2017C164 (Yabukita)	0.17	2	6-8	3.5 leaf stage	Dried leaves	7	9.1
				1 leaf stage		14	2.0
				0.5 leaf stage		21	0.14
					Tea infusion	7	3.0/0.05
						14	0.66
						21	0.040
Saitama, 2017	0.17	2	7	3.5 leaf stage	Dried leaves	7	35

Location, Year Trial ID (Variety)	Application				Sample Analysed	DALA	Methoxyfenozide, mg/kg*
	Rate, kg ai/ha	No.	Int. days	Growth stage at last treatment			
JP2017C164A (Hokumei)				3 leaf stage		14	3.9
				2 leaf stage		21	2.0
					Tea infusion	7	10/0.17
						14	1.2
						21	0.63
Kochi, 2018 JP2018C316B (Yabukita)	0.19	2	7	3 leaf stage	Dried leaves	7	18
					Tea infusion		5.4/0.09
Saitama, 2018 JP2018C316A (Okuharuka)	0.17	2	7	4 leaf stage	Dried leaves	7	29
					Tea infusion		8.5/0.14

In the decline trials, samples were collected on the same date.

* Mean of duplicate analysis run

** Residue concentration for tea infusion was expressed on a dry leaf basis. The value following "/" indicates residue concentration expressed on a tea infusion basis.

Animal Feeds

Eight field trials on rice were conducted in California, USA in 2017 (four trials) and 2018 (four trials) [Samoil, K., 2019; IR-4 PR No. 1197]. Rice straw samples were collected along with rice grain. The detailed methods on the residue trials were described in the food commodity section.

Table 9 Residue concentrations of methoxyfenozide from residue trials on rice (feeds) in the USA

Location, Year Trial ID (Variety)	Application			Portion analysed	DALA	Methoxyfenozide, mg/kg		
	Rate, kg ai/ha	No.	Int. days			Individual value		Mean
Biggs, CA, 2017 CA80 (M206)[a]	0.21-0.22	2	11	Straw	1	13	11	12
					7	15	14	15
					14	8.9	12	11
					21	10	10	10
					28	12	11	12
Biggs, CA, 2017 CA81 (M206)[a]	0.22	2	11	Straw	13	13	12	13
	1.1*	2	11	Bran	13	36		
				Hulls	13	159		
				Polished rice	13	0.46		
Davis, CA, 2017 CA82 (M206)[b]	0.21-0.22	2	8	Straw	13	6.7	8.0	7.4
Davis, CA, 2017 CA83 (M206)[b]	0.21-0.22	2	8	Straw	13	8.0	8.2	8.1
Davis, CA, 2018 CA45 (M206)[c]	0.21	2	9	Straw	14	4.0	5.2	4.6
Davis, CA, 2018	0.21-0.22	2	9	Straw	14	6.2	6.4	6.3

Location, Year Trial ID (Variety)	Application			Portion analysed	DALA	Methoxyfenozide, mg/kg		
	Rate, kg ai/ha	No.	Int. days			Individual value		Mean
CA46 (M206)[c]								
Davis, CA, 2018 CA47 (L207)[c]	0.21	2	9	Straw	14	5.4	5.4	5.4
Davis, CA, 2018 CA48 (L207)[c]	0.21-0.22	2	9	Straw	14	4.3	4.1	4.2

[a] Not independent trials, conducted on the same place and application dates

[b] Not independent trials, conducted on the same place and application dates

[c] Not independent trials, conducted on the same place and application dates

[*] For a processing study

FATE OF RESIDUES IN STORAGE AND PROCESSING

The Meeting received processing studies on rice, sugar cane, coffee bean and tea. The details of the residue trials are described in the section of 'residues resulting from supervised trials on crops'.

Rice (hulls, bran and polished rice)

A sample of rice grain was obtained from a residue trial treated at rates of 1.1 kg ai/ha with two applications and harvest 13 DALA. The grains (14 kg, moisture content of 7%) were separated to hulls, bran and polished rice, simulating commercial practices. Rice grains went through processes of dehulling and polishing using a husker machine and a rice mill, respectively.

Sugar cane (molasses and brown sugar)

Sugar cane samples from two trials treated at rates of 0.060 kg ai/ha with two applications and harvest 60 DALA, were processed. Methoxyfenozide residues in stalks of one trial were detected at below the LOQ value, 0.01 mg/kg. The processing factors were derived from the other trial.

Sugar cane was processed simulating commercial practices. Processing steps of brown sugar were as follows: grinding of sugar cane, concentration of cane juice, crystallization, and drying. For molasses, processing steps were as follows: milling of sugar cane, clarification of cane juice, concentration of clarified juice, concentration of cane juice, cooking and crystallization of sugar mass, and centrifugation.

Coffee bean (roasted bean and soluble coffee)

In coffee bean (green; raw agricultural commodity) obtained from two trials treated at rates of 0.088–0.093 kg ai/ha with two applications and harvest 14 DALA, methoxyfenozide residues were detected at below the LOQ value, 0.01 mg/kg. The processing factors could not be derived.

Tea (infusion)

Infusions were made for all samples obtained from all trials treated at rates of 0.1 kg ai/ha (1998) or 0.17–0.19 kg ai/ha (2016–2018) with two applications and collected at harvest 7 DALA or 7, 14, 21 DALA. Infusions made in trials corresponding to the GAP (7 DALA of 2016–2018) were used for the calculation of processing factors.

For tea infusions, in the 2016–2018 trials, dried tea leaves (9 g) were soaked in boiling water of 540 mL, left for 5 min and then filtered through a cotton plug. In the 1998 trials, dried tea (6 g) was soaked in boiling water of 360 mL and left for 5 min (without filtration).

Table 10 Residue concentrations of methoxyfenozide in processed products

Raw commodity	Methoxyfenozide in raw commodity, mg/kg	Product	Methoxyfenozide in product, mg/kg	Pf
Rice grain	35	Rice, hull	159	4.5
		Rice, bran	36	1.0
		Polished rice	0.46	0.013
Sugar cane, stalk	0.01	Molasses	0.07	7
		Brown sugar	0.06	6
Green tea, dried*	29/28/9.1/35/18/29	Tea infusion*	017/0.13/0.05/0.17/0.09/0.14	0.005 (5), 0.006 (median value, 0.005)

* Data from 7 DALA of the 2016-2017 trials

APPRAISAL

Methoxyfenozide is an insecticide that mimics moulting hormone of Lepidopterous larvae. The JMPR evaluated it first in 2003 for toxicology and residues and in 2006, 2009 and 2012 for additional uses. The 2003 JMPR established an ADI of 0–0.1 mg/kg bw and an ARfD of 0.9 mg/kg bw. The residue definition is methoxyfenozide for compliance with the MRL and dietary risk assessment for plant and animal commodities. The residue is fat-soluble in its distribution between meat muscle and fat, but not in its distribution in milk.

The Forty-third Codex Alimentarius Commission (2020) approved the new work proposals including the priority list of pesticides for evaluation by the current Meeting. The priority list included methoxyfenozide for evaluation of additional uses. The Meeting received information on residue trials on basil, coffee bean, rice, sugar cane and tea.

Methods of analysis

Methods used for analysis of methoxyfenozide were based on Method 02.25 in the basil and rice trials and Method 02.24 in the coffee and sugar cane trials. The two methods, using determination by LC-MS/MS, were considered by the 2012 JMPR as validated. In recovery tests for methoxyfenozide residues in basil, coffee, rice, and sugar cane, recovery results were satisfactory. The LOQ values were 0.01 mg/kg in fresh basil, coffee (green bean, roasted bean and soluble coffee) and sugar cane (stalk, brown sugar and molasses) and 0.05 mg/kg in dried basil and rice (grain, hulls, bran and polished rice).

In the tea trials conducted in 2016–2018, analytical methods for tea leaves and infusion involved extraction with acetonitrile (omitting extraction for infusion samples), clean-up steps by column chromatography, and determination by LC-MS/MS. In recovery tests for methoxyfenozide in dried leaves and infusion, recovery results were satisfactory and the LOQ values were 0.01 mg/kg in both matrices. For the 1998 trials, a different analytical method based on HPLC-UV was used and the Meeting estimated an LOQ of 0.8 mg/kg, based on the lowest fortification level with acceptable recoveries.

Stability of residues in stored analytical samples

Storage stability tests, covering the actual storage days of samples (covering >90% in basil samples) were conducted for basil (fresh, dry), rice (hulls, bran, polished rice) and dried tea leaves, demonstrating that methoxyfenozide residues were stable. For coffee bean and sugar cane samples, the maximum storage interval was 268 days and 200 days, respectively. The previous JMPR (2003) considered that residues were stable for at least for 6–12 months in various plant commodities, when stored frozen. In addition, the 2012 JMPR considered that residues were stable for at least 21 months in spring onion and 15 months in dry pea. Based on the available information, the Meeting considered that methoxyfenozide residues were stable for the sample storage intervals used in the residue field trials considered.

Results of supervised residue trials on crops

Cereal grains – rice

The critical GAP on rice in the USA is two applications at 0.18 kg ai/ha with a minimum re-treatment interval of 10 days and a PHI of 14 days. The Meeting received eight trials (three independent trials) conducted in California, USA, at rates of 0.21–0.22 kg ai/ha with two applications 8–11 days apart, and harvest 13 days after the last application. The residues in rice were (n = 3): 8.3, 9.1 and 12 mg/kg.

The Meeting considered the number of trials insufficient for estimating a maximum residue level for rice.

Grasses for sugar or syrup – sugar cane

The critical GAP on sugar cane in Brazil is two applications at 0.06 kg ai/ha and a PHI of 60 days (the re-treatment interval is specified as, determined by re-infestation). Eight trials were conducted in Brazil, matching the critical GAP. The residues were (n = 8): < 0.01 (7) and 0.01 mg/kg.

The Meeting estimated a maximum residue level of 0.015 mg/kg, an STMR of 0.01 mg/kg and an HR of 0.01 mg/kg for methoxyfenozide in sugar cane.

Seeds for beverages and sweets – Coffee bean

The critical GAP on coffee in Brazil is two applications at 0.09 kg ai/ha with a minimum re-treatment interval of 30 days and a PHI of 7 days. Six trials were conducted in Brazil, matching the critical GAP. The residues in coffee bean (green, dry) were (n = 6): < 0.01, 0.01, 0.02, 0.035, 0.045 and 0.061 mg/kg.

The Meeting estimated a maximum residue level of 0.15 mg/kg and a STMR of 0.0275 mg/kg for methoxyfenozide in coffee bean.

Herbs – basil

The critical GAP on herbs in the USA is an application rate of 0.28 kg ai/ha (1.1 kg ai/ha/year) with a minimum re-treatment interval of 10 days and a PHI of 1 day. Five trials on basil were conducted in the USA, matching the critical GAP. The residues in fresh basil were (n = 5): 9.9, 15, 19, 21 and 43 mg/kg (highest analytical value 47 mg/kg). The residues in dried basil were (n = 5): 60, 92, 100, 110 and 190 mg/kg (highest analytical value 194 mg/kg).

The Meeting estimated a maximum residue level of 80 mg/kg, a STMR of 19 mg/kg and an HR of 47 mg/kg for methoxyfenozide in basil, leaves. In addition, the Meeting estimated a maximum residue level of 400 mg/kg, a STMR of 100 mg/kg and an HR of 194 mg/kg for methoxyfenozide in basil, dry.

Teas – green tea

The critical GAP on tea in Japan is two applications with a dilution rate of 4,000 times (equivalent to 5 g/hL) and a PHI of 7 days. Six trials (2016–2018) on tea were conducted in Japan, matching the critical GAP. The residues in dried tea leaves were (n = 6): 9.1, 18, 28, 29, 29 and 35 mg/kg.

The Meeting estimated a maximum residue level of 80 mg/kg and a STMR of 28.5 mg/kg for methoxyfenozide in tea, green, black (black, fermented and dried).

Residues in animal feeds

A maximum residue level on rice as a food commodity could not be estimated, therefore, the Meeting did not estimate a maximum residue level on rice straw.

Fate of residues during processing

The Meeting received information on the fate of methoxyfenozide during processing of coffee bean, rice, sugar cane and tea. Processing factors for rice, sugar cane and green tea were estimated, and are shown in the table below. For coffee bean, the residue concentration in the raw agricultural commodity was below the limit of quantification, therefore, the processing factors could not be estimated.

Table 1 Processing factors for rice, sugar cane and green tea

Raw agricultural commodity [STMR] (mg/kg)	Processed commodity	Individual processing factors	Mean or best estimate processing factor	STMR-P = $STMR_{RAC}$ ×Pf (mg/kg)
Rice grain	Rice, hull	4.5	4.5	
	Rice, bran	1.0	1.0	
	Polished rice	0.013	0.013	
Sugar cane, stalk [0.01]	Molasses	7	7	0.07
	Brown sugar	6	6	0.06
Dried tea leaves [28.5]	Tea infusion	0.005 (5), 0.006	0.005	0.14

For rice, the maximum residue level on the raw agricultural commodity was not estimated in this Meeting, therefore, the STMR-Ps were not estimated.

A maximum residue level for sugar cane molasses was estimated as 0.1 mg/kg based on the maximum residue level for sugar cane and the estimated processing factor (0.015 mg/kg×Pf, 7).

Animal commodity maximum residue levels

The Meeting noted that of the commodities considered at the current Meeting, only sugar cane molasses is a relevant animal feed item. However, as the inclusion of sugar cane molasses does not significantly increase the animal dietary burden, the Meeting confirmed its previous recommendations for animal commodities.

RECOMMENDATIONS

On the basis of the data obtained from supervised trials, the Meeting concluded that residue levels listed below in Table 2 are suitable for establishing maximum residue limits and for IEDI and IESTI assessments.

Definition of the residue for compliance with the MRL for plant and animal commodities: *methoxyfenozide*

Definition of the residue for dietary risk assessment for plant and animal commodities: *methoxyfenozide*

The residue is fat-soluble in its distribution between meat muscle and fat, but not in its distribution in milk.

Table 2 Recommendations for residues of methoxyfenozide from the 2021 Extra JMPR

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
DH 0722	Basil, dry	400		100	194
HH 0722	Basil, leaves	80		19	47
SB 0716	Coffee bean	0.15		0.0275	
GS 0659	Sugar cane	0.015		0.01	0.01
DM 0659	Sugar cane molasses	0.1		0.07	
DT 1114	Tea, Green, Black (black, fermented and dried)	80		28.5	
	Brown sugar			0.06	
	Tea infusion			0.14	

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for methoxyfenozide is 0–0.1 mg/kg bw. The International Estimated Daily Intakes (IEDIs) for methoxyfenozide were estimated for the 17 GEMS/Food Consumption Cluster Diets using the STMR or STMR-P values estimated by the JMPR. The results are shown in Annex 3 of the 2021 Extra JMPR Report.

The IEDIs ranged from 1–7% of the maximum ADI. The Meeting concluded that long-term dietary exposure to residues of methoxyfenozide from uses considered by the JMPR is unlikely to present a public health concern.

Acute dietary exposure

The ARfD for methoxyfenozide is 0.9 mg/kg bw. The International Estimate of Short Term Intakes (IESTIs) for methoxyfenozide were calculated for the food commodities for which HRs/HR-Ps or STMRs/STMR-Ps were estimated by the present Meeting and for which consumption data were available. The results are shown in Annex 4 of the 2021 Extra JMPR Report.

The IESTIs varied from 0–4% of the ARfD for children and 0–5% of the ARfD for the general population. The Meeting concluded that acute dietary exposure to residues of methoxyfenozide from uses considered by the present Meeting is unlikely to present a public health concern.

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PROTHIOCONAZOLE (232)

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EXPLANATION

Prothioconazole is a broad-spectrum systemic fungicide. It was first evaluated by the JMPR in 2008 (T, R). The latest residue review was done in 2017 (R). It was scheduled at the Fifty-first Session of the CCPR for the evaluation of additional uses by the 2020 JMPR, which was postponed to the 2021 Extra JMPR.

The toxicological review established an ADI of 0–0.05 mg/kg bw and an ARfD of 0.8 mg/kg bw for women of child-bearing age for the parent prothioconazole; an ADI of 0–0.01 mg/kg bw and an ARfD of 0.01 mg/kg bw for women of child-bearing age; and 1 mg/kg bw for general population for prothioconazole-desthio. The residue definition for plant commodities for enforcement and dietary risk assessment is *prothioconazole-desthio*. The residue definition for animal commodities for enforcement is *prothioconazole-desthio* and for dietary risk assessment the *sum of prothioconazole-desthio, prothioconazole-desthio-3-hydroxy, prothioconazole-desthio-4-hydroxy and their conjugates, expressed as prothioconazole-desthio*. The residue covered by the residue definition for compliance is considered not fat-soluble.

For the current Meeting, data were provided on residues in oilseed rape, linseed (flax) and sunflower together with processing studies of rape and sunflower seed as well as currently registered use patterns.

The JMPR has previously noted triazole derived metabolites (1,2,6-triazole, triazole acetic acid, triazole-alanine and triazole lactic acid) can arise from other sources and should be assessed separately. The triazole metabolites while reported were not further considered in the current evaluation.

RESIDUE ANALYSIS

Analytical methods

A number of validated analytical methods for the determination of residues in plant, animal tissue, milk and soils were evaluated by the 2008, 2009 and 2014 JMPR Meetings with additional information made available to the current Meeting on performance relevant to the oilseed commodities under consideration.

Method No. 01013 (Brumhard and Stuke 2008 MR-06/138)

The analytical method 01013 allows determination of residues of prothioconazole-desthio in/on plant materials. Prothioconazole-desthio is extracted from the samples with a mixture of acetonitrile/water (4/1; v/v, containing cysteine hydrochloride) using a blender. After filtration of the extract, stable isotopically labelled analytes are added. The solution is made up to volume, diluted and subjected to reversed phase HPLC-MS/MS without a further clean-up step. Prothioconazole-desthio is detected using electrospray ionization in the positive ion mode (quantification m/z 312 → m/z 70, confirmation m/z 312 → m/z 125). The LOQ was 0.01 mg/kg for all sample materials.

Table 1 provides a summary of the procedural recovery data reported in the oilseed trials considered by the Meeting.

Table 1 Summary of procedural recovery data for method 01013 for prothioconazole-desthio in oilseed commodities (rape 08-2112, 08-2113, 08-2116, 08-3112, 09-2054, 10-2134, 10-2244, 11-2003, 11-2013, 11-

2124, 11-2137, 12-3402, 16-2069, 2616-07, 2618-06, 2619-06; flax 09-2130, 10-2073, 10-2254; sunflower 10-2238, 10-2247, 11-2002, 12-2009, 12-2156)

Matrix	Fortification (mg/kg)	N	Range recoveries (%)	Mean recovery (%)	%RSD
Rape seed	0.01	34	69-118	93.7	12.1
	0.1	22	81-119	98.8	9.0
	0.2	2	96-103		
	0.5	4	73-85	79.3	7.6
	1	8	86-106	96.1	7.8
	1.5	2	91-99		
	5	3	84-88	86	3.3
Rape green material	0.01	32	74-116	99.1	10.7
	0.1	15	81-108	99.8	6.7
	0.3	2	102-108		
	0.5	4	87-92	90	2.9
	1	10	88-104	97.6	5.9
	1.5	2	95-100		
	2	5	70-110	95.3	18.3
	2.5	2	86-102		
	3	7	92-97	94.5	2.0
	3.5	2	104-115		
	4	3	82-91	87.3	5.4
	5	3	84-87	85.5	2.5
Rape pods	0.01	29	74-117	94,1	10.3
	0.1	8	75-107	93.6	13.0
	0.5	4	83-91	87.0	4.6
	1	9	88-103	94.4	6.3
	2	1	107		
	5	4	89-100	92.7	6.9
Rape rest of plant	0.01	15	84-103	95.3	6.9
	0.2	1	106		
	0.5	4	78-90	82.3	8.1
	1	7	94-103	99.8	3.4
	5	4	89-100	93.3	6.3
Oil	0.01	10	83-115	93.8	12.8
	0.1	8	75-96	84.1	9.5
	1	2	89-89		
Pomace	0.01	4	108-109	108.3	0.5
	0.1	6	85-115	98	13.2
	1	2	93-93		
Meal	0.01	4	100-113	108.7	6.9
	0.1	4	98-101	99	1.7
	1	2	88-88		
Flax seed	0.01	10	80-108	92.4	9.2
	0.1	5	95-107	98.2	5.1

Matrix	Fortification (mg/kg)	N	Range recoveries (%)	Mean recovery (%)	%RSD
	1	5	89-94	91.4	2.3
	5	1	92		
Flax green material	0.01	8	96-118	107	7.3
	0.1	4	86-103	97	7.8
	1	5	90-100	95.6	4.2
	2	3	82-99	91.7	9.5
	2.5	2	94-95	94.5	0.7
	5	1	89		
Sunflower seed	0.01	16	88-111	100.3	6.1
	0.1	16	91-108	101.1	4.5
	1	6	89-94	91	2.6
Sunflower partly shelled seed	0.01	4	88-93	90.3	2.5
	0.1	4	94-99	96.3	2.3
Sunflower kernels	0.01	4	78-91	85.5	6.4
	0.1	4	92-95	93.3	1.3

Method No.: 00647 (Heinemann 2001 MR-458/00)

Method 00647 was used in a number of trials to analyse rape commodities. Prothioconazole-desthio is extracted from homogenized samples of rape green material, seed and straw with an acetonitrile/water mixture by high-speed blending. After filtration, the extract is diluted for measurement by HPLC-MS/MS (reversed-phase HPLC on a silica-based C18-column using a gradient acetonitrile/water eluent containing acetic acid). A triple-stage mass spectrometer with an electrospray interface (ESI: TurboIonSpray) operated in the positive ion mode for prothioconazole-desthio under multiple-reaction monitoring conditions (prothioconazole-desthio: m/z 312 → m/z 70). The LOQ for prothioconazole-desthio was 0.01 mg/kg for rape seed and 0.05 mg/kg for rape green material and straw.

Table 2 provides a summary of procedural recovery data reported in the trials on rape.

Table 2 Summary of procedural recovery data for method 00647 for prothioconazole-desthio in rape commodities (2178-01, 2179-01, 2101-02, 2089-00, 2088-00)

Matrix	Fortification (mg/kg)	N	Range recoveries (%)	Mean recovery (%)	%RSD
Rape seed	0.01	9	75-93	86.2	8.1
	0.1	7	79-94	86	6.5
Rape straw	0.05	9	91-103	100	4.1
	0.5	6	94-104	98.3	4.2
Rape green material	0.05	9	95-105	100.3	3.6
	0.5	7	94-102	100.3	2.8
Rape pod	0.05	3	100-103	101.3	1.5
	0.5	2	98-103	100.5	3.5

Method No. 00979/M001 (Freitag 2009 MR-06/023)

The analytical method 00979/M001 was used to determine residues of prothioconazole-desthio- α-hydroxy, prothioconazole-desthio-3-hydroxy, prothioconazole-desthio-4-hydroxy, prothioconazole-desthio-5-hydroxy and prothioconazole-desthio-6-hydroxy expressed as prothioconazole-desthio equivalents in/on matrices of plant origin by LC-MS/MS using matrix matched standards.

Homogenised samples are extracted with a mixture of acetonitrile/water (4/1; v/v) by high-speed blending. After filtration and evaporation, the remaining aqueous phase is diluted and acidified with 5 N hydrochloric acid and refluxed for 2 hours. This hydrolysis step is performed to convert glycosidic bound analogues into the respective hydroxy analytes. An aliquot is neutralized with sodium hydrogen carbonate and purified on a Chromabond® XTR cartridge. The analytes are eluted with cyclohexane/ethyl acetate (85/15; v/v). The eluate is evaporated to dryness and the remainder re-dissolved in acetonitrile. For quantitative analysis, the extract is diluted with acetonitrile and water and subjected to LC-MS/MS. Quantification is done using matrix-matched standard solutions. The LOQ in/on rape (seed and the processed fractions) is 0.01 mg/kg (expressed as prothioconazole-desthio equivalents).

Table 3 provides a summary of procedural recovery data reported in the residue trials that utilised method 00979.

Table 3. Summary of recovery data for method 00979 for prothioconazole-desthio hydroxy metabolites in oilseed commodities (rape 08-2113, 08-3112, 09-2054, 10-2134, 10-2244, 11-2003, 11-2013, 11-2137, 11-2124, 12-3402, 16-2069; flax 09-2130, 10-2073, 10-2254; sunflower 10-2238, 11-2002, 12-2008, 12-2009, 12-2156)

Matrix	Fortification (mg/kg)	N	Range recoveries (%)	Mean recovery (%)	%RSD	N	Range recoveries (%)	Mean recovery (%)	%RSD
		α-OH				3-OH			
Rape seed	0.01	26	67-100	83.5	11.9	23	65-101	84.3	11.6
	0.1	20	67-107	87.4	13.1	19	62-120	85.2	18.9
	0.5	2	79-83	81.0	3.5	2	71-77	74.0	5.7
Rape green material	0.01	24	65-115	91.6	14.0	23	67-120	90.9	19.5
	0.1	23	72-118	90.6	11.0	22	66-117	87.2	15.3
	0.2	2	95-95	95.0		2	89-89	89.0	
	0.5	3	89-90	89.5	0.8	3	78-81	79.5	2.7
	0.8	2	106-106	106.0	0.0	2	118-120	119.0	1.2
	1	2	103-107	105.0	2.7	2	86-101	93.5	11.3
Rape pods	0.01	10	64-105	90.4	16.2	10	76-113	99.8	12.7
	0.1	5	87-105	94.8	8.2	5	70-116	96.2	21.4
	0.2	2	89-98	93.5	6.8	2	76-87	81.5	9.5
	0.3	3	96-100	98.0	2.9	3	85-91	88.0	4.8
	0.4	2	82-86	84.0	3.4	2	82-87	84.5	4.2
	0.5	4	86-97	90.7	6.3	5	77-89	83.8	6.4
	0.8	2	96-103	99.5	5.0	2	108-109	108.5	0.6
Rape rest of plant	0.01	11	78-103	89.6	7.9	11	69-105	83.7	14.6
	0.1	10	71-98	87.3	11.0	10	67-94	83.2	11.2
	0.2	3	89-90	89.5	0.8	3	84-89	86.5	4.1

Matrix	Fortification (mg/kg)	N	Range recoveries (%)	Mean recovery (%)	%RSD	N	Range recoveries (%)	Mean recovery (%)	%RSD
	0.3	5	90-103	95.5	6.1	5	87-91	89.0	1.8
Oil	0.01	8	88-105	95.7	7.2	8	82-94	88.4	5.3
	0.1	7	85-100	93.8	6.2	6	81-96	89.8	6.8
Flax seed	0.01	7	68-88	79.4	10.4	7	81-103	91.4	7.8
	0.1	7	81-108	91.3	9.6	6	66-105	89.3	17.5
	0.4	1	85-85	85.0		1	81		
	0.5	1	85-85	85.0		1	87		
	0.8	2	90-93	91.5	2.3	2	100-102	101.0	1.4
Flax green material	0.01	7	86-106	92.4	7.9	7	72-108	94.7	13.9
	0.1	7	83-115	99.6	10.3	7	79-113	105.1	11.3
	0.4	1	95-95	95.0		2	85-101	93.0	12.2
	0.5	1	96-96	96.0		1	77		
	0.8	2	92-98	95.0	4.5	2	107-114	110.5	4.5
Sunflower seed	0.01	20	74-110	84.8	10.3	20	75-95	86.2	6.7
	0.09	1	87-87	87.0		10	79-94	85.2	5.4
	0.1	19	73-104	88.0	9.6	10	74-99	91.3	8.2
	0.3	1	93-93	93.0		1	96		
Sunflower partly shelled seed	0.01	4	74-83	79.0	5.0	4	69-75	71.8	3.8
	0.1	4	87-96	92.5	4.4	4	77-89	83.8	6.1
Sunflower kernels	0.01	3	72-86	81.3	9.9	4	64-85	76.3	12.3
	0.1	4	84-92	88.2	4.0	4	82-88	85.8	3.4
		4-OH				5-OH			
Rape seed	0.009	2	83-83	83.0	0.0				
	0.01	21	60-112	78.8	16.5	24	61-118	82.4	17.5
	0.09	2	76-85	80.5	7.9				
	0.1	16	69-95	79.8	12.0	19	63-115	84.8	15.9
	0.5	2	68-76	72.0	7.9	2	68-71	69.5	3.1
Rape green material	0.009	4	93-101	95.5	4.0				
	0.01	20	60-104	78.2	16.2	24	60-102	81.5	16.1
	0.09	2	81-93	87.0	9.8	23	63-101	82.0	11.5
	0.1	21	71-101	81.9	10.2	2	89-89	89.0	
	0.2	2	88-88	88.0		3	77-77	77.0	0.0
	0.5	3	76-77	76.5	0.9	2	91-92	91.5	0.8
	0.8	2	92-92	92.0	0.0	2	81-107	94.0	19.6
	0.9	2	82-107	94.5	18.7				
Rape pods	0.01	9	61-100	88.9	13.7	9	63-101	90.1	13.4

Matrix	Fortification (mg/kg)	N	Range recoveries (%)	Mean recovery (%)	%RSD	N	Range recoveries (%)	Mean recovery (%)	%RSD
	0.1	5	64-104	86.2	20.4	6	81-106	92.4	11.9
	0.2					2	87-93	90.0	4.7
	0.3	3	79-84	81.5	4.3	3	92-95	93.5	2.3
	0.4	2	79-84	81.5	4.3	2	67-72	69.5	5.1
	0.5	4	77-88	80.7	7.9	4	76-87	81.7	6.7
	0.8	2	78-82	80.0	3.5	2	78-83	80.5	4.4
Rape rest of plant	0.01	11	64-101	81.8	15.4	7	81-98	88.0	8.9
	0.1	10	67-96	82.0	12.3	5	82-90	87.2	4.1
	0.2	3	84-87	85.5	2.5	3	84-88	86.0	3.3
	0.3	5	83-93	87.5	4.7	5	83-87	85.2	2.4
Oil	0.01	8	83-93	88.0	4.6	8	81-92	87.4	4.6
	0.1	7	78-92	87.7	6.2	7	79-92	87.5	5.9
Flax seed	0.01	7	66-86	78.4	8.9	7	73-82	77.1	4.3
	0.1	7	66-103	81.3	13.6	7	67-105	79.1	15.4
	0.4	1	77-77	77.0		1	77-77		
	0.5	1	83-83	83.0		1	78-78		
	0.8	2	78-80	79.0	1.8	2	75-77	76.0	1.9
Flax green material	0.01	7	79-106	91.0	10.1	7	70-103	89.9	12.9
	0.1	7	74-112	94.3	15.4	7	75-112	94.4	14.8
	0.4	2	81-93	87.0	9.8	2	79-101	90.0	17.3
	0.5	1	90-90	90.0		1	89-89		
	0.8	2	83-89	86.0	4.9	2	82-88	85.0	5.0
Sunflower seed	0.01	20	65-96	81.4	10.2	20	63-97	84.0	10.6
	0.09	3	78-91	85.7	8.0	1	90-90		
	0.1	17	69-94	85.5	6.9	19	72-97	87.0	7.0
	0.3	1	99-99	99.0		1	95-95		
Sunflower partly shelled seed	0.01	4	62-79	71.5	10.2	4	73-77	75.0	2.2
	0.1	4	69-90	81.5	11.0	4	76-88	82.8	6.2
Sunflower kernels	0.01	4	61-74	65.5	8.9	3	67-76	71.3	6.3
	0.1	4	68-76	72.2	4.8	4	80-85	83.0	2.6
			6-OH						
Rape seed	0.01	21	65-116	83.4	16.4				
	0.1	18	64-98	79.7	15.1				
	0.5	2	70-78	74.0	7.6				
Rape green material	0.01	23	63-111	86.4	12.6				
	0.1	22	66-97	79.1	9.8				
	0.2	2	85-85	85.0					

Matrix	Fortification (mg/kg)	N	Range recoveries (%)	Mean recovery (%)	%RSD	N	Range recoveries (%)	Mean recovery (%)	%RSD
	0.5	3	69-71	70.0	2.0				
	0.8	2	85-87	86.0	1.6				
	1	1	102						
Rape pods	0.01	9	69-102	85.8	11.9				
	0.1	5	63-91	76.8	14.7				
	0.2	2	73-81	77.0	7.4				
	0.3	3	75-78	76.5	2.8				
	0.4	2	67-74	70.5	7.0				
	0.5	4	72-86	77.0	10.1				
	0.8	2	75-81	78.0	5.4				
Rape rest of plant	0.01	11	61-99	86.1	13.4				
	0.1	9	62-94	81.9	13.5				
	0.2	3	86-88	87.0	1.6				
	0.3	5	80-90	86.0	4.9				
Oil	0.01	8	69-92	81.1	9.4				
	0.1	7	64-81	76.0	8.2				
Flax seed	0.01	7	71-91	81.7	9.0				
	0.1	7	65-89	73.1	12.4				
	0.4	1	71						
	0.5	1	77						
	0.8	2	74-75	74.5	1.0				
Flax green material	0.01	7	84-110	96.1	10.3				
	0.1	7	77-110	90.0	17.3				
	0.4	2	78-89	83.5	9.3				
	0.5	1	69						
	0.8	2	73-77	75.0	3.8				
Sunflower seed	0.01	18	64-117	95.6	18.0				
	0.09	1	114						
	0.1	19	66-104	86.5	13.4				
	0.3	1	100						
Sunflower partly shelled seed	0.01	4	95-111	105.5	6.8				
	0.1	4	102-119	111.2	6.5				
Sunflower kernels	0.01	3	88-111	100.3	11.6				
	0.1	4	86-99	93.8	5.9				

Method No. 01062/M003 (Westberg 2003 M-259384-01-1; Class and Göcer 2009 M-350517-01-1)

Analytical method 01062/M003 was used for determination of residues of 1,2,4-triazole (T), triazolylalanine (TA), triazole acetic acid (TAA) and triazolyl lactic acid (TLA) in/on plant materials. Residues are extracted with methanol/water (4/1, v/v) using a high-speed homogenizer. After filtration, the final extracts are determined by LC-MS/MS using isotopically labelled internal standards for quantitation. If needed, background level is subtracted.

For the quantitation of 1,2,4-triazole a polar end-capped phase designed for retention of polar compounds and compatible with 100% aqueous mobile phase and one ion transition (MRM) in the positive mode are used. For the quantitation of triazolylalanine, a strong cation exchange HPLC column and MS/MS monitoring is used in the positive mode.

For the quantitation of the two acids triazole acetic acid (TAA) and triazolyl lactic acid (TLA) also the strong cation exchange HPLC column is used and MS/MS monitoring for each analyte with one ion transition (MRM) is done in the negative ion mode.

The LOQ for each analyte was 0.01 mg/kg in rape (seed and the processed fractions).

Table 4 summarises the procedural recovery data for the triazole metabolites measured using method 01062.

Table 4 Summary of recovery data for method 01062 for triazole metabolites in rape commodities (rape 08-2112, 08-2116, 08-3112, 08-3116, 09-2054, 11-2013, 11-2137, 12-3402; flax 9-2130, 10-2073, 10-2254; sunflower 10-2238, 10-2247, 11-2002, 12-2008, 12-2009, 12-2156)

Matrix	Fortification (mg/kg)	N	Range recoveries (%)	Mean recovery (%)	%RSD	N	Range recoveries (%)	Mean recovery (%)	%RSD
	1,2,4-T					TAA			
Rape seed	0.01	26	63-98	81.1	15.9	26	84-111	99.5	7.1
	0.1	14	70-94	81.5	11.7	14	83-112	92.4	10.4
	0.3	2	81-83	82.0	1.7	2	80-88	84.0	6.7
	1.0	13	65-101	87.8	13.9	13	84-113	102.0	10.4
Rape green material	0.01	14	91-109	100.5	6.0	14	82-110	98.3	8.4
	0.1	7	87-101	94.5	5.2	7	91-99	95.2	3.4
	1.0	6	89-100	95.3	4.8	6	85-105	99.2	7.4
Rape pods	0.01	13	80-111	94.6	10.7	13	66-105	91.8	13.5
	0.1	8	83-101	92.3	6.2	8	83-107	95.1	9.3
	1.0	6	92-102	97.2	3.6	6	100-111	105.5	4.5
Rape rest of plant	0.01	13	65-98	79.6	11.6	13	80-109	93.8	10.6
	0.1	14	71-94	81.5	9.9	14	81-103	91.3	7.4
Rape seed oil	0.01	12	78-110	98.6	10.3	12	86-107	99.7	6.8
	0.1	3	67-74	70.5	7.0	3	82-84	83.0	1.7
	1.0	10	72-106	98.7	10.5	10	94-108	104.1	3.9
Rape pomace	0.01	14	67-110	85.9	17.4	14	78-116	100.5	10.6
		5	71-76	72.8	3.3	5	84-86	85.0	1.0

Matrix	Fortification (mg/kg)	N	Range recoveries (%)	Mean recovery (%)	%RSD	N	Range recoveries (%)	Mean recovery (%)	%RSD
	1.0	10	14-108	89.0	33.4	10	82-107	102.2	7.6
Rape seed meal	0.01	5	66-85	72.5	11.8	5	104-115	111.0	4.5
	0.1	5	74-79	75.8	2.9	5	88-92	90.0	1.8
Flax seed	0.01	9	71-118	88.9	20.3	9	83-106	94.7	7.2
	0.1	7	78-100	90.0	10.1	7	84-92	89.4	3.0
	1					1	101		
Flax green material	0.01	8	80-112	94.9	11.4	9	83-112	97.4	8.6
	0.1	7	83-101	92.3	7.0	7	87-113	97.4	9.8
	1					1	100		
Sunflower seed	0.01	22	64-103	87.4	11.3	21	64-113	85.9	17.5
	0.1	23	73-113	87.4	10.3	23	71-120	93.5	11.1
	0.2	3	72-92	83.3	12.3	2	87-90	88.5	2.4
	0.3	3	82-93	86.7	6.6	3	87-102	94.7	7.9
	0.5	1	70						
	1.0	2	85-86	85.5	0.8	2	86-88	87.0	1.6
	1.5					4	73-110	93.2	16.7
Sunflower partially shelled seed	0.01	4	78-91	84.8	6.3	4	92-113	103.2	8.5
	0.1	4	89-94	91.0	2.7	4	90-93	92.0	1.5
	1.0	2	87-88	87.5	0.8	2	90-95	92.5	3.8
Sunflower kernels	0.01	4	71-84	78.0	8.3	4	86-101	93.2	7.9
	0.1	4	93-97	94.8	1.8	4	90-94	92.0	2.0
	1.0	2	85-89	87.0	3.2	2	90-90	90.0	0.0
	TLA					TA			
Rape seed	0.01	26	80-118	100.5	12.8	26	61-109	80.2	17.7
	0.1	14	78-111	88.7	8.7	10	70-98	84.2	11.7
	0.3					2	100-105	102.5	3.5
	1.0	2	82-92	87.0	8.1	12	69-109	87.3	15.1
	1.5					3	86-86	86.0	0.0
	3	13	79-112	101.9	11.2				
Rape green material	0.01	14	85-124	99.3	14.0	12	60-101	89.2	13.3
	0.1	7	93-108	101.2	5.0	6	92-101	97.2	3.4
	0.5					3	103-103	103.0	0.0
	1.0	6	105-109	106.8	1.9	6	95-108	100.7	4.9
Rape pods	0.01	13	87-115	95.4	8.1	11	70-131	101.8	17.4
	0.1	8	80-109	97.3	10.3	5	68-102	87.8	16.4

Matrix	Fortification (mg/kg)	N	Range recoveries (%)	Mean recovery (%)	%RSD	N	Range recoveries (%)	Mean recovery (%)	%RSD
	0.5					3	106-108	107.0	1.3
	1.0	6	103-113	108.3	3.9	6	80-105	88.3	10.5
Rape rest of plant	0.01	13	62-105	88.2	17.2	13	62-116	90.5	20.6
	0.1	14	74-113	93.3	13.0	14	75-119	107.9	12.1
Rape seed oil	0.01	12	87-109	99.3	8.45	12	89-115	104.1	9.4
	0.1	3	82-83	82.50	0.9	3	70-73	71.5	3.0
	1.0	10	99-107	104.3	2.1	10	93-109	104.8	4.6
Rape pomace	0.01	14	73-113	91.2	11.1	7	60-110	95.8	19.4
	0.1	5	79-84	80.5	3.0				
	1.0	10	78-111	101.8	9.4	9	84-100	93.0	6.4
Rape seed meal	0.01	5	71-88	80.2	8.7				
	0.1	5	80-83	81.2	1.8				
Flax seed	0.01	3	92-113	100.7	10.9	8	63-105	80.8	16.7
	0.1	7	86-105	96.3	7.8	7	62-81	70.9	8.5
	1	1	76						
Flax green material	0.01	4	85-107	99.0	9.9	9	68-119	92.4	19.4
	0.1	7	86-102	93.9	6.7	7	84-106	95.3	8.8
	1	1	82						
Sunflower seed	0.01	22	79-112	97.2	8.1	16	67-107	86.6	12.6
	0.1	23	77-119	95.2	11.4	22	67-115	86.3	18.2
	0.2	2	84-89	86.5	4.1	3	89-95	92.0	3.3
	0.3	3	84-95	91.0	6.7	3	72-87	79.7	9.4
	0.5					1	96-96		
	1.0	2	89-90	89.5	0.8	2	107-115	111.0	5.1
Sunflower partially shelled seed	0.01	4	73-94	87.5	11.2	4	79-98	91.0	9.2
	0.1	4	82-88	86.5	3.5	4	77-81	79.8	2.4
	1.0	2	90-91	90.5	0.8	2	86-91	88.5	4.0
Sunflower kernels	0.01	4	65-82	75.2	11.0	4	68-85	78.0	9.9
	0.1	4	84-89	86.0	2.5	4	80-82	81.5	1.2
	1.0	2	90-92	91.0	1.6	2	83-89	86.0	4.9

[a] where required, recoveries for were background corrected.

Method JA/03/01 (M-257444-01-1)

In a number of trials samples were analysed for prothioconazole, prothioconazole-desthio and prothioconazole sulfonic acid using method JA/03/01. Prothioconazole, prothioconazole-desthio and prothioconazole sulfonic acid residues are extracted from sunflower seed, meal and oil samples by refluxing with methanol/hydrogen peroxide. During this process, parent prothioconazole is converted to prothioconazole-desthio and prothioconazole sulfonic acid. An internal standard solution containing radio-labelled prothioconazole-desthio and prothioconazole sulfonic acid is added and an aliquot purified using a C-18 solid phase extraction cartridge. The samples are evaporated to near dryness and made up to volume with 30% methanol/1% acetic acid in water prior to analysis by LC-MS/MS.

The method LOQ for prothioconazole and metabolites prothioconazole-desthio and prothioconazole sulfonic acid is 0.02 mg/kg and the LOD was estimated to be 0.0067 mg/kg for each analyte.

Recovery data for method RPA JA/03/01 on sunflower seed and processed commodities are summarized in Table 5.

Table 5 Summary of method validation and procedural recovery data for method JA/03/01 for prothioconazole-desthio in/on sunflower commodities, combined validation and concurrent recoveries (M-257444-01-1)

Matrix	Fortification (mg/kg)	N	Range desthio recovery	Range sulfonic acid recovery	Range "total" recoveries (%)	Mean "total" recovery (%)	%RSD
Prothioconazole							
Sunflower seed	0.02	9	11.9-48.0	55.5-100	81.4-132	98.7	15.1
	0.04	3	39.0-43.4	37.0-54.0	76-93.5	84.6	10.3
	0.2	7	12.5-26.9	54.0-97.5	76-110.6	91.0	13.7
	0.75	3	17.6-20.1	63.5-64.8	82.4-84.0	83.3	1.0
Sunflower oil	0.02	6	11.8-18.3	59.5-89.0	72.9-105.2	90.3	12.5
	0.04	3	7.7-10.0	65.8-90.3	75.6-100.3	87.6	14.1
	0.2	4	6.6-7.7	82.0-95.5	89.7-102.1	96.0	5.7
	0.5	3	5.5-9.3	87.8-95.0	96.0-100.5	97.9	2.4
Sunflower meal	0.02	6	8.6-15.4	61-85	71.1-100.4	85.0	15.7
	0.04	3	42.8-47.3	47.8-56.5	93.1-103.8	97.5	5.7
	0.2	4	20.3-27.0	50.5-59.5	70.8-86.5	79.5	8.2
	0.5	3	32.3-33.3	44.3-47.0	76.7-80.3	78.5	2.4
Prothioconazole-desthio							
Sunflower seed	0.02	9	74.5-113			91.6	15.2
	0.04	3	80.5-91.0			84.3	6.9
	0.2	7	75.0-91.5			82.1	7.0
	0.75	3	83.9-89.2			86.2	3.2
Sunflower oil	0.02	6	84.0-112			101.3	10.8
	0.04	3	88.5-96.3			91.9	4.4
	0.2	4	86.5-98.0			93.5	5.4
	0.5	3	88.6-89.2			88.9	0.3

Matrix	Fortification (mg/kg)	N	Range desthio recovery	Range sulfonic acid recovery	Range "total" recoveries (%)	Mean "total" recovery (%)	%RSD
Sunflower meal	0.02	6	72.0-99.0			88.2	11.0
	0.04	3	88.0-92.5			89.6	2.8
	0.2	4	82.5-91.0			86.9	4.4
	0.3	3	90.3-95.0			92.3	2.6
Prothioconazole sulfonic acid							
Sunflower seed	0.02	9		78.0-111		93.7	11.7
	0.04	3		89.5-107		96.8	9.4
	0.2	7		69.5-108		90.1	15.5
	0.75	3		73.6-107		92.6	18.5
Sunflower oil	0.02	6		90.0-97.5		94.3	3.6
	0.04	3		87.0-99.3		92.0	7.0
	0.2	4		87.0-95.0		90.1	3.9
	0.5	3		98.6-102		100.9	1.9
Sunflower meal	0.02	6		82.0-118		93.9	14.6
	0.04	3		80.3-88.8		84.4	5.1
	0.2	4		81.0-97.0		88.4	8.0
	0.3	3		86.7-102		92.6	8.9

Stability of pesticides residues in stored analytical samples

Oilseed rape green material and seed at 5 °C

A new storage stability study was conducted for prothioconazole-desthio to investigate stability during refrigerated storage at 5 °C. This is to cover short-term increases in storage temperature which occurred during shipment of trials samples in 2009 (Schöning & Billian, 2010, MR-10/009, M-388450-02-1).

The study was initiated to evaluate the stability of 24 analytes, including prothioconazole-desthio after storage for a period of approximately 5 days at 5 °C. For prothioconazole-desthio, stability investigations were performed in matrices of - high water content: oilseed rape (green material, covering rest of plant and pod), - high oil content: oilseed rape (seed).

Aliquots of 10 g of each homogenized matrix were fortified individually with prothioconazole-desthio at a nominal fortification level of 0.5 mg/kg. The samples were stored in amber glass bottles with plastic caps at an average temperature of +5 °C for up to 5 days. Samples were analysed for prothioconazole-desthio by HPLC-MS/MS using method 01013. Residues in the control samples were <LOQ (<0.01 mg/kg). Overall concurrent recoveries were within the acceptable range of 70–110%, with overall RSDs below 20%. Residues of prothioconazole-desthio were stable in oilseed rape green material and seed for at least 5 days when stored at 5 °C.

Table 6 Stability data for prothioconazole-desthio in rape seed and green material fortified at 0.5 mg/kg and stored at 5 °C for five days

Matrix	Storage Interval (days)	Individual Procedural Recoveries (%)	Mean Procedural Recovery (%)	Residue (mg/kg)	Mean residue (mg/kg)	Mean %remaining
Oilseed rape (seeds)	0	86, 89	88	0.432, 0.465, 0.434, 0.444, 0.454	0.446	-
	5	92, 96	94	0.432, 0.464, 0.458, 0.436, 0.442	0.446	100.1
Oilseed rape (green material)	0	91, 97	94	0.471, 0.488, 0.493, 0.495, 0.476	0.485	-
	5	99, 98	99	0.485, 0.456, 0.460, 0.480, 0.500	0.476	98.3

The stability of prothioconazole, prothioconazole-desthio and prothioconazole sulfonic acid on freezer storage was studied (Ballantine 2013 AAFC08-022R M-527444-01-1). Samples of sunflower seed were fortified with either prothioconazole or and mixture of prothioconazole-desthio and prothioconazole sulfonic acid and stored for intervals of up to 512 days. Residues were measured using method JA/03/01 which converts prothioconazole to a mixture of prothioconazole-desthio and prothioconazole sulfonic acid which are reported as "total prothioconazole". Prothioconazole-desthio and prothioconazole sulfonic acid are not transformed by the method and are measured unchanged.

Table 7 Stability of prothioconazole, prothioconazole-desthio and prothioconazole sulfonic acid residues in sunflower seed fortified at 0.2 mg/kg following freezer storage (Ballantine 2013 AAFC08-022R M-527444-01-1)

Storage interval (days)	Residue (mg/kg)			Procedural recovery (%)			Mean procedural recovery (%)	Mean %remaining
Seed								
"Total"								
28	0.138	0.135	0.136	68.8	72.5	73	71.4	95.5
124	0.143	0.152	0.131	97	93	98	96	74.0
216	0.163	0.205	0.185	84.5	83	80.5	82.7	111
304	0.144	0.142	0.154	85.5	99.4	96.9	93.9	78.2
392	0.165	0.166	0.173	87.5	102	96.5	95.3	88.2
521	0.13	0.127	0.138	89	87.2	91.1	89.1	73.8
Desthio								
28	0.208	0.176	0.173	85.5	88	87	86.8	107
124	0.19	0.187	0.185	96.5	93	113	101	92.8
216	0.193	0.199	0.197	95.5	91	95.5	94	105
304	0.191	0.185	0.181	97	98.5	99.5	98.3	94.4
392	0.192	0.174	0.175	90	89	90.5	89.9	100

Storage interval (days)	Residue (mg/kg)			Procedural recovery (%)			Mean procedural recovery (%)	Mean %remaining
521	0.193	0.201	0.206	102	94	105	100	100
Sulfonic acid								
28	0.215	0.209	0.195	93	102	97.5	97.5	106
124	0.196	0.220	0.194	109	95.5	96.5	100	102
216	0.207	0.203	0.199	99.5	102	94.5	98.7	103
304	0.192	0.203	0.2	100	100	97.5	99.2	100
392	0.201	0.210	0.236	116	113	98	109	99
521	0.191	0.207	0.184	97.5	81	103	93.8	104

Residues of prothioconazole, prothioconazole-desthio and prothioconazole sulfonic acid are stable in sunflower seed on frozen storage for at least 521 days.

The 2008 JMPR reported residues of prothioconazole-desthio are stable on frozen storage of rape pods and rape straw for at least 24 months and rape seed for at least 36 months.

USE PATTERN

Prothioconazole is registered for use in a variety of crops worldwide. Table 8 provides a summary of relevant GAPs.

Table 8 Summary of GAPs for rape, flax and sunflower considered by the Meeting

Crop	Country	Formulation	Method	Rate (g ai/ha)	Water volume (L/ha)	No. or max (g ai/ha/ season)	Spray interval (days)	PHI (days)
Rapeseed	France	250 g ai/L EC	Foliar spray	175	100-400	2 (BBCH 30 to 80)	14-28	56
Oilseed rape	Greece	250 g ai/L EC	Foliar spray	175	100-400	2 (BBCH 30 to 80)	14-28	56
Canola/oilseed rape, black radish	Hungary	125 g ai/L SE	Foliar spray	125	200-400	2 (BBCH 14 to 73)	14	28
Oilseed rape	Ireland	250 g ai/L EC	Foliar spray	175, max 350/season	100-300	2	-	56
Oilseed rape	Ireland	125 g ai/L EC	Foliar spray	125, max 250/season	200-300	2	21	56
Oilseed rape	Serbia	125 g ai/L SE	Foliar spray	125	200-400	2 (BBCH 14 to 73)	-	56
Oilseed rape	Spain	125 g ai/L EC	Foliar spray	125		2 (flower bud stage until 50% pods final size)	28	56
Oilseed rape	UK	125 g ai/L EC	Foliar spray	125	100-400	2	21	56
Flax	France	250 g ai/L EC	Foliar spray	175	100-400	2 (BBCH 30 to 69)	- [a]	56
Flax	Greece	250 g ai/L EC	Foliar spray	175	100-400	2 (BBCH 30 to 69)	14	56

Crop	Country	Formulation	Method	Rate (g ai/ha)	Water volume (L/ha)	No. or max (g ai/ha/ season)	Spray interval (days)	PHI (days)
Mustard	Hungary	125 g ai/L SE	Foliar spray	100-125	200-400	2 (BBCH 57 to 69)	14	56
Poppy	Hungary	125 g ai/L SE	Foliar spray	125	200-400	2 (BBCH 16 to 77)	14	56
Sunflower	Brazil	175 g ai/L SC	Foliar spray	78.75 – 87.5[b]	100-150 (ground) 20-40 (aerial)	2	15	30
Sunflower	Hungary	125 g ai/L SE	Foliar spray	100-125	150-400	2 (BBCH 16 to 69)	14	28
Sunflower	Serbia	125 g ai/L SE	Foliar spray	125	200-400	2 (BBCH 16 to 69)	-	28

[a] Application interval for flax is not specified.

[b] add methylated soya bean oil at 0.25% v/v

RESULTS OF SUPERVISED RESIDUE TRIALS ON CROPS

Rape

Table 9 Residues of prothioconazole and metabolites in oilseed rape

							Residue (mg/kg expressed as prothioconazole-desthio)					
RAPE Location, year, variety	N (int)	Rate g ai/ha	GS Sampling	DALA	Sample	Prothio	Desthio	α-OH	3-OH	4-OH	5-OH	6-OH
Chambourg sur Indre Centre, France, 2008 Flash**	2 (14)	120 120	BBCH82	21	Pod		0.1	0.02	0.18	0.05	0.02	<0.01
			BBCH89	33	Seed		0.02	<0.01	<0.01	<0.01	<0.01	<0.01
Burscheid Nordrhein-Westfalen, Germany, 2008 Titan**	2 (14)	120 120	BBCH89	35	Seed		0.01	<0.01	0.02	<0.01	<0.01	<0.01
Cortil-Noirmont Brabant Wallon, Belgium, 2008 Exocet**	2 (14)	120 120	BBCH87	21	Pod		0.26	0.021	0.29	0.09	0.03	<0.01
			BBCH89	38	Seed		0.03	<0.01	<0.01	<0.01	<0.01	<0.01
Werl-Niederbergstraße Nordrhein-Westfalen, Germany, 2008 Astrid**	2 (14)	120 120	BBCH97	30	Seed		0.04	<0.01	<0.01	<0.01	<0.01	<0.01

							Residue (mg/kg expressed as prothioconazole-desthio)					
RAPE Location, year, variety	N (int)	Rate g ai/ha	GS Sampling	DALA	Sample	Prothio	Desthio	α-OH	3-OH	4-OH	5-OH	6-OH
Cherves, France, 2008 Ovation [a]	2 (14)	120 120	BBCH83	21	Pod		0.14	0.02	0.24	0.08	0.02	<0.01
			BBCH89	31	Seed		0.05	<0.01	0.02	<0.01	<0.01	<0.01
L´Ametlla del Valles, Spain, 2008 Silvia [a]	2 (14)	120 120	BBCH79	21	Pod		0.04	<0.01	0.15	0.05	0.02	<0.01
			BBCH86	30	Seed		0.03	<0.01	0.15	0.05	0.02	<0.01
Fleurieux sur l'Arbesle, Rhone-Alpes, France, 2009 Artiste [b]	2 (13)	120 120	BBCH88	14	pod		1.5	0.01	0.21	0.09	0.02	<0.01
			BBCH89	20	Pod		1.6	0.02	0.26	0.11	0.03	<0.01
			BBCH89	29	Seed		<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
San Giogio Di Piano, Emilia-Romagna, Italy, 2009 PR46W09 [b]	2 (14)	120 120	BBCH89	28	Seed		0.02	<0.01	0.02	<0.01	<0.01	<0.01
Braslou, France, 2009 Arcadia [c]	2 (14)	120 120	BBCH83	14	pod		0.46	0.04	0.37	0.13	0.03	0.01
			BBCH85	21	Pod		0.49	0.04	0.50	0.17	0.04	0.02
			BBCH89	28	Seed		0.03	<0.01	<0.01	<0.01	<0.01	<0.01
Cortil-Noirmont, Belgium, 2009 Monalisa [c]	2 (14)	120 120	BBCH81	14	pod		0.27	0.02	0.21	0.08	0.02	<0.01
			BBCH82	21	Pod		0.25	0.02	0.29	0.11	0.03	<0.01
			BBCH89	28	Seed		0.03	<0.01	<0.01	<0.01	<0.01	<0.01
Nieuw Beerta, Netherlands, 2009 unknown [c]	2 (14)	120 120	BBCH99	28	Seed		0.02	<0.01	<0.01	<0.01	<0.01	<0.01
Bishop Burton UK, 2009 Castille [o] Harvest 13/8/2009	2 (15)	120 120	BBCH83	29	Pod		<0.01	<0.01	<0.01 C0.12	<0.01 C0.04	<0.01 C0.01	<0.01
			BBCH89	44	Seed		0.01	<0.01	0.02	<0.01	<0.01	<0.01
Cortil-Noirmont, Belgium, 2010 Exocet [d]	2 (15)	125 125	BBCH80	14	pod		0.15	0.02	0.19	0.07	0.03	<0.01
			BBCH81	21	Pod		0.09	0.02	0.21	0.07	0.03	<0.01
			BBCH89	28	Seed		0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Royston UK, 2010 Cabernet [d]	2 (13)	125 125	BBCH80	14	pod		0.62	0.07	0.27	0.13	0.03	<0.01
			BBCH85	22	Pod		0.56	0.06	030	0.15	0.03	<0.01
			BBCH89	28	Seed		0.05	<0.01	<0.01	<0.01	<0.01	<0.01

							Residue (mg/kg expressed as prothioconazole-desthio)					
RAPE Location, year, variety	N (int)	Rate g ai/ha	GS Sampling	DALA	Sample	Prothio	Desthio	α-OH	3-OH	4-OH	5-OH	6-OH
Slootdrop The Netherlands, 2010 Haydn [d]	2 (14)	125 125	BBCH85	14	pod		0.48	0.03	0.21	0.09	0.02	<0.01
			BBCH88	21	Pod		0.48	0.05	0.29	0.14	0.04	<0.01
			BBCH89	28	Seed		0.04	<0.01	<0.01	<0.01	<0.01	<0.01
Bouafle France, 2010 Olindigo [d]	2 (14)	125 125	BBCH88	14	pod		3.8	0.04	0.46	0.32	0.07	<0.01
			BBCH89	21	Seed		0.11	<0.01	<0.01	<0.01	<0.01	<0.01
Vilobi d'Onyar Spain, 2010 Artist-Hybrid [d]	2 (11)	125 136	BBCH79	14	pod		0.17	<0.01	0.11	0.04	0.01	<0.01
			BBCH86	21	Seed		0.04	<0.01	<0.01	<0.01	<0.01	<0.01
Vouille France, 2010 Aviator [d]	2 (14)	125 125	BBCH80	14	pod		0.27	0.03	0.19	0.07	0.02	<0.01
			BBCH85	21	Pod		0.18	0.02	0.20	0.06	0.02	<0.01
			BBCH89	27	Seed		0.03	<0.01	<0.01	<0.01	<0.01	<0.01
Maritima Malaga Spain, 2010 Kavel [e]	2 (14)	125 125	BBCH80	14	pod		0.03	<0.01	0.10	0.03	<0.01	<0.01
			BBCH83	21	Pod		0.03	<0.01	0.08	0.02	<0.01	<0.01
			BBCH89	27	Seed		0.03	<0.01	<0.01	<0.01	<0.01	<0.01
Gontaud de Nogaret France, 2010 Hybri Star [e]	2 (13)	125 125	BBCH82	14	pod		0.93	0.07	0.48	0.22	0.02	<0.01
			BBCH85	22	Pod		0.58	0.61	0.60	0.23	0.03	<0.01
			BBCH89	28	Seed		0.09	0.01	0.02	<0.01	<0.01	<0.01
Burscheid Germany, 2011 Elektra [p]	2 (14)	125 125	BBCH80	14	pod		0.14	<0.01	0.075	0.021	<0.01	<0.01
			BBCH81	21	Pod		0.062	<0.01	0.094	0.026	<0.01	<0.01
			BBCH83	29	Pod		0.11	<0.01	0.082	0.024	<0.01	<0.01
			BBCH89	44	Seed		<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Sombreffe (Vielle-Maison) Belgium, 2011 Limone Hybrid restored oil-rich [p]	2 (14)	125 125	BBCH80	14	pod		0.43	<0.01	0.099	0.026	<0.01	<0.01
			BBCH82	21	Pod		0.37	<0.01	0.13	0.039	0.012	<0.01
			BBCH89	29	Seed		0.083	<0.01	<0.01	<0.01	<0.01	<0.01
Royston UK, 2011 FLASH Hybrid [p]	2 (15)	125 125	BBCH80	14	pod		0.21	<0.01	0.064	0.020	<0.01	<0.01
			BBCH84	21	Pod		0.20	<0.01	0.078	0.026	<0.01	<0.01

							Residue (mg/kg expressed as prothioconazole-desthio)					
RAPE Location, year, variety	N (int)	Rate g ai/ha	GS Sampling	DALA	Sample	Prothio	Desthio	α-OH	3-OH	4-OH	5-OH	6-OH
			BBCH89	29	Seed		0.033	<0.01	<0.01	<0.01	<0.01	<0.01
Chambourg sur Indre France, 2011 Dynastie [p]	2 (14)	125 125	BBCH79	14	pod		0.69	<0.01	0.13	0.083	0.015	<0.01
			BBCH87	21	Pod		0.73	<0.01	0.16	0.094	0.016	<0.01
			BBCH89	27	Seed		0.081	<0.01	<0.01	<0.01	<0.01	<0.01
Les Franqueses del Valles Spain, 2011 Pacific [f]	2 (14)	126 120	BBCH81	14	pod		<0.01 c0.15	<0.01 C0.011	<0.01 C0.23	<0.01 C0.13	<0.01 C0.032	<0.01
			BBCH85	21	Pod		0.10	0.024	0.19	0.11	0.029	<0.01
			BBCH87	30	Seed		0.025	<0.01	<0.01	<0.01	<0.01	<0.01
Bouloc France, 2011 Nk Alamir Hybride [f]	2 (13)	120 120	BBCH89	30	Seed		0.026	<0.01	<0.01	<0.01	<0.01	<0.01
Burscheid Germany, 2011 Elektra [f]	2 (14)	120 120	BBCH83	31	Pod		0.075	<0.01	0.16	0.054	0.016	<0.01
			BBCH89	50	Seed		<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Tarquinia Italy, 2011 Hybristar [f]	2 (14)	120 120	BBCH81	14	pod		0.42	0.013	0.34	0.16	0.035	<0.01
			BBCH85	21	Pod		0.41	0.014	0.44	0.20	0.045	<0.01
			BBCH89	30	Seed		0.038	<0.01	<0.01	<0.01	<0.01	<0.01
La Luisiana Spain, 2011 Eswilliams [f]	2 (18)	120 138	BBCH89	31	Seed		0.064	<0.01	<0.01	<0.01	<0.01	<0.01
Velleron France, 2011 Hybrilux; Rape winter [f]	2 (14)	120 120	BBCH89	30	Seed		0.055	<0.01	<0.01	<0.01	<0.01	<0.01
La Luisiana Spain, 2011 Eswilliams [q]	2 (14)	125 125	BBCH86	14	pod		3.9	0.052	0.36	0.28	0.053	0.014
			BBCH88	21	Pod		3.3	0.048	0.32	0.26	0.044	0.015
			BBCH89	28	Seed		0.13	<0.01	<0.01	<0.01	<0.01	<0.01
Bouloc France, 2011 Nk Alamir; Hybride®	2 (14)	125 125	BBCH84	14	pod		0.98	0.053	0.26	0.20	0.033	<0.01
			BBCH87	21	Pod		1.1	0.061	0.35	0.26	0.046	<0.01
			BBCH89	28	Seed		0.043	<0.01	<0.01	<0.01	<0.01	<0.01
Tarquinia (VT) Italy, 2011 Hybristar [q]	2 (14)	125 125	BBCH81	14	pod		2.2	0.022	0.24	0.11	0.019	<0.01
			BBCH85	21	Pod		0.67	0.022	0.31	0.15	0.026	<0.01
			BBCH89	28	Seed		0.060	<0.01	<0.01	<0.01	<0.01	<0.01
Les Franqueses del Valles –	2 (14)	125 125	BBCH81	14	pod		0.14	0.018	0.16	0.083	0.017	<0.01

							Residue (mg/kg expressed as prothioconazole-desthio)					
RAPE Location, year, variety	N (int)	Rate g ai/ha	GS Sampling	DALA	Sample	Prothio	Desthio	α-OH	3-OH	4-OH	5-OH	6-OH
Llerona Spain, 2011 Pacific [q]												
			BBCH85	21	Pod		0.13	0.016	0.29	0.13	0.023	<0.01
			BBCH87	28	Seed		0.03	<0.01	<0.01	<0.01	<0.01	<0.01
Banbury UK, 2011 D.K. Cabernet [m]	2 (14)	120 120	BBCH89	28	Seed		0.054	<0.01	<0.01	<0.01	<0.01	<0.01
Saint Maurice-En-Rivère France, 2016 Manzana [r]	2 (14)	125 138	BBCH83	15	pod		0.061	<0.01	0.038	0.019	<0.01	<0.01
			BBCH88	22	Pod		1.3	0.098	0.55	0.72	0.075	<0.01
			BBCH89	28	Seed		0.056	<0.01	<0.01	0.02	<0.01	<0.01
Faulbach Germany, 2016 PR46W20 [r]	2 (14)	125 125	BBCH88	14	pod		1.3	0.049	0.38	0.16	0.044	<0.01
			BBCH89	20	Pod		2.0	0.090	0.65	0.30	0.083	0.017
			BBCH89	28	Seed		0.14	<0.01	0.01	<0.01	<0.01	<0.01
Mousseaux-Neuville; west of Paris, France, 2002 Capitol [i]	2 (62)	125 125	BBCH89	56	Seed		0.01					
Leichlingen Germany, 2002 Express [i]	2 (58)	125 125	BBCH89	55	Seed		<0.01					
Norton, east of Cambridge, UK, 2002 Madrigal [i]	2 (70)	125 114	BBCH89	54	Seed		<0.01					
Burscheid, Germany, 2002 Licondor [i]	2 (58)	125 125	BBCH87	55	Seed		<0.01					
			BBCH89	62	Seed		<0.01					
Ravenna Italy, 2002 Pegletta [j]	2 (7)	125 125	BBCH87	56	Seed		<0.01					
			BBCH89	63	Seed		<0.01					
Pujalt Spain, 2002 Bristol [j]	2 (39)	125 125	BBCH89	56	Seed		<0.01					
			BBCH89	63	Seed		<0.01					
Braslou (Centre) France, 2007 Grizzly①	2 (26)	125 125	BBCH85	36	Pod	<0.01	0.03					
			BBCH89	50	Seed	<0.01	<0.01					
Burscheid (Nordrhein-Westfalen)	2 (19)	125 125	BBCH85	49	Pod	<0.01	<0.01					

							Residue (mg/kg expressed as prothioconazole-desthio)					
RAPE Location, year, variety	N (int)	Rate g ai/ha	GS Sampling	DALA	Sample	Prothio	Desthio	α-OH	3-OH	4-OH	5-OH	6-OH
Germany, 2007 Elektra [k]												
			BBCH89	69	Seed	<0.01	<0.01					
Werl-Westönnen (Nordrhein-Westfalen) Germany, 2007 Oase [k]	2 (39)	125 125	BBCH85	44	Pod	<0.01	0.02					
			BBCH89	61	Seed	<0.01	<0.01					
Near Eye (Suffolk) UK, 2007 Es Astrid [k]	2 (34)	125 125	BBCH85	46	Pod	<0.01	0.01					
			BBCH89	57	Seed	<0.01	<0.01					
Gargas (Midi-Pyrenees) France, 2007 Corail [l]	2 (20)	125 125	BBCH89	59	Seed	<0.01	<0.01					
Conselice (Ravenna) (Emilia-Romagna) Italy, 2007 Belcanto [l]	2 (29)	125 125	BBCH85	30	Pod	<0.01	0.04					
			BBCH89	40	Seed	<0.01	<0.01					
Burscheid (Nordrhein-Westfalen), Germany, 2006 Talent [g]	2 (19)	125 125	BBCH85	45	Pod	<0.01	0.02					
			BBCH89	57	Seed	<0.01	<0.01					
Chambourg sur Indre (Centre), France, 2006 Standing [g]	2 (15)	125 125	BBCH89	48	Seed	<0.01	<0.01					
Werl-Westönnen (Nordrhein-Westfalen), Germany, 2006 Smart [g]	2 (29)	125 125	BBCH89	50	Seed	<0.01	<0.01					
Lund (Scania), Sweden, 2006 Calypso [g]	2 (23)	125 125	BBCH89	47	Seed	<0.01	<0.01					
Boara (Ferrara) (Emilia-Romagna) Italy, 2006 Molino [h]	2 (24)	125 125	BBCH89	37	Seed	<0.01	<0.01					
Pujalt (Barcelona) (Cataluña) Spain, 2006 Pacific [h]	2 (34)	125 125	BBCH89	37	Seed	<0.01	<0.01					

							Residue (mg/kg expressed as prothioconazole-desthio)					
RAPE Location, year, variety	N (int)	Rate g ai/ha	GS Sampling	DALA	Sample	Prothio	Desthio	α-OH	3-OH	4-OH	5-OH	6-OH
Braslou France, 2008 Grizzly***	2 (14)	100 100	BBCH80	42	Pod		<0.01	<0.01	0.03	<0.01	<0.01	<0.01
			BBCH89	55	Seed		<0.01	<0.01	0.01	<0.01	<0.01	<0.01
Hoofddorp The Netherlands, 2008 Maximus***	2 (14)	100 100	BBCH99	50	Seed		<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Cortil-Noirmont Belgium, 2008 Exocet***	2 (14)	100 100	BBCH85	42	Pod		0.02	<0.01	0.10	0.02	<0.01	<0.01
			BBCH89	56	Seed		<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Werl-Niederbergstraße Germany, 2008 Astrid***	2 (14)	100 100	BBCH97	56	Seed		<0.01	<0.01	0.01	<0.01	<0.01	<0.01
Nieuw Beerta Netherlands, 2009 unknown[&&]	2 (14)	150 150	BBCH99	49	Seed		<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Cortil-Noirmont Belgium, 2009 Monalisa[&&]	2 (15)	150 150	BBCH80	30	Pod		0.03	<0.01	0.12	0.04	0.01	<0.01
			BBCH81	42	Pod		0.03	<0.01	0.13	0.04	0.01	<0.01
			BBCH89	55	Seed		<0.01	<0.01	0.02	<0.01	<0.01	<0.01
Lignieres les Roye France, 2009 Kador[&&]	2 (14)	150 150	BBCH89	55	Seed		0.02	<0.01	0.03	<0.01	<0.01	<0.01
Bishop Burton UK, 2009 Castille [n]	2 (14)	139 150	BBCH75	30	Pod		0.13	<0.01	0.16	0.05	0.01	<0.01
			BBCH80	42	Pod		0.02	<0.01	0.10	0.03	<0.01	<0.01
			BBCH81	56	Seed		<0.01	<0.01	<0.01	<0.01	<0.01	<0.01

* prior to last treatment

[a] 08-2113, method 01013 for prothioconazole-desthio, method 00979 for hydroxy metabolites

[b] 09-2504, method 01013 for prothioconazole-desthio, method 00979 for hydroxy metabolites

[c] 09-2055, method 01013 for prothioconazole-desthio, method 00979 for hydroxy metabolites

[d] 10-2134, method 01013 for prothioconazole-desthio, method 00979 for hydroxy metabolites

[e] 10-2244, method 01013 for prothioconazole-desthio, method 00979 for hydroxy metabolites

[f] 11-2013, method 01013 for prothioconazole-desthio, method 00979 for hydroxy metabolites

[g] 2618/06, method 01013 for prothioconazole and prothioconazole-desthio

[h] 2619/06, method 01013 for prothioconazole and prothioconazole-desthio

[i] 2101/02, method 00647 for desthio

[j] 2102/02, method 00647 for desthio

[k] 2616/07, method 01013 for prothioconazole and prothioconazole-desthio

[l] 2617/07, method 01013 for prothioconazole and prothioconazole-desthio

[m] 11-2137, method 01013 for prothioconazole-desthio, method 00979 for hydroxy metabolites
[n] 09-2244, method 01013 for prothioconazole-desthio, method 00979 for hydroxy metabolites
[o] 09-2245, method 01013 for prothioconazole-desthio, method 00979 for hydroxy metabolites
[p] 11-2003, method 01013 for prothioconazole-desthio, method 00979 for hydroxy metabolites
[q] 11-2124, method 01013 for prothioconazole-desthio, method 00979 for hydroxy metabolites
[r] 16-2069, method 01013 for prothioconazole-desthio, method 00979 for hydroxy metabolites
**08-2112, method 01013 for prothioconazole-desthio, method 00979 for hydroxy metabolites
***08-2116, method 01013 for prothioconazole-desthio, method 00979 for hydroxy metabolites
[&&] 09-2053, method 01013 for prothioconazole-desthio, method 00979 for hydroxy metabolites

Table 10 Residues of triazole metabolites in rape following application of prothioconazole

RAPE		Rate	Vol	Sampling			Residue (mg/kg)			
Location, year, variety	N	g ai/ha	L/ha	GS	DALA	Sample	1,2,4-triazole	TAA	TLA	TA
Fleurieux sur l'Arbesle, Rhone-Alpes, France, 2009 Artiste [a]	2 (13)	120 120	300 300	BBCH88	14	pod	<0.01	0.02 C0.02	0.03 C0.01	0.23 C0.43
				BBCH89	20	Pod	<0.01	0.02	0.01	0.17
				BBCH89	29	Seed	<0.01	<0.01	0.02 C0.03	0.38 C0.72
San Giogio Di Piano, Emilia-Romagna, Italy, 2009 PR46W09 [a]	2 (14)	120 120	300 300	BBCH89	28	Seed	<0.01	<0.01	0.03 C0.02	0.66 C0.49
Braslou, France, 2009 Arcadia [b]	2 (14)	120 120	300 300	BBCH83	14	pod	<0.01	0.08 c0.10	<0.01	<0.01
				BBCH85	21	Pod	<0.01	0.09	<0.01	<0.01
				BBCH89	28	Seed	<0.01	0.21 c0.23	<0.01	0.01 C0.01
Cortil-Noirmont, Belgium, 2009 Monalisa [b]	2 (14)	120 120		BBCH81	14	pod	<0.01	0.09 C0.06	<0.01	<0.01
				BBCH82	21	Pod	<0.01	0.04	<0.01	<0.01
				BBCH89	28	Seed	<0.01	0.13 C0.09	<0.01	<0.01
Nieuw Beerta, Netherlands, 2009 unknown [b]	2 (14)	120 120	300 300	BBCH99	28	Seed	<0.01	0.11 C0.12	<0.01	<0.01
Les Franqueses del Valles Spain, 2011 Pacific [c]	2 (14)	126 120	315 300	BBCH81	14	pod	<0.01	0.084 C0.099	<0.01	<0.01
				BBCH85	21	Pod	<0.01	0.18	<0.01	<0.01
				BBCH87	30	Seed	<0.01	0.16 C0.27	<0.01	<0.01
Bouloc France, 2011 Nk Alamir Hybride [c]	2 (13)	120 120	300 300	BBCH89	30	Seed	<0.01	2.2 C2.2	0.065 C0.08	0.016 C0.019
Burscheid Germany, 2011 Elektra [c]	2 (14)	120 120	300 300	BBCH83	31	Pod	<0.01	0.086 C0.081	<0.01	<0.01

RAPE Location, year, variety	N	Rate g ai/ha	Vol L/ha	Sampling GS	DALA	Sample	Residue (mg/kg) 1,2,4-triazole	TAA	TLA	TA
				BBCH89	50	Seed	<0.01	0.21 C0.18	<0.01	<0.01
Tarquinia Italy, 2011 Hybristar [c]	2 (14)	120 120	300 300	BBCH81	14	pod	<0.01	0.04 C0.024	<0.01	<0.01
				BBCH85	21	Pod	<0.01	0.049	<0.01	<0.01
				BBCH89	30	Seed	<0.01	0.11 C0.057	<0.01	<0.01
La Luisiana Spain, 2011 Eswilliams [c]	2 (18)	120 138	300 344	BBCH89	31	Seed	<0.01	0.87 C0.76	0.022 C0.02	<0.01
Velleron France, 2011 Hybrilux; Rape winter [c]	2 (14)	120 120	300 300	BBCH89	30	Seed	<0.01	0.12 C0.17	<0.01	<0.01
Banbury UK, 2011 D.K. Cabernet [d]	2 (14)	120 120	200 200	BBCH89	28	Seed	<0.01	0.14 C0.24	<0.01	<0.01
Bishop Burton UK, 2009 Castille [e]	2 (14)	139 150	192 200	BBCH75	30	Pod	<0.01	0.14 C0.07	<0.01	0.01
				BBCH80	42	Pod	<0.01	0.10	<0.01	0.01
				BBCH81	56	Seed	<0.01	0.34 C0.27	<0.01	<0.01 C0.01
Bishop Burton UK, 2009 Castille [f] Harvest 13/8/2009	2 (15)	120 120	200 200	BBCH83	29	Pod	<0.01	<0.01	<0.01	0.08 C0.12
				BBCH89	44	Seed	<0.01	<0.01	0.02 C0.01	0.28 C0.01
Nieuw Beerta Netherlands, 2009 unknown [g]	2 (14)	150 150	300 300	BBCH99	49	Seed	<0.01	0.16	<0.01	<0.01 C0.12
Cortil-Noirmont Belgium, 2009 Monalisa [g]	2 (15)	150 150	200 200	BBCH80	30	Pod	<0.01	0.04	<0.01	<0.01 C0.05
				BBCH81	42	Pod	<0.01	0.06	<0.01	<0.01
				BBCH89	55	Seed	<0.01	0.12	<0.01	<0.01 C0.08
Lignieres les Roye France, 2009 Kador [g]	2 (14)	150 150	300 300	BBCH89	55	Seed	<0.01	0.55	<0.01 C0.03	0.02 C0.70

[a] 09-2054, method 01062

[b] 09-2055, method 01062

[c] 11-2013, method 01062

[d] 11-2137, method 01062

[e] 09-2244, method 01062

[f] 09-2245, method 01062

[g] 09-2053, method 01062

C – control sample

Table 11 Residues of prothioconazole in oilseed rape from European trials previously evaluated by the JMPR (2008)

RAPE Location, year, variety	N (int)	Rate g ai/ha	Vol L/ha	Sampling GS	DALA	Sample	Residues (mg/kg) Prothioconazole-desthio
Burscheid (Versuchsgut Höfchen) Germany 2000 Lirajet [a]	2 (38)	175 175	300 300	BBCH79-81	41	Pod	<0.05
				BBCH83-85	56	Pod	<0.05
				BBCH89	67	Seed	<0.01
Eslov Sweden 2000 Maskot [a]	2 (18)	175 175	300 300	BBCH79	42	Pod	<0.05
				BBCH88	56	Pod	<0.05
				BBCH92	64	Seed	<0.01
Villettes France 2000 Carolus [a]	2 (59)	175 175	300 300	BBCH81	42	Pod	0.05
				BBCH89	56	Seed	0.02
Norton UK 2000 Madrigal [a] 421/4	2 (69)	190 175	325 300	BBCH87	42	Pod	<0.05
				BBCH92	56	Seed	0.01
				BBCH93	63	Seed	0.01
Burscheid (Versuchsgut Höfchen) Germany 2001 Express [b]	2 (48)	175 175	300 300	BBCH89	57	Seed	<0.01
				BBCH92	61	Seed	<0.01
Norton Bury St. Edmunds UK 2001 Madrigal [b]	2 (52)	175 163	300 280	BBCH89	56	Seed	<0.01
Etrepagny France 2001 Zenith [b]	2 (49)	175 175	300 300	BBCH89	59	Seed	<0.01
Mousseaux-Neuville France 2001 Capitole [b]	2 (49)	190 175	325 300	BBCH89	56	Seed	0.02
Bouloc France 2000 Olara [c]	2 (37)	175 175	300 300	BBCH80	41	Pod	<0.05
				BBCH87	56	Seed	<0.01
				BBCH89	65	Seed	<0.01
Saint Paul les Romans France 2000 Ebonite [c]	2 (53)	175 175	300 300	BBCH88	42	Pod	0.15
				BBCH89	55	Seed	<0.01
Lusignan France 2000 Capitole [d]	2 (52)	175 175	300 300	BBCH89	56	Seed	0.01

RAPE Location, year, variety	N (int)	Rate g ai/ha	Vol L/ha	Sampling GS	DALA	Sample	Residues (mg/kg) Prothioconazole-desthio
Maire France 2000 Constant [d]	2 (55)	175 175	300 300	BBCH89	56	Seed	0.01

[a] ⑧2088/00, method 00647 for prothioconazole-desthio

[b] ⑤2178/01, method 00647 for prothioconazole-desthio

[c] ⑥2089/00, method 00647 for prothioconazole-desthio

[d] ⑦2179/01, method 00647 for prothioconazole-desthio

Flax

Table 12 Residues of prothioconazole in flax seed following foliar application of prothioconazole

FLAX	N (int)	Rate g ai/ha	sampling GS	DALA	Residue (mg/kg expressed as prothioconazole-desthio)					
					Desthio	α-OH	3-OH	4-OH	5-OH	6-OH
Les Forges Bretagne France, 2009 Everest [a]	2 (13)	175 175	BBCH75	34	0.09	<0.01	<0.01	0.03	<0.01	<0.01
Chaussy France, 2010 Drakkar [b]	2 (14)	175 175	BBCH67	34	0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Villers-Perwin Belgium, 2010 Agatha [b]	2 (14)	175 193	BBCH65	35	0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Melbourne UK, 2010 Aries [c]	2 (14)	175 175	BBCH65	37	0.03	<0.01	<0.01	<0.01	<0.01	<0.01

[a] 09-2130, method 01013 for prothioconazole-desthio, method 00979 for hydroxy metabolites

[b] 10-2073, method 01013 for prothioconazole-desthio, method 00979 for hydroxy metabolites

[c] 10-2254, method 01013 for prothioconazole-desthio, method 00979 for hydroxy metabolites

Table 13 Residues of triazole-metabolites in flax seed following foliar application of prothioconazole

FLAX Location, year, variety	N (int)	Rate g ai/ha	Vol L/ha	sampling GS	DALA	Residue (mg/kg)			
						1,2,4-triazole	TAA	TLA	TA
Melbourne UK, 2010 Aries [a]	2 (14)	175 175	200 200	BBCH89	37	0.03	<0.01	0.027 C0.041	0.09 C0.013
Les Forges Bretagne France, 2009 Everest [b]	2 (13)	175 175	200 200	BBCH89	34	<0.01	<0.01	0.192 C0.164	0.020 C0.018
Chaussy France, 2010 Drakkar [c]	2 (14)	175 175	200 200	BBCH89	34	<0.01	<0.01	0.04 C0.03	<0.01
Villers-Perwin Belgium, 2010 Agatha [c]	2 (14)	175 193	200 221	BBCH89	35	<0.01	<0.01	0.04 C0.03	<0.01

[a] 10-2254, method 01062

[b] 09-2130, method 01062

[c] 10-2073, method 01062

C – control sample

Sunflower

Table 14 Residues of prothioconazole in sunflower seed following foliar application

SUNFLOWER	N	Rate	Sampling	DALA	sample	Residue (mg/kg expressed as prothioconazole-desthio)					
Location, year, variety	(int)	g ai/ha	GS			Desthio	α-OH	3-OH	4-OH	5-OH	6-OH
Burscheid Germany, 2010 Rigasol [c]	2 (15)	125 125	BBCH89	28	Seed	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Frasnes-Lez-Gosselies Belgium, 2010 LG 54.50 HO [c]	2 (15)	125 125	BBCH89	27	Seed	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Gallikos, Kilkis Greece, 2010 Sanay MP [c]	2 (14)	125 125	BBCH89	28	Seed	0.02	<0.01	<0.01	<0.01	<0.01	<0.01
Fuentes de Andalucia Spain, 2010 Transol [e]	2 (14)	125 125	BBCH89	27	Seed	0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Burscheid Germany, 2011 Rigasol [d]	2 (14)	125 125	BBCH76	21	Seed	<0.01	<0.01	<0.01	<0.01	<0.01	
			BBCH79	24	Seed	<0.01	<0.01	<0.01	<0.01	<0.01	
			BBCH81	28	Seed	<0.01	<0.01	<0.01	<0.01	<0.01	
			BBCH83	31	Seed	<0.01	<0.01	<0.01	<0.01	<0.01	
			BBCH89	35	seed	<0.01	<0.01	<0.01	<0.01	<0.01	
			BBCH81	28	Kernel	<0.01	<0.01	<0.01	<0.01	<0.01	
			BBCH81	28	Seed, partly shelled	<0.01	<0.01	<0.01	<0.01	<0.01	
Villers-Perwin Belgium, 2011 P64HE01 (XF4636); hybrid sunflower (experimental) [d]	2 (13)	125 125	BBCH83	21	Seed	<0.01	<0.01	<0.01	<0.01	<0.01	
			BBCH86	23	Seed	<0.01	<0.01	<0.01	<0.01	<0.01	
			BBCH89	28	Seed	<0.01	<0.01	<0.01	<0.01	<0.01	
			BBCH89	30	Seed	<0.01	<0.01	<0.01	<0.01	<0.01	
			BBCH89	35	seed	<0.01	<0.01	<0.01	<0.01	<0.01	
			BBCH89	28	Kernel	<0.01	<0.01	<0.01	<0.01	<0.01	
			BBCH89	28	Seed, partly shelled	<0.01	<0.01	<0.01	<0.01	<0.01	

SUNFLOWER Location, year, variety	N (int)	Rate g ai/ha	Sampling GS	DALA	sample	Residue (mg/kg expressed as prothioconazole-desthio)					
						Desthio	α-OH	3-OH	4-OH	5-OH	6-OH
Kissa, Kozani Greece, 2011 Sanay [d]	2 (14)	125 125	BBCH87	21	Seed	<0.01	<0.01	<0.01	<0.01	<0.01	
			BBCH87	24	Seed	0.011	<0.01	<0.01	<0.01	<0.01	
			BBCH89	28	Seed	0.017	<0.01	<0.01	<0.01	<0.01	
			BBCH89	31	Seed	<0.01	<0.01	<0.01	<0.01	<0.01	
			BBCH89	35	seed	<0.01	<0.01	<0.01	<0.01	<0.01	
			BBCH89	28	Kernel	<0.01	<0.01	<0.01	<0.01	<0.01	
			BBCH89	28	Seed, partly shelled	<0.01	<0.01	<0.01	<0.01	<0.01	
Gargas France, 2011 Tekny [d]	2 (13)	125 125	BBCH87	21	Seed	<0.01	<0.01	<0.01	<0.01	<0.01	
			BBCH87	23	Seed	<0.01	<0.01	<0.01	<0.01	<0.01	
			BBCH89	28	Seed	<0.01	<0.01	<0.01	<0.01	<0.01	
			BBCH89	30	Seed	<0.01	<0.01	<0.01	<0.01	<0.01	
			BBCH89	35	seed	<0.01	<0.01	<0.01	<0.01	<0.01	
			BBCH89	28	Kernel	<0.01	<0.01	<0.01	<0.01	<0.01	
			BBCH89	28	Seed, partly shelled	<0.01	<0.01	<0.01	<0.01	<0.01	
Kiskunfélegyháza Hungary, 2012 NSP IMI [b]	2 (15)	125 125	BBCH90	20	Seed	0.20	<0.01	<0.01	0.024	<0.01	<0.01
			BBCH90	22	Seed	0.10	<0.01	<0.01	<0.01	<0.01	<0.01
			BBCH90	27	Seed	0.11 0.14 (0.13)	<0.01	<0.01	<0.01	<0.01	<0.01
			BBCH90	27	Kernel	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
			BBCH90	27	Seed, partly shelled	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Dánszentmiklós Hungary, 2012 PR36 E82 [b]	2 (16)	125 125	BBCH89	21	Seed	0.014	<0.01	<0.01	<0.01	<0.01	<0.01
			BBCH90	29	Seed	0.042 0.043 (0.043)	<0.01	<0.01	<0.01	<0.01	<0.01
			BBCH90	31	Seed	0.041	<0.01	<0.01	<0.01	<0.01	<0.01
			BBCH90	35	Seed	0.035	<0.01	<0.01	0.014	<0.01	<0.01
			BBCH90	29	Kernel	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
			BBCH90	29	Seed, partly shelled	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01

SUNFLOWER Location, year, variety	N (int)	Rate g ai/ha	Sampling GS	DALA	sample	Residue (mg/kg expressed as prothioconazole-desthio)					
						Desthio	α-OH	3-OH	4-OH	5-OH	6-OH
Tarascon France, 2012 CSF10902 [a]	2 (14)	125 125	BBCH89	21	Seed	0.010	<0.01	<0.01	<0.01	<0.01	<0.01
			BBCH89	24	Seed	0.026	<0.01	<0.01	<0.01	<0.01	<0.01
			BBCH89	28	Seed	0.034 0.036 (0.035)	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01
			BBCH89	31	Seed	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
			BBCH89	35	Seed	0.011	<0.01	<0.01	<0.01	<0.01	<0.01
			BBCH89	29	Kernel	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
			BBCH89	29	Seed, partly shelled	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Dos Hermanas Spain, 2012 PR64H37 [a]	2 (14)	125 125	BBCH85	21	Seed	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
			BBCH85	24	Seed	0.012	<0.01	<0.01	<0.01	<0.01	<0.01
			BBCH87	28	Seed	<0.01 <0.01	<0.01	<0.01	<0.01	<0.01	<0.01
			BBCH87	31	Seed	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
			BBCH89	35	Seed	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
			BBCH87	28	Kernel	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
			BBCH87	28	Seed, partly shelled	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Furbara Cerveteri (RM) Italy, 2012 Starsol [a]	2 (14)	125 125	BBCH87	21	Seed	0.011	<0.01	<0.01	<0.01	<0.01	<0.01
			BBCH89	24	Seed	0.013	<0.01	<0.01	<0.01	<0.01	<0.01
			BBCH89	28	Seed	0.01 0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01
			BBCH89	31	Seed	0.01	<0.01	<0.01	<0.01	<0.01	<0.01
			BBCH89	35	Seed	0.01	<0.01	<0.01	<0.01	<0.01	<0.01
			BBCH89	28	Kernel	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
			BBCH89	28	Seed, partly shelled	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Aramanha-Várzea Portugal, 2012 PR 64H47; Hybrid autoleic [a]	2 (15)	125 125	BBCH87	21	Seed	0.020	<0.01	<0.01	<0.01	<0.01	<0.01
			BBCH87	24	Seed	0.012	<0.01	<0.01	<0.01	<0.01	<0.01
			BBCH89	28	Seed	0.012 0.021 (0.017)	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01
			BBCH89	31	Seed	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01

SUNFLOWER Location, year, variety	N (int)	Rate g ai/ha	Sampling GS	DALA	sample	Residue (mg/kg expressed as prothioconazole-desthio)					
						Desthio	α-OH	3-OH	4-OH	5-OH	6-OH
			BBCH89	35	Seed	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
			BBCH89	28	Kernel	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
			BBCH89	28	Seed, partly shelled	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Kissa, Kozani Greece, 2012 PR64LE20 [a]	2 (14)	125 125	BBCH86	21	Seed	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
			BBCH86	24	Seed	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
			BBCH87	28	Seed	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01
			BBCH89	31	Seed	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
			BBCH89	35	Seed	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
			BBCH87	28	Kernel	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
			BBCH87	28	Seed, partly shelled	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Bologna Italy, 2012 PR64H41 [a]	2 (15)	125 125	BBCH87	20	Seed	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
			BBCH87	23	Seed	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
			BBCH89	27	Seed	<0.01 0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01
			BBCH89	30	Seed	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
			BBCH89	34	Seed	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
			BBCH89	27	Kernel	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
			BBCH89	27	Seed, partly shelled	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Mesnil Milon France, 2012 Vellox [f]	2 (14)	125 125	BBCH87	21	Seed	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
			BBCH87	24	Seed	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
			BBCH89	28	Seed	<0.01 <0.01	<0.01	<0.01	<0.01	<0.01	<0.01
			BBCH89	31	Seed	<0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01
			BBCH92	35	Seed	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
			BBCH89	28	Kernel	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
			BBCH89	28	Seed, partly shelled	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01

SUNFLOWER Location, year, variety	N (int)	Rate g ai/ha	Sampling GS	DALA	sample	Residue (mg/kg expressed as prothioconazole-desthio)					
						Desthio	α-OH	3-OH	4-OH	5-OH	6-OH
Marbais Belgium, 2012 P64HE01 (XF4636); 024 - hybrid sunflower [f]	2 (14)	125 125	BBCH87	21	Seed	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
			BBCH87	25	Seed	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
			BBCH89	28	Seed	<0.01 <0.01	<0.01	<0.01	<0.01	<0.01	<0.01
			BBCH89	32	Seed	<0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01
			BBCH89	35	Seed	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
			BBCH89	28	Kernel	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
			BBCH89	28	Seed, partly shelled	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01

[a] 12-2009, method 01013 for prothioconazole-desthio, method 00979 for hydroxy metabolites

[b] 12-2156, method 01013 for prothioconazole-desthio, method 00979 for hydroxy metabolites

[c] 10-2238, method 01013 for prothioconazole-desthio, method 00979 for hydroxy metabolites

[d] 11-2002, method 01013 for prothioconazole-desthio, method 00979 for hydroxy metabolites

[e] 10-2247, method 01013 for prothioconazole-desthio, method 00979 for hydroxy metabolites

[f] 12-2008, method 01013 for prothioconazole-desthio, method 00979 for hydroxy metabolites

Table 15 Residues of triazole-metabolites in sunflower seed following foliar application of prothioconazole

SUNFLOWER Location, year, variety	N (int)	Rate g ai/ha	Vol L/ha	sampling GS	DALA	Sample	Residue (mg/kg)			
							1,2,4-triazole	TAA	TLA	TA
Tarascon France, 2012 CSF10902 [a]	2 (14)	125 125	300 300	BBCH89	21	Seed	<0.01	<0.01	<0.01	<0.01
				BBCH89	24	Seed	<0.01	<0.01	<0.01	<0.01
				BBCH89	28	Seed	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01
				BBCH89	31	Seed	<0.01	<0.01	<0.01	<0.01
				BBCH89	35	Seed	<0.01	<0.01	<0.01	<0.01
				BBCH89	29	Kernel	<0.01	<0.01	<0.01	<0.01
				BBCH89	29	Seed, partly shelled	<0.01	<0.01	<0.01	<0.01
Dos Hermanas Spain, 2012 PR64H37 [a]	2 (14)	125 125	300 300	BBCH85	21	Seed	<0.01	0.038		<0.01
				BBCH85	24	Seed	<0.01	0.032		<0.01
				BBCH87	28	Seed	<0.01 <0.01	0.041 0.039 C0.013	0.037 0.039	<0.01 <0.01

SUNFLOWER Location, year, variety	N (int)	Rate g ai/ha	Vol L/ha	sampling GS	DALA	Sample	Residue (mg/kg)			
							1,2,4-triazole	TAA	TLA	TA
				BBCH87	31	Seed	<0.01	0.033	0.041	<0.01
				BBCH89	35	Seed	<0.01	0.036	0.044	<0.01
				BBCH87	28	Kernel	<0.01	0.056	0.050	<0.01
				BBCH87	28	Seed, partly shelled	<0.01	0.045	0.043	<0.01
Furbara Cerveteri (RM) Italy, 2012 Starsol [a]	2 (14)	125 125	400 400	BBCH87	21	Seed	<0.01	0.016		<0.01
				BBCH89	24	Seed	<0.01	0.016		<0.01
				BBCH89	28	Seed	<0.01 <0.01	0.017 0.014 C0.031	0.038 0.034	<0.01 <0.01 C0.012
				BBCH89	31	Seed	<0.01	0.019	0.035	<0.01
				BBCH89	35	Seed	<0.01	0.017	0.037	<0.01
				BBCH89	28	Kernel	<0.01	0.024	0.051	<0.01
				BBCH89	28	Seed, partly shelled	<0.01	0.018	0.037	<0.01
Aramanha-Várzea Portugal, 2012 PR 64H47, Hybrid autoleic [a]	2 (15)	125 125	300 300	BBCH87	21	Seed	<0.01	0.015	0.015	<0.01
				BBCH87	24	Seed	<0.01	0.017	0.016	<0.01
				BBCH89	28	Seed	<0.01 <0.01	0.018 0.015	0.018 0.015	<0.01 <0.01
				BBCH89	31	Seed	<0.01	0.017	0.019	<0.01
				BBCH89	35	Seed	<0.01	0.015	0.014	<0.01
				BBCH89	28	Kernel	<0.01	0.023	0.027	<0.01
				BBCH89	28	Seed, partly shelled	<0.01	0.015	0.020	<0.01
Kissa, Kozani Greece, 2012 PR64LE20 [a]	2 (14)	125 125	400 400	BBCH86	21	Seed	<0.01	0.014	<0.01	<0.01
				BBCH86	24	Seed	<0.01	0.015	<0.01	<0.01
				BBCH87	28	Seed	<0.01 <0.01	0.010 0.013	<0.01 <0.01	<0.01 <0.01
				BBCH89	31	Seed	<0.01	0.011	<0.01	<0.01
				BBCH89	35	Seed	<0.01	0.012	<0.01	<0.01
				BBCH87	28	Kernel	<0.01	0.013	<0.01	<0.01
				BBCH87	28	Seed, partly shelled	<0.01	0.013	<0.01	<0.01

SUNFLOWER Location, year, variety	N (int)	Rate g ai/ha	Vol L/ha	sampling GS	DALA	Sample	Residue (mg/kg)			
							1,2,4-triazole	TAA	TLA	TA
Bologna Italy, 2012 PR64H41 [a]	2 (15)	125 125	374 400	BBCH87	20	Seed	<0.01	0.017	0.012	<0.01
				BBCH87	23	Seed	<0.01	0.029	0.020	<0.01
				BBCH89	27	Seed	<0.01 <0.01	0.014 0.034	0.010 0.025	<0.01 <0.01
				BBCH89	30	Seed	<0.01	0.014	0.012	<0.01
				BBCH89	34	Seed	<0.01	0.016	0.013	<0.01
				BBCH89	27	Kernel	<0.01	0.037	0.025	<0.01
				BBCH89	27	Seed, partly shelled	<0.01	0.026	0.023	<0.01
Kiskunfélegyháza Hungary, 2012 NSP IMI [b]	2 (15)	125 125	180 180	BBCH90	20	Seed	<0.01	0.062	0.053	<0.01
				BBCH90	22	Seed	<0.01	0.052	0.052	<0.01
				BBCH90	27	Seed	<0.01 <0.01	0.056 0.050 C0.028	0.047 0.043	<0.01 <0.01 C0.035
				BBCH90	27	Kernel	<0.01	0.072 C0.036	0.054	<0.01 C0.043
				BBCH90	27	Seed, partly shelled	<0.01	0.056 C0.031	0.047	<0.01 C0.034
Dánszentmiklós Hungary, 2012 PR36 E82 [b]	2 (16)	125 125	180 180	BBCH89	21	Seed	<0.01	0.21	0.17	<0.01
				BBCH90	29	Seed	<0.01 <0.01	0.15 0.13 C0.037	0.11 0.092	<0.01 C0.041
				BBCH90	31	Seed	<0.01	0.062	0.049	<0.01
				BBCH90	35	Seed	<0.01	0.077	0.062	<0.01
				BBCH90	29	Kernel	<0.01	0.20	0.15	<0.01
				BBCH90	29	Seed, partly shelled	<0.01	0.16	0.13	<0.01
Burscheid Germany, 2010 Rigasol [c]	2 (15)	125 125	300 300	BBCH89	28	Seed	<0.01	0.074 C0.048	<0.01	0.181 C0.062
Frasnes-Lez-Gosselies Belgium, 2010 LG 54.50 HO [c]	2 (15)	125 125	275 275	BBCH89	27	Seed	<0.01	0.284 C0.205	0.032 C0.025	0.478 C0.232
Gallikos, Kilkis Greece, 2010 Sanay MP [c]	2 (14)	125 125	400 400	BBCH89	28	Seed	<0.01	0.130 C0.086	<0.01	0.105 C0072

SUNFLOWER Location, year, variety	N (int)	Rate g ai/ha	Vol L/ha	sampling GS	DALA	Sample	Residue (mg/kg)			
							1,2,4-triazole	TAA	TLA	TA
Burscheid Germany, 2011 Rigasol [d]	2 (14)	125 125	300 300	BBCH76	21	Seed	<0.01	<0.01	<0.01	0.022
				BBCH79	24	Seed	<0.01	0.014	<0.01	0.033
				BBCH81	28	Seed	<0.01	0.012 c0.015	<0.01	0.025 C0.023
				BBCH83	31	Seed	<0.01	0.012	<0.01	0.033
				BBCH89	35	seed	<0.01	0.015	<0.01	0.036
				BBCH81	28	Kernel	<0.01	0.024 C0.017	<0.01	0.033 C0.020
				BBCH81	28	Seed, partly shelled	<0.01	0.029 C0.023	<0.01	0.031 C0.017
Villers-Perwin Belgium, 2011 P64HE01 (XF4636); hybrid sunflower (experimental) [d]	2 (13)	125 125	275 275	BBCH83	21	Seed	<0.01	0.022	<0.01	0.085
				BBCH86	23	Seed	<0.01	0.020	<0.01	0.085
				BBCH89	28	Seed	<0.01	0.023	<0.01	0.067 C0.017
				BBCH89	30	Seed	<0.01	0.023	<0.01	0.070
				BBCH89	35	seed	<0.01	0.027	<0.01	0.073
				BBCH89	28	Kernel	<0.01	0.032 C0.011	<0.01	0.047 C0.012
				BBCH89	28	Seed, partly shelled	<0.01	0.040 C0.012	<0.01	0.046 C0.010
Kissa, Kozani Greece, 2011 Sanay [d]	2 (14)	125 125	400 400	BBCH87	21	Seed	<0.01	<0.01	<0.01	0.011
				BBCH87	24	Seed	<0.01	<0.01	<0.01	<0.01
				BBCH89	28	Seed	<0.01	<0.01	<0.01	0.014
				BBCH89	31	Seed	<0.01	<0.01	<0.01	<0.01
				BBCH89	35	seed	<0.01	<0.01	<0.01	0.014
				BBCH89	28	Kernel	<0.01	0.403	<0.01	0.236
				BBCH89	28	Seed, partly shelled	<0.01	0.018	<0.01	0.015
Gargas France, 2011 Tekny [d]	2 (13)	125 125	300 300	BBCH87	21	Seed	<0.01	0.683	0.014	0.640
				BBCH87	23	Seed	<0.01	0.594	0.013	0.546

SUNFLOWER Location, year, variety	N (int)	Rate g ai/ha	Vol L/ha	sampling GS	DALA	Sample	Residue (mg/kg)			
							1,2,4-triazole	TAA	TLA	TA
				BBCH89	28	Seed	<0.01	0.658 C0.436	0.015 C0.01	0.597 C0.407
				BBCH89	30	Seed	<0.01	0.669	0.015	0.542
				BBCH89	35	seed	<0.01	0.595	0.016	0.596
				BBCH89	28	Kernel	<0.01	0.788 C0.498	0.010	0.420 C0.293
				BBCH89	28	Seed, partly shelled	<0.01	0.637 C0.402	<0.01	0.370 C0.251
Fuentes de Andalucia Spain, 2010 Transol [e]	2 (14)	125 125	300 300	BBCH89	27	Seed	<0.01	0.253 C0.278	<0.01	0.09 C0.126
Mesnil Milon France, 2012 Vellox [f]	2 (14)	125 125	300 300	BBCH87	21	Seed	<0.01	0.070	<0.01	0.036
				BBCH87	24	Seed	<0.01	0.087	<0.01	0.055
				BBCH89	28	Seed	<0.01 <0.01	0.079 0.091 C0.14	<0.01 <0.01	0.042 0.055 C0.074
				BBCH89	31	Seed	<0.01	0.080	<0.01	0.054
				BBCH92	35	Seed	<0.01	0.080	<0.01	0.048
				BBCH89	28	Kernel	<0.01	0.089 C0.19	<0.01	0.061 C0.15
				BBCH89	28	Seed, partly shelled	<0.01	0.078 C0.16	<0.01	0.049 C0.11
Marbais Belgium, 2012 P64HE01 (XF4636); 024 - hybrid sunflower [f]	2 (14)	125 125	260 275	BBCH87	21	Seed	<0.01	<0.01	<0.01	0.015
				BBCH87	25	Seed	<0.01	0.010	<0.01	0.017
				BBCH89	28	Seed	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	0.014 0.017
				BBCH89	32	Seed	<0.01	0.010	<0.01	0.015
				BBCH89	35	Seed	<0.01	<0.01	<0.01	0.017
				BBCH89	28	Kernel	<0.01	0.011	<0.01	0.016
				BBCH89	28	Seed, partly shelled	<0.01	0.012	<0.01	0.018

[a] 12-2009, method 01062

[b] 12-2156, method 01062

[c] 10-2238, method 01062

[d] 11-2002, method 01062

[e] 10-2247, method 01062

[f] 12-2008, method 01062

C-control sample

Table 16 Residues of prothioconazole in sunflower seed from trials conducted in Brazil¥

Location, year, variety	N	Rate	Vol	Sampling	DALA	Residues (mg/kg as prothioconazole-desthio)		
SUNFLOWER	(int)	g ai/ha	L/ha	GS		Prothioconazole	desthio	Sum of prothioconazole and desthio
Paulínia Brazil, 2012 Catissol 01 [a]	2 (14)	93.1 94.9	150 150	BBCH92	25	<0.009	<0.009	<0.018
				BBCH92	30	<0.009	<0.009	<0.018
				BBCH92	35	<0.009	<0.009	<0.018
Uberlândia Brazil, 2012 Helio 152 [a]	2 (14)	84.4 85.6	150 150	BBCH85	25	<0.009	<0.009	<0.018
				BBCH87	30	0.009	0.009	0.018
				BBCH89	35	<0.009	<0.009	<0.018
Ribeirão Preto Brazil, 2012 BRS 321 [a]	2 (14)	94.0 87.5	150 150	BBCH87	25	0.036	0.063	0.099
				BBCH87	30	<0.009	0.018	0.027
				BBCH87	35	0.027	0.063	0.090
Piracicaba Brazil, 2012 Catissol [a]	2 (14)	91.4 88.7	150 150	BBCH92	30	<0.009	<0.009	<0.018
Jaboticabal Brazil, 2012 BRS 321 [a]	2 (14)	91.4 89.4	150 150	BBCH87	30	0.77	0.32[#]	1.09
Paulínia Brazil, 2013 Catissol [b]	2 (14)	87.5 87.5	150 150	BBCH85	15	<0.009	<0.009	<0.018
				BBCH85	20	<0.009	<0.009	<0.018
				BBCH87	25	<0.009	0.009	<0.018
				BBCH89	30	<0.009	<0.009	<0.018
				BBCH91	35	<0.009	<0.009	<0.018
Uberlândia Brazil, 2013 SYN 042 [b]	2 (14)	88.0 85.1	150 150	BBCH87	15	<0.009	<0.009	<0.018
				BBCH88	20	0.018	0.045	0.063
				BBCH90	25	<0.009	<0.009	<0.018
				BBCH90	30	<0.009	<0.009	<0.018
				BBCH92	35	<0.009	<0.009	<0.018
Ribeirão Preto Brazil, 2013 M-734 [b]	2 (14)	89.4 88.5	150 150	BBCH85	15	<0.009	0.018	0.027
				BBCH85	20	0.045	0.17	0.215

Location, year, variety	N	Rate	Vol	Sampling	DALA	Residues (mg/kg as prothioconazole-desthio)		
SUNFLOWER	(int)	g ai/ha	L/ha	GS		Prothioconazole	desthio	Sum of prothioconazole and desthio
				BBCH87	25	0.009	0.036	0.045
				BBCH87	30	<0.009	0.027	0.036
				BBCH87	35	<0.009	0.036	0.045
Ponta Grossa Brazil, 2013 Catissol [b]	2 (14)	94.2 87.1	150 150	BBCH85	15	<0.009	<0.009	<0.018
				BBCH86	20	<0.009	<0.009	<0.018
				BBCH88	25	<0.009	<0.009	<0.018
				BBCH88	29	<0.009	<0.009	<0.018
				BBCH88	33	<0.009	<0.009	<0.018
Ituverava Brazil, 2013 SYN 042 [b]	2 (14)	87.5 84.3	150 150	BBCH85	15	<0.009	<0.009	<0.018
				BBCH86	20	<0.009	<0.009	<0.018
				BBCH88	25	<0.009	<0.009	<0.018
				BBCH89	30	<0.009	<0.009	<0.018
				BBCH90	34	<0.009	<0.009	<0.018

[¥] in the original report residues were reported in terms of prothioconazole. For consistency, the residues have been converted to prothioconazole-desthio equivalents by making molecular weight corrections (prothioconazole = 344.27 g/mol while prothioconazole-desthio = 312.2 g/mol).

[#] Replicate analyses of the same sample were performed, and the mean value is presented.

[a] F12-030, method 01013 for prothioconazole and prothioconazole-desthio

[b] F13-006, method 01013 for prothioconazole and prothioconazole-desthio

Animal feeds

Rape forage

Table 17 Residues of prothioconazole in oilseed rape plants following foliar application

RAPE PLANT	N	Rate	Sampling	DALA	Sample		Residue (mg/kg expressed as prothioconazole-desthio)					
Location, year, variety	(int)	g ai/ha	GS			Prothio	Desthio	α-OH	3-OH	4-OH	5-OH	6-OH
Chambourg sur Indre Centre, France, 2008 Flash **	2 (14)	120 120	BBCH80	0*	Green material		0.05	<0.01	0.04	0.01	<0.01	<0.01
			BBCH80	0	Green material		0.62	<0.01	0.05	0.02	<0.01	<0.01
			BBCH82	21	Rest of plant		0.04	<0.01	0.10	0.02	<0.01	<0.01
Burscheid Nordrhein-Westfalen,	2 (14)	120 120	BBCH73	0*	Green material		0.04	<0.01	0.03	0.01	<0.01	<0.01

RAPE PLANT Location, year, variety	N (int)	Rate g ai/ha	Sampling GS	DALA	Sample	Residue (mg/kg expressed as prothioconazole-desthio) Prothio	Desthio	α-OH	3-OH	4-OH	5-OH	6-OH
Germany, 2008 Titan												
**			BBCH73	0	Green material		0.57	<0.01	0.04	0.01	<0.01	<0.01
Cortil-Noirmont Brabant Wallon, Belgium, 2008 Exocet	2 (14)	120 120	BBCH80	0*	Green material		0.08	<0.01	0.05	0.02	<0.01	<0.01
**			BBCH80	0	Green material		0.53	<0.01	0.06	0.02	<0.01	<0.01
			BBCH87	21	Rest of plant		0.04	<0.01	0.08	0.02	<0.01	<0.01
Werl-Niederbergstraße Nordrhein-Westfalen, Germany, 2008 Astrid	2 (14)	120 120	BBCH78	0*	Green material		0.10	<0.01	0.07	0.02	<0.01	<0.01
**			BBCH78	0	Green material		0.67	0.01	0.09	0.03	<0.01	<0.01
Cherves, France, 2008 Ovation [a]	2 (14)	120 120	BBCH80	0*	Green material		0.08	<0.01	0.06	0.02	<0.01	<0.01
			BBCH80	0	Green material		0.55	<0.01	0.07	0.02	<0.01	<0.01
			BBCH83	21	Rest of plant		0.03	<0.01	0.08	0.02	<0.01	<0.01
L´Ametlla del Valles, Spain, 2008 Silvia [a]	2 (14)	120 120	BBCH73	0*	Green material		0.05	<0.01	0.04	0.02	<0.01	<0.01
			BBCH73	0	Green material		1.00	<0.01	0.04	0.02	<0.01	<0.01
			BBCH79	21	Rest of plant		0.03	<0.01	0.09	0.03	<0.01	<0.01
Fleurieux sur l'Arbesle, Rhone-Alpes, France, 2009 Artiste [b]	2 (13)	120 120	BBCH85	0*	Green material		0.19	<0.01	0.07	0.03	<0.01	<0.01
			BBCH85	0	Green material		1.3	<0.01	0.07	0.04	<0.01	<0.01
			BBCH88	14	Rest of plant		0.28 0.33	<0.01	0.07	0.03	<0.01	<0.01
			BBCH89	20	Rest of plant		0.18	<0.01	0.06	0.02	<0.01	<0.01
San Giogio Di Piano, Emilia-	2 (14)	120 120	BBCH80	0*	Green material		0.07	<0.01	0.06	0.02	<0.01	<0.01

RAPE PLANT	N	Rate	Sampling	DALA	Sample		Residue (mg/kg expressed as prothioconazole-desthio)					
Location, year, variety	(int)	g ai/ha	GS			Prothio	Desthio	α-OH	3-OH	4-OH	5-OH	6-OH
Romagna, Italy, 2009 PR46W09 [b]												
			BBCH80	0	Green material		0.80	<0.01	0.08	0.03	<0.01	<0.01
Braslou, France, 2009 Arcadia [c]	2 (14)	120 120	BBCH77	0*	Green material		0.06	<0.01	0.06	0.02	<0.01	<0.01
			BBCH77	0	Green material		0.66	<0.01	0.09	0.04	<0.01	<0.01
			BBCH83	14	Rest of plant		0.06	<0.01	0.08	0.03	<0.01	<0.01
			BBCH85	21	Rest of plant		0.04	<0.01	0.10	0.03	<0.01	<0.01
Cortil-Noirmont, Belgium, 2009 Monalisa [c]	2 (14)	120 120	BBCH79	0*	Green material		0.13	<0.01	0.06	0.03	<0.01	<0.01
			BBCH79	0	Green material		0.54	<0.01	0.07	0.03	<0.01	<0.01
			BBCH81	14	Rest of plant		0.08	<0.01	0.11	0.03	0.01	<0.01
			BBCH82	21	Rest of plant		0.05	<0.01	0.15	0.04	0.01	<0.01
Nieuw Beerta, Netherlands, 2009 unknown [c]	2 (14)	120 120	BBCH85	0*	Green material		0.13	0.02	0.16	0.04	0.01	<0.01
			BBCH85	0	Green material		0.84	0.02	0.14	0.04	0.01	<0.01
Bishop Burton UK 2009 Castille [o] Harvest 13/8/2009	2 (15)	120 120	BBCH75	0*	Green material		0.09	<0.01	0.07 C0.03	0.02	<0.01	<0.01
			BBCH75	0	Green material		0.46	<0.01	0.05	0.02	<0.01	<0.01
			BBCH83	29	Rest of plant		0.02 c0.05	<0.01	0.08 C0.01	0.02	<0.01	<0.01
Cortil-Noirmont, Belgium, 2010 Exocet [d]	2 (15)	125 125	BBCH78	0*	Green material		0.10	<0.01	0.03	<0.01	<0.01	<0.01
			BBCH78	0	Green material		0.27	<0.01	0.04	<0.01	<0.01	<0.01
			BBCH80	14	Rest of plant		0.22	<0.01	0.10	0.01	0.01	<0.01
			BBCH81	21	Rest of plant		0.05	<0.01	0.07	0.02	<0.01	<0.01
Royston UK, 2010 Cabernet [d]	2 (13)	125 125	BBCH78	0*	Green material		0.23	<0.01	0.04	0.02	<0.01	<0.01
			BBCH78	0	Green material		0.50	<0.01	0.04	0.02	<0.01	<0.01

RAPE PLANT	N	Rate	Sampling	DALA	Sample		Residue (mg/kg expressed as prothioconazole-desthio)					
Location, year, variety	(int)	g ai/ha	GS			Prothio	Desthio	α-OH	3-OH	4-OH	5-OH	6-OH
			BBCH79	14	Rest of plant		0.28	0.01	0.11	0.04	0.01	<0.01
			BBCH85	22	Rest of plant		0.19	<0.01	0.12	0.04	0.01	<0.01
Slootdrop The Netherlands, 2010 Haydn [d]	2 (14)	125 125	BBCH73	0*	Green material		0.06	<0.01	0.05	0.02	<0.01	<0.01
			BBCH73	0	Green material		0.52	<0.01	0.09	0.03	<0.01	<0.01
			BBCH85	14	Rest of plant		0.09	<0.01	0.09	0.04	<0.01	<0.01
			BBCH88	21	Rest of plant		0.04	<0.01	0.07	0.03	<0.01	<0.01
Bouafle France, 2010 Olindigo [d]	2 (14)	125 125	BBCH82	0*	Green material		0.59	0.02	0.22	0.13	0.02	<0.01
			BBCH82	0	Green material		1.4	<0.01	0.23	0.14	0.02	<0.01
			BBCH88	14	Rest of plant		2.1	0.02	0.24	0.17	0.03	<0.01
Vilobi d'Onyar Spain, 2010 Artist-Hybrid [d]	2 (11)	125 136	BBCH78	0*	Green material		0.04	<0.01	0.03	0.01	<0.01	<0.01
			BBCH78	0	Green material		0.38	<0.01	0.05	0.02	<0.01	<0.01
			BBCH79	14	Rest of plant		0.04	<0.01	0.03	<0.01	<0.01	<0.01
Vouille France, 2010 Aviator [d]	2 (14)	125 125	BBCH78	0*	Green material		0.09	<0.01	0.04	0.01	<0.01	<0.01
			BBCH78	0	Green material		0.43	<0.01	0.05	0.01	<0.01	<0.01
			BBCH80	14	Rest of plant		0.06	<0.01	0.05	0.01	<0.01	<0.01
			BBCH85	21	Rest of plant		0.02	<0.01	0.06	0.01	<0.01	<0.01
Maritima Malaga Spain, 2010 Kavel [e]	2 (14)	125 125	BBCH72	0*	Green material		0.13	<0.01	0.05	0.02	<0.01	<0.01
			BBCH72	0	Green material		0.31	<0.01	0.03	0.01	<0.01	<0.01
			BBCH80	14	Rest of plant		0.05	<0.01	0.09	0.03	<0.01	<0.01
			BBCH83	21	Rest of plant		0.02	<0.01	0.05	0.01	<0.01	<0.01

RAPE PLANT Location, year, variety	N (int)	Rate g ai/ha	Sampling GS	DALA	Sample	Prothio	Residue (mg/kg expressed as prothioconazole-desthio) Desthio	α-OH	3-OH	4-OH	5-OH	6-OH
Gontaud de Nogaret France, 2010 Hybri Star [e]	2 (13)	125 125	BBCH79	0*	Green material		0.26	<0.01	0.12	0.04	<0.01	<0.01
			BBCH79	0	Green material		0.46	<0.01	0.12	0.04	<0.01	<0.01
			BBCH82	14	Rest of plant		0.13	<0.01	0.14	0.05	<0.01	<0.01
			BBCH85	22	Rest of plant		0.05	<0.01	0.20	0.06	<0.01	<0.01
Burscheid Germany, 2011 Elektra [p]	2 (14)	125 125	BBCH79	0*	Green material		0.10	<0.01	0.026	0.01	<0.01	<0.01
			BBCH79	0	Green material		0.34	<0.01	0.027	<0.01	<0.01	<0.01
			BBCH80	14	Rest of plant		0.18	<0.01	0.1	0.031	<0.01	<0.01
			BBCH81	21	Rest of plant		0.099	<0.01	0.099	0.027	<0.01	<0.01
Sombreffe (Vielle-Maison) Belgium, 2011 Limone Hybrid restored oil-rich [p]	2 (14)	125 125	BBCH80	0*	Green material		0.082	<0.01	0.037	0.010	<0.01	<0.01
			BBCH80	0	Green material		0.24	<0.01	0.039	0.012	<0.01	<0.01
			BBCH80	14	Rest of plant		0.081	<0.01	0.064	0.016	<0.01	<0.01
			BBCH82	21	Rest of plant		0.047	<0.01	0.081	0.022	<0.01	<0.01
Royston UK, 2011 FLASH Hybrid [p]	2 (15)	125 125	BBCH79	0*	Green material		0.066	<0.01	0.019	<0.01	<0.01	<0.01
			BBCH79	0	Green material		0.33	<0.01	0.015	<0.01	<0.01	<0.01
			BBCH80	14	Rest of plant		0.059	<0.01	0.035	0.011	<0.01	<0.01
			BBCH84	21	Rest of plant		0.051	<0.01	0.047	0.017	<0.01	<0.01
Chambourg sur Indre France, 2011 Dynastie [p]	2 (14)	125 125	BBCH79	0*	Green material		0.17	<0.01	0.051	0.020	<0.01	<0.01
			BBCH79	0	Green material		0.58	<0.01	0.059	0.025	<0.01	<0.01
			BBCH85	14	Rest of plant		0.099	<0.01	0.065	0.023	<0.01	<0.01
			BBCH87	21	Rest of plant		0.073	<0.01	0.063	0.021	<0.01	<0.01

RAPE PLANT	N	Rate	Sampling	DALA	Sample		Residue (mg/kg expressed as prothioconazole-desthio)					
Location, year, variety	(int)	g ai/ha	GS			Prothio	Desthio	α-OH	3-OH	4-OH	5-OH	6-OH
Les Franqueses del Valles Spain, 2011 Pacific [f]	2 (14)	126 120	BBCH78	0*	Green material		0.029	<0.01	0.034	0.013	<0.01	<0.01
			BBCH78	0	Green material		0.16	<0.01	0.037	0.018	<0.01	<0.01
			BBCH81	14	Rest of plant		0.016	<0.01	0.030	0.012	<0.01	<0.01
			BBCH85	21	Rest of plant		0.039	<0.01	0.046	0.02	<0.01	<0.01
Bouloc France, 2011 Nk Alamir Hybride [f]	2 (13)	120 120	BBCH79	0*	Green material		0.13	<0.01	0.067	0.025	<0.01	<0.01
			BBCH79	0	Green material		1.1	<0.01	0.085	0.039	<0.01	<0.01
Burscheid Germany, 2011 Elektra [f]	2 (14)	120 120	BBCH78	0*	Green material		0.076	<0.01	0.050	0.016	<0.01	<0.01
			BBCH78	0	Green material		0.66	<0.01	0.048	0.015	<0.01	<0.01
Tarquinia Italy, 2011 Hybristar [f]	2 (14)	120 120	BBCH75	0*	Green material		0.060	<0.01	0.051	0.021	<0.01	<0.01
			BBCH75	0	Green material		0.59	<0.01	0.050	0.021	<0.01	<0.01
			BBCH81	14	Rest of plant		0.045	<0.01	0.064	0.023	<0.01	<0.01
			BBCH85	21	Rest of plant		0.042	<0.01	0.097	0.033	<0.01	<0.01
La Luisiana Spain 2011 Eswilliams [f]	2 (18)	120 138	BBCH82	0*	Green material		0.66	0.016	0.15	0.08	0.011	<0.01
			BBCH82	0	Green material		1.4	0.011	0.14	0.065	<0.01	<0.01
Velleron France, 2011 Hybrilux; Rape winter [f]	2 (14)	120 120	BBCH80	0*	Green material		0.13	<0.01	0.06	0.03	<0.01	<0.01
			BBCH80	0	Green material		0.68	<0.01	0.055	0.029	<0.01	<0.01
La Luisiana Spain 2011 Eswilliams [q]	2 (14)	125 125	BBCH82	0*	Green material		0.47	0.020	0.13	0.073	0.014	<0.01
			BBCH82	0	Green material		1.6	0.021	0.13	0.068	0.014	<0.01
			BBCH86	14	Rest of plant		1.0	0.034	0.23	0.11	0.025	<0.01
			BBCH88	21	Rest of plant		1.1	0.034	0.27	0.13	0.032	<0.01

RAPE PLANT Location, year, variety	N (int)	Rate g ai/ha	Sampling GS	DALA	Sample	Residue (mg/kg expressed as prothioconazole-desthio) Prothio	Desthio	α-OH	3-OH	4-OH	5-OH	6-OH
Bouloc France, 2011 Nk Alamir; Hybride [q]	2 (14)	125 125	BBCH79	0*	Green material		0.17	<0.01	0.073	0.038	<0.01	<0.01
			BBCH79	0	Green material		0.88	0.011	0.084	0.048	<0.01	<0.01
			BBCH84	14	Rest of plant		0.17	<0.01	0.12	0.056	0.013	<0.01
			BBCH87	21	Rest of plant		0.15	<0.01	0.12	0.055	0.015	<0.01
Tarquinia (VT) Italy, 2011 Hybristar [q]	2 (14)	125 125	BBCH75	0*	Green material		0.085	<0.01	0.044	0.019	<0.01	<0.01
			BBCH75	0	Green material		0.38	<0.01	0.052	0.022	<0.01	<0.01
			BBCH81	14	Rest of plant		0.076	<0.01	0.10	0.041	<0.01	<0.01
			BBCH85	21	Rest of plant		0.047	<0.01	0.088	0.034	<0.01	<0.01
Les Franqueses del Valles – Llerona Spain, 2011 Pacific [q]	2 (14)	125 125	BBCH78	0*	Green material		0.070	<0.01	0.035	0.018	<0.01	<0.01
			BBCH78	0	Green material		0.15	<0.01	0.036	0.019	<0.01	<0.01
			BBCH81	14	Rest of plant		0.051	<0.01	0.053	0.025	<0.01	<0.01
			BBCH85	21	Rest of plant		0.066	<0.01	0.027	0.012	<0.01	<0.01
Banbury UK, 2011 D.K. Cabernet [m]	2 (14)	120 120	BBCH83	0*	Green material		0.25	0.010	0.11	0.049	<0.01	<0.01
			BBCH83	0	Green material		0.90	0.010	0.12	0.053	0.010	<0.01
Saint Maurice-En-Rivère France, 2016 Manzana [r]	2 (14)	125 138	BBCH79	0*	Green material		0.39	0.020	0.14	0.067	0.015	<0.01
			BBCH79	0	Green material		1.2	0.014	0.13	0.076	0.017	<0.01
			BBCH83	15	Rest of plant		0.085	<0.01	0.056	0.022	<0.01	<0.01
			BBCH88	22	Rest of plant		0.49	0.054	<0.01	0.25	0.019	<0.01
Faulbach Germany, 2016 PR46W20 [r]	2 (14)	125 125	BBCH85	0*	Green material		0.17	0.011	0.079	0.028	<0.01	<0.01
			BBCH85	0	Green material		0.83	0.013	0.086	0.035	0.010	<0.01

RAPE PLANT	N	Rate	Sampling	DALA	Sample		Residue (mg/kg expressed as prothioconazole-desthio)					
Location, year, variety	(int)	g ai/ha	GS			Prothio	Desthio	α-OH	3-OH	4-OH	5-OH	6-OH
			BBCH88	14	Rest of plant		0.12	<0.01	0.090	0.027	<0.01	<0.01
			BBCH89	20	Rest of plant		0.11	<0.01	0.092	0.025	<0.01	<0.01
Mousseaux-Neuville; west of Paris, France, 2002 Capitol [i]	2 (62)	125 125	BBCH73	0	Green material		0.54					
			BBCH89	56	Straw		0.05					
Leichlingen Germany, 2002 Express [i]	2 (58)	125 125	BBCH73	0	Green material		0.52					
			BBCH89	55	Straw		<0.05					
Norton, east of Cambridge, UK, 2002 Madrigal [i]	2 (70)	125 114	BBCH75	0	Green material		0.47					
			BBCH89	54	Straw		<0.05					
Burscheid, Germany, 2002 Licondor [i]	2 (58)	125 125	BBCH73	0	Green material		0.55					
			BBCH87	55	Straw		<0.05					
			BBCH89	62	Straw		<0.05					
Ravenna Italy, 2002 Pegletta [j]	2 (7)	125 125	BBCH63	0	Green material		0.63					
			BBCH87	56	Straw		0.12					
			BBCH89	63	Straw		0.12					
Pujalt Spain, 2002 Bristol [j]	2 (39)	125 125	BBCH65	0	Green material		0.67					
			BBCH89	56	Straw		0.05					
			BBCH89	63	Straw		0.08					
Braslou (Centre) France, 2007 Grizzly [k]	2 (26)	125 125	BBCH73	0*	Green material	0.01	0.04					
			BBCH73	0	Green material	0.58	0.27					
			BBCH79	14	Green material	0.01	0.10					
Burscheid (Nordrhein-Westfalen) Germany, 2007 Elektra [k]	2 (19)	125 125	BBCH73	0*	Green material	0.01	0.07					
			BBCH73	0	Green material	0.62	0.14					

RAPE PLANT	N	Rate	Sampling	DALA	Sample		Residue (mg/kg expressed as prothioconazole-desthio)					
Location, year, variety	(int)	g ai/ha	GS			Prothio	Desthio	α-OH	3-OH	4-OH	5-OH	6-OH
			BBCH79	14	Green material	0.01	0.03					
Werl-Westönnen (Nordrhein-Westfalen) Germany, 2007 Oase [k]	2 (39)	125 125	BBCH73	0*	Green material	<0.01	<0.01					
			BBCH73	0	Green material	0.34	0.13					
			BBCH79	17	Green material	<0.01	0.03					
Near Eye (Suffolk) UK, 2007 Es Astrid [k]	2 (34)	125 125	BBCH73	0*	Green material	<0.01	0.02					
			BBCH73	0	Green material	0.49	0.41					
			BBCH79	25	Green material	<0.01	0.03					
Gargas (Midi-Pyrenees) France, 2007 Corail [l]	2 (20)	125 125	BBCH73	0*	Green material	<0.01	0.06					
			BBCH73	0	Green material	0.10	0.43					
			BBCH79	10	Green material	<0.01	0.05					
Conselice (Ravenna) (Emilia-Romagna) Italy, 2007 Belcanto [l]	2 (29)	125 125	BBCH73	0*	Green material	<0.01	0.03					
			BBCH73	0	Green material	0.19	0.40					
			BBCH79	14	Green material	<0.01	0.11					
Burscheid (Nordrhein-Westfalen), Germany, 2006 Talent [g]	2 (19)	125 125	BBCH73	0*	Green material	0.11	0.11					
			BBCH73	0	Green material	1.5	0.86					
			BBCH79	10	Green material	0.03	0.11					
Chambourg sur Indre (Centre), France, 2006 Standing [g]	2 (15)	125 125	BBCH73	0	Green material	0.56	1.0					
Werl-Westönnen (Nordrhein-Westfalen),	2 (29)	125 125	BBCH63	0*	Green material	<0.01	<0.01					

RAPE PLANT Location, year, variety	N (int)	Rate g ai/ha	Sampling GS	DALA	Sample	Residue (mg/kg expressed as prothioconazole-desthio) Prothio	Desthio	α-OH	3-OH	4-OH	5-OH	6-OH
Germany, 2006 Smart [g]												
			BBCH73	0	Green material	0.69	0.50					
Lund (Scania), Sweden, 2006 Calypso [g]	2 (23)	125 125	BBCH73	0	Green material	0.71	0.66					
Boara (Ferrara) (Emilia-Romagna) Italy, 2006 Molino [h]	2 (24)	125 125	BBCH73	0	Green material	0.94	1.1					
Pujalt (Barcelona) (Cataluña) Spain, 2006 Pacific [h]	2 (34)	125 125	BBCH74	0*	Green material	<0.01	0.03					
			BBCH74	0	Green material	0.49	0.56					
			BBCH79	8	Green material	0.02	0.25					
			BBCH87	27	Green material	0.01	0.19					
Braslou France, 2008 Grizzly ***	2 (14)	100 100	BBCH67	0*	Green material		0.18	<0.01	0.05	0.02	<0.01	<0.01
			BBCH67	0	Green material		0.57	<0.01	0.05	0.02	<0.01	<0.01
			BBCH80	42	Rest of plant		<0.01	<0.01	0.04	<0.01	<0.01	<0.01
Hoofddorp The Netherlands, 2008 Maximus ***	2 (14)	100 100	BBCH69	0*	Green material		0.01	<0.01	0.04	0.01	<0.01	<0.01
			BBCH69	0	Green material		1.2	<0.01	0.04	0.01	<0.01	<0.01
Cortil-Noirmont Belgium, 2008 Exocet ***	2 (14)	100 100	BBCH73	0*	Green material		0.08	<0.01	0.04	0.01	<0.01	<0.01
			BBCH73	0	Green material		0.61	<0.01	0.05	0.01	<0.01	<0.01
			BBCH85	42	Rest of plant		<0.01	<0.01	0.03	<0.01	<0.01	<0.01
Werl-Niederbergstraße Germany, 2008 Astrid ***	2 (14)	100 100	BBCH69	0*	Green material		0.08	<0.01	0.06	0.02	<0.01	<0.01
			BBCH69	0	Green material		0.66	<0.01	0.05	0.02	<0.01	<0.01

RAPE PLANT Location, year, variety	N (int)	Rate g ai/ha	Sampling GS	DALA	Sample	Residue (mg/kg expressed as prothioconazole-desthio) Prothio	Desthio	α-OH	3-OH	4-OH	5-OH	6-OH
Nieuw Beerta Netherlands, 2009 unknown &&	2 (14)	150 150	BBCH77	0*	Green material		0.04	<0.01	0.04	0.01	<0.01	<0.01
			BBCH77	0	Green material		0.70	<0.01	0.03	0.01	<0.01	<0.01
Cortil-Noirmont Belgium, 2009 Monalisa &&	2 (15)	150 150	BBCH73	0*	Green material		0.07	<0.01	0.03	0.01	<0.01	<0.01
			BBCH73	0	Green material		0.61	<0.01	0.04	0.03	<0.01	<0.01
			BBCH80	30	Rest of plant		0.04	<0.01	0.15	0.06	0.02	<0.01
			BBCH81	42	Rest of plant		0.02	<0.01	0.08	0.02	<0.01	<0.01
Lignieres les Roye France, 2009 Kador &&	2 (14)	150 150	BBCH79	0*	Green material		0.07 c0.08	<0.01	<0.01	<0.01	<0.01	<0.01
			BBCH79	0	Green material		0.49	<0.01	0.05	0.02	<0.01	<0.01
Bishop Burton UK 2009 Castille [n]	2 (14)	139 150	BBCH69	0*	Green material		0.29 C0.02	<0.01	0.09 C0.04	0.03 C0.01	<0.01	<0.01
			BBCH69	0	Green material		1.1	<0.01	0.10	0.04	<0.01	<0.01
			BBCH75	30	Rest of plant		0.02	<0.01	0.10	0.03	<0.01	<0.01
			BBCH80	42	Rest of plant		0.10	<0.01	0.08	0.02	<0.01	<0.01

* prior to last treatment

[a] 08-2113, method 01013 for prothioconazole-desthio, method 00979 for hydroxy metabolites

[b] 09-2504, method 01013 for prothioconazole-desthio, method 00979 for hydroxy metabolites

[c] 09-2055, method 01013 for prothioconazole-desthio, method 00979 for hydroxy metabolites

[d] 10-2134, method 01013 for prothioconazole-desthio, method 00979 for hydroxy metabolites

[e] 10-2244, method 01013 for prothioconazole-desthio, method 00979 for hydroxy metabolites

[f] 11-2013, method 01013 for prothioconazole-desthio, method 00979 for hydroxy metabolites

[g] 2618/06, method 01013 for prothioconazole and prothioconazole-desthio

[h] 2619/06, method 01013 for prothioconazole and prothioconazole-desthio

[i] 2101/02, method 00647 for desthio

[j] 2102/02, method 00647 for desthio

[k] 2616/07, method 01013 for prothioconazole and prothioconazole-desthio

[l] 2617/07, method 01013 for prothioconazole and prothioconazole-desthio

[m] 11-2137, method 01013 for prothioconazole-desthio, method 00979 for hydroxy metabolites

[n] 09-2244, method 01013 for prothioconazole-desthio, method 00979 for hydroxy metabolites

[o] 09-2245, method 01013 for prothioconazole-desthio, method 00979 for hydroxy metabolites

[p] 11-2003, method 01013 for prothioconazole-desthio, method 00979 for hydroxy metabolites

[q] 11-2124, method 01013 for prothioconazole-desthio, method 00979 for hydroxy metabolites

[r] 16-2069, method 01013 for prothioconazole-desthio, method 00979 for hydroxy metabolites

** 08-2112, method 01013 for prothioconazole-desthio, method 00979 for hydroxy metabolites

*** 08-2116, method 01013 for prothioconazole-desthio, method 00979 for hydroxy metabolites

[&&] 09-2053, method 01013 for prothioconazole-desthio, method 00979 for hydroxy metabolites

Table 18 Residues of triazole metabolites in rape forage following foliar administration of prothioconazole

RAPE PLANT	N	Rate	Vol	sampling			Residue (mg/kg)			
Location, year, variety	(int)	g ai/ha	L/ha	GS	DALA	Sample	1,2,4-triazole	TAA	TLA	TA
Fleurieux sur l'Arbesle, Rhone-Alpes, France, 2009 Artiste [a]	2 (13)	120 120	300 300	BBCH85	0*	Green material	<0.01	<0.01 C0.01	<0.01 C0.02	0.09 C0.18
				BBCH85	0	Green material	<0.01	<0.01	<0.01	0.10
				BBCH88	14	Rest of plant	<0.01	0.03 C0.03	0.02 C0.02	<0.01
				BBCH89	20	Rest of plant	<0.01	0.02	0.01	<0.01
San Giogio Di Piano, Emilia-Romagna, Italy, 2009 PR46W09 [a]	2 (14)	120 120	300 300	BBCH80	0*	Green material	<0.01	<0.01	<0.01	0.12 C0.04
				BBCH80	0	Green material	<0.01	<0.01	<0.01	0.10
Braslou, France, 2009 Arcadia [b]	2 (14)	120 120	300 300	BBCH77	0*	Green material	<0.01	0.03 c0.03	<0.01	<0.01
				BBCH77	0	Green material	<0.01	0.02	<0.01	<0.01
				BBCH83	14	Rest of plant	<0.01	<0.01	<0.01	<0.01
				BBCH85	21	Rest of plant	<0.01	<0.01	<0.01	<0.01
Cortil-Noirmont, Belgium, 2009 Monalisa [b]	2 (14)	120 120	200 200	BBCH79	0*	Green material	<0.01	0.01	<0.01	<0.01
				BBCH79	0	Green material	<0.01	0.01	<0.01	<0.01
				BBCH81	14	Rest of plant	<0.01	<0.01	<0.01	<0.01
				BBCH82	21	Rest of plant	<0.01	<0.01	<0.01	<0.01
Nieuw Beerta, Netherlands, 2009 unknown [b]	2 (14)	120 120	300 300	BBCH85	0*	Green material	<0.01	0.02 C0.02	<0.01	<0.01
				BBCH85	0	Green material	<0.01	0.02	<0.01	<0.01
Les Franqueses del Valles Spain, 2011 Pacific [c]	2 (14)	126 120	315 300	BBCH78	0*	Green material	<0.01	0.055 C0.11	<0.01	<0.01
				BBCH78	0	Green material	<0.01	0.058	<0.01	<0.01
				BBCH81	14	Rest of plant	<0.01	0.041 C0.010	<0.01	<0.01
				BBCH85	21	Rest of plant	<0.01	0.019	<0.01	<0.01

RAPE PLANT Location, year, variety	N (int)	Rate g ai/ha	Vol L/ha	sampling GS	DALA	Sample	Residue (mg/kg) 1,2,4-triazole	TAA	TLA	TA
Bouloc France, 2011 Nk Alamir Hybride [c]	2 (13)	120 120	300 300	BBCH79	0*	Green material	<0.01	0.37 C0.53	0.027 C0.041	0.011 C0.015
				BBCH79	0	Green material	<0.01	0.36	0.028	0.012
Burscheid Germany, 2011 Elektra [c]	2 (14)	120 120	300 300	BBCH78	0*	Green material	<0.01	0.024 C0.025	<0.01	<0.01
				BBCH78	0	Green material	<0.01	0.029	<0.01	<0.01
Tarquinia Italy, 2011 Hybristar [c]	2 (14)	120 120	300 300	BBCH75	0*	Green material	<0.01	0.018 C0.013	<0.01	<0.01
				BBCH75	0	Green material	<0.01	0.017	<0.01	<0.01
				BBCH81	14	Rest of plant	<0.01	<0.01	<0.01	<0.01
				BBCH85	21	Rest of plant	<0.01	<0.01	<0.01	<0.01
La Luisiana Spain, 2011 Eswilliams [c]	2 (18)	120 138	300 344	BBCH82	0*	Green material	<0.01	0.3 C0.23	0.015 C0.016	<0.01
				BBCH82	0	Green material	<0.01	0.25	0.015	0.01
Velleron France, 2011 Hybrilux; Rape winter [c]	2 (14)	120 120	300 300	BBCH80	0*	Green material	<0.01	0.042 C0.048	<0.01	<0.01
				BBCH80	0	Green material	<0.01	0.044	<0.01	<0.01
Banbury UK, 2011 D.K. Cabernet [d]	2 (14)	120 120	200 200	BBCH83	0*	Green material	<0.01	0.045 C0.083	<0.01	<0.01
				BBCH83	0	Green material	<0.01	0.048	<0.01	<0.01
Bishop Burton UK, 2009 Castille [e]	2 (14)	139 150	192 200	BBCH69	0*	Green material	<0.01	0.04 C0.06	<0.01	<0.01
				BBCH69	0	Green material	<0.01	0.04	<0.01	<0.01
				BBCH75	30	Rest of plant	<0.01	<0.01	<0.01	<0.01
				BBCH80	42	Rest of plant	<0.01	<0.01	<0.01	<0.01
Bishop Burton UK, 2009 Castille [f] Harvest 13/8/2009	2 (15)	120 120	200 200	BBCH75	0*	Green material	<0.01	<0.01	<0.01	0.03 C0.06
				BBCH75	0	Green material	<0.01	<0.01	<0.01	0.03
				BBCH83	29	Rest of plant	<0.01	<0.01	<0.01	<0.01
Nieuw Beerta Netherlands, 2009 unknown &&	2 (14)	150 150	300 300	BBCH77	0*	Green material	<0.01	<0.01	<0.01	<0.01
				BBCH77	0	Green material	<0.01	<0.01	<0.01	<0.01
Cortil-Noirmont Belgium, 2009 Monalisa &&	2 (15)	150 150	200 200	BBCH73	0*	Green material	<0.01	<0.01	<0.01	<0.01
				BBCH73	0	Green material	<0.01	<0.01	<0.01	<0.01

RAPE PLANT Location, year, variety	N (int)	Rate g ai/ha	Vol L/ha	sampling GS	DALA	Sample	Residue (mg/kg) 1,2,4-triazole	TAA	TLA	TA
				BBCH80	30	Rest of plant	<0.01	<0.01	<0.01	<0.01
				BBCH81	42	Rest of plant	<0.01	<0.01	<0.01	<0.01
Lignieres les Roye France, 2009 Kador &&	2 (14)	150 150	300 300	BBCH79	0*	Green material	<0.01	0.02	<0.01	<0.01 C0.03
				BBCH79	0	Green material	<0.01	0.03	<0.01	<0.01

[a] 09-2504, method 01062

[b] 09-2055, method 01062

[c] 11-2013, method 01062

[d] 11-2137, method 01062

[e] 09-2244, method 01062

[f] 09-2245, method 01062

[&&] 09-2053, method 01062

C – control sample

Table 19 Residues of prothioconazole in oilseed rape forage and fodder from European trials previously evaluated by JMPR (2008)

RAPE Location, year, variety	N (int)	Rate g ai/ha	Vol L/ha	Sampling GS	DALA	Sample	Residues (mg/kg) Prothioconazole-desthio
Burscheid (Versuchsgut Höfchen) Germany 2000 Lirajet [a]	2 (38)	175 175	300 300	BBCH69-71	0*	Forage	<0.05
				BBCH69-71	0	Forage	0.75
				BBCH79-81	41	Rest of plant	<0.05
				BBCH83-85	56	Rest of plant	<0.05
Eslov Sweden 2000 Maskot [a]	2 (18)	175 175	300 300	BBCH65-67	0*	Forage	0.21
				BBCH65-67	0	Forage	0.85
				BBCH79	42	Rest of plant	<0.05
				BBCH88	56	Rest of plant	0.05
Villettes France 2000 Carolus [a]	2 (59)	175 175	300 300	BBCH72	0	Forage	<0.05
				BBCH72	0*	Forage	0.78
				BBCH81	42	Rest of plant	<0.05
				BBCH89	56	Straw	<0.05
Norton UK 2000 Madrigal [a] 421/4	2 (69)	190 175	325 300	BBCH77	0	Forage	<0.05
				BBCH77	0*	Forage	0.54
				BBCH87	42	Rest of plant	<0.05

RAPE Location, year, variety	N (int)	Rate g ai/ha	Vol L/ha	Sampling GS	DALA	Sample	Residues (mg/kg) Prothioconazole-desthio
				BBCH92	56	straw	0.07
Burscheid (Versuchsgut Höfchen) Germany 2001 Express [b]	2 (48)	175 175	300 300	BBCH69-70	0	Forage	0.70
				BBCH89	57	Straw	0.07
Norton Bury St. Edmunds UK 2001 Madrigal [b]	2 (52)	175 163	300 280	BBCH78	0	Forage	0.93
				BBCH89	56	Straw	0.10
Etrepagny France 2001 Zenith [b]	2 (49)	175 175	300 300	BBCH73	0	Forage	0.80
				BBCH89	59	Straw	0.05
Mousseaux-Neuville France 2001 Capitole [b]	2 (49)	190 175	325 300	BBCH73	0	Forage	1.1
				BBCH89	56	Straw	0.05
Bouloc France 2000 Olara [c]	2 (37)	175 175	300 300	BBCH67	0*	Forage	0.10
				BBCH67	0	Forage	0.64
				BBCH80	41	Rest of plant	<0.05
				BBCH87	56	Straw	<0.05
Saint Paul les Romans France 2000 Ebonite [c]	2 (53)	175 175	300 300	BBCH78	0*	Forage	<0.05
				BBCH78	0	Forage	0.94
				BBCH88	42	Rest of plant	<0.05
				BBCH89	55	Straw	0.09
Lusignan France 2000 Capitole [d]	2 (52)	175 175	300 300	BBCH69	0	Forage	0.85
				BBCH89	56	Straw	0.08
Maire France 2000 Constant [d]	2 (55)	175 175	300 300	BBCH72	0	Forage	0.97
				BBCH89	56	Straw	0.20

[a] 2088/00, method 00647 for prothioconazole-desthio

[b] 2178/01, method 00647 for prothioconazole-desthio

[c] 2089/00, method 00647 for prothioconazole-desthio

[d] 2179/01, method 00647 for prothioconazole-desthio

Table 20 Residues of prothioconazole in flax green material

FLAX PLANT Location, year, variety	N (int)	Rate g ai/ha	Sampling GS	DALA	Residue (mg/kg as prothioconazole-desthio)					
					Desthio	α-OH	3-OH	4-OH	5-OH	6-OH
Les Forges Bretagne France, 2009 Everest [a]	2 (13)	175 175	BBCH75	0	1.9	0.02	0.13	0.24	0.06	<0.01
Chaussy France, 2010 Drakkar [b]	2 (14)	175 175	BBCH67	0	2.0	<0.01	0.03	0.38	0.20	0.02
Villers-Perwin Belgium, 2010 Agatha [b]	2 (14)	175 193	BBCH65	0	1.8	<0.01	0.10	0.17	0.06	<0.01
Melbourne UK, 2010 Aries [c]	2 (14)	175 175	BBCH65	0	0.96	<0.01	0.03	0.17	0.09	0.01

[a] 09-2130, method 01013 for prothioconazole-desthio, method 00979 for hydroxy metabolites

[b] 10-2073, method 01013 for prothioconazole-desthio, method 00979 for hydroxy metabolites

[c] 10-2254, method 01013 for prothioconazole-desthio, method 00979 for hydroxy metabolites

Table 21 Residues of triazole metabolites in flax forage (green material) following application of prothioconazole

Location, year, variety	N (int)	Rate g ai/ha	Vol L/ha	Sampling GS	DALA	Residue (mg/kg)			
						1,2,4-triazole	TAA	TLA	TA
Melbourne UK, 2010 Aries [a]	2 (14)	175 175	200 200	BBCH65	0	0.96	<0.01	0.027 C0.108	0.178 C0.013
Les Forges Bretagne France, 2009 Everest [b]	2 (13)	175 175	200 200	BBCH75	0	<0.01	<0.01	0.301 C0.291	0.026 C0.016
Chaussy France, 2010 Drakkar [c]	2 (14)	175 175	200 200	BBCH67	0	<0.01	<0.01	0.02 C0.01	<0.01
Villers-Perwin Belgium, 2010 Agatha [c]	2 (14)	175 193	200 221	BBCH65	0	<0.01	<0.01	0.02 C0.02	<0.01

[a] 10-2254, method 01062

[b] 09-2130, method 01062

[c] 10-2073, method 01062

C – control sample

FATE OF RESIDUES DURING PROCESSING

Four additional oilseed rape processing trials conducted in Europe are summarised below (Hoffmann & Teubner, 2013, 09-3245, M-449117-02-1; Freitag & Hoffmann, 2011, 08-3116, M-393067-02-1; Glaubitz & Czaja, 2013, 12-3402, M-474270-01-1). Seed samples were processed into screw-pressed oil, pomace, meal, solvent extracted oil, crude oil, pre-clarified crude oil, neutralised crude oil and refined oil. Batch processing

procedures simulated small-scale industrial (commercial) processes. All processed fractions were stored frozen (-18 °C) prior to analysis. Seed was analysed within 356 days of sampling, and all processed fractions were analysed within 694 days of processing. Residues of prothioconazole-desthio were determined by LC-MS/MS using method 01013 (LOQ 0.01 mg/kg in all matrices).

Very low residue levels were found in seed (≤0.01–0.01 mg/kg) in the processing trials from rape treated at 100–120 g ai/ha, compared to the exaggerated rate trials treated at the 3× rate of 360 g ai/ha, which contained residues of 0.13–0.28 mg/kg in seed. Consequently, the exaggerated rate trials (12-3402-01 and 12-3402-02) are considered more representative.

After defrosting, the rape seeds were dried at 35 °C for 30 hours to a moisture content of 6–10%. The conditioned seed was cleaned manually using a sieve to remove parts of coarse stalks and weed seeds.

Pressing: The conditioned and cleaned rape seeds were pressed in a screw press yielding oil, screw-pressed and pomace. Dependent on pressing ability of the seed, the processing procedure was conducted with a heated press head.

Extraction: An aliquot of the pomace was milled (distance of the rolls: 2.0 mm). The milled pomace was divided into 6 almost equal parts and the extraction was done for each of these parts separately. The pomace for the extraction was manually transferred in special vessels at a small technical extraction device. The extraction was conducted in two steps. The first extraction step started with addition of n-hexane to the pomace. The n-hexane circulated through the pomace for about 2 h. During the circulation, the n-hexane was heated up to approx. 60 °C.

To remove the rest solvent out of the solvent-oil-mixture (miscella) a rotary evaporator was used. In the rotary evaporator the extracted solvent oil and the remainder of the n-hexane were heated up to approx. 50 °C and were distilled until no solvent was recoverable (visual assessment), yielding oil, solvent extracted. The solvent-extracted meal was sampled after storing at room temperature for approximately one day.

The two obtained oil fractions (oil, screw-pressed and oil, solvent extracted) were mixed yielding the sample of crude oil.

Refining

Filtration: The crude oil was filtered, yielding the sample crude oil, pre-clarified.

Hydration: The pre-clarified crude oil was heated up to 60–70 °C while stirring. After addition of 10% water, the mixture of water and crude oil was heated up to 85–90 °C and stayed then for approx. 45 min. at 85 °C while stirring. Then the stirring was stopped and after phase separation the watery phase including mucilage was removed.

Desliming (degumming): To the oily phase approx. 1% of concentrated phosphorus acid was added at 60 °C while stirring. The mixture was heated up to approx. 85 °C and stayed then for approx. 45 min at that temperature while stirring. Immediately before switching off the stirrer, 10% water was added. After switching off the stirrer, the mixture of water and oily phase remained at approx. 85 °C until separation of the phases. Then the watery phase including flocculated precipitation was removed.

Neutralisation: After determination of the acid-number, the oily phase was heated up to approx. 90 °C while stirring. Then a few grams of a 7% sodium hydroxide solution (amount depending on acidity of the oil phase) were added, and the mixture was stirred for approx. 20 min. After addition of 10% water and further 5 min of stirring the stirrer was switched off and the phases were allowed to separate. After phase separation the watery phase was removed. The acid-number of the oil was above 0.12, so the process had

to be repeated. After the second neutralisation process, the acid value was below 0.12 and as such, an aliquot of crude oil, neutralised could be sampled.

Washing: The remaining crude oil, neutralised was heated to 90 °C while stirring and washed by the addition of 10% water. The mixture was stirred for 20 minutes at 90 °C. After phase separation, the watery phase was removed. These steps were repeated until the pH of the washing water was in the range 7–8 and the soap content of the oil was ≤ 0.01%.

Drying: The oil was heated up to approx. 95 °C while stirring. After addition of citric acid (60 mg/kg oil) drying was done using a vacuum until no more water escaped from the oil.

Bleaching: The oil was heated up to 95 °C while stirring. After addition of 1% podsol (referring to the used oil) the oil was bleached for 5 min without vacuum and 20 min with vacuum.

Filtration: The podsol was removed using a special filter equipment under vacuum.

Deodorization: The oil was heated under vacuum up to 240 °C while stirring. After reaching 160 °C, steam was transferred through the oil in order to expel fatty acids, odour and taste influencing compounds as well as other volatile compounds. The mixture stayed at 240 °C for about 20 minutes. After cooling to 160 °C the steam supply was stopped and then the oil was dried under vacuum until a temperature of ≤ 80 °C was reached. The refined oil was sampled.

Table 22 Residues of prothioconazole-desthio and hydroxy metabolites in processed rape seed commodities

RAPE	N (int)	Rate g ai/ha	DALA	Sample	Residue (mg/kg as prothioconazole-desthio)					
					Desthio	α-OH	3-OH	4-OH	5-OH	6-OH
Burscheid Germany, 2008 Titan [a] 9.8 kg	2 (14)	120 120	35	Seed	0.01	<0.01	0.02	<0.01	<0.01	<0.01
				Oil screw-pressed	0.01	<0.01	<0.01	<0.01	<0.01	<0.01
				Pomace	0.01	<0.01	0.02	<0.01	<0.01	<0.01
				Meal	<0.01	<0.01	0.03	<0.01	<0.01	<0.01
				Oil solv ext	0.02	<0.01	<0.01	<0.01	<0.01	<0.01
				Oil crude	0.02	<0.01	<0.01	<0.01	<0.01	<0.01
				Crude oil pre-clarified	0.02	<0.01	<0.01	<0.01	<0.01	<0.01
				Crude oil neutralised	0.01	<0.01	<0.01	<0.01	<0.01	<0.01
				Oil refined	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Bishop Burton UK, 2009 Castille [d] 5.6 kg	2 (15)	120 120	44	Seed	0.01	<0.01	<0.01	<0.01	<0.01	<0.01
				Oil screw-pressed	0.01	<0.01	<0.01	<0.01	<0.01	<0.01
				Pomace	0.01	<0.01	<0.01	<0.01	<0.01	<0.01
				Meal	0.02	<0.01	<0.01	<0.01	<0.01	<0.01
				Oil solv ext	0.02	<0.01	<0.01	<0.01	<0.01	<0.01

RAPE	N (int)	Rate g ai/ha	DALA	Sample	Residue (mg/kg as prothioconazole-desthio)					
					Desthio	α-OH	3-OH	4-OH	5-OH	6-OH
				Oil crude	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
				Crude oil pre-clarified	0.01	<0.01	<0.01	<0.01	<0.01	<0.01
				Crude oil neutralised	0.02	<0.01	<0.01	<0.01	<0.01	<0.01
				Oil refined	0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Hoofddorp Netherlands, 2008 Maximus [b] Controls only analysed for seed 7.6 kg	2 (14)	100 100	64	Seed	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
				Oil screw-pressed	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
				Pomace	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
				Meal	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
				Oil solv ext	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
				Oil crude	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
				Crude oil pre-clarified	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
				Crude oil neutralised	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
				Oil refined	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Burscheid Germany, 2012 Vision [c] 10.8 kg	2 (15)	120 120	32	Seed	0.13 0.13 (0.045)	0.016 0.017 (0.0165)	0.080 0.071 (0.0751)	0.027 0.025 (0.026)	0.013 0.010 (0.0115)	<0.01 <0.01
				Oil screw-pressed	0.27	<0.01	<0.01	<0.01	<0.01	<0.01
				Pomace	0.18	0.015	0.077	0.025	0.014	<0.01
				Meal	0.061	0.011	0.094	0.030	0.019	<0.01
				Oil solv ext	0.39	0.016	<0.01	<0.01	<0.01	<0.01
				Oil crude	0.37	0.013	<0.01	<0.01	<0.01	<0.01
				Crude oil pre-clarified	0.37	0.013	<0.01	<0.01	<0.01	<0.01
				Crude oil neutralised	0.32	<0.01	<0.01	<0.01	<0.01	<0.01
				Oil refined	0.29	<0.01	<0.01	<0.01	<0.01	<0.01
Velleron France, 2012 Hybrilux [c] 12.7 kg	2 (14)	120 120	40	Seed	0.29 0.27	0.017 0.019 (0.018)	0.047 0.039 (0.043)	0.026 0.022 (0.024)	0.016 0.013 (0.0145)	0.010 0.011 (0.0105)
				Oil screw-pressed	0.60	<0.01	<0.01	<0.01	<0.01	<0.01
				Pomace	0.35	0.018	0.046	0.027	0.016	0.014
				Meal	0.12	0.015	0.059	0.031	0.024	0.016
				Oil solv ext	0.86	0.016	<0.01	<0.01	<0.01	<0.01
				Oil crude	0.74	0.012	<0.01	<0.01	<0.01	<0.01

RAPE	N (int)	Rate g ai/ha	DALA	Sample	Residue (mg/kg as prothioconazole-desthio)					
					Desthio	α-OH	3-OH	4-OH	5-OH	6-OH
				Crude oil pre-clarified	0.68	0.010	<0.01	<0.01	<0.01	<0.01
				Crude oil neutralised	0.68	<0.01	<0.01	<0.01	<0.01	<0.01
				Oil refined	0.59	<0.01	<0.01	<0.01	<0.01	<0.01

[a] 08-3112

[b] 08-3116

[c] 12-3402

[d] 09-3245

Table 23 Residues of triazole metabolites in processed rape seed commodities

RAPE Location, year, variety	N (int)	Rate g ai/ha	DALA	Sample	Residue (mg/kg)			
					1,2,4-triazole	TA	TAA	TLA
Burscheid Germany, 2008 Titan [a] 9.8 kg	2 (14)	120 120	35	Seed	<0.01	0.27 C0.31	<0.01	<0.01
				Oil screw-pressed	<0.01	<0.01	<0.01	<0.01
				Pomace	<0.01	0.21 C0.19	0.01	0.02 C0.02
				Meal	<0.01	0.22 C0.16	0.02 C0.02	0.03 C0.01
				Oil solv ext	<0.01	<0.01	<0.01	<0.01
				Oil crude	<0.01	<0.01	<0.01	<0.01
				Crude oil pre-clarified	<0.01	<0.01	<0.01	<0.01
				Crude oil neutralised	<0.01	<0.01	<0.01	<0.01
				Oil refined	<0.01	<0.01	<0.01	<0.01
Bishop Burton UK, 2009 Castille [d] 5.6 kg	2 (15)	120 120	44	Seed	<0.01	0.28 C0.25	<0.01	0.02 c0.01
				Oil screw-pressed	<0.01	<0.01	<0.01	<0.01
				Pomace	<0.01	0.37 C0.34	<0.01	0.02 C0.01
				Meal	<0.01	0.37 C0.24	<0.01	0.03 C0.01
				Oil solv ext	<0.01	<0.01	<0.01	<0.01
				Oil crude	<0.01	<0.01	<0.01	<0.01
				Crude oil pre-clarified	<0.01	<0.01	<0.01	<0.01

RAPE Location, year, variety	N (int)	Rate g ai/ha	DALA	Sample	Residue (mg/kg)			
					1,2,4-triazole	TA	TAA	TLA
				Crude oil neutralised	<0.01	<0.01	<0.01	<0.01
				Oil refined	<0.01	<0.01	<0.01	<0.01
Hoofddorp Netherlands, 2008 Maximus [b] Controls only analysed for seed 7.6 kg	2 (14)	100 100	64	Seed	<0.01	0.41 C0.26	<0.01	<0.01
				Oil screw-pressed	<0.01	<0.01	<0.01	<0.01
				Pomace	<0.01	0.55	<0.01	<0.01
				Meal	<0.01	0.78	<0.01	0.02
				Oil solv ext	<0.01	<0.01	<0.01	<0.01
				Oil crude	<0.01	<0.01	<0.01	<0.01
				Crude oil pre-clarified	<0.01	<0.01	<0.01	<0.01
				Crude oil neutralised	<0.01	<0.01	<0.01	<0.01
				Oil refined	<0.01	<0.01	<0.01	<0.01
Burscheid Germany, 2012 Vision [c] 10.8 kg	2 (15)	120 120	32	Seed	<0.01 <0.01	0.26 0.28 C0.055	<0.01 <0.01	0.01 <0.01
				Oil screw-pressed	<0.01	<0.01	<0.01	<0.01
				Pomace	<0.01	HC	0.011	0.013
				Meal	<0.01	HC	0.017	0.018
				Oil solv ext	<0.01	<0.01	<0.01	<0.01
				Oil crude	<0.01	<0.01	<0.01	<0.01
				Crude oil pre-clarified	<0.01	<0.01	<0.01	<0.01
				Crude oil neutralised	<0.01	<0.01	<0.01	<0.01
				Oil refined	<0.01	<0.01	<0.01	<0.01
Velleron France, 2012 Hybrilux [c] 12.7 kg Controls for seed only	2 (14)	120 120	40	Seed	<0.01	<0.01 C0.048	<0.01	<0.01
				Oil screw-pressed	<0.01	<0.01	<0.01	<0.01
				Pomace	<0.01	<0.01	<0.01	<0.01
				Meal	<0.01	<0.01	<0.01	<0.01
				Oil solv ext	<0.01	<0.01	<0.01	<0.01
				Oil crude	<0.01	<0.01	<0.01	<0.01
				Crude oil pre-clarified	<0.01	<0.01	<0.01	<0.01
				Crude oil neutralised	<0.01	<0.01	<0.01	<0.01
				Oil refined	<0.01	<0.01	<0.01	<0.01

[a] 08-3112

[b] 08-3116

[c] 12-3402

[d] 09-3245

HC = high residue in untreated samples used for procedural recoveries and controls

Sunflower

A processing study on sunflower seed was made available to the Meeting. An SC formulation containing 480 g/L prothioconazole was applied to sunflower at BBCH 65 at an exaggerated rate of 1009 g ai/ha. Duplicate seed samples were collected for processing at BBCH 99 (44–day PHI). Seed samples were processed into de-oiled meal and crude sunflower oil. Processing procedures conducted on a lab scale simulated industrial (commercial) processes. All processed fractions were stored frozen (<-10 °C) prior to analysis. Seed was analysed within 515 days of sampling, and oil and meal were analysed after 333 and 343 days, respectively. These sample storage durations fall within the stability period of 3 years as confirmed by previous studies with prothioconazole and prothioconazole-desthio on canola seed and oil, mustard greens, tomato fruit, turnip root, wheat flour, forage and straw.

Residues of prothioconazole (determined after conversion to prothioconazole-desthio and prothioconazole-sulfonic acid) and prothioconazole-desthio were determined by LC-MS/MS using method JA/03/01. The LOQ was 0.02 mg/kg in all matrices. Procedural recoveries at fortification levels of 0.02–0.2 mg/kg were within the acceptable range of 70–120%, RSD <20%, for prothioconazole-desthio in each matrix.

A summary of the processing trial is provided in Table 24, and a summary of the processing factors is presented in Table 25.

Sunflower seed for processing had a moisture content of 6.5–9.5%. Due to the small quantity of seed processed, de-hulling was not carried out, instead seed was cracked by passing through a series of corrugated rolls and then passed through flaking rolls. Flaking was not successful for one control sample which was pressed as whole seed. Cracked seed was heated in a microwave for 2.5–3 minutes and subsequently transferred to an oven maintained at 75 °C prior to pressing. The resulting crude press oil was centrifuged. The press cake was extracted with iso-hexane (55–60 °C) for 3–3.5 hours. The solvent was removed on a rotary evaporator and the crude press oil was combined with the solvent recovered oil and filtered. The solvent extracted meal was spread thinly on a tray in a fume hood and left for approximately 24 hours or until no hexane odour was detected.

Table 24 Residues of prothioconazole and metabolites in processed sunflower commodities (Ballantine, 2013, AFFFC08-022R, M-527444-01-1)

SUNFLOWER Location, year, variety	Rate g ai/ha	DALA	Commodity	Residue (mg/kg as prothioconazole-desthio) Desthio	Prothioconazole sulfonic acid	"Total"
Scott, SK, Canada, 2008 Pioneer 63A21 24.3 kg, 24.1 kg	1×1009	44	Seed	0.29 0.27 (0.28)	0.081 0.077 (0.079)	0.371 0.347 (0.359)
			Meal	0.19 0.20 (0.205)	0.055 0.060 (0.0575)	0.245 0.260 (0.2625)
			Oil	0.38	0.022	0.402

SUNFLOWER Location, year, variety	Rate g ai/ha	DALA	Commodity	Residue (mg/kg as prothioconazole-desthio)		
				Desthio	Prothioconazole sulfonic acid	"Total"
				0.39 (0.385)	0.026 (0.024)	0.416 (0.409)

Note: The analytical method converts prothioconazole residues to a mixture of prothioconazole-desthio and prothioconazole sulfonic acid.

As residues of prothioconazole and prothioconazole sulfonic acid may be converted to prothioconazole-desthio on processing the processing factor for prothioconazole-desthio is (prothioconazole-desthio in processed commodity)/(prothioconazole+prothioconazole-sulfonic acid + prothioconazole-desthio in the raw commodity). The error introduced by part of prothioconazole residues being counted as prothioconazole-desthio in the processed commodity is expected to be small as prothioconazole residues are anticipated to be present at levels much lower that prothioconazole-desthio. Table 25 summarises the processing factors for prothioconazole in sunflower commodities.

Table 25 Processing factors for prothioconazole-desthio in sunflower processed fractions

Processed Fractions	Residue (mg/kg as prothioconazole-desthio)	Processing factor
Sunflower seed	0.359	-
Meal	0.205	0.57
Crude oil	0.385	1.07

APPRAISAL

Prothioconazole is a broad-spectrum systemic fungicide. It was first evaluated by the JMPR in 2008 (T, R). The latest residue review was done in 2017 (R). It was scheduled at the Fifty-first Session of the CCPR for the evaluation of additional uses by the 2020 JMPR, which was postponed to the 2021 Extra JMPR.

The toxicological review established an ADI of 0–0.05 mg/kg bw and an ARfD of 0.8 mg/kg bw for women of child-bearing age for the parent prothioconazole; an ADI of 0–0.01 mg/kg bw and an ARfD of 0.01 mg/kg bw for women of child-bearing age; and 1 mg/kg bw for general population for prothioconazole-desthio. The residue definition for plant commodities for enforcement and dietary risk assessment is prothioconazole-desthio. The residue definition for animal commodities for enforcement is prothioconazole-desthio and for dietary risk assessment the sum of prothioconazole-desthio, prothioconazole-desthio-3-hydroxy, prothioconazole-desthio-4-hydroxy and their conjugates, expressed as prothioconazole-desthio. The residue covered by the residue definition for compliance is considered not fat-soluble.

For the current Meeting, data were provided on residues in oilseed rape, linseed (flax) and sunflower together with processing studies of rape and sunflower seed as well as currently registered use patterns.

Methods of analysis

The Meeting received additional validation information on analytical methods previously evaluated by the 2008, 2009 and 2014 JMPRs for prothioconazole and metabolites in oilseeds (rape, linseed, sunflower). The Meeting concluded that the presented methods were sufficiently validated and are suitable to measure

prothioconazole, prothioconazole-desthio as well as the metabolites prothioconazole-desthio-α-hydroxy (α-OH), prothioconazole-desthio-3-hydroxy (3-OH), prothiocopnazole-desthio-4-hydroxy (4-OH), prothioconazole-desthio-5-hydroxy (5-OH) and prothioconazole-desthio-6-hydroxy (6-OH) as well as the triazole metabolites 1,2,4-triazole (1,2,4-T), triazole acetic acid (TAA), triazole lactic acid (TLA) and triazole alanine (TA). The LOQs are 0.01 mg/kg individually for prothioconazole and metabolites in the oilseeds (rape, linseed, sunflower) as well as in processed commodities. The LOQs for the triazole metabolites 1,2,4-triazole (1,2,4-T), triazole acetic acid (TAA), triazole lactic acid (TLA) and triazole alanine (TA) in the same commodities were also 0.01 mg/kg for each compound. One method measured prothioconazole, prothioconazole-desthio and prothioconazole sulfonic acid in sunflower seed and processed commodities with LOQs of 0.01 mg/kg for each compound.

Stability of pesticide residues in stored analytical samples

A storage stability study was provided to cover the short-term increase in temperature that occurred during shipment for rape samples in one trial prior to frozen storage. Residues of prothioconazole were stable in rape seed and rape green material when stored at 5 °C for five days. The Meeting considered the short-term increase in temperature of samples during shipment in one trial to have no impact on the residues measured.

The demonstrated stability intervals for prothioconazole and metabolites in frozen storage of at least 36 months for rape seed and 24 months for rape pods and plant material reported by the 2008 and 2014 JMPR encompass the duration of storage in the residue trials evaluated. An additional storage stability study for prothioconazole, prothioconazole-desthio, and prothioconazole sulfonic acid on sunflower seed demonstrated stability for at least 521 days.

The periods of demonstrated stability adequately cover the frozen storage intervals of samples in supervised trials considered by the current Meeting.

Results of supervised residue trials on crops

Supervised trials were available for the use of prothioconazole on a number of oilseed crops; rape, flax (linseed) and sunflower.

Rape

The critical GAP for prothioconazole on rape in Hungary is two foliar applications at 125 g ai/ha at a minimum interval of 14 days and a PHI of 28 days.

In thirty-two trials approximating GAP residues were (n = 32): < 0.01, 0.01, 0.01, 0.02, 0.02, 0.02, 0.025, 0.026, 0.03, 0.03, 0.03, 0.03, 0.03, 0.03, 0.033, 0.038, 0.04, 0.04, 0.043, 0.05, 0.05, 0.054, 0.055, 0.056, 0.06, 0.064, 0.081, 0.083, 0.09, 0.11, 0.13, 0.14 mg/kg.

The Meeting estimated a maximum residue level of 0.2 mg/kg and an STMR of 0.039 mg/kg for prothioconazole in rape seed.

Linseed (flax)

The Meeting received supervised residue trials on flax. In Greece, the cGAP is two foliar applications at 175 g ai/ha, with a minimum re-treatment interval of 14 days and a PHI of 56 days. No trials matching cGAP in Greece were made available to the Meeting. However, as rapeseed is the representative commodity for the

subgroup "Small seed oilseeds", the Meeting agreed to utilise trials on rapeseed matching the GAP for flax to estimate residues and to extrapolate the results to flax seed.

In four trials approximating cGAP in Greece, residues in rape seed were:

2× 150 g ai/ha: < 0.01, < 0.01, < 0.01, 0.02 mg/kg.

In a further seven trials at lower application rates residues were:

2×125 g ai/ha: < 0.01, < 0.01, < 0.01 mg/kg.

2×100 g ai/ha: < 0.01, < 0.01, < 0.01, < 0.01 mg/kg.

The Meeting agreed to use proportionality to scale the residues.

Rate (g ai/ha)	Residue (mg/kg)	Proportionality scaling factor	Scaled residues
2×100	< 0.01 (4)	1.75	< 0.0175 (4)
2×125	< 0.01 (3)	1.4	< 0.014 (3)
2×150	< 0.01 (3), 0.02	1.17	< 0.0117 (3), 0.0234

After scaling, residues approximating cGAP are (n = 11): < 0.0117 (3), < 0.014 (3), < 0.0175 (4) and 0.0234 mg/kg.

The Meeting estimated a maximum residue level of 0.03 mg/kg and an STMR of 0.014 mg/kg for prothioconazole in linseed.

Sunflower

The Meeting received supervised residue trials on sunflower. In Hungary, cGAP is two foliar applications at 125 g ai/ha at an interval of 14 days with a PHI of 28 days.

In eighteen trials on sunflower matching cGAP residues at harvest were (n = 18): < 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, 0.017, 0.017, 0.02, 0.035, 0.043, 0.13 mg/kg.

The critical GAP for prothioconazole on sunflower in Brazil is two foliar applications at 87.5 g ai/ha at a minimum interval of 15 days and a PHI of 30 days.

In ten trials from Brazil approximating GAP residues of prothioconazole-desthio were (n = 10): < 0.009, < 0.009, < 0.009, < 0.009, < 0.009, < 0.009, 0.009, 0.036, 0.063, 0.32 mg/kg.

The dataset from Brazil leads to the higher maximum residue level and the Meeting decided to utilise this dataset to make a recommendation for a maximum residue level of 0.5 mg/kg and an STMR of 0.009 mg/kg for prothioconazole in sunflower seed.

Residues in animal feeds

Rape forage (green) = green material prior to BBCH 60

Rape forage corresponds to green material prior to BBCH 60. No samples of plant material were analysed at growth stages before BBCH 60. The data are not suitable to estimate residues in rape forage.

Fate of residues during processing

The Meeting received information on the fate of prothioconazole residues during processing of rape and sunflower seed. Residues of prothioconazole metabolites were measured using method 00979 (desthio,

alpha-OH, 3-OH, 4-OH, 5-OH, 6-OH). As prothioconazole residues may be converted to prothioconazole-desthio, the processing factor for prothioconazole-desthio is calculated as (prothioconazole-desthio in the processed commodity)/(sum of prothioconazole and prothioconazole-desthio in the raw commodity). To calculate processing factors, residues of prothioconazole should be measured in the raw commodity or evidence is required that prothioconazole residues are not detected. In previous trials on rape seed assessed by the 2008 and 2014 JMPR, prothioconazole was converted to a mixture of prothioconazole sulfonic acid and prothioconazole-desthio with residues reported for both the sulfonic acid and prothioconazole-desthio and for the sum of prothioconazole sulfonic acid and prothioconazole-desthio which is referred to as "total prothioconazole". In these trials it is not possible to determine whether prothioconazole is present at levels above the LOD and it is therefore not appropriate to use the processing studies provided to derive processing factors. However, the processing factors reported by the 2008 JMPR for rape seed for total prothioconazole are relevant as the sum of prothioconazole and prothioconazole-desthio was measured and are < 0.7 for both rape seed meal and refined oil.

Table 1 Processing factors and median residue values for prothioconazole-desthio used for estimation of maximum residue levels.

Processed commodity	Raw commodity [STMR/ median residue]	Processing factor	STMR-P (mg/kg)	Median residue-P (mg/kg)
Rape meal	0.039	< 0.7	0.0273	0.04641
Rape oil – refined		< 0.7	0.0273	
Sunflower meal	0.009	0.57	0.00513	0.008721
Sunflower crude oil		1.07	0.00963	

STMR-P = for plant commodity risk assessment residue definition – prothioconazole-desthio

Median residue-P = for compounds relevant for the livestock dietary burden for the livestock residue definition for risk assessment

Conversion factor for median residue for use in livestock burden calculations = 1.7

The Meeting applied the rape seed refined oil processing factor (0.7) to the estimated maximum residue level for rape seed (0.2 mg/kg) to estimate a maximum residue level of 0.15 mg/kg for rape seed oil, edible.

The Meeting **considered** the sunflower crude oil processing factor (1.07) and the estimated maximum residue level for sunflower seed (0.5 mg/kg) to estimate a maximum residue level of 0.5 mg/kg for sunflower seed oil, edible.

Residues in animal commodities

Farm animal dietary burden

The residue definition for risk assessment for livestock commodities differs from that for compliance for plant and livestock commodities. To estimate inputs for dietary intake calculations it is necessary to account for the transfer of compounds relevant to the livestock commodity residue definition from plants to livestock tissues. Metabolism data reported in the 2008 JMPR were used to estimate conversion factors to convert prothioconazole-desthio residues to those that may be converted on feeding to livestock to components in the risk assessment residue definition (prothioconazole, prothioconazole sulfonic acid M02, prothioconazole-desthio-3-hydroxy M14 and prothioconazole-desthio-4-hydroxy M15 and their conjugates

M21, M22, M54, M55). As foliar application leads to the highest residues, in considering conversion factors, the average of values available following foliar application were used, i.e. data for seed treatment were not considered relevant. Conversion factors for wheat were extrapolated to all forage and fodders except peanut hay while factors for sugar beet tops and sugar beet roots were extrapolated to potatoes.

The following conversion factors were obtained:

Cereal forage 1.6

Cereal hay 2.1

Cereal straw 2.6

Grains/seeds 1.7

Peanut hay 2.4

Sugar beet tops 1.6

Sugar beet roots/potato tubers 1.0

The feed commodities listed in the Table below were used in estimating livestock dietary burdens.

Some processed and forage commodities do not appear in the Recommendations Table (because no maximum residue level is needed), but they are used in estimating livestock dietary burdens. Those commodities are included in the list below.

Table 2 Potential livestock feed items and residues of compounds in feed relevant to livestock risk assessment

Codex classification	Prothioconazole-desthio		Conversion factor [A]	Residues relevant to livestock risk assessment dietary burden	
	Median residue (mg/kg)	Highest residue (mg/kg)		Median residue (mg/kg)	Highest residue (mg/kg)
Barley grain	0.035		1.7	0.0595	
Barley forage	1.2	5.4	1.6	1.92	8.64
Cotton seed	0.052		1.7	0.0884	
Cotton gin by-products	1.1	1.8	2.6	2.86	4.68
Cotton seed meal	0.005		1.7	0.0085	
Cotton seed hulls	0.008		1.7	0.0136	
Fodder of cereal grains (dry)	1.5	4.8	2.1	3.15	10.08
Linseed/flax seed	0.014		1.7	0.0238	
Maize	0.018		1.7	0.0306	
Maize forage/silage	2.15	4.08	1.6	3.44	6.528
Maize fodder	3.48	6.7	2.1	7.308	14.07
Oat grain	0.01		1.7	0.017	
Peanut fodder (hay)	4.08	11.6	2.4	9.792	27.84
Potato culls	0.01		1	0.01	
Pulses (grain)	0.05		1.7	0.085	
Rape seed	0.039		1.7	0.0663	
Rape seed meal	0.0273		1.7	0.04641	
Rye grain	0.01		1.7	0.017	
Soya bean (dry)	0.05		1.7	0.085	

Soya bean aspirated grain fractions	3.75		1.7	6.375	
Soya bean hulls	0.025		1.7	0.0425	
Straw and fodder (dry) of cereals	0.65	1.9	2.6	1.69	4.94
Sugar beet	0.05		1	0.05	
Sugar beet tops	1.5	3.9	1.6	2.4	6.24
Sweet corn fodder	3.48	6.7	2.1	7.308	14.07
Triticale grain	0.01		1.7	0.017	
Wheat grain	0.02		1.7	0.034	
Wheat bran	0.048		1.7	0.0816	
Wheat milled by-products	5		1.7	8.5	

[a] While the Meeting noted conversion factors for processed commodities might be different to those of the RAC, the Meeting agreed, in the absence of better information, to also use the conversion factors derived for the RAC for livestock feed commodities derived from processing.

Dietary burdens were calculated for beef cattle, dairy cattle, broilers and laying poultry based on feed items evaluated by the JMPR by the current Meeting. The dietary burdens were estimated using the most recent version of the OECD livestock dietary burden calculator, are presented in Annex 6 and summarised below.

Table 3 Estimated maximum and mean dietary burdens of farm animals (prothioconazole-desthio)

	Animal dietary burden: prothioconazole-desthio, ppm of dry matter diet							
	US-Canada		EU		Australia		Japan	
	max	mean	Max	mean	max	mean	max	mean
Beef cattle	4.1	3.4	13.9	5.7	15.8	5.5	3.1	3.1
Dairy cattle	10.0	4.8	14.6	5.8	15.8a b	5.5	7.7	5.2
Broilers	2.9	2.9	1.2	1.2	1.2	1.2	0.3	0.3
Layers	2.9c d	2.9	2.1	1.5	1.2	1.2	1.7	1.7

[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 maximum broiler or laying hen dietary burden suitable for MRL estimates for poultry tissues

[d] Highest maximum laying hen dietary burden suitable for MRL estimates for eggs.

Table 4 Estimated maximum and mean dietary burdens of farm animals (residues relevant to estimation of livestock residues according to the risk assessment definition)

	Animal dietary burden: relevant to risk assessment, ppm of dry matter diet							
	US-Canada		EU		Australia		Japan	
	max	mean	Max	mean	max	mean	Max	mean
Beef cattle	7.1	5.7	22.2	9.3	31.2	10.8	5.3	5.3
Dairy cattle	17.8	9.4	23.3	9.5	31.2 [a]	10.8 [b]	12.5	8.7
Broilers	4.9	4.9	2.0	2.0	2.0	2.0	0.5	0.5
Layers	4.9 [c]	4.9 [d]	3.6	2.6	2.0	2.0	2.9	2.9

[a] Highest maximum beef or dairy cattle dietary burden suitable for HR estimates for mammalian tissues and milk

[b] Highest mean beef or dairy cattle dietary burden suitable for STMR estimates for mammalian tissues and milk.

[c] Highest maximum broiler or laying hen dietary burden suitable for HR estimates for poultry tissues and eggs

[d] Highest mean broiler or laying hen dietary burden suitable for STMR estimates for poultry tissues and eggs.

Animal commodity maximum residue levels

Cattle

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

Table 5 Anticipated residues of prothioconazole-desthio in mammalian commodities

	Feed Level (ppm) for milk residues	Prothioconazole-desthio residues (mg eq/kg) in milk	Feed Level (ppm) for tissue residues	Prothioconazole-desthio residues (mg eq/kg)			
				Muscle	Liver	Kidney	Fat
Highest residue for maximum residue level estimation (beef or dairy cattle)							
Feeding Study	29 [a]	< 0.004	5.1 29 [a]	< 0.01	0.03 0.18	0.01 0.03	0.01
Dietary burden and estimate of highest residue	15.8	< 0.0022	15.8	< 0.005	0.129	0.023	0.0054

[a] The nominal feeding levels reported by the 2008 JMPR were 4, 25 and 100 ppm, however, the actual feed levels were 5.1, 29 and 125 ppm. The actual feed levels are used here.

	Feed Level (ppm) for milk residues	Total residues (mg eq/kg) in milk	Feed Level (ppm) for tissue residues	Total residues [A] (mg eq/kg)			
				Muscle	Liver	Kidney	Fat
HR Determination (beef or dairy cattle)							
Feeding Study			29[b] 125[b]	0.03	0.26 1.6	0.17 1.1	0.02 0.14
Dietary burden and estimate of highest residue			31.2	0.007	0.291	0.191	0.023
STMR Determination (beef or dairy cattle)							
Feeding Study	125 [b c]	0.011	5.5[b] 29[b]	< 0.01	0.04 0.22	0.023 0.14	0.013
Dietary burden and estimate of highest residue	10.8	0.001	10.8	< 0.004	0.077	0.049	0.005

No residues detected above LOQ for the 5.1 and 29 ppm dose groups, mean residues were 0.011 mg/kg for the 125 ppm dose group.

[a] Total residues = sum of prothioconazole, prothioconazole-desthio, prothioconazole-desthio-3-hydroxy, prothioconazole-desthio-4-hydroxy and their conjugates expressed as prothioconazole-desthio.

[b] The nominal feeding levels reported by the 2008 JMPR were 4, 25 and 100 ppm, however, the actual feed levels were 5.1, 29 and 125 ppm. The actual feed levels are used here.

[c] Residues were <LOQ in milk at the 5.1 and 29 ppm feed levels. As residues in milk were detected at the 125 ppm feed level, the Meeting decided to utilise the results from the 125 ppm feed level to estimate residues in milk.

The current Meeting has estimated livestock dietary burdens based on the different residue definitions for compliance and risk assessment, including accounting for the different compounds that need to be included in the estimation of the livestock dietary burdens. Additionally, the OECD livestock dietary burden calculator has been updated several times since 2009. The Meeting has taken the opportunity to make revised recommendations with the newly developed livestock dietary burdens and most recent version of the OECD livestock dietary burden calculator.

The Meeting estimated the following maximum residue levels: mammalian fat (except milk fat) 0.01 mg/kg and edible offal 0.15 mg/kg to replace its previous recommendations and confirmed its previous estimations for milk (0.004* mg/kg) and meat (mammalian except marine mammals) (0.01 mg/kg). The Meeting estimated the following HRs: muscle 0.007 mg/kg; fat 0.023 mg/kg; liver 0.291 mg/kg and kidney 0.191 mg/kg and STMRs: muscle 0.004 mg/kg; fat 0.005 mg/kg; liver 0.077 mg/kg, kidney 0.049 mg/kg and milk 0.001 mg/kg.

For poultry, the Meeting noted that the 2008 JMPR had concluded that the poultry feeding study designs did not reflect the residue composition in feed and that the results could not be used for estimating maximum residue limits or STMR values. The 2017 JMPR noted in a metabolism study on laying hens with the parent prothioconazole conducted at an exaggerated rate of 171 ppm feed and slaughter at 5 hours after the last dose, residues of prothioconazole-desthio were 0.167, 0.007, 0.006 and 0.13 mg eq/kg in liver, eggs, muscle and fat, respectively. Total radioactive residues (TRR) were 4.017, 0.036, 0.089 and 0.45 mg/kg for liver, eggs, muscle and fat, respectively. Assuming residues are proportional to the level in feed, residues of prothioconazole-desthio following exposure to feed at 2.9 ppm would be 0.0028, 0.0001, 0.0001 and 0.0022 mg/kg in liver, eggs, muscle and fat, below the LOQs of 0.005 mg/kg for eggs and 0.01 mg/kg for tissues. Total residues on feeding at 4.9 ppm would be 0.118, 0.001, 0.0026 and 0.0129 mg/kg residue in liver, eggs, muscle and fat, respectively.

The Meeting estimated the following maximum residue levels for poultry commodities: poultry fat 0.01 mg/kg and poultry edible offal 0.01 mg/kg to replace its previous recommendations and confirmed its previous estimations for eggs (0.005(*) mg/kg) and poultry meat (0.01(*) mg/kg). The Meeting estimated the following HRs: poultry meat 0.0026 mg/kg; poultry fat 0.0129 mg/kg; liver 0.118 mg/kg and eggs 0.001 mg/kg and STMRs at the same levels.

RECOMMENDATIONS

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

Definition of the residue for compliance with the MRL and dietary risk assessment for plant commodities: *prothioconazole-desthio*.

Definition of the residue for compliance with the MRL for animal commodities: *prothioconazole-desthio*; and

Definition of the residue for dietary risk assessment for animal commodities: the *sum of prothioconazole-desthio, prothioconazole-desthio-3-hydroxy, prothioconazole-desthio-4-hydroxy and their conjugates expressed as prothioconazole-desthio*.

The residue is not fat-soluble.

Table 6 Recommendations for residues of prothioconazole from the 2021 Extra JMPR

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)	0.15	0.3	0.077 L 0.049 K	0.291 L 0.191 K
PE 0112	Eggs	0.005*	0.005*	0.001	0.001
SO 0693	Linseed	0.03		0.014	
MF 0100	Mammalian fats (except milk fats)	0.01	0.02	0.005	0.023
MM 0095	Meat (from mammals other than marine mammals)	0.01	0.01	0.004 M 0.005 F	0.007 M 0.023 F
ML 0106	Milks	0.004*	0.004*	0.001	
PO 0111	Poultry, Edible offal of	0.01	0.1	0.118	0.118
PF 0111	Poultry, fats	0.01	0.01*	0.0129	0.0129
PM 0110	Poultry meat	0.01*	0.01*	0.0026 M 0.0129 F	0.0026 M 0.0129 F
SO 0495	Rape seed	0.2	0.1	0.039	
OR 0495	Rape seed oil, Edible	0.15		0.0273	
SO 2091	Sunflower seeds, Subgroup of	0.5		0.009	
OC 0702	Sunflower seed oil, crude	0.5		0.0096	

(as) – as received; (dw) – dry weight

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for prothioconazole-desthio is 0–0.01 mg/kg bw. The International Estimated Daily Intakes (IEDIs) for prothioconazole-desthio were estimated for the 17 GEMS/Food Consumption Cluster Diets using the STMR or STMR-P values estimated by the JMPR. The results are shown in Annex 3 of the 2021 Extra JMPR Report.

The IEDIs ranged from 1–3% of the maximum ADI. The Meeting concluded that long-term dietary exposure to residues of prothioconazole from uses considered by the JMPR is unlikely to present a public health concern.

Acute dietary exposure

The ARfD for prothioconazole-desthio is 0.01 mg/kg bw for women of child-bearing age and 1 mg/kg bw for the general population. The International Estimate of Short-Term Intakes (IESTIs) for prothioconazole-desthio were calculated for the food commodities and their processed commodities for which HRs/HR-Ps

or STMRs/STMR-Ps were estimated by the present Meeting and for which consumption data were available. The results are shown in Annex 4 of the 2021 Extra JMPR Report.

The IESTIs varied from 0–8% of the ARfD for women of child-bearing age and 0% of the ARfD for children and the general population. The Meeting concluded that acute dietary exposure to residues of prothioconazole from uses considered by the present Meeting is unlikely to present a public health concern.

References

Reference Number	Author(s)	Year	Study Title
08-2112	Freitag, T., Reineke, A., Krusell, L.	2010	Determination of the residues of BYF 00587 and prothioconazole in/on winter rape after spraying of bixafen & prothioconazole EC 225 in the field in Belgium, France (North) and Germany Bayer CropScience AG, D-40789, Germany. Report No. 08-2112 and Amendments 0001 and 0002 Bayer Ref: M-385740-03-1 GLP; Unpublished 07 July 2010 (Report) 26 October 2010 (Amendment 0001) 10 April 2012 (Amendment 0002)
08-2113	Freitag, T., Krusell, L.	2010a	Determination of the residues of BYF 00587 and prothioconazole in/on winter rape after spraying of Bixafen & Prothioconazole EC 225 in the field in France (South) and Spain Bayer CropScience AG, D-40789, Germany. Report No. 08-2113 Bayer Ref: M-385556-01-1 GLP; Unpublished 01 July 2010
09-2054	Freitag, T.	2010a	Determination of the residues of BYF 00587 and prothioconazole in/on winter rape after spraying of Bixafen & Prothioconazole EC 225 in the field in France (South) and Italy Bayer CropScience AG, D-40789, Germany. Report No. 09-2054 Bayer Ref: M-393231-01-1 GLP; Unpublished 25 October 2010
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10-2134	Bomke, S., Ruhl, S., Bauer, J.	2011	Determination of the residues of fluopyram and prothioconazole in/on summer rape and winter rape after spraying of AE C656948 & JAU 6476 SE 250 in the field in Belgium, the United Kingdom, the Netherlands, France (North), Spain and France (South) Report No. 10-2134 and Amendments 001 Bayer Ref: M-414866-02-1 GLP; Unpublished 27 September 2011 (Report) 06 December 2011 (Amendment 001)
10-2244	Noss, G., Bauer, J.	2011	Determination of the residues of AE C656948 and prothioconazole in/on rape, winter after spraying of AE C656948 & JAU 6476 SE 250 in the field in Spain and France (South) Report No. 10-2244 Bayer Ref: M-414553-01-1 GLP; Unpublished 22 September 2011

Reference Number	Author(s)	Year	Study Title
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11-2013	Bomke, S.	2012	Determination of the residues of BYF 00587 and prothioconazole in/on winter rape after spray application of Bixafen & Prothioconazole EC 225 in Spain, southern France, Germany and Italy Report No. 11-2013 Bayer Ref: M-433723-01-1 GLP; Unpublished 19 June 2012
11-2124	Bomke, S., Ballmann, C.	2012	Determination of the residues of AE C656948 and prothioconazole in/on winter rape after spray application of AE C656948 & JAU 6476 SE 250 in Spain, southern France and Italy Report No. 11-2124 Bayer Ref: M-442130-01-1 GLP; Unpublished 20 November 2012
11-2137	Noss, G., Ballmann, C.	2012	Determination of the residues of BYF 00587 and prothioconazole in/on winter rape after spray application of Bixafen & Prothioconazole EC 225 in the United Kingdom Report No. 11-2137 Bayer Ref: M-429606-01-1 GLP; Unpublished 23 April 2012
16-2069	Glaubitz, J., Kowalski, N.	2018	Determination of the residues of fluopyram and prothioconazole in/on rape after spray application of AE C656948 & JAU 6476 SE 250 in France and Germany Report No. 16-2069 Bayer Ref: M-612253-01-1 GLP; Unpublished 16 January 2018
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RA-2102/02	Heinemann, O.	2003b	Determination of Residues of Tebuconazole and Prothioconazole-desthio in/on Rape after Spray Application of HWG1608 & JAU6476 250 EC in the Field in Italy and Spain Report No. RA-2102/02 Bayer Ref: M-087511-01-1 GLP; Unpublished 14 March 2003
RA-2616/07	Stuke, S., Schmeer, K.	2008a	Determination of the residues of AE C656948 and JAU 6476 in/on winter rape after spraying of AE C656948 & JAU 6476 (250 SC) in the field in Northern France, Germany and the United Kingdom Report No. RA-2616/07 Bayer Ref: M-308224-01-1 GLP; Unpublished 29 September 2008
RA-2617/07	Stuke, S., Schmeer, K.	2008b	Determination of the residues of AE C656948 and JAU 6476 in/on winter rape after spraying of AE C656948 & JAU 6476 (250 SC) in the field in Southern France and Italy Report No. RA-2617/07 Bayer Ref: M-306557-01-1 GLP; Unpublished 26 August 2008
RA-2618/06	Freitag, T., Telscher, M.	2007	Determination of the residues of JAU 6476 and Tebuconazole in/on winter rape after spraying of HWG 1608 & JAU 6476 (250 EC) in the field in Germany, Northern France, and Sweden. Report No. RA-2618/06 Bayer Ref: M-294425-01-1 GLP; Unpublished 12 November 2007
RA-2619/06	Freitag, T., Räcker, T., Telscher, M.	2007	Determination of the residues of JAU 6476 and Tebuconazole in/on summer rape and winter rape after spraying of HWG 1608 & JAU 6476 (250 EC) in the field in Italy and Spain Report No. RA-2619/06 Bayer Ref: M-295744-01-1 GLP; Unpublished 17 December 2007
08-2116	Freitag, T., Reineke, A., Krusell, L.	2012	Determination of the residues of BYF 00587, prothioconazole and tebuconazole in/on winter rape after spraying of Bixafen & Prothioconazole & Tebuconazole EC 275 in the field in Belgium,

Reference Number	Author(s)	Year	Study Title
			France (North), Germany and the Netherlands Report No. 08-2116 and Amendment No 0001 Bayer Ref: M-385576-02-1 GLP; Unpublished 05 July 2010 (Report) 10 April 2012 (Amendment No 0001)
09-2053	Freitag, T.	2010b	Determination of the residues of BYF 00587 and prothioconazole in/on Rape, Winter after spraying of bixafen & prothioconazole EC 225 in the field in Belgium, France (North), and the Netherlands Report No. 09-2053 Bayer Ref: M-390359-01-1 GLP; Unpublished 16 September 2010
09-2244	Noss, G., Krusell, L., Reineke, A.	2010	Determination of the residues of BYF 00587 and prothioconazole in/on winter rape after spraying of Bixafen & Prothioconazole EC 225 in the field in the United Kingdom Report No. 09-2244 Bayer Ref: M-388919-01-1 GLP; Unpublished 25 August 2010
09-2130	Noss, G., Krusell, L., Bauer, J.	2011	Determination of the residues of prothioconazole in/on flax/linseed after spraying of JAU 6476 EC 250 in the field in France (North) Report No. 09-2130 Bayer Ref: M-409378-01-1 GLP; Unpublished 15 June 2011
10-2073	Bomke, S.	2011	Determination of the residues of prothioconazole in/on flax/linseed after spraying of JAU 6476 EC 250 in the field in France (North) and Belgium Report No. 10-2073 and Amendment No 1 Bayer Ref: M-410275-02-1 GLP; Unpublished 29 June 2011 (Report) 25 July 2011 (Amendment No 1)
10-2254	Noss, G., Reineke, A., Ballmann, C.	2011	Determination of the residues of prothioconazole in/on flax/linseed after spraying of JAU 6476 EC 250 in the field in the United Kingdom Report No. 10-2254 Bayer Ref: M-411484-01-1 GLP; Unpublished 28 July 2011
10-2238	Bomke, S., Bauer, J., Ballmann, C.	2011	Determination of the residues of fluopyram and prothioconazole in/on sunflower after spraying of AE C656948 & JAU 6476 SE 250 in the field in Germany, Belgium and Greece Report No. 10-2238 Bayer Ref: M-420654-01-1 GLP; Unpublished 14 December 2011
10-2247	Noss, G., Ruhl, S.	2011	Determination of the residues of AE C656948 and prothioconazole in/on sunflower after spraying of AE C656948 & JAU 6476 SE 250 in the field in Spain Report No. 10-2247 Bayer Ref: M-416717-01-1 GLP; Unpublished 26 October 2011
11-2002	Glaubitz, J., Bomke, S., Diehl, P.	2013	Determination of the residues of AE C656948 and prothioconazole in/on sunflower after spray application of AE C656948 & JAU 6476 SE 250 in Germany, Belgium, Greece and southern France Report No. 11-2002 and Amendment No 1 Bayer Ref: M-447536-02-1 GLP; Unpublished 11 February 2013 (Report) 10 April 2013 (Amendment No 1)
12-2156	Noss, G., Diehl, P.	2013	Determination of the residues of AE C656948 and prothioconazole in/on sunflower after spray application of AE C656948 & JAU 6476 SE 250 in Hungary Report No. 12-2156 Bayer Ref: M-473235-01-1 GLP; Unpublished 17 December 2013
12-2009	Glaubitz, J., Diehl, P.	2013	Determination of the residues of AE C656948 and prothioconazole in/on Sunflower after Spray Application of AE C656948 & JAU 6476 SE 250 in the Field in Southern France, Spain, Italy, Portugal and Greece Report No. 12-2009 Bayer Ref: M-469299-01-1 GLP; Unpublished 05 November 2013

Reference Number	Author(s)	Year	Study Title
12-2008	Glaubitz, J.	2013	Determination of the residues of AE C656948 and prothioconazole in/on sunflower after spray application of AE C656948 & JAU 6476 SE 250 in Northern France and Belgium Report No. 12-2008 Bayer Ref: M-468618-0101 GLP, Unpublished, 30 October 2013
F12-030	Resende, G.	2016	Determinação de resíduos de Bixafen, Prothioconazole e Trifloxystrobin e seus respectivos metabólitos na cultura do girassol após a pulverização de Bixafen & Prothioconazole & Trifloxystrobin 450 SC juntamente com o adjuvante óleo metilado de soja em ensaios no Brasil Report No. F12-030 Bayer Ref: M-449336-03-1 GLP; Unpublished 14 March 2013 (Report) 17 April 2013 (Amendment No 1) 10 March 2016 (Amendment No 2)
F13-006	Resende, G.	2016	Determinação de resíduos de Bixafen, Prothioconazole e Trifloxystrobin e seus respectivos metabólitos na cultura do Girassol, após aplicação de Bixafen & Prothioconazole & Trifloxystrobin 450 SC, juntamente com o adjuvante óleo metilado de soja em ensaios no Brasil Report No. F13-006 Bayer Ref: M-487633-02-1 GLP; Unpublished 09 June 2014 (Report) 10 March 2016 (Amendment No 1)
RA-2088/00	Heinemann, O.	2002a	Determination of Residues of JAU6476-desthio on Rape after Spray Application of JAU6476 250 EC in Germany, Sweden. Northern France and Great Britain Report No. RA-2088/00 Bayer Ref: M-091148-01-1 GLP; Unpublished 14 January 2002
RA-2178/01	Heinemann, O.	2002b	Determination of Residues of JAU6476-desthio on Rape after Spray Application of JAU6476 250 EC in Germany, Northern France and Great Britain Report No. RA-2178/01 Bayer Ref: M-035525-01-1 GLP; Unpublished 08 February 2002
RA-2089/00	Heinemann, O.	2001	Determination of Residues of JAU6476-desthio on Rape after Spray Application of JAU6476 250 EC in Southern France Report No. RA-2089/00 Bayer Ref: M-074984-01-1 GLP; Unpublished 27 September 2001
RA-2179/01	Heinemann, O.	2002c	Determination of Residues of JAU6476-desthio on Rape after Spray Application of JAU6476 250 EC in Southern France. Report No. RA-2179/01, Bayer Ref: M-033374-01-1, GLP; Unpublished, 28 January 2002
08-3112	Freitag, Th., Hoffmann, M.	2011	Determination of the residues of BYF 00587 and prothioconazole in the processed fractions of winter rape (oil, screwpressed; pomace; meal; oil, solvent extracted; oil, crude; crude oil, preclarified; crude oil, neutralized and oil, refined) after spraying of Bixafen & Prothioconazole EC 225 in the field in Germany. Report No. 08-3112 and Amendment No 1 Bayer Ref: M-392874-02-1 GLP; Unpublished 12 October 2010 (Report) 02 May 2011 (Amendment No 1)
09-3245	Hoffmann, M., Teubner, L.	2013	Determination of the residues of BYF 00587 and prothioconazole in/on rape, winter and the processed fractions (oil, screwpressed; pomace; extracted meal; oil, solv. extracted; oil, crude; crude oil, preclarified; crude oil, neutralised and oil, refined) after spraying of Bixafen & Prothioconazole EC 225 in the field in the United Kingdom Report No. 09-3245 and Amendment No 1 Bayer Ref: M-449117-02-1 GLP; Unpublished 13 March 2013 (Report) 10 April 2013 (Amendment No 1)
08-3116	Freitag, Th., Hoffmann, M.,	2011	Determination of the residues of BYF 00587 and prothioconazole in the processed fractions of winter rape (oil, screwpressed; pomace; meal; oil, solvent extracted; oil, crude; crude oil, preclarified; crude

Reference Number	Author(s)	Year	Study Title
			oil, neutralized and oil, refined) after spraying of Bixafen & Prothioconazole & Tebuconazole EC 275 in the field in the Netherlands Report No. 08-3116 and Amendment No 1 Bayer Ref: M-393067-02-1 GLP; Unpublished 12 October 2010 (Report) 24 May 2011 (Amendment No 1)
12-3402	Glaubitz, J., Czaja, C.	2013	Determination of the residues of BYF 00587 and prothioconazole in/on Rape, winter and the processed fractions (oil, screwpressed; pomace; extracted meal; oil, solv. extracted; oil, crude; crude oil, preclarified; crude oil, neutralised and oil, refined) after Spray Application of Bixafen & Prothioconazole EC 225 in the Field in Germany and Southern France Report No. 12-3402 Bayer Ref: M-474270-01-1 GLP; Unpublished 18 December 2013
AFFC08-022R	Ballantine, J.	2013	Prothioconazole: Magnitude of the Residue on Sunflower Report No. AFFC08-022R Bayer Ref: M-527444-01-1 GLP; Unpublished 15 March 2013

PYDIFLUMETOFEN (309)

First draft prepared by Ms M Thomas, Pest Management Regulatory Agency, Canada

EXPLANATION

Pydiflumetofen is a broad-spectrum fungicide belonging to the carboxamide group. It acts through inhibition of succinate dehydrogenase in complex II of fungal mitochondrial respiration.

Pydiflumetofen was first evaluated for toxicology and residues by the JMPR in 2018. An ADI of 0–0.1 mg/kg bw and an ARfD of 0.3 mg/kg bw were established. The residue definition for compliance with the MRL for plant and animal commodities, and dietary risk assessment for plant commodities is *pydiflumetofen*. The residue definition for dietary risk assessment for animal commodities other than mammalian liver and kidney is the *sum of pydiflumetofen and 2,4,6-TCP (2,4,6-Trichlorophenol) and its conjugates, expressed as pydiflumeto*fen and for dietary risk assessment for mammalian liver and kidney is the *sum of pydiflumetofen, 2,4,6-trichlorophenol (2, 4, 6-TCP) and its conjugates, and SYN547897 and its conjugates, expressed as pydiflumetofen. The residue is fat-soluble*.

The 2018 JMPR noted that pydiflumetofen residues are very persistent in soil (up to 2380 days DT_{50}) and may be taken up by rotational crops.

At the Fifty-first Session of the CCPR, pydiflumetofen was scheduled for evaluation by the 2020 JMPR for several new uses, which was postponed to the 2021 Extra JMPR.

The Meeting received information from the manufacturer on use patterns, supervised residue trials on lemons, oranges, grapefruits, apples, pears, peaches, cherries, plums, blueberries, strawberries, bulb onions, green onions, cabbage, cauliflower, broccoli, beans with pods, peas with pods, beans without pods, peas without pods, sugar beet, carrots, radish, sorghum, almonds, pecans, sunflower and cottonseed, and processing studies.

RESIDUE ANALYSIS

Analytical methods

The LC-MS/MS analytical method GRM061.03A reviewed by the 2018 JMPR was used to quantify all pydiflumetofen residues in the supervised crop field trials and processing studies submitted to the current meeting. The pydiflumetofen recoveries determined during the concurrent method validation are summarized in Table 1. Based on these results, the method is considered to be sufficiently validated

Table 1 Concurrent recoveries of pydiflumetofen from various crop matrices

Matrix	Fortification level [mg/kg]	Sample size (n)	Individual recoveries [%]	Range of recoveries [%]	Mean recovery [%]	RSD [%]	Reference
Orange	0.01	6	85, 94, 109, 82, 108, 117	82-117	99	14.1	Report No. TK0268661
	1.0	5	133, 125, 122, 96, 104	96-133	116	13.3	
	10.0	3	100, 109, 121	100-121	110	9.9	
Tangerine	0.01	3	103, 83, 105	83-105	97	12.6	

Matrix	Fortification level [mg/kg]	Sample size (n)	Individual recoveries [%]	Range of recoveries [%]	Mean recovery [%]	RSD [%]	Reference
	1.0	2	129, 75	75-129	102	N/A	
	5.0	3	125, 128, 127	125-128	127	1.1	
Lemon	0.01	4	99, 77, 81, 99	77-99	89	12.6	
	1.0	3	90, 122, 131	90-131	114	8.9	
	5.0	3	113, 108, 108	108-113	110	2.7	
Grapefruit	0.01	3	106, 123, 97	97-123	109	12.5	
	1.0	3	123, 85, 97	85-123	102	19.3	
Orange oil	0.01	1	85	N/A	N/A	N/A	
	1.0	1	83	N/A	N/A	N/A	
	75	3	103, 103, 92	92-103	99	6.5	
Orange dried pulp	0.01	1	116	N/A	N/A	N/A	
	1.0	1	121	N/A	N/A	N/A	
	5.0	3	118, 126, 109	109-126	118	7.3	
Orange peel	0.01	2	126, 101	101-126	114	N/A	
	1.0	2	113, 108	108113	110	N/A	
	10	3	83, 85, 89	83-89	86	3.5	
Orange pulp	0.01	1	92	N/A	N/A	N/A	
	1.0	1	121	N/A	N/A	N/A	
Orange juice	0.01	1	120	N/A	N/A	N/A	
	1.0	1	104	N/A	N/A	N/A	
Tangerine peel	0.01	1	116	N/A	N/A	N/A	
	1.0	1	91	N/A	N/A	N/A	
Tangerine pulp	0.01	1	89	N/A	N/A	N/A	
	1.0	1	102	N/A	N/A	N/A	
Lemon peel	0.01	1	103	N/A	N/A	N/A	
	1.0	1	92	N/A	N/A	N/A	
Lemon pulp	0.01	1	110	N/A	N/A	N/A	
	1.0	1	115	N/A	N/A	N/A	
Grapefruit peel	0.01	1	81	N/A	N/A	N/A	
	1.0	1	96	N/A	N/A	N/A	
Grapefruit pulp	0.01	1	91	N/A	N/A	N/A	
	1.0	1	93	N/A	N/A	N/A	
Apple	0.01	3	104, 100, 105	100-105	103	2.6	Report No. TK0103850
	0.10	3	100, 102, 104	100-104	102	2.0	
	0.20	3	111, 105, 104	104-111	107	3.6	
Pear	0.01	3	109, 99, 105	99-109	104	4.8	
	0.10	3	100, 101, 103	100-103	101	1.5	
	0.20	3	112, 110, 110	110-112	111	1.1	
Apple	0.01	7	74, 90, 90, 93, 72, 108, 80	72-108	87	14.4	Report TK0103855
	1.0	7	90, 83, 73, 102, 87, 95, 82	73-102	88	10.8	

Matrix	Fortification level [mg/kg]	Sample size (n)	Individual recoveries [%]	Range of recoveries [%]	Mean recovery [%]	RSD [%]	Reference
Pear	0.01	4	76, 75, 72, 93	72-93	79	12.0	
	1.0	4	90, 99, 78, 93	78-99	90	10.0	
Apple canned	0.01	1	73	N/A	N/A	N/A	
	1.0	1	81	N/A	N/A	N/A	
Apple wet pomace	0.01	1	120	N/A	N/A	N/A	
	1.0	2	117, 96	96-117	107	N/A	
	3.0	1	98	N/A	N/A	N/A	
Apple juice	0.01	1	72	N/A	N/A	N/A	
	1.0	1	81	N/A	N/A	N/A	
Apple sauce	0.01	1	80	N/A	N/A	N/A	
	1.0	1	71	N/A	N/A	N/A	
Apple dried	0.01	1	92	N/A	N/A	N/A	
	1.0	1	107	N/A	N/A	N/A	
Pear canned	0.01	1	99	N/A	N/A	N/A	
	0.1	1	96	N/A	N/A	N/A	
Pear wet pomace	0.01	1	101	N/A	N/A	N/A	
	1.0	2	119, 82	101	N/A	N/A	
	3.0	1	100	N/A	N/A	N/A	
Pear dried fruit	0.01	1	96	N/A	N/A	N/A	
	1.0	1	105	N/A	N/A	N/A	
Pear juice	0.01	1	87	N/A	N/A	N/A	
	1.0	1	79	N/A	N/A	N/A	
Cherry	0.01	12	93, 100, 93, 93, 84, 73, 78, 87, 82, 75, 82, 82	73-100	85	10	Report No. AAFC16-047R
	1.0	12	91, 92, 94, 94, 90, 84, 89, 88, 87, 84, 85, 82	82-94	8	5	
	5	1	90	N/A	N/A	N/A	
Peach	0.01	8	88, 80, 80, 79, 89, 97, 88, 82	82-97	86	7	Report No. AAFC16-048R
	1.0	6	93, 89, 94, 86, 93, 85	85-94	90	4	
Fresh plum	0.01	7	73, 75, 70, 78, 82, 78, 82	70-82	77	6	Report No. AAFC16-049R
	1.0	3	88, 88, 93	88-93	90	3	
	5.0	1	90	N/A	N/A	N/A	
Dry plum	0.01	5	103, 84, 82, 91, 88	82-103	90	9	
	1.0	1	98	N/A	N/A	N/A	
	5.0	1	92	N/A	N/A	N/A	
Peach fresh fruit	0.01	3	92, 100, 98	92-100	97	4.3	

Matrix	Fortification level [mg/kg]	Sample size (n)	Individual recoveries [%]	Range of recoveries [%]	Mean recovery [%]	RSD [%]	Reference
	0.1	2	95, 91	91-95	93	N/A	Report No. TK0270167
	0.5	2	98, 98	N/A	98	N/A	
Plum fresh fruit	0.01	3	105, 98, 94	94-105	99	5.6	
	0.1	2	99, 99	N/A	99	N/A	
	1.5	2	89, 93	89-93	91	N/A	
Plum juice	0.01	1	106	N/A	N/A	N/A	
	0.1	1	87	N/A	N/A	N/A	
Plum puree	0.01	1	83	N/A	N/A	N/A	
	0.1	1	86	N/A	N/A	N/A	
	0.5	2	104, 102	102-104	103	N/A	
Prunes	0.01	2	94, 86	86-94	90	N/A	
	0.1	2	89, 91	89-91	90	N/A	
	4.0	2	114, 113	113-114	114	N/A	
Blueberry	0.01	3	85, 91, 90	85-91	89	3	Report No. 11763
	0.10	2	99, 98	98-99	99	N/A	
	10.0	3	90, 93, 90	90-93	92	1.6	
Strawberry	0.01	3	88, 82, 99	82-99	90	7.8	Report No. 11159
	0.10	3	90, 88, 82	82-90	87	3.9	
Onion bulb	0.01	3	115, 85, 88	85-115	96	17.6	Report No. TK0268909
	1.0	3	105, 95, 100	95-105	100	5.0	
Green onion	0.01	3	82, 87, 92	82-92	87	5.5	
	1.0	2	89, 90	89-90	89	N/A	
	5.0	3	113, 91, 95	91-113	100	12.0	
Cabbage	0.01	5	92, 112, 84, 117, 77	77-117	96	17.9	Report No. TK0268910
	0.25	3	99, 73, 75	73-99	83	17.5	
	1.0	1	84	N/A	N/A	N/A	
	10.0	3	115, 111, 112	111-115	113	1.9	
Broccoli	0.01	2	84, 92	84-92	88	N/A	
	0.25	1	90	N/A	N/A	N/A	
	5.0	3	80, 86, 89	80-89	85	5.5	
Cauliflower	0.01	1	110	N/A	N/A	N/A	
	0.25	1	117	N/A	N/A	N/A	
Bean without pod	0.01	3	102, 86, 85	85-102	91	8.6	Report No. TK0268911
	0.10	1	116	N/A	N/A	N/A	
	0.25	1	89	N/A	N/A	N/A	
	0.50	1	98	N/A	N/A	N/A	
Pea with pod	0.01	2	88, 84	84-88	86	N/A	
	0.25	2	92, 84	84-92	88	N/A	
	2.5	2	94, 94	94	N/A	N/A	
Bean with pod	0.01	4	97, 88, 101, 85	85-101	93	7.0	
	0.10	1	98	N/A	N/A	N/A	

Matrix	Fortification level [mg/kg]	Sample size (n)	Individual recoveries [%]	Range of recoveries [%]	Mean recovery [%]	RSD [%]	Reference
	0.50	3	90, 98, 98	90-98	95	4.0	
Pea without pod	0.01	2	97, 92	92-97	95	N/A	
	0.10	2	94, 91	91-94	93	N/A	
Bean with pod	0.01	4	103, 100, 96, 81	81-103	95	10.2	Report No. TK0269135
	0.10	3	100, 97, 99	97-100	99	1.3	
Pea with pod	0.01	4	90, 89, 93, 86	86-93	90	2.8	
	0.10	3	99, 89, 100	89-100	96	6.7	
Bean without pod	0.01	3	96, 92, 99	92-99	96	4.0	
	0.10	3	98, 102, 99	98-102	99	2.4	
Pea without pod	0.01	3	88, 91, 84	84-91	88	4.0	
	0.10	3	98, 93, 93	93-98	95	3.0	
Carrot roots	0.01	3	104, 93, 78	78-104	92	13.8	Report No. TK0268658
	1.0	2	82, 97	82-97	90	N/A	
	10	1	107	N/A	N/A	N/A	
Radish roots	0.01	3	91, 80, 100	80-100	90	11.2	
	1.0	2	99, 101	99-101	100	N/A	
	10	3	113, 111, 113	111-113	112	1.0	
Radish leaves	0.01	3	104, 101, 78	78-104	94	15.1	
	1.0	2	82, 94	82-94	88	N/A	
	150	3	94, 92, 88	88-94	91	3.5	
Sugar beet roots	0.01	6	118, 101, 132, 79, 79, 113	79-132	104	20.6	
	1.0	4	90, 90, 130, 94	90-130	101	19.4	
	10	6	112, 114, 115, 112, 114, 115	112-115	114	1.1	
Sugar beet leaves	0.01	4	109, 122, 84, 105	84-122	105	14.9	
	1.0	3	85, 79, 90	79-90	85	6.8	
	260	6	86, 90, 86, 104, 85, 84	84-104	89	8.4	
Sugar beet refined sugar	0.01	1	85	N/A	N/A	N/A	
	10	1	108	N/A	N/A	N/A	
Sugar beet molasses	0.01	1	88	N/A	N/A	N/A	
	1.0	1	98	N/A	N/A	N/A	
Sugar beet dried pulp	0.01	2	93, 113	93-113	98	N/A	
	1.0	1	132	N/A	N/A	N/A	
	10	4	104, 100, 99, 82	82-104	96	10.1	
Sugar beet roots	0.01	3	85, 95, 87	85-95	89	5.6	Report No. TK0269131
	0.10	3	101, 97, 96	96-101	98	2.5	
Sugar beet leaves	0.01	3	89, 88, 93	88-93	89	2.6	
	0.10	3	94, 101, 100	94-101	98	4.2	

Matrix	Fortification level [mg/kg]	Sample size (n)	Individual recoveries [%]	Range of recoveries [%]	Mean recovery [%]	RSD [%]	Reference
Sorghum forage	0.01	7	100, 110, 109, 105, 114, 119, 84	84-119	106	11.0	Report No. TK0294708
	1.0	3	90, 89, 94	89-94	91	3.0	
	2.0	3	94, 93, 91	91-94	92	2.0	
Sorghum grain	0.01	4	92, 74, 77, 114	74-114	89	20.0	
	1.0	3	119, 81, 88	81-119	96	21.0	
	5.0	3	93, 97, 101	93-101	97	1.0	
Sorghum stover	0.01	3	120, 85, 103	85-120	103	14.0	
	1.0	3	92, 117, 104	92-117	104	12.0	
	10.0	1	96	N/A	N/A	N/A	
Sorghum flour	0.01	2	108, 109	108-109	109	13.0	
	1.0	1	88	N/A	N/A	N/A	
	5.0	3	94, 79, 88	79-94	87	5.0	
Sorghum aspirated grain fractions	0.1	4	97, 93, 107, 90	90-107	97	8.0	
	160	1	101	N/A	N/A	N/A	
	300	3	94, 90, 91	90-94	92	2.0	
Almond nutmeat	0.01	2	106, 85	85-106	96	N/A	Report No. TK0173383
	0.10	2	94, 114	94-114	104	N/A	
Almond hulls	0.01	2	110, 93	93-110	102	N/A	
	0.10	2	95, 115	95-115	105	N/A	
	10	1	113	N/A	N/A	N/A	
Almond oil	0.01	1	99	N/A	N/A	N/A	
	0.10	1	100	N/A	N/A	N/A	
Pecan nutmeat	0.01	3	104, 85, 97	85-104	95	8.2	
	0.10	3	97, 114, 77	77-114	96	15.8	
Cotton undelinted seed	0.01	9	87, 80, 128, 93, 107, 99, 99, 106, 109	80-128	101	13.9	Report No. TK0265622
	0.10	12	87, 92, 96, 93, 98, 104, 107, 98, 100, 94, 104, 94	87-107	97	5.8	
	5.0	3	110, 110, 108	108-110	109	1.2	
Gin by-products	0.01	3	113, 119, 118	113-119	117	2.7	
	0.10	3	112, 106, 121	106-121	113	6.4	
	5.0	3	75, 78, 88	75-88	80	8.5	
Cotton hulls	0.01	2	99, 109	99-109	104	N/A	
	0.10	1	100	N/A	N/A	N/A	
	1.0	3	115, 109, 120	109-120	115	4.6	
Cotton meal	0.01	1	101	N/A	N/A	N/A	
	1.0	3	106, 111, 108	106-111	1.8	2.4	
Cotton refined oil	0.01	1	90	N/A	N/A	N/A	
	1.0	3	90, 120, 128	90-128	113	17.9	

Matrix	Fortification level [mg/kg]	Sample size (n)	Individual recoveries [%]	Range of recoveries [%]	Mean recovery [%]	RSD [%]	Reference
Sunflower seed	0.01	6	83, 70, 77, 79, 79, 81	70-83	78	5.8	Report No. TK0265867
	0.10	1	96	N/A	N/A	N/A	
	0.50	4	90, 102, 108, 78	78-108	95	14	
	1.25	2	98, 103	98-103	101	N/A	
Sunflower alkali refined oil	0.01	1	70	N/A	N/A	N/A	
	0.50	1	70	N/A	N/A	N/A	
Sunflower meal	0.01	1	67	N/A	N/A	N/A	
	0.50	1	111	N/A	N/A	N/A	
Sunflower seed	0.01	4	95, 100, 95, 96	95-100	97	3.0	Report No. TK0269138
	0.10	3	95, 92, 93	92-95	93	1.6	

USE PATTERNS

Pydiflumetofen is a broad-spectrum fungicide of the chemical group *N*-methoxy-(phenyl-ethyl)-pyrazole-carboxamide and belongs to the SDHI (Succinate Dehydrogenase Inhibitors) fungicide group. It inhibits succinate dehydrogenase in complex II of fungal mitochondrial respiration. The use patterns of the suspension concentrate formulations registered in the USA are summarised in Table 2.

Table 2 Summary of registered use patterns from the USA

Crop (CCN)	Formulation Type	Application						PHI (days)
		Method	No.	Rate/ application (g ai/ha)	Re-treatment interval (days)	Water (L/ha)	Rate/season (g ai/ha)	
Citrus Fruit Crop Group 10-10[a]	SC	Foliar	4	85	7	Ground (NS) Aerial (Min 94)	340	0
Pome Fruit Crop Group 111-10	SC	Foliar	4	50	7	Ground (NS) Aerial (Min 94)	200	30
Stone Fruit Crop Group 12-12[c]	SC	Foliar	4	75	7	Ground (NS) Aerial (Min 94)	300	0
Bushberry Crop Subgroup 13-07B[d]	SC	Foliar	2	150	7	NS	300	0
	SC	Foliar	4	75	7	NS	300	0
Berry, Low Growing	SC	Foliar	2	150	7	NS	300	0

Crop (CCN)	Formulation Type	Application						PHI (days)
		Method	No.	Rate/ application (g ai/ha)	Re-treatment interval (days)	Water (L/ha)	Rate/season (g ai/ha)	
Subgroup 13-07G (except cranberry)[e]	SC	Foliar	4	75	7	NS	300	0
Bulb Vegetables Crop Group 3-07[f]	SC	Foliar	3	125	7	Ground (Min 140) Aerial (Min 94)	375	7
Bulb Onions [g]	SC	Foliar	4	75	7	NS	300	7
Greens onions [g]		Foliar	3	75	7	NS	225	7
Brassica Head and Stem Vegetables Crop Group 5-16[h]	SC	Foliar	3	125	7	Ground (NS) Aerial (Min 28)	375	0
	SC	Foliar	4	75	7	NS	300	1
Legume Vegetables, Dry and Succulent, Crop Group 6[i]	SC	Foliar	2	200	14	NS	400	14 (includes peas/beans and pea vines and hay) Do not feed or harvest cowpea forage or hay
Dried Shelled Pea and Bean Crop Subgroup 6C[j]	SC	Foliar	4 (2 for pea vines and hay)	78	14	NS	314 (157 for pea vines and hay)	14 (includes peas/beans and pea vines and hay) Do not feed or harvest cowpea forage or hay
Root Vegetables Crop Subgroup 1A[k]	SC	Foliar	4	75	7	NS	300	7 Do not harvest tops of carrots or radish for feed or food.

Crop (CCN)	Formulation Type	Application						PHI (days)
		Method	No.	Rate/ application (g ai/ha)	Re-treatment interval (days)	Water (L/ha)	Rate/season (g ai/ha)	
Sorghum	SC	Foliar	2	126	7	NS	252	21 (grain and stover) 30 (forage)
Tree Nuts Crop Group 14-12[l]	SC	Foliar	3	100	7	Ground (NS) Aerial (Min 94)	300	14
	SC	Foliar	4	75	14	Ground (NS) Aerial (Min 94)	300	14
Cotton	SC	Foliar	3	75	10	NS	225	45
Sunflower (Sunflower Seed Subgroup 20B)[m]	SC	Foliar	2	200	14	NS	400	30

Note: An adjuvant (type not specified) may be added at recommended rates.

[a] Crops including cultivars, varieties, and/or hybrids of Australian desert lime; Australian finger-lime; Australian round lime; Brown River finger lime; Calamondin; Citron; Citrus hybrids; Grapefruit; Japanese summer grapefruit; Kumquat; Lemon; Lime; Mediterranean mandarin; Mount white lime; New Guinea wild lime; Orange, sour; Orange, sweet; Pummelo; Russell River lime; Satsuma mandarin; Sweet lime; Tachibana orange; Tahiti lime; Tangelo; Tangerine (mandarin); Tangor; Trifoliate orange and Uniq fruit

[b] Crops including cultivars, varieties, and/or hybrids apple; azarole; crabapple; loquat; mayhaw; medlar pear; pear, Asian; quince; quince, Chinese; quince, Japanese; tejocote

[c] Crops including cultivars, varieties, and/or hybrids of apricot; apricot, Japanese; capulin; cherry, black; cherry, Nanking; cherry, sweet; cherry, tart; jujube, Chinese; nectarine; peach; plum; plum, American; plum, beach; plum, Canada; plum, cherry; plum, chickasaw; plum, damson; plum, Japanese; plum, klamath; plumcot; plum, prune; sloe

[d] Crops including cultivars, varieties, and/or hybrids of aronia berry; blueberry, highbush; blueberry, lowbush; Buffalo currant; Chilean guava; cranberry, highbush; currant, black; currant, red; elderberry; European barberry; gooseberry; honeysuckle, edible; huckleberry; jostaberry; juneberry (Saskatoon berry); lingonberry; native currant; salal; sea buckthorn

[e] Crops including cultivars, varieties, and/or hybrids of bearberry bilberry cloudberry muntries partridgeberry strawberry

[f] Crops including cultivars, varieties, and/or hybrids of Bulb onion subgroup 3-07A: Daylily, bulb Fritillaria, bulb Garlic, bulb Garlic, great-headed, bulb Garlic, serpent, bulb Lily, bulb Onion, bulb Onion, Chinese, bulb Onion, pearl Onion, potato, bulb Shallot, bulb AND Green onion subgroup 3-07B: Chive, fresh leaves Chive, Chinese, fresh leaves Elegans hosta Fritillaria, leaves Kurrat Lady's leek, Leek, wild Onion, Beltsville bunching Onion, fresh Onion, green Onion, macrostem Onion, tree, tops Onion, Welsh, tops Shallot, fresh leaves

[g] Crops including cultivars, varieties, and/or hybrids of broccoli, cabbage, cauliflower, Brussels sprouts and Cabbage, Chinese napa

[h] Crops including cultivars, varieties, and/or hybrids of beet, garden; beet, sugar; burdock, edible; carrot; cassava, bitter and sweet; celeriac (celery root); chervil, turnip-rooted; chicory; dasheen (taro); parsnip; radish; radish, oriental (daikon); rutabaga; salsify, black; sweet potato; tanier (cocoyam); turnip; yam, true

[i] Crops including cultivars, varieties, and/or hybrids of Bean (Lupinus spp.): grain lupin; sweet lupin; white lupin; white sweet lupin; Bean (Phaseolus spp.): field bean; kidney bean; lima bean; navy bean; pinto bean; runner bean; snap bean; tepary

bean; wax bean; Bean (Vigna spp.): adzuki bean; blackeyed pea; catjang; Chinese longbean; cowpea; crowder pea; moth bean; mung bean; rice bean; southern pea; urd bean; yardlong bean; Pea (Pisum spp.): dwarf pea; edible-podded pea; English pea; field pea; garden pea; green pea; snowpea; sugar snap pea; broad bean (fava bean); chickpea (garbanzo bean); guar; jackbean; lablab bean (hyacinth bean); lentil; pigeon pea; soy (immature seed); sword bean

[j] Crops including cultivars, varieties, and/or hybrids of Bean (Lupinus spp.): grain lupin, sweet lupin, white lupin, white sweet lupin; Bean (Phaseolus spp.): field bean, kidney bean, lima bean (dry), navy bean, pinto bean, tepary bean; Bean (Vigna spp.): adzuki bean, blackeyed pea, catjang cowpea, crowder pea, moth bean, mung bean, rice bean, southern pea, urd bean, broad bean (dry), chickpea (garbanzo bean), guar, lablab bean (hyacinth bean), lentil, pigeon pea; Pea (Pisum spp.): field pea

[k] Crops including cultivars, varieties, and/or hybrids of beet, garden; burdock, edible; carrot; celeriac; chervil, turnip-rooted; chicory; ginseng; horseradish; parsley, turnip-rooted; parsnip; radish; radish, oriental; rutabaga; salsify; salsify, black; salsify, Spanish; skirret; turnip

[l] Crops including cultivars, varieties, and/or hybrids of African nut-tree; beechnut; Brazil nut; Brazilian pine; bunya; bur oak; butternut; cajou nut; candlenut; cashew; chestnut; chinquapin; coconut; coquito nut; dika nut; ginkgo; Guiana chestnut; heartnut; hickory nut; Japanese horse-chestnut; macadamia nut; mongongo nut; monkey-pot; monkey puzzle nut; okari nut; pachira nut; peach palm nut; pequi; pili nut; pine nut; sapucaia nut; tropical almond; walnut, black; walnut, English; yellowhorn

[m] Crops including cultivars, varieties, and/or hybrids of calendula; castor oil plant; Chinese tallowtree; euphorbia; evening primrose; jojoba; niger seed; rose hip; safflower; stokes aster; sunflower; tallowwood; tea oil plant; Vernonia

Rotational Crop Restrictions

Crop, Crop Group, or Crop Subgroup	Plant-Back Interval
Bushberry Crop, Subgroup 13-07B Low Growing Berry, Crop Subgroup 13-07G, except cranberry Brassica Head and Stem Vegetable, Crop Group 5-16 Brassica Leafy Greens, Crop Subgroup 4-16B Bulb Vegetables, Crop Goup 3-07 Canola (Rapeseed Crop Subgroup 20A) Carrots Cereals (barley, oats, wheat, triticale, rye) Citrus Fruit, Crop Group 10-10 Corn, field Corn, sweet Cotton (Cottonseed, Crop Group 20C) Cucurbit Vegetables, Crop Group 9 Fruiting Vegetables, Crop Group 8-10 Grape and Small Fruit Vine Climbing Subgroup (except Fuzzy Kiwifruit), Crop Subgroup 13-07F Leaf Petiole Vegetables, Crop Subgroup 22B Leafy Greens, Crop Subgroup 4-16A Leaves of Root and Tuber Vegetables, Crop Group 2 Legume Vegetables, Succulent or Dried, Crop Group 6, except cowpea forage and cowpea hay Peanut Peppers Pome Fruit, Crop Group 11-10 Potato Quinoa Root Vegetables, Crop Subgroup 1A	0 day

Crop, Crop Group, or Crop Subgroup	Plant-Back Interval
Sorghum Soybeans, excluding soybean forage, hay, and silage Stone Fruit, Crop Group 12-12 Sunflower, Crop Subgroup 20B Tomatoes and tomatillos Tree Nuts, Crop Group 14-12 Tuberous and Corm Vegetables, Crop Subgroup 1C	
Grasses Grown for Seed Non-grass Animal Feeds, Crop Group 18 Rice Tobacco	30 days
All other crops Intended for Food and Feed	365 days

RESULTS OF SUPERVISED RESIDUE TRIALS ON CROPS

The Meeting received information on supervised field trials for pydiflumetofen on the following crops or crop groups:

Crop		Table No.
Citrus fruits		3
Pome fruits		4
Stone fruits		5
Bushberries		
	Blueberries	6
Low growing berries		
	Strawberries	7
Bulb vegetables		8
Brassica vegetables (except Brassica leafy vegetables)		9
Legume vegetables		
	Beans with pods	10
	Peas with pods	11
	Beans without pods	12
	Peas without pods	13
Root vegetables		14
Cereal grains		
	Sorghum	15
Tree nuts		16
Oilseeds		
	Sunflower	17
	Cotton	18

Crop	Table No.
Leaves of root vegetables	19
Straw, Fodder and Forage of Cereal Grains and Grasses (Including Buckwheat Fodder) (Forage)	
Sorghum forage	20
Straw, Fodder and Forage of Cereal Grains and Grasses (Including Buckwheat Fodder) (Straws and Fodder Dry)	
Sorghum stover	21
Almond hulls	22
Cotton gin by-products	23

Trials were well documented with laboratory and field reports. Laboratory reports included method validation with procedural recoveries from spiking at residue levels bracketing 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. Residue data are recorded unadjusted for recovery.

Residue values from the trials conducted in accordance with the critical 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.

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.

Citrus fruits

Twenty-seven independent field trials were conducted on citrus fruits (oranges: 10 trials; grapefruits: 7 trials; lemons: 6 trials and tangerine: 4 trials) during the 2016 and 2017 growing seasons in the USA (Crawford, 2018, Report No. TK0268661).

Each trial site consisted of one untreated control plot and two treated plots. The treated plots received four foliar airblast applications of a suspension concentrate (SC) formulation of pydiflumetofen (nominal concentration 20% w/w) at 82–91 g ai/ha per application, with retreatment intervals of 6–9 days, totaling 336–345 g ai/ha. The applications were made with concentrate spray volumes (385–845 L/ha) and with dilute spray volumes (1130–2989 L/ha). A non-ionic surfactant (NIS) or crop oil concentrate (COC) was added to the spray mixture for all applications. For all trials, samples were collected 0 days after the last application (DALA). For the decline trials, mature orange samples were collected at 7, 14, 21, and 28 DALA.

Samples were analyzed using the LC-MS/MS Method GRM061.03A. Acceptable concurrent recoveries were reported for all citrus fruits at fortification levels of 0.01 to 10.0 mg/kg, thus validating the method. The limit of quantitation (LOQ), determined as the lowest level of method validation (LLMV), was 0.01 mg/kg.

Treated samples were maintained frozen until analysis for a maximum storage interval of 197 days. Adequate storage stability data are available to support the storage conditions and intervals for samples in the current trials.

Table 3 Residues of pydiflumetofen in citrus fruits from trials conducted in the USA following application of an SC formulation

Location; Year, Variety (Trial ID)	Application rate per treatment				Growth Stage (BBCH)	Portion Analysed	DALA	Pydiflumetofen Residues mg/kg (average)
	No.	Rate (g ai/ha)	RTI	Water (L/ha)				
US GAP	4	85	7	Ground (NS) Aerial (Min 94)			0	
LEMONS								
Oviedo, FL; USA 2016 Meyer (860.1500-16-433-01A-11)	4	85 84 85 85	- 7 7 7	712 716 714 707	BBCH 77 BBCH 79 BBCH 79 BBCH 83	Fruit	0	0.390, 0.423, 0.321 (0.378)
Fresno, CA; USA 2016 Lisbon (860.1500-16-433-01A-15)	4	85 85 85 84	- 6 8 7	712 716 714 707	BBCH 85 BBCH 85 BBCH 85 BBCH 89	Fruit	0	0.530, 0.383 (0.457)
Porterville, CA; USA 2016/2017 Pryor (860.1500-16-433-01A-17)[a]	4	83 84 85 84	- 7 6 7	502 496 518 503	BBCH 81 BBCH 81 BBCH 83 BBCH 83	Fruit	0	0.310, 0.187 (0.248)
Reedley, CA; USA 2016 Meyer (860.1500-16-433-01A-19)	4	87 87 86 85	- 8 7 6	1181 1190 1167 1160	BBCH 83 BBCH 83 BBCH 84 BBCH 88	Fruit	0	0.257, 0.324 (0.290)
San Luis Obispo, CA; USA 2017 Lisbon (860.1500-16-433-01A-21)	4	84 84 84 84	- 8 7 6	953 1110 1133 1045	BBCH 81 BBCH 81 BBCH 81 BBCH 85	Fruit	0	0.036, 0.016, 0.012 (0.021)
Porterville, CA; USA 2017 Meyer (860.1500-16-433-01A-24)[a]	4	84 85 84 84	- 7 7 7	764 785 800 780	BBCH 81 BBCH 81 BBCH 83 BBCH 89	Whole fruit	0	0.139, 0.147 (0.143)
						Flesh	0	< 0.01
						Peel	0	0.393
TANGERINES								

Location; Year, Variety (Trial ID)	Application rate per treatment				Growth Stage (BBCH)	Portion Analysed	DALA	Pydiflumetofen Residues mg/kg (average)
	No.	Rate (g ai/ha)	RTI	Water (L/ha)				
US GAP	4	85	7	Ground (NS) Aerial (Min 94)			0	
Clermont, FL; USA 2016 Clementine (860.1500-16-433-01A-01)	4	85 83 84 85	- 7 7 7	385 403 387 404	BBCH 81 BBCH 81 BBCH 85 BBCH 89	Fruit	0	0.598, 0.521 (0.560)
Oviedo, FL; USA 2016 Murcott (860.1500-16-433-01A-09)	4	84 83 85 85	- 7 7 7	1599 1573 1606 1601	BBCH 79 BBCH 79 BBCH 81 BBCH 83	Fruit	0	0.281, 0.283 (0.282)
Fresno, CA; USA 2016 Mandarin (860.1500-16-433-01A-14)	4	86 85 86 85	- 6 8 7	2381 2352 2380 2351	BBCH 85 BBCH 85 BBCH 85 BBCH 89	Fruit	0	0.238, 0.239, 0.255 (0.244)
Raymondville, TX; USA 2017 Dancy (860.1500-16-433-01A-27)	4	85 85 86 87	- 6 8 7	2932 2930 2951 2989	BBCH 79-81 BBCH 79-81 BBCH 81-83 BBCH 83-89	Whole fruit	0	0.162, 0.186 (0.174)
						Flesh	0	< 0.01
						Peel	0	0.606
ORANGES								
Groveland, FL; USA 2016 Hamlin (860.1500-16-433-01A-02)	4	84 83 84 84	- 7 7 7	2338 1536 1513 1679	BBCH 81 BBCH 81 BBCH 85 BBCH 89	Fruit	0	0.301, 0.312 (0.307)
Clermont, FL; USA 2017 Valencia (860.1500-16-433-01A-03)	4	84 84 86 83	- 7 7 7	413 416 430 464	BBCH 81 BBCH 81 BBCH 83 BBCH 85	Fruit	0	0.290, 0.176 (0.233)
Lake Hamilton, FL; USA 2017 Valencia (860.1500-16-433-01A-04)	4	84 85 84 85	- 7 7 7	1258 1240 1254 1321	BBCH 81 BBCH 81 BBCH 83 BBCH 85	Fruit	0	0.178, 0.155, 0.158 (0.164)

Location; Year, Variety (Trial ID)	Application rate per treatment				Growth Stage (BBCH)	Portion Analysed	DALA	Pydiflumetofen Residues mg/kg (average)
	No.	Rate (g ai/ha)	RTI	Water (L/ha)				
US GAP	4	85	7	Ground (NS) Aerial (Min 94)			0	
Fort Pierce, FL; USA 2016 Hamlin (860.1500-16-433-01A-07)	4	84 84 84 84	- 7 7 6	748 747 746 750	BBCH 85 BBCH 85 BBCH 85 BBCH 85	Fruit	0	0.548, 0.765, 0.714 (0.676)
Oviedo, FL; USA 2016 Navel (860.1500-16-433-01A-10)[b]	4	85 86 86 84	- 7 7 7	702 712 715 694	BBCH 79 BBCH 79 BBCH 81 BBCH 83	Fruit	0	0.186, 0.207 (0.197)
							7	0.188, 0.166 (0.177)
							14	0.186, 0.236 (0.211)
							20	0.094, 0.154 (0.124)
							28	0.103, 0.098 (0.100)
Raymondville, TX; USA 2016 Valencia (860.1500-16-433-01A-12)	4	86 84 87 84	- 7 7 7	724 733 736 709	BBCH 79 BBCH 79 BBCH 79 BBCH 81-83	Fruit	0	0.220, 0.507 (0.364)
Richgrove, CA; USA 2016 Atwood (860.1500-16-433-01A-16)	4	85 84 84 84	- 7 7 7	1724 1698 1685 1695	BBCH 81 BBCH 81 BBCH 81 BBCH 83	Fruit	0	0.152, 0.140 (0.146)
							7	0.122, 0.117 (0.120)
							14	0.206, 0.138 (0.172)
							21	0.140, 0.153 (0.146)
							28	0.153, 0.127 (0.140)
Oviedo, FL; USA 2016 Valencia (860.1520-16-433-01A-22)[b]	4	84 85 85 84	- 7 7 9	700 715 700 709	BBCH 83 BBCH 83 BBCH 83 BBCH 83	Fruit	0	0.103, 0.116 (0.110)
Reedley, CA; USA 2016 Valencia (860.1520-16-433-01A-23)	4	87 87 86 84	- 8 7 6	1179 1185 1171 1140	BBCH 83 BBCH 84 BBCH 84 BBCH 88	Fruit	0	0.435, 0.358 (0.397)
Richgrove, CA; USA 2017 Atwood (860.1500-16-433-01A-25)	4	84 84 84 84	- 7 7 7	1651 1633 1627 1638	BBCH 81 BBCH 81 BBCH 83 BBCH 89	Whole fruit	0	0.125, 0.232 (0.179)
						Flesh	0	0.042
						Peel	0	0.465
GRAPEFRUITS								

Location; Year, Variety (Trial ID)	Application rate per treatment				Growth Stage (BBCH)	Portion Analysed	DALA	Pydiflumetofen Residues mg/kg (average)
	No.	Rate (g ai/ha)	RTI	Water (L/ha)				
US GAP	4	85	7	Ground (NS) Aerial (Min 94)			0	
Clermont, FL; USA 2016 Flaming Red (860.1500-16-433-01A-05)	4	84 84 85 85	- 7 7 7	478 503 484 508	BBCH 81 BBCH 81 BBCH 85 BBCH 89	Fruit	0	0.171, 0.157, 0.142 (0.157)
Lake Hamilton, FL, USA 2017 Redblush Ruby (860.1500-16-433-01A-06)	4	84 84 83 85	- 7 7 7	1416 1417 1322 1343	BBCH 81 BBCH 81 BBCH 82 BBCH 85	Fruit	0	0.172, 0.136 (0.154)
Fort Pierce, FL; USA 2016 Ruby Red, X-639 (860.1500-16-433-01A-08)	4	84 84 82 84	- 7 6 7	845 841 825 843	BBCH 83 BBCH 83 BBCH 83 BBCH 83	Fruit	0	0.516, 0.646 (0.581)
Raymondville, TX; USA 2016 Rio Red (860.1500-16-433-01A-13)	4	85 84 87 86	- 7 7 7	2413 2428 2461 2418	BBCH 79 BBCH 79-81 BBCH 81 BBCH 81-83	Fruit	0	0.122, 0.105, 0.145 (0.124)
Porterville, CA; USA 2016 Mellow Gold (860.1500-16-433-01A-18)	4	91 84 84 83	- 7 7 7	762 707 710 691	BBCH 81 BBCH 81 BBCH 83 BBCH 83	Fruit	0	0.129, 0.102 (0.115)
Orange Cove, CA; USA 2016 Star Ruby (860.1500-16-433-01A-20)	4	84 85 84 86	- 6 7 8	1130 1144 1131 1151	BBCH 83 BBCH 83 BBCH 85 BBCH 88	Fruit	0	0.091, 0.096 (0.093)
Raymondville, TX; USA 2017 Rio Red (860.1500-16-433-01A-26)	4	86 85 84 86	- 7 6 8	481 483 479 490	BBCH 79-81 BBCH 79-81 BBCH 81-83 BBCH 83-89	Whole fruit	0	0.157, 0.101 (0.129)
						Peel	0	0.617
						Pulp	0	<0.01

[a] Applications were separated by 11 months, rendering the trials independent.

[b] Applications were separated by 5 months, rendering the trials independent

Pome fruits

Twenty-six independent field trials were carried out in Canada and the United States on apples (15) and pears (11) during the 2014 and 2015 growing seasons (Salzman, 2017, Report No. TK003855; Oakes, 2018, Report No. TK0103850).

Each trial site consisted of one untreated control plot and two treated plots. The treated plots received four foliar airblast applications of an SC formulation of pydiflumetofen (nominal concentration 20% w/w) at 44–54 g ai/ha per application, with retreatment intervals of 5–11 days, totaling 191–206 g ai/ha. The applications were made with concentrate spray volumes (234–898 L/ha) and with dilute spray volumes (1000–3012 L/ha). A non-ionic surfactant (NIS) or crop oil concentrate (COC) was added to the spray mixture for all applications. For all trials, samples were collected 0 and 30 days after the last application (DALA). For the decline trials, mature apple and pear samples were collected at 0, 3, 7, 10 and 14 DALA. At least 24 or more fruit for a minimum sample weight of 2 kg were collected from each plot.

Samples were analyzed using the LC-MS/MS Method GRM061.03A. Acceptable concurrent recoveries were reported for apples at fortification levels of 0.01 to 1.0 mg/kg, thus validating the method. The limit of quantitation (LOQ), determined as the lowest level of method validation (LLMV), was 0.01 mg/kg.

Treated samples were maintained frozen until analysis for a maximum storage interval of 435 days. Adequate storage stability data are available to support the storage conditions and intervals for samples in the current trials.

Table 4 Residues of pydiflumetofen in pome fruits from trials conducted in North America following application of an SC formulation

Location; Year, Variety (Trial ID)	Application rate per treatment				Growth Stage (BBCH)	DALA	Portion Analysed	Pydiflumetofen Residues mg/kg (average)
	No.	Rate (g ai/ha)	RTI	Water (L/ha)				
US GAP	4	50	7	Ground (NS) Aerial (Min 94)		30		
APPLES								
Berwick, NS Canada 2014 Cortland (TK0103850-T604)	4	51 50 50 51	- 7 8 7	824 769 788 775	BBCH 77 - 84 BBCH 78 - 85 BBCH 79 - 87 BBCH 87	0	Fruit	0.083, 0.092 (0.088)
	4	50 50 49 49	- 7 7 7	847 825 843 806	BBCH 75 - 77 BBCH 75 - 81 BBCH 76 - 82 BBCH 76 - 82	30	Fruit	0.051, 0.075 (0.063)
Branchton, ON; Canada 2014 Spartan (TK0103850-T605)	4	49 48 47 51	- 8 6 8	1000 1000 1000 1000	BBCH 85 - 87 BBCH 85 - 87 BBCH 87 - 89 BBCH 87 - 89	0	Fruit	0.058, 0.082 (0.070)
	4	50 50	- 8	1000 1000	BBCH 81 - 85 BBCH 81 - 85	29	Fruit	0.058, 0.056 (0.057)

Location; Year, Variety (Trial ID)	Application rate per treatment				Growth Stage (BBCH)	DALA	Portion Analysed	Pydiflumetofen Residues mg/kg (average)
	No.	Rate (g ai/ha)	RTI	Water (L/ha)				
US GAP	4	50	7	Ground (NS) Aerial (Min 94)		30		
		50 50	7 7	1000 1000	BBCH 81 - 85 BBCH 81 - 85			
Simcoe, ON, Canada 2014 MacIntosh (TK0103850-T606)[A]	4	48 49 53 54	- 7 8 7	1000 1000 1000 1000	BBCH 85 - 87 BBCH 85 - 87 BBCH 85 - 87 BBCH 87 - 89	0	Fruit	0.083, 0.12 (0.102)
	4	50 53 48 48	- 8 7 7	1000 1000 1000 1000	BBCH 75 - 77 BBCH 75 - 77 BBCH 81 - 85 BBCH 81 - 85	29	Fruit	0.074, 0.083 (0.079)
Simcoe, ON, Canada 2014 Mutsu (TK0103850-T607)[a]	4	48 48 52 54	- 7 8 7	1000 1000 1000 1000	BBCH 85 - 87 BBCH 85 - 87 BBCH 85 - 87 BBCH 87 - 89	0	Fruit	0.13, 0.16 (0.15)
	4	50 53 50 48	- 8 7 7	1000 1000 1000 1000	BBCH 75 - 77 BBCH 75 - 77 BBCH 81 - 85 BBCH 81 - 85	29	Fruit	0.096, 0.110 (0.103)
Okanagan Falls, BC; Canada 2014 Spartan (TK0103850-T608)	4	51 50 50 50	- 7 8 7	1000 1000 1000 1000	BBCH 81 - 85 BBCH 85 - 89 BBCH 87 - 89 BBCH 87 - 89	0	Fruit	0.10, 0.095 (0.098)
	4	51 50 51 53	- 7 8 5	1000 1000 1000 1000	BBCH 76 - 81 BBCH 77 - 81 BBCH 78 - 81 BBCH 79 - 85	32	Fruit	0.058, 0.065 (0.062)
Alton, NY; USA 2014 Rome (TK0103855-02)	4	50 50 50 50	- 7 8 6	748 748 748 748	BBCH 78 BBCH 79 BBCH 81 BBCH 85-87	0	Fruit	0.070, 0.072 (0.071)
	4	50 50 50 50	- 8 6 8	748 748 748 748	BBCH 75 - 76 BBCH 76 BBCH 77 BBCH 77	29	Fruit	0.086, 0.088 (0.087)
North Rose, NY; USA 2014 Ida Red	4	50 51 50 50	- 7 7 7	1871 1880 1871 1871	BBCH 81-85 BBCH 81-85 BBCH 85-87 BBCH 87-89	0	Fruit	0.089, 0.123 (0.106)

Location; Year, Variety (Trial ID)	Application rate per treatment				Growth Stage (BBCH)	DALA	Portion Analysed	Pydiflumetofen Residues mg/kg (average)
	No.	Rate (g ai/ha)	RTI	Water (L/ha)				
US GAP	4	50	7	Ground (NS) Aerial (Min 94)		30		
(TK0103855-03)	4	51 51 51 51	- 7 7 7	1908 1880 1880 1889	BBCH 81 BBCH 81 BBCH 81 BBCH 81 - 85	29	Fruit	0.081, 0.085 (0.083)
Cana, VA; USA 2014 Rome (TK0103855-04)	4	51 51 51 51	- 8 7 6	711 720 720 720	BBCH 79-80 BBCH 82-83 BBCH 87 BBCH 89	0	Fruit	0.189, 0.210 (0.200)
	4	51 51 51 51	- 6 7 7	692 823 683 702	BBCH 75 BBCH 75 - 76 BBCH 77 - 78 BBCH 78 - 79	28	Fruit	0.084, 0.127 (0.101)
Dix, IL; USA 2014 Jonathan (TK0103855-05)	4	50 51 50 51	- 7 7 6	674 674 692 692	BBCH 78-79 BBCH 81 BBCH 85 BBCH 89	0	Fruit	0.136, 0.169 (0.152)
	4	52 50 50 49	- 11 7 7	683 664 664 645	BBCH 74 - 75 BBCH 75 - 76 BBCH 76 - 77 BBCH 77 - 78	29	Fruit	0.041, 0.09 (0.066)
Centralia, IL; USA 2014 Jonathan (TK0103855-06)	4	49 49 50 50	- 7 7 7	2030 1674 1768 1843	BBCH 80-81 BBCH 80-81 BBCH 81-85 BBCH 81-85	0 3 7 10 14	Fruit	0.084, 0.080 (0.082) 0.112 0.074 0.051 0.053
	4	51 52 52 52	- 7 7 7	1646 2058 2020 2039	BBCH 75 - 77 BBCH 75 - 77 BBCH 79 - 80 BBCH 79 - 80	30	Fruit	0.038, 0.054 (0.046)
Canon City, CO; USA 2014 Red Delicious (TK0103855-07)	4	50 51 52 52	- 8 5 8	243 243 243 253	BBCH 87 BBCH 87 BBCH 87 BBCH 89	0	Fruit	0.191, 0.205 (0.198)
	4	51 51 51 50	- 8 5 8	243 234 243 243	BBCH 75 - 76 BBCH 75 - 76 BBCH 76 - 77 BBCH 77 - 78	30	Fruit	0.053, 0.057 (0.055)

Location; Year, Variety (Trial ID)	Application rate per treatment				Growth Stage (BBCH)	DALA	Portion Analysed	Pydiflumetofen Residues mg/kg (average)
	No.	Rate (g ai/ha)	RTI	Water (L/ha)				
US GAP	4	50	7	Ground (NS) Aerial (Min 94)		30		
Madera, CA; USA 2014 Anna (TK0103855-08)	4	51 50 51 51	- 6 8 7	1600 1590 1600 1600	BBCH 77-78 BBCH 78-79 BBCH 85-87 BBCH 81-85	0	Fruit	0.060, 0.068 (0.064)
	4	51 51 52 51	- 7 7 7	1609 1618 1637 1628	BBCH 75 - 76 BBCH 75 - 76 BBCH 76 - 77 BBCH 76 - 77	30	Fruit	0.093, 0.070 (0.084)
Parkdale, OR; USA 2014 Gala (TK0103855-10)	4	51 52 51 50	- 7 7 7	599 617 655 664	BBCH 81 BBCH 81 BBCH 85 BBCH 85	0	Fruit	0.072, 0.065 (0.069)
	4	48 49 48 50	- 6 7 7	589 533 524 571	BBCH 76 BBCH 76 BBCH 76 BBCH 81	29	Fruit	0.046, 0.05 (0.048)
Ephrata, WA; USA 2014 Gala (TK0103855-11)[b]	4	51 51 51 51	- 7 7 7	617 608 608 608	BBCH 79-80 BBCH 82-83 BBCH 84-85 BBCH 86-87	0	Fruit	0.238, 0.263 (0.251)
	4	51 51 51 52	- 7 7 7	608 608 608 608	BBCH 75 - 76 BBCH 75 - 76 BBCH 76 - 77 BBCH 77 - 78	30	Fruit	0.102, 0.122 (0.112)
Ephrata, WA; USA 2014 Red Delicious (TK0103855-12)[b]	4	51 52 52 52	- 7 7 7	1871 1880 1880 1889	BBCH 84-85 BBCH 85-86 BBCH 86-87 BBCH 87-88	0	Fruit	0.087, 0.111 (0.099)
	4	52 51 51 52	- 7 7 7	1880 1871 1861 1880	BBCH 76 - 77 BBCH 77 - 78 BBCH 78 - 79 BBCH 79 - 80	30	Fruit	0.06, 0.043, 0.071 (0.058)
PEARS								
Branchton, ON; Canada 2014 Harrow Gold (TK0103850-T609)	4	49 51 49 44	- 7 6 7	1000 1000 1000 1000	BBCH 75 - 77 BBCH 81 - 85 BBCH 81 - 85 BBCH 87 - 89	0	Fruit	0.17, 0.15 (0.16)
	4	49 50 50	- 7 7	1000 1000 1000	BBCH 73 - 74 BBCH 74 - 75 BBCH 74 - 75	29	Fruit	0.035, 0.056 (0.046)

Location; Year, Variety (Trial ID)	Application rate per treatment				Growth Stage (BBCH)	DALA	Portion Analysed	Pydiflumetofen Residues mg/kg (average)
	No.	Rate (g ai/ha)	RTI	Water (L/ha)				
US GAP	4	50	7	Ground (NS) Aerial (Min 94)		30		
		50	7	1000	BBCH 75 - 77			
St. Catharines, ON; Canada 2014 Bosc (TK0103850-T611)[c]	4	48 52 47 48	- 7 7 8	1200 1200 1200 1200	BBCH 81 - 85 BBCH 81 - 85 BBCH 85 - 87 BBCH 87 - 89	0	Fruit	0.15, 0.16 (0.16)
	4	49 49 46 48	- 7 6 7	1200 1200 1200 1200	BBCH 74 - 75 BBCH 75 - 77 BBCH 75 - 77 BBCH 77 - 81	29	Fruit	0.077, 0.078 (0.078)
St. Catharines, ON; Canada 2014 Bartlett (TK0103850-T612)[c]	4	48 52 47 49	- 7 7 8	1200 1200 1200 1200	BBCH 81 - 85 BBCH 81 - 85 BBCH 87 - 89 BBCH 89	0	Fruit	0.13, 0.12 (0.13)
	4	47 50 47 48	- 7 6 7	1200 1200 1200 1200	BBCH 74 - 75 BBCH 75 - 77 BBCH 75 - 77 BBCH 77 - 81	29	Fruit	0.040, 0.034 (0.037)
Okanagan Falls, BC; Canada 2014 Anjou (TK0103850-T613)	4	50 50 50 51	- 9 6 7	1000 1000 1000 1000	BBCH 79 - 81 BBCH 81 - 85 BBCH 81 - 85 BBCH 87	0	Fruit	0.13, 0.11 (0.12)
	4	51 50 51 49	- 7 8 6	1000 1000 1000 1000	BBCH 75-76 BBCH 76-77 BBCH 79-81 BBCH 79-81	31	Fruit	0.100, 0.130 (0.115)
Williamson, NY; USA 2014 Bartlett (TK0103855-13)	4	51 51 51 50	- 6 7 8	889 898 889 886	BBCH 75 BBCH 77 BBCH 77-81 BBCH 85-87	0	Fruit	0.018, 0.019 (0.019)
	4	51 51 51 51	- 7 6 7	889 898 889 889	BBCH 72-73 BBCH 73-74 BBCH 74 BBCH 74-75	29	Fruit	0.018, 0.019 (0.019)
Madera, CA; USA 2014 Hosui Asian (TK0103855-14)	4	51 52 51 51	- 7 7 7	1871 1889 1871 1871	BBCH 85 BBCH 85 BBCH 87 BBCH 89	0	Fruit	0.096, 0.125 (0.111)

Location; Year, Variety (Trial ID)	Application rate per treatment				Growth Stage (BBCH)	DALA	Portion Analysed	Pydiflumetofen Residues mg/kg (average)
	No.	Rate (g ai/ha)	RTI	Water (L/ha)				
US GAP	4	50	7	Ground (NS) Aerial (Min 94)		30		
	4	51 51 51 51	- 7 7 7	1880 1861 1871 1861	BBCH 79 BBCH 81 BBCH 81 BBCH 81	30	Fruit	0.060, 0.079 (0.070)
Lindsay, CA; USA 2014 Olympic (TK0103855-15)	4	50 51 50 50	- 7 7 7	2965 3012 2647 3012	BBCH 77-80 BBCH 77-81 BBCH 85-87 BBCH 87-89	0	Fruit	0.089, 0.088 (0.089)
						3		0.121
						7		0.103
						10		0.088
						14		0.079
	4	50 50 50 51	- 8 7 7	2694 2694 2853 2769	BBCH 72 - 74 BBCH 72 - 74 BBCH 73 - 75 BBCH 73 - 76	30	Fruit	0.09, 0.095 (0.092)
Ephrata, WA; USA 2014 D'Anjou (K0103855-16)[d]	4	51 51 51 50	- 7 7 7	608 608 608 608	BBCH 74-75 BBCH 76-77 BBCH 79-80 BBCH 81-82	0	Fruit	0.142, 0.135 (0.139)
	4	51 52 52 51	- 7 7 7	617 617 608 608	BBCH 70 - 71 BBCH 70 - 71 BBCH 71 - 72 BBCH 72 - 73	30	Fruit	0.056, 0.041 (0.049)
Hood River, OR; USA 2014 D'Anjou (TK0103855-17)	4	51 50 52 51	- 7 7 7	552 552 617 617	BBCH 81 BBCH 81 BBCH 81 BBCH 85	0	Fruit	0.097, 0.077 (0.087)
	4	50 50 51 51	- 6 7 7	571 505 505 543	BBCH 76 BBCH 76 BBCH 76 BBCH 79	29	Fruit	0.031, 0.038 (0.035)
Ephrata, WA; USA 2014 Red D'Anjou (K0103855-18)[d]	4	51 51 51 52	- 7 7 7	1861 1871 1852 1880	BBCH 80-81 BBCH 81-82 BBCH 83-84 BBCH 85-86	0	Fruit	0.153, 0.142 (0.148)
	4	52 51 52 52	- 7 7 7	1889 1843 1889 1899	BBCH 75 - 76 BBCH 76 - 77 BBCH 77 - 78 BBCH 78 - 79	30	Fruit	0.041, 0.055 (0.048)
Alton, NY; USA 2014	4	50 50	- 7	748 748	BBCH 77 BBCH 78	0	Fruit	0.019, 0.017 (0.018)

Location; Year, Variety (Trial ID)	Application rate per treatment				Growth Stage (BBCH)	DALA	Portion Analysed	Pydiflumetofen Residues mg/kg (average)
	No.	Rate (g ai/ha)	RTI	Water (L/ha)				
US GAP	4	50	7	Ground (NS) Aerial (Min 94)		30		
Earligold (TK0103855-19)		50 50	8 6	748 748	BBCH 81 BBCH 85-87			
	4	50 50 50 50	- 8 6 7	748 748 748 748	BBCH 73 BBCH 74 BBCH 75 BBCH 76	30	Fruit	0.02, 0.017 (0.019)
Blackfoot, ID; USA 2014 Honey Crisp (TK0103855-20)	4	51 52 50 51	- 7 8 7	748 795 795 720	BBCH 79-84 BBCH 79-83 BBCH 79-83 BBCH 79-84	0	Fruit	0.021, 0.023 (0.022)
	4	51 51 50 50	- 7 7 7	823 823 786 814	BBCH 76 - 81 BBCH 79 - 82 BBCH 79 - 83 BBCH 79 - 80	30	Fruit	0.012, 0.017 (0.015)

[a] Applications were made on the same day, rendering the trials dependent.

[b] Applications were separated by 29 days, rendering the trials independent.

[c] Applications were made on the same day, rendering the trials dependent.

[d] Applications were separated by 20 days, rendering the trials independent.

Stone fruits

Thirty-four independent field trials were conducted on stone fruits (peaches: 12 trials; plums: 9 trials; cherries: 13 trials) during the 2016 and 2017 growing seasons in Canada and the United States (Hampton, 2018, Report No. TK0270167; Courcelles, 2018, Report Nos. AAFC16-047R/048R/049R).

Each trial site consisted of one untreated control plot and one treated plot. The treated plots received four foliar airblast applications of SC formulations (nominal concentration 200 g pydiflumetofen/L or nominal concentration of 75 g/L pydiflumetofen and 125 g/L of difenoconazole) at 72–83 g ai/ha per application, with retreatment intervals of 5–9 days, totaling 294–345 g ai/ha. One cherry trial received five foliar spray applications for a total seasonal application rate of 389 g ai/ha. The applications were made with concentrate spray volumes (524–960 L/ha) or with dilute spray volumes (1366–1889 L/ha). A non-ionic surfactant (NIS) or crop oil concentrate (COC) was added to the spray mixture for all applications. For all trials, samples were collected 0 days after the last application (DALA). For the decline trials, mature peaches, plums and cherries were collected at 1, 2/3, 6/7, 9/10, 13, 20 and 27 DALA. Each RAC sample consisted of at least 24 pieces of fruit and weighed a minimum of 2 kg.

Samples were analyzed using the LC-MS/MS Method GRM061.03A. Acceptable concurrent recoveries were reported for all stone fruits at fortification levels of 0.01 to 5 mg/kg, thus validating the method. The limit of quantitation (LOQ) was 0.01 mg/kg.

Treated samples were maintained frozen at the testing facility, during shipping to the laboratory, and were stored frozen until analysis. The maximum storage interval for samples between harvest and analysis was 409 days. Adequate storage stability data are available to support the storage conditions and intervals for samples in the current trials.

Table 5 Residues of pydiflumetofen in stone fruits from trials conducted in North America following application of SC formulations

Location; Year, Variety (Trial ID)	Application rate per treatment				Growth Stage (BBCH)	DALA	Portion Analysed	Pydiflumetofen Residues mg/kg (average)
	No.	Rate (g ai/ha)	RTI	Water (L/ha)				
US GAP	4	75	7	Ground (NS) Aerial (Min 94)		0		
PEACHES								
Williamson, NY; USA 2016 Vine Gold (TK0270167-01)	4	75 75 75 75	- 7 7 7	1880 1861 1880 1880	BBCH 76 - 77 BBCH 77 BBCH 81 BBCH 87 - 89	0	Fruit without stone	0.292, 0.286 (0.289)
Monetta, SC; USA 2016 Autumn Prince (TK0270167-02)	4	75 76 76 76	- 7 7 7	655 683 655 655	BBCH 75 - 76 BBCH 75 - 76 BBCH 76 - 77 BBCH 79 - 81	0	Fruit without stone	0.210, 0.189 (0.200)
Athens, GA; USA 2016 Contender (TK0270167-03)	4	76 74 74 75	- 7 7 7	1366 1375 1384 1366	BBCH 74 - 75 BBCH 75 - 76 BBCH 77 - 81 BBCH 84 - 87	0	Fruit without stone	0.140, 0.258 (0.199)
Chula, GA; USA 2016 Hawthorn (TK0270167-04)	4	75 75 75 76	- 5 7 8	533 561 543 552	BBCH 74 - 76 BBCH 75 - 76 BBCH 76 - 77 BBCH 79 - 81	0 6 13 20 27	Fruit without stone	0.190, 0.227 (0.209) 0.100, 0.204 (0.152) 0.163, 0.162 (0.163) 0.092, 0.125 (0.109) 0.113, 0.071 (0.092)
Conklin, MI; USA 2016 Red Haven (TK0270167-05)	4	75 75 75 75	- 7 7 7	1787 1889 1777 1787	BBCH 75 - 77 BBCH 77 - 81 BBCH 79 - 85 BBCH 85 - 88	0	Fruit without stone	0.245, 0.192 (0.219)
Stonewall, TX; USA 2016 Parade (TK0270167-06RR)	4	76 77 75 77	- 7 7 7	524 580 627 655	BBCH 81 BBCH 81 BBCH 83 BBCH 85	0	Fruit without stone	0.250, 0.170 (0.210)
Dinuba, CA; USA 2016 Ivory Duchess (TK0270167-07)	4	74 75 76 74	- 6 7 7	1412 1403 1412 1384	BBCH 85 BBCH 85 - 89 BBCH 85 - 89 BBCH 89	0	Fruit without stone	0.248, 0.202 (0.225)
Porterville, CA; USA 2016 Fay Elberta	4	74 74 75	- 7 7	552 552 571	BBCH 75 - 76 BBCH 76 - 77 BBCH 78 - 79	0	Fruit without stone	0.092, 0.086 (0.089)

Location; Year, Variety (Trial ID)	Application rate per treatment				Growth Stage (BBCH)	DALA	Portion Analysed	Pydiflumetofen Residues mg/kg (average)
	No.	Rate (g ai/ha)	RTI	Water (L/ha)				
US GAP	4	75	7	Ground (NS) Aerial (Min 94)		0		
(TK0270167-08)		74	7	552	BBCH 89			
Hickman, CA; USA 2016 Summerset (TK0270167-09)	4	75 74 76 75	- 7 7 7	1843 1815 1852 1833	BBCH 85 BBCH 85 - 87 BBCH 85 - 87 BBCH 89	0	Fruit without stone	0.185, 0.199 (0.192)
Jordan Station, ON; Canada 2016 Harrow Diamond (AAFC16-048R-204)[a]	4	75 76 76 75	- 7 7 7	601 611 608 600	green fruit green fruit Coloured fruit Coloured fruit - mature	0	Fruit without stone	0.264, 0.235 (0.250)
Jordan Station, ON, Canada 2016 Loring (AAFC16-048R-205)[a]	4	75 75 75 76	- 7 6 7	802 803 805 809	green fruit Colouring fruit Colouring fruit Colouring fruit	0 1 3 7 10	Fruit without stone	0.158, 0.240 (0.199) 0.191, 0.223 (0.207) 0.194, 0.222 (0.208) 0.173, 0.127 (0.150) 0.071, 0.068 (0.070)
Jordan Station, ON; Canada 2016 Glowing Star (AAFC16-048R-206)[a]	4	82 77 79 79	- 7 7 7	658 614 631 630	BBCH 77 - 81 BBCH 78 - 79 BBCH 81 BBCH 87 - 89	0	Fruit without stone	0.805, 0.758 (0.782)
Jordan Station, ON; Canada 2016 Brighton (AAFC16-048R-207)[a]	4	81 80 76 77	- 6 7 7	645 643 610 615	BBCH 76 - 78 BBCH 77 - 81 BBCH 81 - 85 BBCH 87 - 89	0	Fruit without stone	0.714, 0.499 (0.606)
Summerland, BC; Canada 2016 Glohaven (AAFC16-048R-208)	4	75 75 76 76	- 7 6 7	952 949 955 960	BBCH 77 BBCH 81 BBCH 85 ripe fruit	0	Fruit without stone	0.193, 0.132 (0.162)
PLUMS								
Conklin, MI; USA 2016 Stanley (TK0270167-10)	4	75 75 75 75	- 7 7 7	608 608 617 617	BBCH 77 - 81 BBCH 78 - 83 BBCH 79 - 85 BBCH 85 - 87	0	Fruit without stone	0.354, 0.296 (0.325)
Dinuba, CA; USA 2016 Angelina (TK0270167-11)	4	76 76 75 75	- 8 7 7	1347 1356 1347 1347	BBCH 85 - 87 BBCH 85 - 87 BBCH 87 - 89 BBCH 87 - 89	0 6 13 20	Fruit without stone	0.108, 0.138 (0.123) 0.096, 0.152 (0.124) 0.115, 0.120 (0.118) 0.082, 0.122 (0.102)

Location; Year, Variety (Trial ID)	Application rate per treatment				Growth Stage (BBCH)	DALA	Portion Analysed	Pydiflumetofen Residues mg/kg (average)
	No.	Rate (g ai/ha)	RTI	Water (L/ha)				
US GAP	4	75	7	Ground (NS) Aerial (Min 94)		0		
						27		0.103, 0.059 (0.081)
Farmersville, CA; USA 2016 French Prune (TK0270167-12)	4	71 75 75 74	- 7 7 7	524 543 543 552	BBCH 87 - 89 BBCH 88 - 89 BBCH 88 - 89 BBCH 88 - 89	0	Fruit without stone	0.082, 0.175 (0.129)
Terra Bella, CA; USA 2016 French Prune (TK0270167-13)	4	75 76 75 76	- 6 7 6	533 552 543 552	BBCH 81 - 85 BBCH 85 - 87 BBCH 87 - 89 BBCH 89	0	Fruit without stone	0.334, 0.373 (0.354)
Hickman, CA; USA 2016 Grand Rosa (TK0270167-14)	4	75 76 75 75	- 7 7 7	1833 1861 1843 1843	BBCH 85 - 87 BBCH 87 BBCH 87 BBCH 89	0	Fruit without stone	0.052, 0.067 (0.060)
Roseburg, OR; USA 2016 Moyer (TK0270167-15)	4	75 76 76 76	- 7 7 7	1721 1730 1740 1740	BBCH 85 BBCH 86 BBCH 87 BBCH 89	0	Fruit without stone	0.174, 0.129 (0.152)
Greenlane Farms, ON; Canada 2016 Shiro (AAFC16-049R-199)[b]	4	75 76 76 75	- 6 7 7	752 755 764 753	green fruit green fruit Coloured fruit Coloured fruit - mature	0	Fruit without stone	0.122, 0.104 (0.113)
Summerland, BC; Canada 2016 Early Italian (AAFC16-049R-200)	4	78 79 78 77	- 6 8 6	980 992 987 975	BBCH 85 BBCH 85 Commercial Ripeness ripe fruit	0 1 2 6 9	Fruit without stone	0.138, 0.171 (0.155) 0.161, 0.156 (0.158) 0.152, 0.120 (0.136) 0.099, 0.095 (0.097) 0.104, 0.085 (0.095)
Greenland Farms, ON; Canada 2016 Stanley (AAFC16-049R-201)[b]	4	76 77 76 76	- 7 7 8	761 766 755 764	Colouring fruit - immature Colouring fruit - immature mature fruit mature fruit	0	Fruit without stone	0.233, 0.186 (0.210)
CHERRIES								
Vineland, ON; Canada 2016 Cherry, Sweet (Hedelfinger) (AAFC16-047R-220)[c]	4	76 75 75 76	- 6 8 7	1529 1494 1504 1517	fruiting Coloured fruit Red fruit Coloured fruit	0	Fruit without stone	0.451, 0.584 (0.518)
Vineland, ON; Canada	4	77	-	824	fruiting	0	Fruit without stone	0.558, 0.669 (0.614)

Location; Year, Variety (Trial ID)	Application rate per treatment				Growth Stage (BBCH)	DALA	Portion Analysed	Pydiflumetofen Residues mg/kg (average)
	No.	Rate (g ai/ha)	RTI	Water (L/ha)				
US GAP	4	75	7	Ground (NS) Aerial (Min 94)		0		
2016 Cherry, Tart (Meteor) (AAFC16-047R-221)[c]		76 76 76	8 6 6	809 809 806	Coloured fruit Red fruit Red fruit			
Branchton, ON; Canada 2016 Cherry, Tart (Mateor) (AAFC16-047R-222)	5	78 78 78 77 77	- 6 7 7 7	626 622 624 618 618	BBCH 72 NR NR NR BBCH 87	0	Fruit without stone	0.856, 0.935 (0.896)
Summerland, BC; Canada 2016 Cherry, Sweet (Lapin) (AAFC16-047R-231)[d]	4	83 77 78 77	- 7 6 7	825 845 859 842	BBCH 81 BBCH 85 BBCH 85 BBCH 87 - 89	0	Fruit without stone	0.244, 0.234 (0.239)
Summerland, BC; Canada 2016 Cherry, Sweet (Sentennia) (AAFC16-047R-232)[d]	4	77 79 77 76	- 7 6 7	2021 2067 2026 1997	BBCH 85 BBCH 85 - 87 BBCH 87 BBCH 87	0	Fruit without stone	0.196, 0.167 (0.182)
Fennville, MI; USA 2016 Cherry, Tart (Montmorency) (AAFC16-047R-223)[e]	4	73 73 76 76	- 7 7 7	919 927 956 955	fruiting fruiting fruiting fruiting	0 1 2 7 13	Fruit without stone	0.341, 0.324 (0.333) 0.379, 0.282 (0.331) 0.341, 0.343 (0.342) 0.293, 0.267 (0.280) 0.226, 0.250 (0.238)
Fennville, MI; USA 2016 Cherry, Tart (Balaton) (AAFC16-047R-224)[e]	4	75 74 74 74	- 7 7 7	715 707 699 720	fruiting fruiting fruiting fruiting	0	Fruit without stone	0.348, 0.379 (0.364)
Fennville, MI; USA 2016 Cherry, Tart (Balaton) (AAFC16-047R-225)[e]	4	75 74 75 75	- 7 7 7	943 936 937 944	fruiting fruiting fruiting fruiting	0	Fruit without stone	0.447, 0.425 (0.436)
Clarksville, MI; USA 2016 Cherry, Tart (Montmorency)	4	72 74 73 74	- 7 7 7	914 941 930 939	fruiting fruiting fruiting fruiting	0	Fruit without stone	0.678, 0.770 (0.724)

Location; Year, Variety (Trial ID)	Application rate per treatment				Growth Stage (BBCH)	DALA	Portion Analysed	Pydiflumetofen Residues mg/kg (average)
	No.	Rate (g ai/ha)	RTI	Water (L/ha)				
US GAP	4	75	7	Ground (NS) Aerial (Min 94)		0		
(AAFC16-047R-226)								
Fennville, MI; USA 2016 Cherry, Sweet (Hedelfinger) (AAFC16-047R-227)[e]	4	77 75 76 76	- 7 7 7	937 958 963 959	fruiting fruiting fruiting fruiting	0	Fruit without stone	0.263, 0.404 (0.334)
Hotchkiss, CO; USA 2016 Cherry, Tart (Montmorency) (AAFC16-047R-228)	4	76 78 81 76	- 7 7 7	564 581 606 565	fruiting fruiting fruits near maturity mature fruit	0	Fruit without stone	1.571, 1.725 (1.648)
Parlier, CA; USA 2016 Cherry, Sweet (Brooks) (AAFC16-047R-229)	4	75 77 77 77	- 8 8 6	988 1005 1002 1032	fruiting fruiting fruiting fruiting	0	Fruit without stone	0.514, 0.544 (0.529)
Courtland, CA; USA 2016 Cherry, Sweet (Burgundy Pearl) (AAFC16-047R-230)	4	74 77 77 76	- 6 6 9	1340 1402 1395 1389	fruiting fruiting fruiting fruiting	0	Fruit without stone	0.137, 0.149 (0.143)
Grandview, WA; USA 2016 Cherry, Sweet (Bony) (AAFC16-047R-233)	4	77 75 75 75	- 8 7 7	902 898 909 907	fruiting fruiting fruiting mature fruit	0 1 3 7 13	Fruit without stone	0.201, 0.166 (0.184) 0.142, 0.184 (0.163) 0.134, 0.165 (0.150) 0.116, 0.145 (0.131) 0.089, 0.130 (0.110)
Filer, ID; USA 2016 Cherry, Sweet (Bing) (AAFC16-047R-234)	4	75 77 75 75	- 6 7 7	502 515 501 502	maturing maturing maturing mature fruit	0	Fruit without stone	0.428, 0.382 (0.415)
Moxee, WA; USA 2016 Cherry, Sweet (Bing) (AAFC16-047R-235)	4	74.4 76.0 75.6 73.4	- 6 6 7	1497 1528 1523 1470	fruiting fruiting fruiting fruiting	0	Fruit without stone	0.376, 0.368 (0.372)
Hood River, OR; USA 2016 Cherry, Sweet (Regina)	4	71.8 80.8 76.1	- 7 7	1528 1660 1564	Fruit yellow - Fruit pink Fruit turning red	0	Fruit without stone	0.198, 0.230 (0.214)

Location; Year, Variety (Trial ID)	Application rate per treatment				Growth Stage (BBCH)	DALA	Portion Analysed	Pydiflumetofen Residues mg/kg (average)
	No.	Rate (g ai/ha)	RTI	Water (L/ha)				
US GAP	4	75	7	Ground (NS) Aerial (Min 94)		0		
(AAFC16-047R-236)		76.1	7	1564	Fruit turning red Mature, ripe fruit			
Lyons, NY; USA 2017 Cherry, Tart (Montmorency) (AAFC16-047R-292)	4	75.6 75.9 75.8 76.9	- 7 7 7	938 942 941 954	BBCH 76 BBCH 78 BBCH 85 BBCH 87	0	Fruit without stone	0.245, 0.271 (0.258)

[a] For trials 206 and 207, applications were separated by 6 days rendering the trials dependent. For trials 204 and 207 and trials 205 and 206, applications were separated by 11 days rendering the trials dependent. Therefore, only trials 204 and 206 were considered independent and used in the MRL calculation.

[b] Applications were separated by 49 days, rendering the trials independent.

[c] Applications were made on the same day, rendering the trials dependent.

[d] Applications were separated by 21 days, rendering the trials independent.

[e] Applications were separated by 1-8 days, rendering the trials dependent

Bushberries

Blueberries

Seventeen independent field trials were conducted on blueberries during the 2015 and 2016 growing seasons in Canada and the USA (Lennon, 2017, Report No. 11763; Oakes, 2018, Report Nos. TK0256279 and TK0269133).

Each trial site in the USA consisted of one untreated control plot and one treated plot. The treated plots received two foliar airblast applications of an SC formulation (nominal concentration 200 g pydiflumetofen/L) at 147–154 g ai/ha/application with re-treatment intervals of 6–8 days, resulting in total seasonal application rates of 297–305 g ai/ha. Spray volumes ranged from 299–543 L/ha. For all trials, samples were collected 0 days after the last application (DALA) and for one of the trials, samples were also collected 1, 3, 7 and 10 DALA to monitor residue decline.

Each trial site in Canada consisted of one untreated control plot and two treated plots. For seven of the trials, one of the treated plots received two foliar airblast spray applications of the same SC formulation as that of the US trials at 53–58 g ai/ha per application with re-treatment intervals of 10–11 days, resulting in total seasonal application rates of 108–115 g ai/ha. The spray volume was 350 L/ha. Blueberries were harvested 77–98 DALA. For three of the trials, two foliar spray applications of the same formulation were made to one of the treated plots at 147–162 g ai/ha with re-treatment intervals of 6–22 days, resulting in seasonal application rates of 298–320 g ai/ha. Spray volumes were 300 or 500 L/ha and blueberries were harvested immediately following the last application (0-DALA). For all ten trials, the second treated plot received two foliar spray applications of the same SC formulation at 54–62 g ai/ha,

followed by 2 spray applications of another SC formulation (nominal concentrations of 150 g/L pydiflumetofen and 250 g/L of fludioxonil), at 117–125 g ai/ha, totaling 349–360 g ai/ha per season. The first two applications were separated from the last two applications by a minimum of 60 days. Spray volumes ranged from 300–500 L/ha and blueberries were harvested 0 DALA. Two samples from the treated plots were collected at normal commercial harvest to obtain at least 0.5 kg of fruit per sample.

Samples were analyzed using the LC-MS/MS Method GRM061.03A. Acceptable concurrent recoveries were reported for blueberries at fortification levels of 0.01 to 10.0 mg/kg, thus validating the method. The limit of quantitation (LOQ) was 0.01 mg/kg.

Treated samples were maintained frozen until analysis for a maximum storage interval of 330 days. Adequate storage stability data are available to support the storage conditions and intervals for samples in the current trials.

Table 6 Residues of pydiflumetofen in blueberries from trials conducted in North America following applications of various SC formulations

Location; Year, Variety (Trial ID)	Application rate per treatment				Growth Stage (BBCH)	DALA	Portion Analysed	Pydiflumetofen Residues mg/kg (average)
	No.	Rate (g ai/ha)	RTI	Water (L/ha)				
US GAP	2	150	7	NS		0		
Alapaha, GA; USA 2016 TH-653 (16-GA*145)	2	154 151	- 6	365 355	fruiting fruiting	0	Fruit	0.737, 0.647 (0.692)
Castle Hayne, NC; USA 2016 Croatan (16-NC231)	2	150 150	- 7	477 477	Some blue fruit Blue fruit	0	Fruit	1.014, 0.899 (0.956)
						1		0.895, 0.822 (0.858)
						3		0.652, 0.715 (0.684)
						7		0.520, 0.490 (0.505)
						10		0.515, 0.536 (0.526)
Cream Ridge, NJ; USA 2016 Duke (16-NJ254)	2	151 149	- 7	543 543	green fruit - blue fruit fruiting	0	Fruit	0.882, 0.845 (0.864)
Chatsworth, NJ; USA 2016 Bluecrop (16-NJ255)	2	150 150	- 6	514 533	green fruit - Blue fruit fruiting	0	Fruit	0.693, 0.631 (0.662)
Aurora, OR; USA 2016 Bluecrop (16-OR320)	2	148 150	- 8	468 477	Blue fruit - green fruit Fully ripe - Semi-mature	0	Fruit	0.448, 0.362 (0.405)
Helenville, WI; USA 2016	2	147 150	- 7	346 355	fruiting fruiting	0	Fruit	3.894, 3.206 (3.550)[c]

Location; Year, Variety (Trial ID)	Application rate per treatment				Growth Stage (BBCH)	DALA	Portion Analysed	Pydiflumetofen Residues mg/kg (average)
	No.	Rate (g ai/ha)	RTI	Water (L/ha)				
Bluecrop (16-WI505)								
Bayfield, WI; USA 2016 Duke (16-WI506)[a]	2	151 153	- 6	421 421	fruiting fruiting	0	Fruit	0.752, 0.568 (0.660)
Bayfield, WI; USA 2016 Patriot (16-WI507)[a]	2	149 149	- 6	299 299	fruiting fruiting	0	Fruit	0.709, 0.617 (0.663)
Upper Stewiacke, NS; Canada 2015 Wild– lowbush (TK0256279-T720)	2	58 57	- 10	350 350	BBCH 51 - 52 BBCH 51 - 53	91	Fruit	<0.01, <0.01 (<0.01)
	4	57 57 120 123	- 10 84 7	350 350 350 350	BBCH 51 - 52 BBCH 51 - 53 BBCH 86 - 89 BBCH 85 - 89	0	Fruit	1.50, 1.30 (1.40)
Mt Thom, NS; Canada 2015 Wild– lowbush (TK0256279-T721)	2	57 56	- 11	350 350	BBCH 51 - 52 BBCH 51 - 53	88	Fruit	<0.01, <0.01 (<0.01)
	4	56 56 118 121	- 11 80 8	350 350 350 350	BBCH 51 - 52 BBCH 51 - 53 BBCH 85 - 89 BBCH 85 - 89	0	Fruit	1.30, 1.50 (1.40)
East Village, NS; Canada 2015 Wild– lowbush (TK0256279-T722)	2	57 54	- 10	350 350	BBCH 51 - 52 BBCH 51 - 53	87	Fruit	<0.01, <0.01 (<0.01)
	4	57 56 122 117	- 10 81 6	350 350 350 350	BBCH 51 - 52 BBCH 51 - 53 BBCH 85 - 89 BBCH 85 - 89	0	Fruit	1.50, 1.40 (1.45)
Upper Rawdon, NS; Canada 2015 Wild - lowbush (TK0256279-T723-D)	2	53 55	- 10	350 350	BBCH 51 - 52 BBCH 51 - 53	81	Fruit	<0.01, <0.01 (<0.01)
	4	56 56 119 121	- 10 73 8	350 350 350 350	BBCH 51 - 52 BBCH 51 - 53 BBCH 83 - 89 BBCH 83 - 89	0 1 3 6 10	Fruit	0.57, 0.87 (0.72) 0.82 0.78 0.69 0.55
East Grove, NS, Canada 2016 Lowbush	2	54 55	- 10	350 350	BBCH 03-07 BBCH 57-60	77	Fruit	0.01, 0.011 (0.011)
	4	54 55	- 10	350 350	BBCH 03 - 07 BBCH 57 - 60	0	Fruit	1.10, 1.20 (1.15)

Location; Year, Variety (Trial ID)	Application rate per treatment				Growth Stage (BBCH)	DALA	Portion Analysed	Pydiflumetofen Residues mg/kg (average)
	No.	Rate (g ai/ha)	RTI	Water (L/ha)				
(TK0256279-T800)		121 120	69 8	350 350	BBCH 81 - 89 BBCH 85 - 89			
Mt. Thom, NS; Canada 2016 Lowbush (TK0256279-T801)	2	56 56	- 10	350 350	BBCH 03-08 BBCH 55-57	98	Fruit	<0.01, <0.01 (<0.01)
	4	55 56 119 119	- 10 90 8	350 350 350 350	BBCH 03 - 08 BBCH 55 - 57 BBCH 83 - 89 BBCH 83 - 89	0	Fruit	1.10, 1.70 (1.40)
Dean, NS; Canada 2016 Lowbush (TK0256279-T802)	2	56 53	- 10	350 350	BBCH 03-07 BBCH 55-57	96	Fruit	<0.01, <0.01 (<0.01)
	4	54 55 121 122	- 10 90 6	350 350 350 350	BBCH 03 - 07 BBCH 55 - 57 BBCH 83 - 89 BBCH 83 - 89	0	Fruit	2.20, 2.00 (2.10)
Langton, ON; Canada 2016 Bluecrop (TK0269133-T803)[b]	2	151 147	- 7	500 500	BBCH 81 - 89 BBCH 81 - 89	0	Fruit	2.10, 1.70 (1.90)
	4	52 62 128 118	- 11 81 7	300 300 500 500	BBCH 07 - 09 BBCH 55 - 56 BBCH 81 - 89 BBCH 81 - 89	0	Fruit	1.00, 1.00 (1.00)
Langton, ON; Canada 2016 Duke (TK0269133-T804)[b]	2	151 153	- 22	500 500	BBCH 81 - 87 BBCH 79 - 81	0	Fruit	0.88, 0.92 (0.90)
	4	56 58 125 121	- 11 50 58	300 300 500 500	BBCH 01 - 09 BBCH 55 - 56 BBCH 79 - 81 BBCH 81 - 87	0	Fruit	0.75, 0.84 (0.80)
Madeleine, QC; Canada 2016 Patriot (TK0269133-T805-D)	2	162 158	- 6	300 300	BBCH 78 - 85 BBCH 78 - 89	0	Fruit	1.40, 1.50 (1.45)
	4	57 57 122 120	- 10 79 6	300 300 300 300	BBCH 07 - 09 BBCH 55 - 56 BBCH 81 - 89 BBCH 81 - 89	0	Fruit	1.20
						1		1.00
						3		0.94
						6		1.00
						9		0.82

[a] Applications were made on the same day, rendering the trials dependent.

[b] Applications were separated by 30 days, rendering the trials independent.

[c] The study report noted that there was reduced foliage (30-40% reduction) due to a late frost. The berries were small and all were exposed during application.

Low growing berries

Strawberries

Ten independent field trials were conducted on strawberries during the 2015 growing season in the Canada and the USA (Homa, 2016, Report No. 11159).

Each trial site consisted of one untreated control plot and one treated plot. The treated plots received two airblast applications of an SC formulation, containing 200 g ai/L, at 143–161 g ai/ha per application, with retreatment intervals of 6–8 days, totaling 292–312 g ai/ha. The applications were made with spray volumes of 309–620 L/ha. A non-ionic surfactant (NIS) or crop oil concentrate (COC) was added to the spray mixture for most applications. For all trials, samples were collected 0 days after the last application (DALA). For the decline trials, mature strawberries were collected at 1, 3, 6 and 9 DALA. Each RAC sample consisted of 40–50 berries and weighed a minimum of 1.3 kg.

Samples were analyzed using the LC-MS/MS Method GRM061.03A. Acceptable concurrent recoveries were reported for all strawberry samples at fortification levels of 0.01 and 0.10 mg/kg, thus validating the method. The limit of quantitation (LOQ) was 0.01 mg/kg.

Treated samples were maintained frozen until analysis for a maximum storage interval of 345 days. Adequate storage stability data are available to support the storage conditions and intervals for samples in the current trials.

Table 7 Residues of pydiflumetofen in strawberries from trials conducted in North America following application of an SC formulation

Location; Year, Variety (Trial ID)	Application rate per treatment				Growth Stage (BBCH)	DALA	Portion Analysed	Pydiflumetofen Residues mg/kg (average)
	No.	Rate (g ai/ha)	RTI	Water (L/ha)				
US GAP	2	150	7	NS		0		
Waterville, NS; Canada 2015 Wendy (1159.15-NS311)	2	143 149	- 7	478 496	fruiting fruiting	0	Fruit	0.168, 0.217 (0.193)
Harrow, ON; Canada 2015 Sapphire (11159.15-ON350)[a]	2	149 149	- 6	397 398	mature fruit mature fruit	0	Fruit	0.202, 0.134 (0.168)
Harrow, ON; Canada 2015 Tribute (11159.15-ON353)[a]	2	148 161	- 7	349 380	fruiting fruiting	0	Fruit	0.197, 0.157 (0.177)
L'Acadie, QC; Canada 2015 Albion (11159.15-QC395)	2	148 148	- 7	494 492	mature fruit mature fruit	0	Fruit	0.045, 0.100 (0.072)
						1		0.056, 0.088 (0.072)
						3		0.089, 0.074 (0.082)
						6		0.086, 0.078 (0.082)
						9		0.060, 0.072 (0.066)
Salinas, CA; USA	2	149	-	404	Flowering and fruiting	0	Fruit	0.413, 0.468 (0.441)

Location; Year, Variety (Trial ID)	Application rate per treatment				Growth Stage (BBCH)	DALA	Portion Analysed	Pydiflumetofen Residues mg/kg (average)
	No.	Rate (g ai/ha)	RTI	Water (L/ha)				
2015 Albion (11159.15-CA*23)		150	7	502	Flowering and fruiting			
Parlier, CA; USA 2015 San Andreas (11159.15-CA24)[b]	2	155 155	- 7	610 607	fruiting fruiting	0	Fruit	0.617, 0.496 (0.556)
						1		0.454, 0.453 (0.454)
						3		0.416, 0.354 (0.385)
						7		0.328, 0.193 (0.260)
						10		0.129, 0.187 (0.158)
Parlier, CA; USA 2015 San Andreas (11159.15-CA57)[b]	2	151 154	- 7	464 477	fruiting fruiting	0	Fruit	0.530, 0.356 (0.443)
Citra, FL; USA 2015 Festival (11159.15-FL164)	2	147 152	- 6	595 620	fruiting fruiting	0	Fruit	0.401, 0.512 (0.456)
East Lansing, MI; USA 2015 Jewell (11159.15-MI259)	2	148 150	- 7	368 369	fruiting fruiting - ripe	0	Fruit	0.087, 0.101 (0.094)
Cream Ridge, NJ; USA 2015 All Star (11159.15-NJ293)	2	151 150	- 8	309 314	fruiting fruiting	0	Fruit	0.290, 0.316 (0.303)
Freeville, NY; USA 2015 Jewell (11159.15-NY499)	2	149 149	- 6	442 407	fruiting - bloom fruiting - bloom	0	Fruit	0.089, 0.113 (0.101)
Aurora, OR; USA 2015 Totem (11159.15-OR363)	2	155 157	- 8	470 476	green fruit - ripe fruit green fruit - ripe fruit	0	Fruit	0.178, 0.147 (0.162)

[a] Applications were separated by 7 days, rendering the trials dependent.

[b] Applications were separated by 4 days, rendering the trials dependent.

Bulb vegetables

Twelve independent field trials were conducted on dry bulb (8) and green onions (4) during the 2016 growing season in the USA (Wai Hoi, 2018, Report No. TK0268909).

Each trial site consisted of one untreated control plot and one treated plot. The treated plots received three broadcast foliar spray applications of an SC formulation, containing 200 g ai/L, at 120–130 g ai/ha/application, with retreatment intervals of 6–8 days, totaling 372–384 g ai/ha. The applications were

made with spray volumes of 140–346 L/ha. A non-ionic surfactant (NIS) or crop oil concentrate (COC) was added to the spray mixture for most applications. For all trials, samples were collected 7 days after the last application (DALA). For the decline trials, mature bulb vegetables were collected at 0, 3, 7, 10 and 13/14 DALA. At least 24 bulbs, or 12 very large bulbs, without roots or tops, and 24 whole plants without roots were collected, having a minimum sample weight of 2 kg.

Samples were analyzed using the LC-MS/MS Method GRM061.03A. Acceptable concurrent recoveries were reported for all bulb vegetables at fortification levels of 0.01 to 5.0 mg/kg, thus validating the method. The limit of quantitation (LOQ) was 0.01 mg/kg.

Treated samples were maintained frozen until analysis for a maximum storage interval of 217 days. Adequate storage stability data are available to support the storage conditions and intervals for samples in the current trials.

Table 8 Residues of pydiflumetofen in bulb vegetables from trials conducted in the USA following application of an SC formulation

Location; Year, Variety (Trial ID)	Application rate per treatment				Growth Stage (BBCH)	DALA	Portion Analysed	Pydiflumetofen Residues mg/kg (average)
	No.	Rate (g ai/ha)	RTI	Water (L/ha)				
US GAP	3	125	7	NS		7		
BULB ONION								
North Rose, NY; USA 2016 Safrane, F1 (TK0268909-01)	3	128 127 129	- 7 7	234 234 234	BBCH 42 - 43 BBCH 43 - 45 BBCH 46 - 48	7	Bulb	0.113, 0.129 (0.121)
Fitchburg, WI; USA 2016 Ebenezer (TK0268909-02)	3	126 125 126	- 7 6	234 234 243	BBCH 43 BBCH 45 BBCH 48	7	Bulb	<0.01, <0.01 (<0.01)
San Angelo, TX; USA 2016 White Bermuda (TK0268909-03)	3	125 120 127	- 7 7	140 140 149	BBCH 43 - 45 BBCH 45 - 47 BBCH 48	7	Bulb	<0.01, <0.01 (<0.01)
Levelland, TX; USA 2016 Candy (TK0268909-04)	3	127 125 126	- 7 7	140 140 140	BBCH 46 BBCH 46 BBCH 46	6	Bulb	0.054, 0.037 (0.046)
King City, CA; USA 2016 Merengue (TK0268909-05)	3	127 124 126	- 8 7	253 243 253	BBCH 47 - 48 BBCH 47 - 48 BBCH 48 - 49	6	Bulb	0.054, 0.056 (0.055)
Porterville, CA; USA 2016	3	126 125 127	- 6 8	234 234 234	BBCH 47 - 48 BBCH 47 - 48 BBCH 48 - 49	0 3 7	Bulb	0.036, 0.020 (0.028) 0.015, 0.013 (0.014) <0.01, <0.01 (<0.01)

Location; Year, Variety (Trial ID)	Application rate per treatment				Growth Stage (BBCH)	DALA	Portion Analysed	Pydiflumetofen Residues mg/kg (average)
	No.	Rate (g ai/ha)	RTI	Water (L/ha)				
US GAP	3	125	7	NS		7		
Yellow Sweet Spanish (TK0268909-06)						10		0.013, <0.01 (0.012)
						14		<0.01, <0.01 (<0.01)
Nampa, ID; USA 2016 Nunhems Vaquero (TK0268909-07)	3	123 130 120	- 6 8	187 196 178	BBCH 45 - 47 BBCH 45 - 47 BBCH 47 - 49	8	Bulb	0.040, 0.056 (0.048)
Oregon City, OR; USA 2016 Vaquero (TK0268909-08)	3	129 129 125	- 7 7	243 253 234	BBCH 42 - 44 BBCH 43 - 45 BBCH 43 - 46	7	Bulb	0.060, 0.054 (0.057)
GREEN ONION								
Delavan, WI; USA 2016 White (TK0268909-09)	3	122 124 124	- 7 7	178 187 178	BBCH 15 BBCH 17 BBCH 19	7	Whole plant	0.324, 0.393 (0.359)
San Angelo, TX; USA 2016 Evergreen White Bunching (TK0268909-10)	3	125 123 124	- 7 7	187 187 187	BBCH 15 - 41 BBCH 17 - 41 BBCH 41	0	Whole plant	2.30, 1.95 (2.12)
						3		1.81, 1.73 (1.77)
						7		1.32, 0.814 (1.067)
						10		0.765, 0.920 (0.842)
						13		0.842, 0.692 (0.767)
King City, CA; USA 2016 Bunching (TK0268909-11)	3	127 127 127	- 7 7	271 346 346	BBCH 15 - 19 BBCH 15 - 19 BBCH 19 - 41	7	Whole plant	0.293, 0.303 (0.298)
Oregon City, OR; USA 2016 Parade (TK0268909-12)	3	126 123 123	- 7 7	234 234 234	BBCH 13 - 16 BBCH 14 - 16 BBCH 15 - 16	7	Whole plant	0.355, 0.197 (0.276)

Brassica vegetables (except Brassica leafy vegetables)

Fourteen independent field trials were conducted on cabbage (6), cauliflower (4) and broccoli (4) during the 2016 growing season in the USA (Kelley, 2018, Report No. TK0268910).

Each trial site consisted of one untreated control plot and one treated plot. The treated plots received three broadcast foliar spray applications of an SC formulation, containing 200 g ai/L, at 120–131 g ai/ha per application, with retreatment intervals of 5–8 days, totaling 370–387 g ai/ha. The applications were made with spray volumes of 168–356 L/ha. A methylated seed oil (MSO), non-ionic surfactant (NIS) or crop oil concentrate (COC) was added to the spray mixture for most applications. For all trials, samples were collected 0 days after the last application (DALA). For the decline trials, mature brassica vegetables

were collected at 0, 3, 7, 10 and 14 DALA. For the cabbage trials, mature fresh heads were collected with the wrapper leaves included and with the wrapper leaves removed as separate samples. Each cabbage sample consisted of a minimum of 12 plants. For the broccoli trials, mature flower head and stem samples were collected, each weighing a minimum of 1 kg. For cauliflower trials, mature flower head and stem samples were collected consisting of a minimum of 12 plants.

Samples were analyzed using the LC-MS/MS Method GRM061.03A. Acceptable concurrent recoveries were reported for all brassica vegetables at fortification levels of 0.01 to 10 mg/kg, thus validating the method. The limit of quantitation (LOQ) was 0.01 mg/kg.

Treated samples were maintained frozen until analysis for a maximum storage interval of 351 days. Adequate storage stability data are available to support the storage conditions and intervals for samples in the current trials.

Table 9 Residues of pydiflumetofen in Brassica vegetables from trials conducted in the USA following application of an SC formulation

Location; Year, Variety (Trial ID)	Application rate per treatment				Growth Stage (BBCH)	DALA	Portion Analysed	Pydiflumetofen Residues mg/kg (average)
	No.	Rate (g ai/ha)	RTI	Water (L/ha)				
US GAP	3	125	7	Ground (NS) Aerial (Min 28)	NS	0		
CABBAGE								
Lyons, NY; USA 2016 Farao F1 OG (TK0268910-01)	3	126 124 126	- 7 7	281 281 281	BBCH 45 - 47 BBCH 47 - 48 BBCH 48 - 49	0	Head w/o wrapper leaves	0.111, 0.133 (0.122)
						3		0.060, 0.068 (0.064)
						7		0.049, 0.048 (0.049)
						10		0.035, 0.045 (0.040)
						14		0.028, 0.032 (0.030)
						0	Head with wrapper leaves	1.199, 0.786 (0.992)
						3		0.836, 0.884 (0.860)
						7		0.580, 0.537 (0.559)
						10		0.681, 0.498 (0.590)
						14		0.236, 0.399 (0.318)
Seven Springs, NC; USA 2016 Early Jersey Wakefield (TK0268910-02)	3	126 124 127	- 7 7	243 234 309	BBCH 45 BBCH 47 BBCH 49	0	Head w/o wrapper leaves	0.035, 0.025 (0.030)
						0	Head with wrapper leaves	0.387, 0.338 (0.363)
Greenville, FL; USA 2016 Cheers (TK0268910-03)	3	126 123 126	- 6 7	206 206 206	BBCH 44 - 46 BBCH 47 - 48 BBCH 48 - 49	0	Head w/o wrapper leaves	0.040, 0.063 (0.052)
						0	Head with wrapper leaves	0.178, 0.425 (0.302)

Location; Year, Variety (Trial ID)	Application rate per treatment				Growth Stage (BBCH)	DALA	Portion Analysed	Pydiflumetofen Residues mg/kg (average)
	No.	Rate (g ai/ha)	RTI	Water (L/ha)				
US GAP	3	125	7	Ground (NS) Aerial (Min 28)	NS	0		
Richland, IA; USA 2016 Late Flat Dutch (TK0268910-04)	3	126 124 124	- 7 6	234 225 234	BBCH 42 BBCH 45 BBCH 48	0	Head w/o wrapper leaves	<0.01, <0.01 (<0.01)
						0	Head with wrapper leaves	1.094, 0.812 (0.953)
Hinton, OK; USA 2016 Stonehead (TK0268910-05)	3	126 120 124	- 7 7	206 196 178	BBCH 44 - 45 BBCH 45 - 46 BBCH 47 - 49	0	Head w/o wrapper leaves	0.019, 0.029 (0.024)
						0	Head with wrapper leaves	0.290, 0.361 (0.326)
Hughson, CA; USA 2016 Primo Vantage (TK0268910-06)	3	124 123 124	- 7 7	281 281 281	BBCH 48 BBCH 48 - 49 BBCH 49	0	Head w/o wrapper leaves	0.040, 0.039 (0.040)
						0	Head with wrapper leaves	0.626, 1.144 (0.885)
CAULIFLOWER/BROCCOLI								
Hinton, OK; USA 2016 Destiny (TK0268910-07)	3	128 128 131	- 7 5	215 178 187	BBCH 43 - 47 BBCH 47 - 49 BBCH 49	0	Broccoli head and stem	1.505, 1.359 (1.432)
Guadalupe, CA; USA 2016 Imperial (TK0268910-09)	3	126 127 124	- 8 7	290 290 281	BBCH 41 - 43 BBCH 43 - 46 BBCH 46 - 49	0	Broccoli head and stem	0.701, 0.635 (0.668)
Oregon City, OR; USA 2016 Marathon (TK0268910-10)	3	122 120 122	- 7 7	234 225 234	BBCH 41 - 43 BBCH 43 - 48 BBCH 46 - 49	0	Broccoli head and stem	0.405, 0.428 (0.417)
King City, CA; USA 2016 Marathon (TK0268910-15)	3	127 128 130	- 6 7	346 346 356	BBCH 45 - 47 BBCH 45 - 47 BBCH 47 - 49	0	Broccoli head and stem	0.858, 1.039 (0.948)
						4		0.578, 0.449 (0.514)
						7		0.391, 0.575 (0.483)
						10		0.300, 0.325 (0.313)
						14		0.246, 0.260 (0.253)
North Rose, NY; USA 2016 Apex (TK0268910-11)	3	126 123 124	- 7 6	206 206 206	BBCH 41 - 43 BBCH 43 - 45 BBCH 47 - 49	0	Cauliflower head and stem	0.340, 0.286 (0.313)
Carlyle, IL; USA 2016	3	126 126	- 7	178 168	BBCH 41 - 42 BBCH 45 - 46	0	Cauliflower head and stem	0.359, 0.371 (0.365)

Location; Year, Variety (Trial ID)	Application rate per treatment				Growth Stage (BBCH)	DALA	Portion Analysed	Pydiflumetofen Residues mg/kg (average)
	No.	Rate (g ai/ha)	RTI	Water (L/ha)				
US GAP	3	125	7	Ground (NS) Aerial (Min 28)	NS	0		
Snow Crown (TK0268910-12)		126	7	178	BBCH 49			
Porterville, CA; USA 2016 Absolute (TK0268910-13)	3	124 128 126	- 7 7	290 299 290	BBCH 43 - 45 BBCH 46 - 47 BBCH 48 - 49	0 3 7 9 14	Cauliflower head and stem	0.016, 0.013 (0.015) 0.026, 0.021 (0.024) 0.014, 0.030 (0.022) 0.068, 0.028 (0.048) 0.034, 0.014 (0.024)
Aurora, OR; USA 2016 Bishop (TK0268910-14)	3	127 126 126	- 6 8	243 234 234	BBCH 43 - 46 BBCH 45 - 46 BBCH 44 - 47	0	Cauliflower head and stem	0.040, 0.038 (0.039)

Legume Vegetables

Beans with pods

Ten independent field trials were conducted on beans with pods during the 2016 growing season in Canada and the USA (Hampton, 2018, Report No. TK0268911; Oakes, 2018, Report No. TK0269135).

Each trial site consisted of one untreated control plot and one treated plot. The treated plots received two broadcast applications of an SC formulation, containing 200 g ai/L, at 193–209 g ai/ha per application, with retreatment intervals of 6–9 days, totaling 398–429 g ai/ha. The applications were made with spray volumes of 159–290 L/ha. A methylated seed oil (MSO), non-ionic surfactant (NIS) or crop oil concentrate (COC) was added to the spray mixture for most applications. For all trials, samples were collected 13–14 days after the last application (DALA). For the decline trials, beans with pods were collected at 0, 7, 14 and 21 DALA. Each bean sample weighed a minimum of 1.0 kg with the exception of trial TK0269135-T808-D, where samples weighed 0.5 kg.

Samples were analyzed using the LC-MS/MS Method GRM061.03A. Acceptable concurrent recoveries were reported for all samples of beans with pods at fortification levels of 0.01 to 0.50 mg/kg, thus validating the method. The limit of quantitation (LOQ) was 0.01 mg/kg.

Treated samples were maintained frozen until analysis for a maximum storage interval of 310 days. Adequate storage stability data are available to support the storage conditions and intervals for samples in the current trials.

Table 10 Residues of pydiflumetofen in beans with pods from trials conducted in North America following application of an SC formulation

Location; Year, Variety (Trial ID)	Application rate per treatment				Growth Stage (BBCH)	DALA	Portion Analysed	Pydiflumetofen Residues mg/kg (average)
	No.	Rate (g ai/ha)	RTI	Water (L/ha)				
US GAP	2	200	14	NS		14		
Lyons, NY, USA 2016 Blue Lake Bush (TK0268911-10R)	2	204 203	- 8	281 281	BBCH 65 - 71 BBCH 71 - 74	14	Bean with pods	<0.01, 0.016 (0.013)
Rebecca, GA; USA 2016 Opportunes (TK0268911-11)	2	209 217	- 7	168 159	BBCH 51 BBCH 63	14	Beans with pods	0.022, 0.025 (0.024)
Oviedo, FL; USA 2016 Prevail (TK0268911-12)	2	205 193	- 7	290 271	BBCH 65 BBCH 69	14	Beans with pods	0.098, 0.185 (0.142)
Richland, IA; USA 2016 Provider OG (TK0268911-13)	2	198 201	- 7	168 225	BBCH 64 BBCH 69	13	Beans with pods	0.089, 0.044 (0.076)
Delavan, WI; USA 2016 Blue Lake Bush (TK0268911-14)	2	200 204	- 7	206 206	BBCH 69 - 70 BBCH 74	0	Beans with pods	0.124, 0.093 (0.109)
						7		0.066, 0.055 (0.061)
						14		0.015, 0.019 (0.017)
						21		<0.01, 0.013 (0.012)
						28		<0.01, <0.01 (<0.01)
Ephrata, WA; USA 2016 Improved Tendergreen (TK0268911-15)	2	202 202	- 8	187 187	BBCH 51 - 55 BBCH 61 - 63	14	Beans with pods	0.028, 0.025 (0.027)
Alma, ON; Canada 2016 Valentino (TK0269135-T806)	2	193 205	- 6	200 200	BBCH 55 - 59 BBCH 60 - 61	14	Beans with pods	0.01, 0.011 (0.011)
Bright, ON; Canada 2016 Oakley (TK0269135-T807)	2	216 212	- 7	200 200	BBCH 65 - 71 BBCH 65 - 75	14	Beans with pods	0.061, 0.055 (0.058)
St-Marc-sur-Richelieu, QC; Canada 2016 Gold Rush (TK0269135-T808-D)	2	207 205	- 8	200 200	BBCH 65 - 71 BBCH 65 - 79	0	Beans with pods	0.58
						6		0.67
						13		0.47, 0.39 (0.43)
						19		0.24
						24		0.24

Location; Year, Variety (Trial ID)	Application rate per treatment				Growth Stage (BBCH)	DALA	Portion Analysed	Pydiflumetofen Residues mg/kg (average)
	No.	Rate (g ai/ha)	RTI	Water (L/ha)				
Abbotsford, BC; Canada 2016 Gold Rush (TK0269135-T809)	2	196 206	- 9	200 200	BBCH 64 - 66 BBCH 72 - 76	13	Beans with pods	0.23, 0.28 (0.26)

Peas with pods

Seven independent field trials were conducted on peas with pods during the 2016 growing season in Canada and the USA (Hampton, 2018, Report No. TK0268911; Oakes, 2018, TK0269135).

Each trial site consisted of one untreated control plot and one treated plot. The treated plots received two broadcast applications of an SC formulation, containing 200 g ai/L, at 197–209 g ai/ha per application, with retreatment intervals of 6–9 days, totaling 402–413 g ai/ha. The applications were made with spray volumes of 178–234 L/ha. A methylated seed oil (MSO), non-ionic surfactant (NIS) or crop oil concentrate (COC) was added to the spray mixture for most applications. For all trials, samples were collected 13–14 days after the last application (DALA). For the decline trials, peas with pods were collected at 0, 7, 14, 21 and 28 DALA. Each pea sample weighed a minimum of 1.0 kg.

Samples were analyzed using the LC-MS/MS Method GRM061.03A. Acceptable concurrent recoveries were reported for all samples of peas with pods at fortification levels of 0.01 to 2.5 mg/kg, thus validating the method. The limit of quantitation (LOQ) was 0.01 mg/kg.

Treated samples were maintained frozen until analysis for a maximum storage interval of 353 days. Adequate storage stability data are available to support the storage conditions and intervals for samples in the current trials.

Table 11 Residues of pydiflumetofen in peas with pods from trials conducted in North America following application of an SC formulation

Location; Year, Variety (Trial ID)	Application rate per treatment				Growth Stage (BBCH)	DALA	Portion Analysed	Pydiflumetofen Residues mg/kg (average)
	No.	Rate (g ai/ha)	RTI	Water (L/ha)				
US GAP	2	200	14	NS		14		
Delavan, WI; USA 2016 Sugar Snap (TK0268911-07)	2	201 202	- 7	187 178	BBCH 50 BBCH 65	14	Peas with pods	<0.01, 0.012 (0.011)
Porterville, CA; USA 2016 Oregon Giant (TK0268911-08)	2	204 204	- 7	196 234	BBCH 69 - 79 BBCH 69 - 79	0	Peas with pods	1.797, 1.399 (1.598)
						7		0.725, 1.10 (0.913)
						14		0.435, 0.841 (0.638)
						21		0.296, 0.337 (0.317)
						28		0.038, 0.108 (0.073)

Location; Year, Variety (Trial ID)	Application rate per treatment				Growth Stage (BBCH)	DALA	Portion Analysed	Pydiflumetofen Residues mg/kg (average)
	No.	Rate (g ai/ha)	RTI	Water (L/ha)				
Ephrata, WA; USA 2016 Sugar pod (TK0268911-09)	2	201 204	- 7	187 187	BBCH 59 - 60 BBCH 63 - 65	14	Peas with pods	0.054, 0.053 (0.054)
New Glasgow, PEI; Canada 2016 Oregon Sugar Pod II (TK0269135-T810)	2	203 209	- 6	200 200	BBCH 51 - 61 BBCH 61 - 63	13	Peas with pods	0.10, 0.13 (0.12)
Portage la Prairie, MB; Canada 2016 Super Sugar Snap (TK0269135-T811)	2	205 197	- 7	200 200	BBCH 39 BBCH 57 - 59	14	Peas with pods	0.011, <0.01 (0.011)
Carlisle, ON; Canada 2016 Little Sweetie (TK0269135-T812)	2	209 198	- 6	200 200	BBCH 65 - 71 BBCH 65 - 73	13	Peas with pods	0.16, 0.15 (0.16)
St-Marc-sur-Richelieu, QC; Canada 2016 Sugar Queen (TK0269135-T813-D)	2	206 207	- 7	200 200	BBCH 65 - 69 BBCH 67 - 73	0	Peas with pods	0.79
						7		0.34
						14		0.64, 0.64 (0.64)
						22		0.021

Beans without pods

Nine independent field trials were conducted on beans without pods during the 2016 growing season in Canada and the USA (Hampton, 2018, Report No. TK0268911; Oakes, 2018, TK0269135).

Each trial site consisted of one untreated control plot and one treated plot. The treated plots received two broadcast applications of an SC formulation, containing 200 g ai/L, at 159–204 g ai/ha per application, with retreatment intervals of 6-8 days, except trial TK0268911-05, where the RTI was 14 days. Total seasonal rates ranged from 318–406 g ai/ha. The applications were made with spray volumes of 159–290 L/ha. A methylated seed oil (MSO), non-ionic surfactant (NIS) or crop oil concentrate (COC) was added to the spray mixture for most applications. For all trials, samples were collected 14–15 days after the last application (DALA). For the decline trials, beans without pods were collected at 0, 7, 14, 21 and 27 DALA. Each bean sample weighed a minimum of 1.0 kg with the exception of trials TK0269135-T815/T817-D, where samples weighed 0.3–0.9 kg.

Samples were analyzed using the LC-MS/MS Method GRM061.03A. Acceptable concurrent recoveries were reported for all samples of beans without pods at fortification levels of 0.01 to 0.50 mg/kg, thus validating the method. The limit of quantitation (LOQ) was 0.01 mg/kg.

Treated samples were maintained frozen until analysis for a maximum storage interval of 316 days. Adequate storage stability data are available to support the storage conditions and intervals for samples in the current trials.

Table 12 Residues of pydiflumetofen in beans without pods from trials conducted in North America following application of an SC formulation

Location; Year, Variety (Trial ID)	Application rate per treatment				Growth Stage (BBCH)	DALA	Portion Analysed	Pydiflumetofen Residues mg/kg (average)
	No.	Rate (g ai/ha)	RTI	Water (L/ha)				
US GAP	2	200	14	NS		14		
Seven springs, NC; USA 2016 Jackson Wonder Bush (TK0268911-01)	2	203 203	- 8	262 243	BBCH 75 - 80 BBCH 75 - 81	15	Beans without pods	0.050, 0.079 (0.065)
Chula, GA; USA 2016 Henderson (TK0268911-03)	2	159 159	- 6	206 207	BBCH 75 - 76 BBCH 77 - 79	14	Beans without pods	0.030, 0.038 (0.034)
Delavan, WI; USA 2016 Henderson Bush (TK0268911-04)	2	201 201	- 7	206 206	BBCH 72 - 74 BBCH 73 - 75	14	Beans without pods	0.021, 0.013 (0.017)
Porterville, CA; USA 2016 Fordhook 242 (TK0268911-05)	2	202 204	- 14	281 281	BBCH 84 - 86 BBCH 87 - 89	0	Beans without pods	0.013, 0.019 (0.016)
						7		0.025, 0.017 (0.021)
						14		0.013, 0.013 (0.013)
						21		0.012, 0.011 (0.012)
						27		<0.01, <0.01 (<0.01)
Ephrata, WA; USA 2016 Kingston, green baby lima (TK0268911-06)	2	202 203	- 7	187 187	BBCH 65 - 70 BBCH 65 - 72	14	Beans without pods	0.014, 0.012 (0.013)
Blackville, SC; USA 2016 Jackson Wonder (TK0268911-02R)	2	202 203	- 7	140 140	BBCH 66 - 71 BBCH 66 - 76	14	Beans without pods	0.018, 0.018 (0.018)
Carlisle, ON; Canada 2016 Eastland (TK0269135-T815)	2	202 192	- 6	200 200	BBCH 63 - 71 BBCH 65 - 73	15	Beans without pods	0.01, <0.01 (0.011)
Branchton, ON; Canada 2016 Zorro (TK0269135-T816)	2	204 202	- 6	200 200	BBCH 63 - 65 BBCH 67 - 69	15	Beans without pods	<0.01, <0.01 (<0.01)
St-Marc-sur-Richelieu, QC; Canada 2016 Sorano	2	202 201	- 7	200 200	BBCH 64 - 73 BBCH 65 - 75	0	Beans without pods	0.013
						8		<0.01
						15		<0.01, <0.01 (<0.01)
						21		<0.01

Location; Year, Variety (Trial ID)	Application rate per treatment				Growth Stage (BBCH)	DALA	Portion Analysed	Pydiflumetofen Residues mg/kg (average)
	No.	Rate (g ai/ha)	RTI	Water (L/ha)				
(TK0269135-T817-D)						28		<0.01

Peas without pods

Ten independent field trials were conducted on peas without pods during the 2016 growing season in Canada and the USA (Hampton, 2018, Report No. TK0268911; Oakes, 2018, TK0269135).

Each trial site consisted of one untreated control plot and one treated plot. The treated plots received two broadcast applications of an SC formulation, containing 200 g ai/L, at 198–212 g ai/ha per application, with retreatment intervals of 6–9 days, totalling 397–418 g ai/ha. The applications were made with spray volumes of 168–281 L/ha. A methylated seed oil (MSO), non-ionic surfactant (NIS) or crop oil concentrate (COC) was added to the spray mixture for most applications. For all trials, samples were collected 13–15 days after the last application (DALA). For the decline trials, peas without pods were collected at 0, 7, 15, 21 and 28 DALA. Each pea sample weighed a minimum of 1.0 kg.

Samples were analyzed using the LC-MS/MS Method GRM061.03A. Acceptable concurrent recoveries were reported for all samples of peas without pods at fortification levels of 0.01 and 0.10 mg/kg, thus validating the method. The limit of quantitation (LOQ) was 0.01 mg/kg.

Treated samples were maintained frozen until analysis for a maximum storage interval of 332 days. Adequate storage stability data are available to support the storage conditions and intervals for samples in the current trials.

Table 13 Residues of pydiflumetofen in peas without pods from trials conducted in North America following application of an SC formulation

Location; Year, Variety (Trial ID)	Application rate per treatment				Growth Stage (BBCH)	DALA	Portion Analysed	Pydiflumetofen Residues mg/kg (average)
	No.	Rate (g ai/ha)	RTI	Water (L/ha)				
US GAP	2	200	14	NS		14		
Lyons, NY; USA 2016 Oregon Sugar Pod II (TK0268911-16)	2	204 203	- 7	281 281	BBCH 71 - 73 BBCH 73 - 75	14	Peas without pods	0.011, 0.014 (0.013)
Oregon, WI; USA 2016 Sweet Savor EX08540797 (TK0268911-17)	2	205 202	- 7	168 178	BBCH 65 BBCH 69 - 71	14	Peas without pods	<0.01, <0.01 (<0.01)
Delavan, WI; USA 2016 Wando (TK0268911-18)	2	201 201	- 9	187 187	BBCH 71 - 72 BBCH 73 - 74	0	Peas without pods	0.013, 0.01 (0.012)
						7		<0.01, <0.01 (<0.01)
						15		<0.01, <0.01 (<0.01)
						21		<0.01, <0.01 (<0.01)
						28		<0.01, 0.013 (0.012)

Location; Year, Variety (Trial ID)	Application rate per treatment				Growth Stage (BBCH)	DALA	Portion Analysed	Pydiflumetofen Residues mg/kg (average)
	No.	Rate (g ai/ha)	RTI	Water (L/ha)				
Richland, IA; USA 2016 Sienna (TK0268911-19)	2	202 199	- 7	178 178	BBCH 68 - 69 BBCH 71	14	Peas without pods	0.014, 0.022 (0.018)
Ephrata, WA; USA 2016 Naches (TK0268911-20)	2	199 202	- 7	187 187	BBCH 65 - 67 BBCH 69 - 73	14	Peas without pods	<0.01, <0.01 (<0.01)
Grants-Pass, OR; USA 2016 Oregon Giant (TK0268911-21)	2	206 207	- 7	187 187	BBCH 71 BBCH 73 - 74	15	Peas without pods	<0.01, <0.01 (<0.01)
Bright, ON; Canada 2016 Jumbo (TK0269135-T819)	2	212 201	- 7	200 200	BBCH 67 - 75 BBCH 69 - 78	14	Peas without pods	<0.01, <0.01 (<0.01)
Alma, CO; USA 2016 Jumpstart (TK0269135-T820)	2	212 206	- 6	200 200	BBCH 61 - 62 BBCH 67 - 72	13	Peas without pods	0.016, <0.01 (0.013)
St-Marc-sur-Richelieu, QC ; Canada 2016 Petit Merveille (TK0269135-T821-D)	2	211 205	- 8	200 200	BBCH 65 - 71 BBCH 65 - 79	0	Peas without pods	0.049
						6		0.019
						13		<0.01, 0.011 (0.011)
						19		<0.01
						24		<0.01
Taber, AB; Canada 2016 Strike (TK0269135-T822)	2	198 199	- 7	200 200	BBCH 51 - 59 BBCH 65 - 69	13	Peas without pods	<0.01, <0.01 (<0.01)

Root vegetables

Twenty-eight independent field trials were conducted on sugar beets (17), carrot (6) and radish (5) during the 2016 growing season in Canada and the USA (Crawford, 2018, Report No. TK0268658; Oakes, 2018, TK0269131).

Each trial site consisted of one untreated control plot and one treated plot. For the trials conducted in the USA, the treated plots received four broadcast applications of an SC formulation, containing 75 g pydiflumetofen/L and 125 g difenoconazole/L, at 69–78 g ai/ha per application, with retreatment intervals of 6–8 days, totalling 295–308 g ai/ha. The applications were made with spray volumes of 87–462 L/ha. A non-ionic surfactant (NIS) or crop oil concentrate (COC) was added to the spray mixture for all applications. For all trials, samples were collected 6–7 days after the last application (DALA).

For the sugar beet trials conducted in Canada, the treated plots received four broadcast applications of an SC formulation, containing 200 g pydiflumetofen/L, at 73–82 g ai/ha per application, with retreatment intervals of 5–8 days, totalling 302–312 g ai/ha. The applications were made with spray volumes of 196–218 L/ha. A non-ionic surfactant (NIS) or crop oil concentrate (COC) was added to the spray mixture for all applications. For all trials, samples were collected 7 days after the last application (DALA).

For the decline trials, root and tuber vegetables were collected at 0, 7, 13/14, 21 and 28/29 DALA to monitor residue decline. All samples weighed a minimum of 2.0 kg.

Samples were analyzed using the LC-MS/MS Method GRM061.03A. Acceptable concurrent recoveries were reported for all samples of root and tuber vegetables at fortification levels of 0.01 to 10.0 mg/kg, thus validating the method. The limit of quantitation (LOQ) was 0.01 mg/kg.

Treated samples were maintained frozen until analysis for a maximum storage interval of 453 days. Adequate storage stability data are available to support the storage conditions and intervals for samples in the current trials.

Table 14 Residues of pydiflumetofen in root vegetables from trials conducted in North America following applications of various SC formulations

Location; Year, Variety (Trial ID)	Application rate per treatment				Growth Stage (BBCH)	DALA	Portion Analysed	Pydiflumetofen Residues mg/kg (average)
	No.	Rate (g ai/ha)	RTI	Water (L/ha)				
US GAP	4	75	7	NS		7		
SUGAR BEETS								
Carrington, ND; USA 2016 BetaShield PB BTS 89RR83 RP (860.1500-16-433-08A-07)	4	75 75 75 75	- 7 7 7	140 141 141 141	BBCH 37 BBCH 38 BBCH 39 BBCH 39	7	Root	0.059, 0.059 (0.059)
Kisksville, MO; USA 2016 NR (860.1500-16-433-08A-08)	4	75 74 77 77	- 7 7 7	171 185 180 183	BBCH 45-46 BBCH 46 BBCH 46-47 BBCH 48-49	7	Root	0.039, 0.033 (0.036)
Richland, IA; USA 2016 NR (860.1500-16-433-08A-09)	4	75 75 75 76	- 7 7 7	181 144 143 215	BBCH 46 BBCH 47 BBCH 47-48 BBCH 48-49	0 7 14 21 28	Root	0.050, 0.052 (0.051) 0.040, 0.046 (0.043) 0.033, 0.045 (0.039) 0.055, 0.040 (0.048) 0.035, 0.081 (0.058)
St. John, KS; USA 2016 BTS 8512 Pro 200 (860.1500-16-433-08A-10)	4	77 77 77 75	- 7 7 7	213 212 211 207	BBCH 36 BBCH 37 BBCH 38 BBCH 38	7	Root	0.034, 0.036 (0.035)
Larned, KS; USA 2016 VTS 8RR52 RP (860.1500-16-433-08A-11)	4	73 73 73 76	- 7 7 7	201 201 201 209	BBCH 35 BBCH 37 BBCH 38 BBCH 39	7	Root	0.095, 0.171 (0.133)
Delavan, WI; USA	4	73	-	202	BBCH 38	7	Root	0.090, 0.091 (0.091)

Location; Year, Variety (Trial ID)	Application rate per treatment				Growth Stage (BBCH)	DALA	Portion Analysed	Pydiflumetofen Residues mg/kg (average)
	No.	Rate (g ai/ha)	RTI	Water (L/ha)				
US GAP	4	75	7	NS		7		
2016 HR (860.1500-16-433-08A-12)		73 74 74	7 7 7	204 208 202	BBCH 42 BBCH 45 BBCH 48			
American Falls, ID; USA 2016 BJS28R (860.1500-16-433-08A-18)	4	74 69 75 77	- 7 6 8	91 87 93 96	BBCH 37 BBCH 38 BBCH 39 BBCH 39-49	7	Root	0.061, 0.063 (0.062)
Northwood, ND; USA 2016 BTS 89RR83 (860.1500-16-433-08A-19)	4	76 75 76 75	- 7 7 7	141 140 141 140	BBCH 38 BBCH 38 BBCH 39 BBCH 39	7	Root	0.058, 0.037 (0.048)
United Porterville, CA; USA 2016 HH142 (860.1500-16-433-08A-20)	4	75 75 75 75	- 7 7 7	343 347 451 462	BBCH 38 BBCH 39 BBCH 39 BBCH 49	7	Root	0.018, 0.019 (0.019)
Elm Creek, MB; Canada 2016 SV36152RR (TK0269131-T827)	4	75 78 74 80	- 7 7 7	199 207 196 213	BBCH 39 BBCH 39 BBCH 39 BBCH 39	7	Root	0.094, 0.097 (0.096)
Portage la Prairie, MB; Canada 2016 HM 9221RR (TK0269131-T828)	4	78 76 78 77	- 7 7 7	208 203 208 205	BBCH 40 BBCH 40 BBCH 49 BBCH 49	7	Root	0.016, 0.015 (0.016)
Elie, MB; Canada 2016 9221RR (TK0269131-T829)	4	77 74 77 77	- 7 7 7	205 196 206 204	BBCH 49 BBCH 49 BBCH 49 BBCH 49	7	Root	0.098, 0.150 (0.124)
Taber, AB; Canada 2016 SV36152RR (TK0269131-T830)	4	73 75 77 77	- 5 7 7	201 200 205 206	BBCH 39 - 49 BBCH 39 - 49 BBCH 39 - 49 BBCH 39 - 49	0 7 13 21 29	Root	0.066 0.050, 0.054 (0.052) 0.058 0.057 0.052
Bow Island, AB; Canada 2016 BTS 49RR Pro 200 (TK0269131-T831)	4	82 74 75 76	- 5 7 7	218 196 200 202	BBCH 39 - 49 BBCH 39 - 49 BBCH 39 - 49 BBCH 39 - 49	7	Root	0.088, 0.078 (0.083)
Grassy Lake, AB: Canada 2016 9221RR	4	79 77 76	- 5 7	210 205 202	BBCH 39 - 49 BBCH 39 - 45 BBCH 39 - 45	7	Root	0.15, 0.12 (0.14)

Location; Year, Variety (Trial ID)	Application rate per treatment				Growth Stage (BBCH)	DALA	Portion Analysed	Pydiflumetofen Residues mg/kg (average)
	No.	Rate (g ai/ha)	RTI	Water (L/ha)				
US GAP	4	75	7	NS		7		
(TK0269131-T832)		80	7	212	BBCH 39 - 49			
Outlook, SK; Canada 2016 9221RR (TK0269131-T833)	4	77 77 75 77	- 7 7 7	205 204 199 206	BBCH 37 - 38 BBCH 37 - 38 BBCH 37 - 38 BBCH 37 - 38	7	Root	0.044, 0.085 (0.065)
Kipp, AB; Canada 2016 SV36152RR (TK0269131-T834)	4	78 77 77 76	- 5 8 6	207 206 204 203	BBCH 39 - 40 BBCH 39 - 40 BBCH 39 - 49 BBCH 39 - 49	7	Root	0.097, 0.10 (0.101)
CARROTS								
Hobe sound, FL; USA 2016 Apache (860.1500-16-433-08A-02)	4	75 75 75 74	- 7 7 7	335 330 337 326	BBCH 45 BBCH 46 BBCH 48 BBCH 48	7	Root	0.067, 0.080 (0.074)
Fisk, MO; USA 2016 Notable (860.1500-16-433-08A-05)	4	75 75 76 76	- 7 7 7	94 93 95 94	BBCH 46 BBCH 42 BBCH 48 BBCH 44	7	Root	0.062, 0.060 (0.061)
Raymondville, TX; USA 2016 Danvers 126 (860.1500-16-433-08A-13)	4	78 75 78 77	- 7 7 7	195 187 194 192	BBCH 43-45 BBCH 45 BBCH 45-47 BBCH 47-49	7	Root	0.109, 0.094 (0.102)
Marysville, CA: USA 2016 Red Cored Chantenay (860.1500-16-433-08A-14)	4	75 76 75 76	- 7 7 7	282 284 281 286	BBCH 46 BBCH 47 BBCH 48 BBCH 48	7	Root	0.016, 0.014 (0.015)
Porterville, CA; USA 2016 Danvers (860.1500-16-433-08A-16)	4	75 76 75 75	- 7 7 7	195 194 192 193	BBCH 44 BBCH 46 BBCH 47 BBCH 48	0 7 14 21 28	Root	0.017, 0.017 (0.017) 0.029, 0.024 (0.027) 0.032, 0.023 (0.028) 0.018, 0.021 (0.020) 0.013, 0.010 (0.012)
Arroyo Grande, CA; USA 2016 KF Mini (860.1500-16-433-08A-17)	4	75 75 74 75	- 7 7 7	283 282 279 282	BBCH 41 BBCH 42 BBCH 43 BBCH 45	6	Root	0.057, 0.102 (0.080)
RADISH								
North Rose, NY; USA 2016 Champion (860.1500-16-433-08A-01)	4	78 77 77 75	- 7 7 7	243 239 239 234	BBCH 09 BBCH 15 BBCH 20 BBCH 85	7	Root	0.132, 0.149 (0.141)

Location; Year, Variety (Trial ID)	Application rate per treatment				Growth Stage (BBCH)	DALA	Portion Analysed	Pydiflumetofen Residues mg/kg (average)
	No.	Rate (g ai/ha)	RTI	Water (L/ha)				
US GAP	4	75	7	NS		7		
Hobe Sound, FL; USA 2016 Redsilk (860.1500-16-433-08A-03)	4	73 75 75 75	- 7 7 7	324 337 332 342	BBCH 12 BBCH 13 BBCH 43 BBCH 48	7	Root	0.177, 0.154 (0.166)
Oviedo, FL; USA 2016 Rover F1 (860.1500-16-433-08A-04)	4	75 74 75 76	- 7 7 7	282 279 281 285	BBCH 19 BBCH 41 BBCH 44 BBCH 47	7	Root	0.170, 0.163 (0.167)
Northwood, ND; USA 2016 Cherry Belle (860.1500-16-433-08A-06)	4	75 74 76 75	- 7 7 7	190 185 191 190	BBCH 11-12 BBCH 13-14 BBCH 42 BBCH 44-45	7	Root	0.014, 0.012 (0.013)
Yuba City, CA; USA 2016 Crimson Giant (860.1500-16-433-08A-15)	4	74 75 74 76	- 7 7 7	92 94 92 95	BBCH 12 BBCH 14 BBCH 41 BBCH 45	7	Root	0.029, 0.018 (0.024)

Cereal Grains

Sorghum

Twelve independent field trials were conducted on sorghum during the 2016 growing season in the USA (Seastrum, 2018, Report No. TK0294708).

Each trial site consisted of one untreated control plot and two treated plots, one for the harvest of forage and the second for the harvest of grain and stover. The treated plot, intended for the harvest of grain, received two broadcast applications of an SC formulation, containing 200 g ai/L, at 122–134 g ai/ha per application, with retreatment intervals of 4–6 days, totalling 245–262 g ai/ha. The applications were made with spray volumes of 94–215 L/ha. A non-ionic surfactant (NIS) or crop oil concentrate (COC) was added to the spray mixture for most applications. For all trials, samples were collected 18–23 days after the last application (DALA). For the decline trials, sorghum grain was collected at 15, 18, 21, 25 and 27 DALA.

Samples were analyzed using the LC-MS/MS Method GRM061.03A. Acceptable concurrent recoveries were reported for all samples of sorghum grain at fortification levels of 0.01 to 5.0 mg/kg, thus validating the method. The limit of quantitation (LOQ) was 0.01 mg/kg.

Treated samples were maintained frozen until analysis for a maximum storage interval of 143 days. Adequate storage stability data are available to support the storage conditions and intervals for samples in the current trials.

Table 15 Residues of pydiflumetofen in sorghum grain from trials conducted in the USA following application of an SC formulation

Location; Year, Variety (Trial ID)	Application rate per treatment				Growth Stage (BBCH)	DALA	Portion Analysed	Pydiflumetofen Residues mg/kg (average)
	No.	Rate (g ai/ha)	RTI	Water (L/ha)				
US GAP	2	126	7	NS		21		
Richlands, NC; USA 2017 SP7715 (TK0294708-01)	2	124 126	- 6	215 215	BBCH 85 - 87 BBCH 86 - 88	18	Grain	0.620, 0.654 (0.637)
Proctor, AR; USA 2017 Pioneer 84P80 (TK0294708-02)	2	124 123	- 5	140 140	BBCH 86 - 87 BBCH 87 - 88	21	Grain	0.495, 0.393 (0.444)
Cresco, IA; USA 2017 Martin Milo (TK0294708-03)	2	123 122	- 6	187 94	BBCH 83 BBCH 83	21	Grain	0.095, 0.130 (0.112)
Richland, IA; USA 2017 AG 1401 (TK0294708-04)	2	123 125	- 4	150 178	BBCH 80 BBCH 82	20	Grain	0.105, 0.111 (0.108)
Gardner, ND; USA 2017 WGF Grain Sorghum (TK0294708-05)	2	134 128	- 5	150 150	BBCH 78 - 80 BBCH 85 - 87	15	Grain	0.348, 0.343 (0.346)
						18		0.341, 0.267 (0.304)
						21		0.344, 0.259 (0.302)
						25		0.260, 0.305 (0.283)
						27		0.290, 0.296 (0.293)
Northwood, ND; USA 2017 LGS 5001T (TK0294708-06)	2	125 124	- 5	187 187	BBCH 87 BBCH 87	21	Grain	0.740, 0.658 (0.699)
Waller, TX; USA 2017 DKS53-53 (TK0294708-07)	2	127 122	- 4	140 140	BBCH 69 BBCH 73	21	Grain	0.116[a], 0.105[a] (0.110)
Uvalde, TX; USA 2017 DKS37-07 (TK0294708-08)	2	122 125	- 5	159 168	BBCH 87 BBCH 87	20	Grain	1.028, 1.095 (1.062)
Grand Island, NE; USA 2017 7PO6 (TK0294708-09)	2	124 124	- 6	187 178	BBCH 87 BBCH 87	19	Grain	0.469, 0.425 (0.447)
Levelland, TX; USA 2017	2	123 125	- 5	140 140	BBCH 77 - 83 BBCH 84 - 85	21	Grain	1.851, 1.823 (1.837)

Location; Year, Variety (Trial ID)	Application rate per treatment				Growth Stage (BBCH)	DALA	Portion Analysed	Pydiflumetofen Residues mg/kg (average)
	No.	Rate (g ai/ha)	RTI	Water (L/ha)				
DKS37-07 (TK0294708-10)								
San Angelo, TX; USA 2017 Golden Acres 3960B (TK0294708-11)	2	127 126	- 5	178 187	BBCH 85 - 87 BBCH 87	23	Grain	0.671, 0.490 (0.580)
Edmonson, TX; USA 2017 Pioneer 84P80 (TK0294708-12)	2	130 125	- 4	150 140	BBCH 86 - 87 BBCH 86 - 87	15	Grain	0.752, 0.733 (0.743)
						17		0.574, 0.633 (0.604)
						21		0.552, 0.630 (0.591)
						25		0.645, 0.644 (0.645)
						29		0.496, 0.513 (0.505)

[a] Values presented are the average of three analyses.

Tree Nuts

Ten independent field trials were conducted on almonds (5) and pecans (5) during the 2014 growing season in the USA (McDonald, 2018, Report No. TK0173383).

Each trial site consisted of one untreated control plot and one treated plot. The treated plot received three broadcast applications of an SC formulation, containing 200 g ai/L, at 98–103 g ai/ha per application, with retreatment intervals of 7 days, totalling 301–307 g ai/ha. The applications were made using concentrated spray volumes of 468–786 L/ha and dilute spray volumes of 1403–1880 L/ha. A non-ionic surfactant (NIS) was added to the spray mixture for all applications. For all trials, samples were collected 14 days after the last application (DALA). For the decline trials, tree nuts were collected at 7, 10, 14, 17/18 and 20/21 DALA. Whole nuts were collected from several locations on the trees to generate a minimum sample weight of 1.4 kg of nutmeat.

Samples were analyzed using the LC-MS/MS Method GRM061.03A. Acceptable concurrent recoveries were reported for all samples of nutmeats at fortification levels of 0.01 and 0.10 mg/kg, thus validating the method. The limit of quantitation (LOQ) was 0.01 mg/kg.

Treated samples were maintained frozen until analysis for a maximum storage interval of 420 days. Adequate storage stability data are available to support the storage conditions and intervals for samples in the current trials.

Table 16 Residues of pydiflumetofen in tree nuts from trials conducted in the USA following application of an SC formulation

Location; Year, Variety (Trial ID)	Application rate per treatment				Growth Stage (BBCH)	DALA	Portion Analysed	Pydiflumetofen Residues mg/kg (average)
	No.	Rate (g ai/ha)	RTI	Water (L/ha)				
US GAP	3	100	7	Ground (NS) Aerial (Min 94)		14		
ALMONDS								
Strathmore, CA; USA 2014 Fritz (TK0173383-01)	3	102 101 101	- 7 7	505 496 514	BBCH 81 - 85 BBCH 81 - 85 BBCH 85 - 89	7	Nutmeat	<0.01
						10		<0.01
						14		<0.01, <0.01 (<0.01)
						17		<0.01
						21		<0.01
Fresno, CA; USA 2014 Aldrich (TK0173383-02)	3	101 102 101	- 7 7	1871 1880 1861	BBCH 81 BBCH 83 BBCH 84	14	Nutmeat	0.033, 0.031 (0.032)
Wasco, CA; USA 2014 Butte (TK0173383-03)	3	101 102 98	- 7 7	1506 1506 1403	BBCH 89 BBCH 89 BBCH 89	14	Nutmeat	<0.01, <0.01 (<0.01)
Kerman, CA; USA 2014 Monterey (TK0173383-04)	3	102 103 102	- 7 7	468 477 468	BBCH 85 - 87 BBCH 85 - 87 BBCH 85 - 89	14	Nutmeat	<0.01, <0.01 (<0.01)
Madera, CA; USA 2014 Nonpareil (TK0173383-05)	3	101 101 102	- 7 7	1600 1600 1609	BBCH 81 - 85 BBCH 81 - 85 BBCH 81 - 85	14	Nutmeat	0.026, 0.028 (0.027)
PECANS								
Weston, GA; USA 2014 Byrd (TK0173383-06)	3	101 101 101	- 7 7	1469 1469 1525	BBCH 78 - 79 BBCH 82 - 85 BBCH 79 - 80	14	Nutmeat	<0.01, <0.01 (<0.01)
Mystic, GA; USA 2014 Summer (TK0173383-07)	3	100 100 101	- 7 7	1525 1487 1469	BBCH 85 - 87 BBCH 87 - 89 BBCH 87 - 89	7	Nutmeat	<0.01
						10		<0.01
						14		<0.01, <0.01 (<0.01)
						18		<0.01
						20		<0.01
Opelosuas, LA; USA 2014 Jackson (TK0173383-08)	3	103 102 99	- 7 7	655 636 627	BBCH 73 - 74 BBCH 78 - 79 BBCH 84 - 85	13	Nutmeat	0.018, 0.012 (0.015)
Pearsall, TX; USA	3	101	-	683	BBCH 85 - 87	14	Nutmeat	<0.01, <0.01 (<0.01)

Location; Year, Variety (Trial ID)	Application rate per treatment				Growth Stage (BBCH)	DALA	Portion Analysed	Pydiflumetofen Residues mg/kg (average)
	No.	Rate (g ai/ha)	RTI	Water (L/ha)				
US GAP	3	100	7	Ground (NS) Aerial (Min 94)		14		
2014 Cheyanne (TK0173383-09)		102 101	7 7	599 599	BBCH 87 BBCH 87 - 89			
Dill City, OK; USA 2014 Kanza (TK0173383-10)	3	102 103 101	- 7 7	748 758 786	BBCH 79 - 80 BBCH 80 - 83 BBCH 80 - 83	15	Nutmeat	<0.01, <0.01 (<0.01)

Oilseeds

Sunflower

Eleven independent field trials were conducted on sunflower during the 2016 growing season in Canada and the USA (Hampton, 2018, Report No. TK0265867; McDonald, 2018, Report No. TK0269138).

Each trial site consisted of one untreated control plot and one treated plot. The treated plot received two broadcast applications of an SC formulation, containing 200 g ai/L, at 195–208 g ai/ha per application, with retreatment intervals of 11–15 days, totalling 399–412 g ai/ha. The applications were made using spray volumes of 122–253 L/ha. A non-ionic surfactant (NIS) crop oil concentrate (COC) or methylated seed oil (MSO)/MSO blend was added to the spray mixture for all applications. For all trials, samples were collected 28–33 days after the last application (DALA). For the decline trials, sunflower seeds were collected at 20/21, 24/25, 30, 35/36 and 39/40 DALA. All seed samples weighed a minimum of 1.0 kg.

Samples were analyzed using the LC-MS/MS Method GRM061.03A. Acceptable concurrent recoveries were reported for all samples of nutmeats at fortification levels of 0.01 to 1.25 mg/kg, thus validating the method. The limit of quantitation (LOQ) was 0.01 mg/kg.

Treated samples were maintained frozen until analysis for a maximum storage interval of 341 days. Adequate storage stability data are available to support the storage conditions and intervals for samples in the current trials.

Table 17 Residues of pydiflumetofen in sunflower seeds from trials conducted in North America following application of an SC formulation

Location; Year, Variety (Trial ID)	Application rate per treatment				Growth Stage (BBCH)	DALA	Portion Analysed	Pydiflumetofen Residues mg/kg (average)
	No.	Rate (g ai/ha)	RTI	Water (L/ha)				
US GAP	2	200	14	NS		30		
Branchton, ON; Canada 2016 Falcon	2	208 195	- 14	208 195	BBCH 63 - 65 BBCH 69 - 71	33	Seed	0.13, 0.10 (0.12)

Location; Year, Variety (Trial ID)	Application rate per treatment				Growth Stage (BBCH)	DALA	Portion Analysed	Pydiflumetofen Residues mg/kg (average)
	No.	Rate (g ai/ha)	RTI	Water (L/ha)				
(TK0269138-T823)								
Bright, ON; Canada 2016 Dakota (TK0269138-T824)	2	199 200	- 14	199 200	BBCH 65 - 75 BBCH 69 - 79	30	Seed	0.029, 0.23 (0.130)
Killarney, MB; Canada 2016 Falcon (TK0269138-T826_	2	205 207	- 14	205 207	BBCH 81 - 83 BBCH 83 - 85	28	Seed	0.015, 0.02 (0.018)
Geneva, MN; Canada 2016 Royal Hybrid 1121 F1 (TK0265867-01)	2	204 204	- 14	187 178	BBCH 71 - 73 BBCH 75 - 79	20	Seed	0.064, 0.045 (0.055)
						25		0.037, 0.059 (0.048)
						30		0.052, 0.051 (0.052)
						35		0.041, 0.051 (0.046)
						40		0.076, 0.061 (0.069)
Aurora, SD: USA 2016 Dove CL blend (TK0265867-02)	2	203 203	- 11	253 243	BBCH 65 - 69 BBCH 80 - 85	32	Seed	0.071, 0.072 (0.072)
Northwood, ND; USA 2016 Cobalt II (TK0265867-03_	2	204 201	- 14	187 140	BBCH 79 - 80 BBCH 84 - 86	29	Seed	0.018, 0.02 (0.019)
Velva, ND; USA 2016 SC410CL (TK0265867-04)	2	208 201	- 14	122 122	BBCH 69 - 73 BBCH 71 - 75	31	Seed	0.075, 0.076 (0.076)
Carrington, ND; USA 2016 Cobalt II (TK0265867-05)	2	202 200	- 14	140 140	BBCH 79 - 80 BBCH 84 - 86	28	Seed	0.014, 0.018 (0.016)
Kenesaw, NE; USA 2016 2C5302C-Maxx (TK0265867-06)	2	202 204	- 14	224 206	BBCH 75 - 79 BBCH 85 - 87	21	Seed	0.334, 0.387 (0.361)
						24		0.255, 0.294 (0.275)
						30		0.329, 0.410 (0.370)
						36		0.130, 0.129 (0.130)
						39		0.211, 0.249 (0.230)
Grand Island, NE; USA 2016 8N668S (TK0265867-07)	2	201 202	- 15	187 187	BBCH 81 BBCH 83	30	Seed	0.151, 0.115 (0.133)
Larned, KS; USA 2016 63M91 (TK0265867-08)	2	204 208	- 14	206 178	BBCH 65 - 67 BBCH 79	28	Seed	0.077, 0.067 (0.072)

Cottonseed

Twelve independent field trials were conducted on cottonseed during the 2016 growing season in the USA (Lenz, 2018, Report No. TK0265622).

Each trial site consisted of one untreated control plot and one treated plot. The treated plot received two broadcast applications of an SC formulation, containing 200 g ai/L, at 113–134 g ai/ha per application, with retreatment intervals of 7–12 days, totalling 237–259 g ai/ha. The applications were made using spray volumes of 94–196 L/ha. A non-ionic surfactant (NIS) or crop oil concentrate (COC) was added to the spray mixture for all applications. For all trials, samples were collected 28–33 days after the last application (DALA). For the decline trials, cottonseeds were collected at 20, 24/26, 30/31, 35/38 and 40 DALA. All seed samples weighed a minimum of 1.0 kg.

Samples were analyzed using the LC-MS/MS Method GRM061.03A. Acceptable concurrent recoveries were reported for all samples of cotton seeds at fortification levels of 0.01 to 5.0 mg/kg, thus validating the method. The limit of quantitation (LOQ) was 0.01 mg/kg.

Treated samples were maintained frozen until analysis for a maximum storage interval of 341 days. Adequate storage stability data are available to support the storage conditions and intervals for samples in the current trials.

Table 18 Residues of pydiflumetofen in cotton seeds from trials conducted in the USA following application of an SC formulation

Location; Year, Variety (Trial ID)	Application rate per treatment				Growth Stage (BBCH)	DALA	Portion Analysed	Pydiflumetofen Residues mg/kg (average)
	No.	Rate (g ai/ha)	RTI	Water (L/ha)				
US GAP	3	75	10	NS		45		
Chula, GA; USA 2016 PHY 444 (TK0265622-01)	2	129 128	- 8	159 168	BBCH 83-84 BBCH 85	30	Undelinted seed	0.031, 0.044 (0.038)
Cheneyville, LA; USA 2016 ST 4946 GLB2 (TK0265622-02)	2	125 134	- 8	178 196	BBCH 79-80 BBCH 81-82	28	Undelinted seed	0.015, <0.01 (0.012)
Proctor, AR; USA 2016 ST 4946 GLB2 (TK0265622-03)	2	124 125	- 10	94 94	BBCH 80-81 BBCH 84-85	33	Undelinted seed	0.073, 0.095 (0.084)
Fisk, MO; USA 2016 Croplan 3475 Xtend (TK0265622-04)	2	127 128	- 9	187 187	BBCH 78-79 BBCH 81-82	20	Undelinted seed	<0.01, <0.01 (<0.01)
						26		0.018, <0.01 (0.014)
						30		<0.01, <0.01 (<0.01)
						35		<0.01, <0.01 (<0.01)
						40		<0.01, <0.01 (<0.01)
Waller, TX; USA	2	126	-	140	BBCH 76-77	29	Undelinted seed	0.083, 0.088 (0.086)

Location; Year, Variety (Trial ID)	Application rate per treatment				Growth Stage (BBCH)	DALA	Portion Analysed	Pydiflumetofen Residues mg/kg (average)
	No.	Rate (g ai/ha)	RTI	Water (L/ha)				
2016 PHY 499 (TK0265622-05)		123	11	140	BBCH 83-84			
Hinton, OK; USA 2016 A1020045 (TK0265622-06)	2	126 124	- 11	122 196	BBCH 81-82 BBCH 82-83	27	Undelinted seed	0.048, 0.091 (0.070)
Levelland, TX; USA 2016 NexGen 3406 (TK0265622-07)	2	124 113	- 10	140 140	BBCH 69 BBCH 78-80	29	Undelinted seed	0.10, 0.087 (0.094)
Groom, TX; USA 2016 PHY 222 (TK0265622-08)	2	129 126	- 7	196 187	BBCH 86 BBCH 87	33	Undelinted seed	0.026, 0.032 (0.029)
San Angelo, TX; USA 2016 FM 2334 GLT (TK0265622-09)	2	125 126	- 12	150 150	BBCH 81 BBCH 85	20	Undelinted seed	0.036, 0.040 (0.038)
						24		0.039, 0.024 (0.032)
						31		0.036, 0.015 (0.026)
						38		0.027, 0.028 (0.028)
						40		0.017, 0.017 (0.017)
Paso Robles, CA; USA 2016 DP 358 (TK0265622-10)	2	128 127	- 11	196 187	BBCH 81-83 BBCH 81-85	29	Undelinted seed	0.102, 0.144 (0.120)
Fresno, CA; USA 2016 Acala (TK0265622-11)	2	124 124	- 10	187 187	BBCH 82 BBCH 85	30	Undelinted seed	0.060, 0.024 (0.042)
Sanger, CA; USA 2016 PHY 725 RF – Acala (TK0265622-12)	2	124 126	- 10	131 131	BBCH 61-65 BBCH 61-65	32	Undelinted seed	<0.01, <0.01 (<0.01)

Animal Feeds

Leaves of root vegetables

Twenty-two independent field trials were conducted on sugar beets (17) and radish (5) during the 2016 growing season in Canada and the USA (Crawford, 2018, Report No. TK0268658; Oakes, 2018, TK0269131).

Each trial site consisted of one untreated control plot and one treated plot. For the trials conducted in the United States, the treated plots received four broadcast applications of an SC formulation, containing 75 g pydiflumetofen/L and 125 g difenoconazole/L, at 69–78 g ai/ha per application, with retreatment intervals of 6–8 days, totalling 295–308 g ai/ha. The applications were made with spray volumes of 87–

462 L/ha. A non-ionic surfactant (NIS) or crop oil concentrate (COC) was added to the spray mixture for all applications. Leaves were collected 7 days after the last application (DALA).

For the sugar beet trials conducted in Canada, the treated plots received four broadcast applications of an SC formulation, containing 200 g pydiflumetofen/L, at 73–82 g ai/ha per application, with retreatment intervals of 5–8 days, totalling 302–312 g ai/ha. The applications were made with spray volumes of 196–218 L/ha. A non-ionic surfactant (NIS) or crop oil concentrate (COC) was added to the spray mixture for all applications. Leaves were collected 7 days after the last application (DALA).

For the decline trials, sugar beet and radish tops were collected at 0, 7, 13/14, 21 and 28/29 DALA to monitor residue decline. All samples weighed a minimum of 2.0 kg.

Samples were analyzed using the LC-MS/MS Method GRM061.03A. Acceptable concurrent recoveries were reported for all samples of leaves of root and tuber vegetables at fortification levels of 0.01 to 150 mg/kg, thus validating the method. The limit of quantitation (LOQ) was 0.01 mg/kg.

Treated samples were maintained frozen until analysis for a maximum storage interval of 453 days. Adequate storage stability data are available to support the storage conditions and intervals for samples in the current trials.

Table 19 Residues of pydiflumetofen in sugar beet tops and radish tops from trials conducted in North America following application of various SC formulations

Location; Year, Variety (Trial ID)	Application rate per treatment				Growth Stage (BBCH)	DALA	Portion Analysed	Pydiflumetofen Residues mg/kg (average)
	No.	Rate (g ai/ha)	RTI	Water (L/ha)				
US GAP	4	75	7	NS		7		
SUGAR BEETS								
Carrington, ND; USA 2016 BetaShield PB BTS 89RR83 RP (860.1500-16-433-08A-07)	4	75 75 75 75	- 7 7 7	140 141 141 141	BBCH 37 BBCH 38 BBCH 39 BBCH 39	7	Top (leaves)	1.02, 0.810 (0.915)
Kisksville, MO; USA 2016 NR (860.1500-16-433-08A-08)	4	75 74 77 77	- 7 7 7	171 185 180 183	BBCH 45-46 BBCH 46 BBCH 46-47 BBCH 48-49	7	Top (leaves)	6.37, 4.99 (5.68)
Richland, IA; USA 2016 NR (860.1500-16-433-08A-09)	4	75 75 75 76	- 7 7 7	181 144 143 215	BBCH 46 BBCH 47 BBCH 47-48 BBCH 48-49	0 7 14 21 28	Top (leaves)	6.14, 5.39 (5.77) 5.44, 4.29 (4.86) 5.68, 5.00 (5.34) 0.999, 0.934 (0.967) 0.306, 0.525 (0.416)
St. John, KS; USA 2016 BTS 8512 Pro 200 (860.1500-16-433-08A-10)	4	77 77 77 75	- 7 7 7	213 212 211 207	BBCH 36 BBCH 37 BBCH 38 BBCH 38	7	Top (leaves)	1.59, 1.56 (1.58)
Larned, KS; USA 2016	4	73 73	- 7	201 201	BBCH 35 BBCH 37	7	Top (leaves)	2.08, 1.28 (1.68)

Location; Year, Variety (Trial ID)	Application rate per treatment				Growth Stage (BBCH)	DALA	Portion Analysed	Pydiflumetofen Residues mg/kg (average)
	No.	Rate (g ai/ha)	RTI	Water (L/ha)				
US GAP	4	75	7	NS		7		
VTS 8RR52 RP (860.1500-16-433-08A-11)		73 76	7 7	201 209	BBCH 38 BBCH 39			
Delavan, WI; USA 2016 HR (860.1500-16-433-08A-12)	4	73 73 74 74	- 7 7 7	202 204 208 202	BBCH 38 BBCH 42 BBCH 45 BBCH 48	7	Top (leaves)	0.831, 0.718 (0.774)
American Falls, ID; USA 2016 BJS28R (860.1500-16-433-08A-18)	4	74 69 75 77	- 7 6 8	91 87 93 96	BBCH 37 BBCH 38 BBCH 39 BBCH 39-49	7	Top (leaves)	0.937, 0.589 (0.763)
Northwood, ND; USA 2016 BTS 89RR83 (860.1500-16-433-08A-19)	4	76 75 76 75	- 7 7 7	141 140 141 140	BBCH 38 BBCH 38 BBCH 39 BBCH 39	7	Top (leaves)	1.26, 0.769 (1.01)
United Porterville, CA; USA 2016 HH142 (860.1500-16-433-08A-20)	4	75 75 75 75	- 7 7 7	343 347 451 462	BBCH 38 BBCH 39 BBCH 39 BBCH 49	7	Top (leaves)	4.49, 8.05 (6.27)
Elm Creek, MB; Canada 2016 SV36152RR (TK0269131-T827)	4	75 78 74 80	- 7 7 7	199 207 196 213	BBCH 39 BBCH 39 BBCH 39 BBCH 39	7	Top (leaves)	1.00, 1.10 (1.05)
Portage la Prairie, MB; Canada 2016 9221RR (TK0269131-T828)	4	78 76 78 77	- 7 7 7	208 203 208 205	BBCH 40 BBCH 40 BBCH 49 BBCH 49	7	Top (leaves)	1.60, 1.40 (1.50)
Elie, MB; Canada 2016 9221RR (TK0269131-T829)	4	77 74 77 77	- 7 7 7	205 196 206 204	BBCH 49 BBCH 49 BBCH 49 BBCH 49	7	Top (leaves)	1.60, 1.40 (1.50)
Taber, AB; Canada 2016 SV36152RR (TK0269131-T830)	4	73 75 77 77	- 5 7 7	201 200 205 206	BBCH 39 - 49 BBCH 39 - 49 BBCH 39 - 49 BBCH 39 - 49	0 7 13 21 29	Top (leaves)	1.80 1.3, 1.5 (1.4) 0.83 0.52 0.45
Bow Island, AB; Canada 2016 BTS 49RR Pro 200 (TK0269131-T831)	4	82 74 75 76	- 5 7 7	218 196 200 202	BBCH 39 - 49 BBCH 39 - 49 BBCH 39 - 49 BBCH 39 - 49	7	Top (leaves)	1.30, 1.60 (1.45)
Grassy Lake, AB: Canada 2016	4	79 77	- 5	210 205	BBCH 39 - 49 BBCH 39 - 45	7	Top (leaves)	3.70, 3.60 (3.65)

Location; Year, Variety (Trial ID)	Application rate per treatment				Growth Stage (BBCH)	DALA	Portion Analysed	Pydiflumetofen Residues mg/kg (average)
	No.	Rate (g ai/ha)	RTI	Water (L/ha)				
US GAP	4	75	7	NS		7		
9221RR (TK0269131-T832)		76 80	7 7	202 212	BBCH 39 - 45 BBCH 39 - 49			
Outlook, SK; Canada 2016 9221RR (TK0269131-T833)	4	77 77 75 77	- 7 7 7	205 204 199 206	BBCH 37 - 38 BBCH 37 - 38 BBCH 37 - 38 BBCH 37 - 38	7	Top (leaves)	0.96, 0.98 (0.97)
Kipp, AB; Canada 2016 SV36152RR (TK0269131-T834)	4	78 77 77 76	- 5 8 6	207 206 204 203	BBCH 39 - 40 BBCH 39 - 40 BBCH 39 - 49 BBCH 39 - 49	7	Top (leaves)	3.80, 3.00 (3.40)
RADISH								
North Rose, NY; USA 2016 Champion (860.1500-16-433-08A-01)	4	78 77 77 75	- 7 7 7	243 239 239 234	BBCH 09 BBCH 15 BBCH 20 BBCH 85	7	Top (leaves)	5.57, 4.59 (5.08)
Hobe Sound, FL; USA 2016 Redsilk (860.1500-16-433-08A-03)	4	73 75 75 75	- 7 7 7	324 337 332 342	BBCH 12 BBCH 13 BBCH 43 BBCH 48	7	Top (leaves)	2.54, 3.24 (2.89)
Oviedo, FL; USA 2016 Rover F1 (860.1500-16-433-08A-04)	4	75 74 75 76	- 7 7 7	282 279 281 285	BBCH 19 BBCH 41 BBCH 44 BBCH 47	7	Top (leaves)	3.10, 2.85 (2.98)
Northwood, ND; USA 2016 Cherry Belle (860.1500-16-433-08A-06)	4	75 74 76 75	- 7 7 7	190 185 191 190	BBCH 11-12 BBCH 13-14 BBCH 42 BBCH 44-45	7	Top (leaves)	0.847, 0.941 (0.894)
Yuba City, CA; USA 2016 Crimson Giant (860.1500-16-433-08A-15)	4	74 75 74 76	- 7 7 7	92 94 92 95	BBCH 12 BBCH 14 BBCH 41 BBCH 45	7	Top (leaves)	0.136, 0.106 (0.121)

Sorghum forage

Twelve independent field trials were conducted on sorghum during the 2016 growing season in the USA (Seastrum, 2018, Report No. TK029708).

Each trial site consisted of one untreated control plot and two treated plots, one for the harvest of forage and the second for the harvest of grain and stover. The treated plot, intended for the harvest of forage, received two broadcast applications of an SC formulation, containing 200 g ai/L, at 121–127 g ai/ha per application, with retreatment intervals of 4–7 days, totalling 245–254 g ai/ha. The applications were

made with spray volumes of 70–271 L/ha. A non-ionic surfactant (NIS) or crop oil concentrate (COC) was added to the spray mixture for most applications. For all trials, samples were collected 28–32 days after the last application (DALA). For the decline trials, sorghum forage was collected at 24, 27/28, 30, 33/34 and 36/37 DALA. Each sample weighed a minimum of 1.0 kg.

Samples were analyzed using the LC-MS/MS Method GRM061.03A. Acceptable concurrent recoveries were reported for all samples of sorghum forage at fortification levels of 0.01 to 2.0 mg/kg, thus validating the method. The limit of quantitation (LOQ) was 0.01 mg/kg.

Treated samples were maintained frozen until analysis for a maximum storage interval of 181 days. Adequate storage stability data are available to support the storage conditions and intervals for samples in the current trials.

Table 20 Residues of pydiflumetofen in sorghum forage (as received) from trials conducted in the USA following application of an SC formulation

Location; Year, Variety (Trial ID)	Application rate per treatment				Growth Stage (BBCH)	DALA	Portion Analysed	Pydiflumetofen Residues mg/kg (average)
	No.	Rate (g ai/ha)	RTI	Water (L/ha)				
US GAP	2	126	7	NS		30		
Richlands, NC; USA 2017 SP7715 (TK0294708-01)	2	126 128	- 5	271 234	BBCH 37 - 41 BBCH 55 - 56	28	Forage	0.082, 0.098 (0.090)
Proctor, AR; USA 2017 Pioneer 84P80 (TK0294708-02)	2	124 124	- 5	40 140	BBCH 35 - 37 BBCH 41 - 43	30	Forage	0.027, 0.070 (0.048)
Cresco, IA; USA 2017 Martin Milo (TK0294708-03)	2	126 125	- 5	187 187	BBCH 58 BBCH 61	30	Forage	0.165, 0.198 (0.182)
Richland, IA; USA 2017 AG 1401 (TK0294708-04)	2	123 124	- 4	206 206	BBCH 19 BBCH 39	30	Forage	0.476, 0.430 (0.453)
Gardner, ND; USA 2017 WGF Grain Sorghum (TK0294708-05)	2	126 125	- 5	150 150	BBCH 58 - 61 BBCH 62 - 65	24	Forage	0.510, 0.473 (0.492)
						27		1.18, 0.969 (1.08)
						30		0.575, 0.632 (0.604)
						33		0.729, 0.666 (0.698)
						36		0.559, 0.413 (0.486)
Northwood, ND; USA 2017 LGS 5001T (TK0294708-06)	2	123 124	- 5	187 187	BBCH 71 BBCH 73	29	Forage	0.604, 0.607 (0.606)
Waller, TX; USA 2017 DKS53-53	2	127 127	- 7	140 140	BBCH 41 - 43 BBCH 53 - 54	31	Forage	0.145, 0.128 (0.136)

Location; Year, Variety (Trial ID)	Application rate per treatment				Growth Stage (BBCH)	DALA	Portion Analysed	Pydiflumetofen Residues mg/kg (average)
	No.	Rate (g ai/ha)	RTI	Water (L/ha)				
(TK0294708-07)								
Uvalde, TX; USA 2017 DKS37-07 (TK0294708-08)	2	125 123	- 4	206 140	BBCH 16 - 17 BBCH 19 - 37	32	Forage	0.076, 0.109 (0.091)
Grand Island, NE; USA 2017 7PO6 (TK0294708-09)	2	124 123	- 5	187 187	BBCH 67 - 71 BBCH 75	32	Forage	0.119, 0.164 (0.142)
Levelland, TX; USA 2017 DKS37-07 (TK0294708-10)	2	121 124	- 6	140 140	BBCH 45 - 47 BBCH 51	29	Forage	0.505, 0.399 (0.452)
San Angelo, TX; USA 2017 Golden Acres 3960B (TK0294708-11)	2	124 125	- 5	187 187	BBCH 39 - 43 BBCH 58 - 61	28	Forage	0.578 0.475 (0.527)
Edmonson, TX; USA 2017 Pioneer 84P80 (TK0294708-12)	2	121 124	- 5	140 150	BBCH 35 - 37 BBCH 35 - 37	24	Forage	0.504, 0.366 (0.435)
						28		0.253, 0.438 (0.346)
						30		0.244, 0.247 (0.246)
						34		0.309, 0.241 (0.275)
						37		0.249, 0.183 (0.216)

Sorghum stover

Twelve independent field trials were conducted on sorghum during the 2016 growing season in the USA (Seastrum, 2018, Report No. TK029708).

Each trial site consisted of one untreated control plot and two treated plots, one for the harvest of forage and the second for the harvest of grain and stover. The treated plot, intended for the harvest of stover, received two broadcast applications of an SC formulation, containing 200 g ai/L, at 122–134 g ai/ha per application, with retreatment intervals of 4–6 days, totalling 245–262 g ai/ha. The applications were made with spray volumes of 94–215 L/ha. A non-ionic surfactant (NIS) or crop oil concentrate (COC) was added to the spray mixture for most applications. For all trials, samples were collected 19–23 days after the last application (DALA). For the decline trials, sorghum grain was collected at 15, 18, 21, 25 and 27 DALA.

Samples were analyzed using the LC-MS/MS Method GRM061.03A. Acceptable concurrent recoveries were reported for all samples of sorghum stover at fortification levels of 0.01 to 10.0 mg/kg, thus validating the method. The limit of quantitation (LOQ) was 0.01 mg/kg.

Treated samples were maintained frozen until analysis for a maximum storage interval of 144 days. Adequate storage stability data are available to support the storage conditions and intervals for samples in the current trials.

Table 21 Residues of pydiflumetofen in sorghum stover (as received) from trials conducted in the USA following application of an SC formulation

Location; Year, Variety (Trial ID)	Application rate per treatment				Growth Stage (BBCH)	DALA	Portion Analysed	Pydiflumetofen Residues mg/kg (average)
	No.	Rate (g ai/ha)	RTI	Water (L/ha)				
US GAP	2	1266	7			21		
Richlands, NC; USA 2017 SP7715 (TK0294708-01)	2	124 126	- 6	215 215	BBCH 85 - 87 BBCH 86 - 88	21	Stover	0.676, 0.637 (0.657)
Proctor, AR; USA 2017 Pioneer 84P80 (TK0294708-02)	2	124 123	- 5	140 140	BBCH 86 - 87 BBCH 87 - 88	21	Stover	0.252, 0.249 (0.251)
Cresco, IA; USA 2017 Martin Milo (TK0294708-03)	2	123 122	- 6	187 94	BBCH 83 BBCH 83	21	Stover	0.183, 0.119 (0.151)
Richland, IA; USA 2017 AG 1401 (TK0294708-04)	2	123 125	- 4	150 178	BBCH 80 BBCH 82	20	Stover	0.082, 0.064 (0.073)
Gardner, ND; USA 2017 WGF Grain Sorghum (TK0294708-05)	2	134 128	- 5	150 150	BBCH 78 - 80 BBCH 85 - 87	15	Stover	0.334, 0.188 (0.261)
						18		0.181, 0.182 (0.181)
						21		0.128, 0.257 (0.192)
						25		0.210, 0.174 (0.192)
						27		0.113, 0.184 (0.148)
Northwood, ND; USA 2017 LGS 5001T (TK0294708-06)	2	125 124	- 5	187 187	BBCH 87 BBCH 87	21	Stover	5.16, 4.78 (4.97)
Waller, TX; USA 2017 DKS53-53 (TK0294708-07)	2	127 122	- 4	140 140	BBCH 69 BBCH 73	21	Stover	0.186, 0.233 (0.210)
Uvalde, TX; USA 2017 DKS37-07 (TK0294708-08)	2	122 125	- 5	159 168	BBCH 87 BBCH 87	20	Stover	1.29, 1.20 (1.25)
Grand Island, NE; USA 2017 7PO6	2	124 124	- 6	187 178	BBCH 87 BBCH 87	19	Stover	0.324, 0.440 (0.382)

Location; Year, Variety (Trial ID)	Application rate per treatment				Growth Stage (BBCH)	DALA	Portion Analysed	Pydiflumetofen Residues mg/kg (average)
	No.	Rate (g ai/ha)	RTI	Water (L/ha)				
(TK0294708-09)								
Levelland, TX; USA 2017 DKS37-07 (TK0294708-10)	2	123 125	- 5	140 140	BBCH 77 - 83 BBCH 84 - 85	21	Stover	7.55, 7.42, 4.41, 4.56 (5.98)
San Angelo, TX; USA 2017 Golden Acres 3960B (TK0294708-11)	2	127 126	- 5	178 187	BBCH 85 - 87 BBCH 87	23	Stover	3.45, 3.39 (3.42)
Edmonson, TX; USA 2017 Pioneer 84P80 (TK0294708-12)	2	130 125	- 4	150 140	BBCH 86 - 87 BBCH 86 - 87	15	Stover	0.852, 0.530 (0.691)
						17		0.788, 0.379 (0.584)
						21		0.625, 0.622 (0.624)
						25		0.529, 0.482 (0.506)
						29		0.280, 0.292 (0.286)

Almond Hulls

Five independent field trials were conducted on almonds during the 2014 growing season in the USA (McDonald, 2018, Report No. TK0173383).

Each trial site consisted of one untreated control plot and one treated plot. The treated plot received three broadcast applications of an SC formulation, containing 200 g ai/L, at 98–103 g ai/ha per application, with retreatment intervals of 7 days, totalling 301–307 g ai/ha. The applications were made using concentrated spray volumes of 468–514 L/ha and dilute spray volumes of 1403–1880 L/ha. A non-ionic surfactant (NIS) was added to the spray mixture for all applications. For all trials, almond hull samples were collected 14 days after the last application (DALA). For the decline trials, almonds were collected at 7, 10, 14, 17 and 21 DALA.

Samples were analyzed using the LC-MS/MS Method GRM061.03A. Acceptable concurrent recoveries were reported for all samples of almond hulls at fortification levels of 0.01 to 10 mg/kg, thus validating the method. The limit of quantitation (LOQ) was 0.01 mg/kg.

Treated samples were maintained frozen until analysis for a maximum storage interval of 420 days. Adequate storage stability data are available to support the storage conditions and intervals for samples in the current trials.

Table 22 Residues of pydiflumetofen in almond hulls from trials conducted in the USA following application of an SC formulation

Location; Year, Variety (Trial ID)	Application rate per treatment				Growth Stage (BBCH)	DALA	Portion Analysed	Pydiflumetofen Residues mg/kg (average)
	No.	Rate (g ai/ha)	RTI	Water (L/ha)				
US GAP	3	100	7	Ground (NS)		14		

Location; Year, Variety (Trial ID)	Application rate per treatment				Growth Stage (BBCH)	DALA	Portion Analysed	Pydiflumetofen Residues mg/kg (average)
	No.	Rate (g ai/ha)	RTI	Water (L/ha)				
				Aerial (Min 94)				
Strathmore, CA; USA 2014 Fritz (TK0173383-01)	3	102 101 101	- 7 7	505 496 514	BBCH 81 - 85 BBCH 81 - 85 BBCH 85 - 89	7 10 14 17 21	Hulls	0.69 0.79 0.28, 0.46 (0.37) 0.71 0.78
Fresno, CA; USA 2014 Aldrich (TK0173383-02)	3	101 102 101	- 7 7	1871 1880 1861	BBCH 81 BBCH 83 BBCH 84	14	Hulls	3.98, 3.87 (3.92)
Wasco, CA; USA 2014 Butte (TK0173383-03)	3	101 102 98	- 7 7	1506 1506 1403	BBCH 89 BBCH 89 BBCH 89	14	Hulls	1.70, 1.40 (1.55)
Kerman, CA; USA 2014 Monterey (TK0173383-04)	3	102 103 102	- 7 7	468 477 468	BBCH 85 - 87 BBCH 85 - 87 BBCH 85 - 89	14	Hulls	1.30, 1.40 (1.35)
Madera, CA; USA 2014 Nonpareil (TK0173383-05)	3	101 101 102	- 7 7	1600 1600 1609	BBCH 81 - 85 BBCH 81 - 85 BBCH 81 - 85	14	Hulls	3.60, 3.60 (3.60)

Cotton gin by-products

Twelve independent field trials were conducted on cottonseed during the 2016 growing season in the USA (Lenz, 2018, Report No. TK0265622).

Each trial site consisted of one untreated control plot and one treated plot. The treated plot received two broadcast applications of an SC formulation, containing 200 g ai/L, at 113–126 g ai/ha per application, with retreatment intervals of 10–12 days, totalling 237–251 g ai/ha. The applications were made using spray volumes of 122–196 L/ha. A non-ionic surfactant (NIS) or crop oil concentrate (COC) was added to the spray mixture for all applications. At seven trial sites, cotton samples were collected using mechanical picker equipment and at five trial sites, cotton samples were collected using mechanical stripper equipment. For all trials, samples were collected 27–29 days after the last application (DALA). For the decline trials, cotton seeds were collected at 20, 24, 31, 38 and 40 DALA. After harvest, cotton samples were ginned. All samples of cotton gin by-products weighed a minimum of 0.5 kg.

Samples were analyzed using the LC-MS/MS Method GRM061.03A. Acceptable concurrent recoveries were reported for all samples of cotton gin by-products at fortification levels of 0.01 to 5.0 mg/kg, thus validating the method. The limit of quantitation (LOQ) was 0.01 mg/kg.

Treated samples were maintained frozen until analysis for a maximum storage interval of 341 days. Adequate storage stability data are available to support the storage conditions and intervals for samples in the current trials.

Table 23 Residues of pydiflumetofen in cotton gin by-products from trials conducted in the USA following application of an SC formulation

Location; Year, Variety (Trial ID)	Application rate per treatment				Growth Stage (BBCH)	DALA	Portion Analysed	Pydiflumetofen Residues mg/kg (average)
	No.	Rate (g ai/ha)	RTI	Water (L/ha)				
Waller, TX; USA 2016 PHY 499 (TK0265622-05)	2	126 123	- 11	140 140	BBCH 76-77 BBCH 83-84	29	Gin by-product	1.64, 2.01 (1.83)
Hinton, OK; USA 2016 A1020045 (TK0265622-06)	2	126 124	- 11	122 196	BBCH 81-82 BBCH 82-83	27	Gin by-product	1.69, 1.48 (1.585)
Levelland, TX; USA 2016 NexGen 3406 (TK0265622-07)	2	124 113	- 10	140 140	BBCH 69 BBCH 78-80	29	Gin by-product	3.28, 3.62 (3.45)
San Angelo, TX; USA 2016 FM 2334 GLT (TK0265622-09)	2	125 126	- 12	150 150	BBCH 81 BBCH 85	20	Gin by-product	1.20, 1.20 (1.20)
						24		0.940, 0.799 (0.870)
						31		0.830, 0.820 (0.825)
						38		0.947, 1.27 (1.11)
						40		1.15, 1.08 (1.11)

FATE OF RESIDUES IN STORAGE AND PROCESSING

Residues after processing

Citrus fruits - Orange

Two processing trials were conducted in conjunction with residue field trials on oranges grown in the USA during the 2016 growing season (Crawford, 2018, Report No. TK0268661). Pydiflumetofen, formulated as an SC formulation (containing 200 g ai/L) was applied four times at 411–432 g ai/ha, with application intervals of 6–8 days totalling 1676–1695 g ai/ha/season. Each application was made in conjunction with either a non-ionic surfactant (NIS) or crop oil concentrate (COC) adjuvant.

Samples of oranges were collected 0 days after the last application (DALA) from both trial sites. Oranges were processed into juice, peel, dried pulp and oil using methods which are representative of commercial practice.

Briefly, washed oranges (6.7 kg) were hand peeled to generate the washed peel sample. A separate sample of washed oranges (196 kg) was then abraded to scarify the flavedo for oil recovery. The scarified

fruit was hand peeled to generate the peel after oil extraction sample. The remaining peeled, scarified fruit was used to make dried pulp.

To make dried pulp, the peeled and scarified fruit was chopped using a food processor and fed through a pulper finisher. The juice from the finisher was used to make marmalade. The pulp recovered from the finisher was pressed using a fruit press to form the wet pulp. A portion of the wet pulp was dried in an air dryer to < 10% moisture and the dried pulp was milled to a finished moisture content of 2.25–2.87%.

For the juice, scarified oranges (36 kg) were transferred to a juice extractor. The collected juice was transferred to a pulper finisher and screened to remove the vesicular membranes, seeds, segment membranes, and peel fragments from the juice.

To make the oil, the collected oil-water emulsion from the scarification process (181 kg) was transferred to a sifter and screened to separate any flavedo fractions from the oil-water emulsion. The scarified flavedo fraction was used to make pomace. The oil-water emulsion was processed through a cream separator and centrifuged to separate the oil from the emulsion.

Once processed, samples of orange fruit (RAC), peel, dried pulp, oil and juice were stored frozen for a maximum of 197 days and then analysed for residues of pydiflumetofen using the concurrently validated method GRM061.03A.

Table 24 Pydiflumetofen residues in orange and processed commodities

Trial Identification (City, State/Region, Country, Year)	Variety	Commodity or Matrix	Total Rate (g ai/ha)	DALA	Pydiflumetofen Residues (Average), mg/kg	Processing Factor
860.1520-16-433-01A-22 (Oviedo, FL, USA/2016)	Valencia	Whole fruit RAC, pre-processing	1676	0	0.470, 0.448, 0.374 (0.430)	-
		Peel			0.833	1.94
		Juice			<0.01	<0.02
		Dried pulp			1.45	3.38
		Oil			14.50	33.7
860.1520-16-433-01A-23 (Reedley, CA; USA/2016)	Valencia	Whole fruit RAC, pre-processing	1695	0	0.994, 0.894, 1.018 (0.968)	-
		Peel			1.74	1.78
		Juice			<0.01	<0.02
		Oil			51.0	52.7

Pome fruits

Two processing trials were conducted in conjunction with residue field trials on pome fruits (apple and pear) in the USA during 2014 (Salzman, 2017, Repot No. TK0103855). Pydiflumetofen, formulated as an SC formulation (containing 200 g ai/L) was applied four times at application rates of 253–258 g ai/ha, with an application interval of 7 days, totalling 1014–1030 g ai/ha. Each application was made in conjunction with either a non-ionic surfactant (NIS) or crop oil concentrate (COC) adjuvant.

Samples of apple and pear were collected 0 days after the last application (DALA) from both trial sites. Fruits were processed into juice, canned fruit, wet pomace, dried fruit and apple sauce using methods which are representative of commercial practice.

One sample of apples and pears (60–65 kg) was passed through a hammer mill to produce mash. The mash was layered into cloth sacks on a hydraulic press and pressed to separate juice from wet pomace. While the mash was pressed, juice samples were collected. After pressing the wet pomace stacks were broken up, combined, mixed and wet pomace samples collected. A second sample of apples and pears was peeled, sliced and cored prior to being dehydrated. Some of the sliced fruit were heated to make sauce (apple) and for canning.

Samples of fruit (RAC), juice, canned fruit, wet pomace, dried fruit and apple sauce were stored frozen for a maximum of 322 days and then analysed for residues of pydiflumetofen using the concurrently validated method GRM061.03A.

Table 25 Pydiflumetofen residues in pome fruit and pome fruit processed commodities

Trial Identification (City, State/Region, Country, Year)	Crop / Variety	Commodity or Matrix	Total Rate (g ai/ha)	DALA	Pydiflumetofen Residues (Average), mg/kg	Processing Factor
TK0103855-03 (North Rose, NY, USA/2014)	Apple/ Ida Red	Whole fruit RAC, pre-processing	1014	0	0.465, 0.732, 0.506 (0.568)	-
		Canned apple			0.019, 0.018 (0.019)	0.03
		Apple wet pomace			2.26, 2.02 (2.14)	3.77
		Apple juice			0.034, 0.037 (0.036)	0.06
		Apple sauce			0.048, 0.022 (0.035)	0.06
		Dried apple			0.223, 0.239 (0.231)	0.41
TK0103855-18 (Ephrata, WA/2014)	Pear/ Red D'Anjou	Whole fruit RAC, pre-processing	1030	0	0.598, 0.682, 0.654 (0.645)	-
		Canned fruit			0.062, 0.060 (0.061)	0.09
		Dried fruit			0.351, 0.449 (0.400)	0.62
		Wet pomace			1.94, 1.92 (1.93)	3.0
		Juice			0.075, 0.067 (0.071)	0.11

Stone fruits

Plum

Two processing trials were conducted in conjunction with residue field trials on plums in the USA during the 2016 growing season (Hampton, 2018, Report No. TK0270167). Pydiflumetofen, formulated as an SC formulation (containing 200 g ai/L) was applied four times at application rates of 372–382 g ai/ha, with application intervals of 6–7 days, totalling 1501–1512 g ai/ha. Only one trial included the application of a methylated seed oil (MSO) adjuvant.

Samples of plums were collected 0 days after the last application (DALA) from both trial sites. Plums were processed into juice, puree and prunes using methods which are representative of commercial practice.

Briefly, fresh plums (34 kg) were washed for 5 minutes of manual agitation in a stainless steel wash cart or kettle. For dried prunes, washed plums (10 kg) were spread onto lined drying trays and placed in a

dryer set at 68–79 °C for 11.5–17 hours. The prunes were allowed to cool for approximately 20 minutes, and were hand-pitted by slitting them with a knife and squeezing the pits out.

For puree processing, a sample of washed plums (9 kg) was blanched in 96 °C water for 1 minute and then chopped to break up the skins. The chopped fruit was fed through a pulper finisher to separate the puree from the skins, stones and fibrous waste.

For juicing, the washed fruit (15 kg) was chopped in a food processor, then transferred to a kettle and heated to 40–50 °C. Pectic enzyme was added to the fruit and blended and allowed to set and react for ~2 hours. The heated, enzyme treated fruit was then pressed. The fresh juice was centrifuged to separate the heavy solids. The juice was decanted prior to being blended with diatomaceous earth and filtered using a vacuum pump, vacuum flask and Buchner funnels lined with filter paper.

Samples of plum (RAC), juice, puree and prunes were stored frozen for a maximum of 195 days then were analysed for residues of pydiflumetofen using the concurrently validated method GRM061.03A.

Table 26 Pydiflumetofen residues in plum and processed commodities

Trial Identification (City, State/Region, Country, Year)	Variety	Commodity or Matrix	Total Rate (g ai/ha)	DALA	Pydiflumetofen Residues (Average), mg/kg	Processing Factor
TK0270167-12 (Farmersville, CA, USA/2016)	French Prune	Pre-processing fruit	1501	0	0.922, 0.962, 0.917 (0.93)	-
		Juice			0.013, 0.014 (0.014)	0.01
		Puree			0.245, 0.246 (0.246)	0.27
		Prune			1.68, 1.71 (1.70)	1.8
TK0270167-12 (Terra Bella, CA, USA/2016)	French Prune	Pre - processing fruit	1512	0	1.12, 1.20, 1.18 (1.17)	-
		Juice			0.025, 0.021 (0.023)	0.02
		Puree			0.172, 0.173 (0.173)	0.15
		Prune			0.325, 0.325 (0.325)	2.8

Root vegetables

Sugar beets

Two processing trials were conducted in conjunction with residue field trials on sugar beet in the USA during the 2016 growing season (Crawford, 2018, TK0268658). Pydiflumetofen, formulated as an SC formulation (containing 75 g ai/L pydiflumetofen + 125 g ai/L difenoconazole) was applied four times at 374–379 g ai/ha, at an application interval of 7 days and totalling 1500–1506 g ai/ha. Each application was made in conjunction with either a non-ionic surfactant (NIS)

Samples of sugar beet root were collected 7 days after the last application (DALA) from both trial sites. Sugar beet roots were processed into refined sugar, molasses and dried pulp using methods which are representative of commercial practice.

Sugar beet roots were washed in a stainless-steel tub filled with warm water to remove excess field dirt. The larger cleaned beets were cut into thick pieces while the remaining cleaned beets were sliced into cossettes. Sugar was extracted from the cossettes in a series of steam heated kettles with a mixture of fresh water and pulp press water. The kettles were heated to 65–80 °C. The cossettes and water were transferred counter current to each other through a series of vessels. Extracted beet pulp was pressed to

recover sugar solution. Pressed pulp was dried at 81–83 °C to a moisture content of 2.33–9.8% and subsequently milled.

Raw juice from the diffuser was purified in a kettle by adding lime and carbon dioxide. The temperature was maintained at an average of 83 °C. The precipitated impurities were coagulated by adding settling acid and allowed to settle and clarify. Clear juice was decanted and vacuum filtered using Buchner funnels. The clarified liquid was further purified by a second carbonation with carbon dioxide gas. Carbonated liquor was vacuum filtered using Buchner funnels. The clarified thin juice was concentrated in a steam jacketed stainless steel kettle. The concentrated juice was filtered and the filtered thick juice was warmed to approximately 75 °C and placed in a steam jacketed stainless steel kettle. The massecuite (wet sugar) was filtered after which a sample of molasses was collected. The remaining massecuite was heated. The washed sugar was removed as a filter cake from the Buchner funnel and placed in a mixing bowl. Hot air was used to heat the bowl while the wet sugar was stirred and dried.

Samples of root (RAC), refined sugar, molasses and dried pulp were stored frozen for a maximum of 419 days and then analysed for residues of pydiflumetofen using the concurrently validated method GRM061.03A.

Table 27 Pydiflumetofen residues in sugar beet and processed commodities

Trial Identification (City, State/Region Country, Year)	Variety	Commodity or Matrix	Total Rate (g ai/ha)	DALA	Pydiflumetofen Residues (Average), mg/kg	Processing Factor
860.1500-16-433-08A-19 (Northwood, ND, USA/2016)	BTS 89RR83 RP	Root pre-processing (RAC)	1506	7	0.410, 0.654, 0.506 (0.523)	-
		Refined sugar			0.037	0.07
		Molasses			0.357	0.68
		Dried pulp			3.12	6.0
860.1500-16-433-08A-20 (Porterville CA, USA/2016)	HH 142	Root pre-processing (RAC)	1500	7	0.159, 0.175, 0.081 (0.138)	-
		Refined sugar			<0.01	<0.07
		Molasses			0.068	0.50
		Dried pulp			0.247	1.8

Cereals

Sorghum

Two processing trials were conducted in conjunction with residue field trials on sorghum in the USA during the 2017 growing season (Seastrum, 2018, Report No. TK0294708).

Pydiflumetofen, formulated as an SC formulation (containing 200 g ai/L) was applied twice at 610–632 g ai/ha, with application intervals of 4–6 days, totalling 1225–1253 g ai/ha. Each application was made in conjunction with either a non-ionic surfactant (NIS) or crop oil concentrate (COC) adjuvant.

Samples of sorghum grain were collected 21 days after the last application (DALA) and processed into flour and aspirated grain fraction (AGF) using methods which are representative of commercial practice.

To generate aspirated grain fractions, grain samples (337 kg) 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). Each batch was moved for 120 minutes. Light impurities were classified according to different particle sizes. The 2360-micron material was classified as aspirated grain fraction.

A separate sample of grain sorghum (23 kg) was cleaned by aspiration and screening to separate large and small foreign particles (screenings) from the cleaned grain sorghum sample. Cleaned grain sorghum (19 kg) was ground in a mill. Ground material was screened with a rotating sifter equipped with a mesh sieve. Material passing through the screen was grain sorghum flour.

Samples of grain, flour and AGF were stored frozen for a maximum of 189 days and then analysed for residues of pydiflumetofen using the concurrently validated method GRM061.03A.

Table 28 Pydiflumetofen residues in sorghum and processed commodities

Trial Identification (City, State/Region, Country, Year)	Variety	Commodity or Matrix	Total Rate (g ai/ha)	DALA	Pydiflumetofen Residues (Average), mg/kg	Processing Factor
TK0294708-06 (Northwood, ND, USA/2017)	LGS 5001T	Pre-processing grain	1253	21	5.07, 3.83, 4.63 (4.51)	-
		Aspirated grain fraction			289, 276 (283)	63
		Flour			3.67, 3.68 (3.68)	0.82
TK0294708-08 (Uvalde, TX, USA/2017)	DKS37-07	Pre-processing grain	1225	21	4.22, 4.14, 4.52 (4.30)	-
		Aspirated grain fraction			267, 258 (263)	61
		Flour			3.85, 4.01 (3.93)	0.91

Tree nuts

Almonds

A single processing trial was conducted in conjunction with a residue field trial on almond in the USA during the 2014 growing season (McDonald, 2018, Report No. TK0173383). Pydiflumetofen, formulated as an SC formulation (containing 200 g ai/L) was applied three times at 500–504 g ai/ha, with an application interval of 7 days, totalling 1506 g ai/ha. Each application was made in conjunction with a non-ionic surfactant (NIS).

Samples of almond nutmeat were collected 14 days after the last application and processed into roasted almond and oil using methods which are representative of commercial practice.

For almond oil, nutmeats (6 kg) were cracked/broken into smaller pieces and dried in an oven for 1 hour with a maximum temperature of 146 °C. After drying, nutmeat material was fed through an expeller to mechanically remove a majority of the oil. Cold pressing produced crude oil and presscake (meal with residual oil). Crude oil was filtered.

For light roasting, whole nutmeats (1.5 kg) were dry roasted at temperature of 130–150 °C for 45 minutes in a roaster.

Samples of nutmeat (RAC), roasted almond and oil were stored frozen for a maximum of 386 days and then analysed for residues of pydiflumetofen using the concurrently validated method GRM061.03A.

Table 29 Pydiflumetofen residues in almond and processed commodities

Trial Identification (City, State/Region, Country, Year)	Variety	Commodity or Matrix	Total Rate (g ai/ha)	DALA	Pydiflumetofen Residues (Average), mg/kg	Processing Factor
TK0173383-03 (Wasco, CA, USA/2014)	Butt	Pre-processing nutmeat	1506	14	0.12, 0.13, 0.072 (0.107)	-
		Roasted			0.041	0.37
		Oil			0.038	0.34

Oilseeds

Sunflower

Two processing trials were conducted in conjunction with residue field trials on sunflower in the USA during the 2016 growing season (Hampton, 2018, Report No. TK 0265867). Pydiflumetofen, formulated as an SC formulation (containing 200 g ai/L) was applied twice at 1003–1017 g ai/ha, at an application interval of 10–14 days, totalling 2012–2024 g ai/ha. Each application was made in conjunction with either a non-ionic surfactant (NIS) or crop oil concentrate (COC) adjuvant.

Samples of sunflower seed were collected 29–32 days after the last application (DALA). Seeds were processed into meal and oil using methods which are representative of commercial practice.

Sunflower seeds (31 kg) were dried, cleaned by aspiration and screened. Light impurities were removed from the whole seed using an aspirator. After aspiration, the whole seed was screened in a two-screen cleaner. Large and small particles (screenings) not removed by aspiration were separated from the sunflower seed.

Cleaned sunflower seeds were fed into a huller to crack the hull and liberate the kernel. Cracked material was passed through the aspirator to separate the hull from the kernel material. The kernel was moisture conditioned and expelled (mechanically pressed) in an expeller to liberate a portion of the crude oil. Presscake exiting the expeller was ground with a mill resulting in expelled crude oil and ground presscake with residual crude oil. Residual oil in the presscake was removed during solvent extraction.

The presscake was placed in stainless steel batch extractors and submerged in heated hexane. After 30 minutes, the miscella (hexane and crude oil) was drained. Heated hexane was added to repeat the cycle two more times.

After the final draining, solvent-extracted presscake (meal) was desolventized using warm air.

Miscella was passed through a laboratory vacuum evaporator to separate the crude oil and hexane. Crude oil was heated to remove any remaining hexane.

After the crude oil was filtered, the free fatty acid (FFA) content was determined. Based on the FFA content and weight of the crude oil to be refined, sodium hydroxide was added to the crude oil in a water bath and mixed. Neutralized oil was centrifuged to separate refined oil and soapstock. Refined oil was decanted from the soapstock and filtered.

Refined oil was mixed and heated. An activated bleaching earth was added and the solution was placed under vacuum and heated for 10–15 minutes. Heating was stopped and the oil allowed to cool prior to breaking the vacuum and filtering the solution to separate bleached oil and spent bleaching earth.

Bleached oil was further deodorized and steam bathed. During the cooling period a 0.5% citric acid solution was added.

Samples of seed, meal and oil were stored frozen for a maximum of 258 days and then analysed for residues of pydiflumetofen using the validated method GRM061.03A

Table 30 Pydiflumetofen residues in sunflower and processed commodities

Trial Identification (City, State/Region, Country, Year)	Variety	Commodity or Matrix	Total Rate (g ai/ha)	DALA	Pydiflumetofen Residues (Average), mg/kg	Processing Factor
TK0265867-02 (Aurora, SD, USA/2016)	Dove CL blend	Seed	2024	32	0.735, 0.364, 0.363 (0.487)	-
		Meal			<0.01, <0.01 (<0.01)	<0.02
		Oil (refined)			0.01, 0.01 (0.01)	0.02
TK0265867-03 (Northwood, ND, USA/2016)	Cobalt II	Seed	2012	29	0.555, 0.408, 0.458 (0.474)	-
		Meal			0.030, 0.028 (0.029)	0.06
		Oil (refined)			0.043, 0.042 (0.043)	0.09

Cotton

Two processing trials were conducted in conjunction with a residue field trial on cotton in the USA during the 2016 growing seasons (Lenz, 2018, Report No. TK0265622).

Pydiflumetofen, formulated as an SC formulation (containing 200 g ai/L) was applied twice at 623–643 g ai/ha, with application intervals of 7–10 days, totalling 1247–1277 g ai/ha. Each application was made in conjunction with either a non-ionic surfactant (NIS) or crop oil concentrate (COC) adjuvant.

Samples of cotton seed were collected 31–33 days after the last application (DALA). Seeds were processed into meal and oil using methods which are representative of commercial practice.

Seed cotton (70 kg) was cleaned with a stick extractor to remove the gin by-products (gin trash) and ginned to separate ginned cottonseed (undelinted cottonseed) and lint.

With approximately 11–15% remaining lint, the ginned cottonseed samples were mechanically delinted to remove most of the remaining lint and produce delinted cottonseed with approximately 3% lint remaining on the seed.

The resulting delinted cottonseed (31 kg) was cracked in a roller mill. Kernel and hull material were separated with a cleaner equipped with screens. The moisture content of the kernel was adjusted by adding water and mixing. Following mixing, samples were allowed to equilibrate for a minimum of 2 hours.

Kernel material was heated in a steam heated mixer to 80–100 °C and held for 28–32 minutes. After heating, the material was flaked in flaking roll. Flaked material was fed into a continuous extruder. As the material moved through the extruder, steam was injected directly on the product. Collets exiting the extruder were ground placed in stainless steel batch extractors and submerged in heated hexane. After 30 minutes, the miscella (hexane and crude oil) was drained. Heated hexane was added to the collets to repeat the cycle two more times.

After extraction, meal was toasted after which it was removed from the toaster, allowed to cool and then screened with a screen cleaner equipped with a sieve. Toasted cottonseed meal passing through the sieve was collected.

Miscella was passed through a laboratory vacuum evaporator to separate the crude oil and hexane. Crude oil was heated to remove any remaining hexane.

After the crude oil was filtered, the free fatty acid (FFA) content was determined. Based on the FFA content and amount of crude oil to be refined, weighed amounts of sodium hydroxide and crude oil were placed in a water bath and mixed. Neutralized oil was centrifuged to separate refined oil and soapstock. Refined oil was decanted from the soapstock and filtered. Refined oil was further bleached.

Refined oil was mixed and heated. An activated bleaching earth was added and the solution was placed under vacuum and heated for 10–15 minutes. Heating was stopped and the oil allowed to cool prior to breaking the vacuum and filtering the solution to separate bleached oil and spent bleaching earth. Bleached oil was further deodorized and steam bathed. During the cooling period a 0.5% citric acid solution was added.

Samples of seed, meal and oil were stored frozen for a maximum of 355 days and then analysed for residues of pydiflumetofen using the concurrently validated method GRM061.03A.

Table 31 Pydiflumetofen residues in cotton and processed commodities

Trial Identification (City, State/Region, Country, Year)	Variety	Commodity or Matrix	Total Rate (g ai/ha)	DALA	Pydiflumetofen Residues (Average), mg/kg	Processing Factor
TK0265622-03 (Proctor, AR, USA/2016)	ST 4946 GLB2	Seed	1247	33	0.882, 1.05, 0.860 (0.93)	-
		Meal			<0.01, <0.01 (<0.01)	<0.01
		Hull			0.131, 0.101 (0.116)	0.12
		Oil (refined)			0.018, 0.017 (0.018)	0.02
TK0265622-08 (Groom, TX, USA/2016)	PHY 222	Seed	1277	33	2.88, 2.76, 3.00 (2.88)	-
		Meal			<0.01, <0.01 (<0.01)	<0.01
		Hull			0.336, 0.427 (0.381)	0.13
		Oil (refined)			0.142, 0.108 (0.125)	0.04

APPRAISAL

Pydiflumetofen is a broad-spectrum fungicide belonging to the carboxamide group. It acts through inhibition of succinate dehydrogenase in complex II of fungal mitochondrial respiration.

Pydiflumetofen was first evaluated for toxicology and residues by the JMPR in 2018. An ADI of 0–0.1 mg/kg bw and an ARfD of 0.3 mg/kg bw were established. The residue definition for compliance with the MRL for plant and animal commodities and dietary risk assessment for plant commodities is pydiflumetofen. The residue definition for dietary risk assessment for animal commodities other than mammalian liver and kidney is the sum of pydiflumetofen and 2,4,6-TCP (2,4,6-Trichlorophenol) and its conjugates, expressed as pydiflumetofen and for dietary risk assessment for mammalian liver and kidney is the sum of pydiflumetofen, 2,4,6-trichlorophenol (2, 4, 6-TCP) and its conjugates and SYN547897 and its conjugates, expressed as pydiflumetofen. The residue is fat-soluble.

The 2018 JMPR noted that pydiflumetofen residues are very persistent in soil (up to 2380 days DT_{50}) and may be taken up by rotational crops.

At the Fifty-first Session of the CCPR, pydiflumetofen was scheduled for evaluation by the 2020 JMPR for several new uses, which was postponed to the 2021 Extra JMPR.

The Meeting received information from the manufacturer on use patterns, supervised residue trials on lemons, oranges, grapefruits, apples, pears, peaches, cherries, plums, blueberries, strawberries, bulb onions, green onions, cabbage, cauliflower, broccoli, beans with pods, peas with pods, beans without pods, peas without pods, sugar beet, carrots, radish, sorghum, almonds, pecans, sunflower and cottonseed and processing studies.

Methods of analysis

The LC-MS/MS analytical method (GRM061.03A) used for analysis of residues of pydiflumetofen in plant commodities, with n LOQ of 0.01 mg/kg, was reviewed by the 2018 JMPR. All samples collected from the supervised residue trials provided to the current Meeting were analysed using the same method. The methods are fit for purpose based on the recoveries from the concurrent method validation.

Stability of pesticide residues in stored analytical samples

The stability of residues of pydiflumetofen during frozen storage was evaluated by the 2018 JMPR. Pydiflumetofen was determined to be stable when stored frozen for at least 23 months at -20 ºC in commodities representative of the high water, high acid, high starch, high protein and high oil commodity groups. The periods of demonstrated stability adequately covered the frozen storage intervals of the samples in the supervised residue trials on crops considered by the current Meeting.

Results of supervised residue trials on crops

For maximum residue level estimation of pydiflumetofen residues in primary crop commodities, the addition of residues arising from direct treatment in combination with root uptake of pydiflumetofen from previous years, as determined at the 2019 Meeting, must be taken into account. The crop groups for plant food and feed established in the Codex Classification of Foods and Animal Feeds were used to give recommendations on the overall residue levels of pydiflumetofen expected in these commodities. As the seasonal application rates for the primary crop uses considered by the current Meeting are equivalent to or less than those considered at the 2019 Meeting, new scaling of the rotational crop residues is not required.

The Meeting noted that the use of statistical methods for the estimation of maximum residue levels is not possible when considering potential carryover of residues in succeeding crops.

The Meeting recognised that for permanent crops and crops cultivated in/on culture soil/medium and water, the contribution from carryover is not needed, as those crops are not expected to be subject to significant uptake of pydiflumetofen from the soil.

Citrus Fruits

The critical GAP for citrus fruits is from the USA; 4×85 g ai/ha, 7-day RTI, 0-day PHI. The Meeting received supervised residue trials conducted on lemon, mandarin, orange and grapefruit in the USA matching the critical GAP.

Pydiflumetofen residues in whole lemons in ranked order were (n = 6): 0.02, 0.14, 0.25, 0.29, 0.38 and 0.46 mg/kg.

Pydiflumetofen residues in whole mandarins in ranked order were (n = 4): 0.17, 0.24, 0.28 and 0.56 mg/kg.

Pydiflumetofen residues in whole oranges in ranked order were (n = 10): 0.11, 0.16, 0.17, 0.18, 0.21, 0.23, 0.31, 0.36, 0.40 and 0.68 mg/kg.

Pydiflumetofen residues in whole grapefruits in ranked order were (n = 7): 0.09, 0.12 (2), 0.13, 0.15, 0.16 and 0.58 mg/kg.

The Meeting noted that the GAP covers the group of citrus fruits and that median residues of lemons, mandarins, oranges and grapefruits are within a 5-fold difference. The Kruskal-Wallis H-test also determined that the datasets were from the same population. Therefore, the Meeting decided to combine the four datasets of lemons, mandarins, oranges and grapefruits.

Combined pydiflumetofen residues in lemons, mandarins, oranges and grapefruits were (n = 27): 0.02, 0.09, 0.11, 0.12 (2), 0.13, 0.14, 0.15, 0.16 (2), 0.17 (2), 0.18, 0.21, 0.23, 0.24, 0.25, 0.28, 0.29, 0.31, 0.36, 0.38, 0.40, 0.46, 0.56, 0.58 and 0.68 mg/kg (highest single value of 0.76 mg/kg).

Citrus fruits are cultivated as permanent crops and are not expected to be subject to significant uptake of pydiflumetofen from the soil. The Meeting estimated a maximum residue level of 0.9 mg/kg for the Group of citrus fruits.

For dietary risk assessment, the Meeting calculated a ratio of residues in flesh (0.042 mg/kg) to whole orange (0.179 mg/kg) and applied this ratio of 0.24 to all samples of citrus fruits, where combined pydiflumetofen residues were (n = 27): 0.01, 0.02, 0.03 (5), 0.04 (6), 0.06 (4), 0.07 (3), 0.09 (2), 0.10, 0.11, 0.13, 0.14 and 0.16 mg/kg, from which an HR of 0.16 and STMR of 0.05 mg/kg were estimated for citrus flesh. The Meeting also estimated an HR of 0.76 mg/kg and an STMR of 0.21 mg/kg for kumquats. An HR of 0.76 mg/kg and a median residue of 0.21 mg/kg were estimated for whole citrus fruits and used for estimation of HR-Ps and STMR-Ps for processed commodities.

Pome Fruits

The critical GAP for pome fruits is from the USA; 4×50 g ai/ha, 7-day RTI, 30-day PHI. The Meeting received supervised residue trials conducted on apple and pear in Canada and the USA matching the critical GAP.

Pydiflumetofen residues in apples in ranked order were (n = 14): 0.05 (2), 0.06 (5), 0.07, 0.08 (2), 0.09, 0.10 (2) and 0.11 mg/kg (highest value of 0.13 mg/kg from replicate samples).

Pydiflumetofen residues in pears in ranked order were (n = 11): 0.02 (3), 0.04, 0.05 (3), 0.07, 0.08, 0.09 and 0.12 mg/kg (highest value of 0.13 mg/kg from replicate samples).

The Meeting noted that the GAP covers the group of pome fruits except persimmon, Japanese and that median residues in apples and pears are within a 5-fold difference. The Mann-Whitney U-test also determined that the datasets were from the same population. Therefore, the Meeting decided to combine the two datasets of apples and pears.

Combined pydiflumetofen residues in apples and pears were (n = 25): 0.02 (3), 0.04, 0.05 (5), 0.06 (5), 0.07 (2), 0.08 (3), 0.09 (2), 0.10 (2), 0.11 and 0.12 mg/kg.

Pome fruits are cultivated as permanent crops and are not expected to be subject to a potential uptake of pydiflumetofen from the soil. The Meeting estimated a maximum residue level of 0.2 mg/kg, an HR of 0.13 mg/kg and an STMR of 0.06 mg/kg for the Group of pome fruits, except persimmon, Japanese.

Stone fruits

The critical GAP for stone fruits is from the USA; 4×75 g ai/ha, 7-day RTI, 0-day PHI. The Meeting received supervised residue trials on peaches, cherries and plums conducted in Canada and the USA matching the critical GAP.

Peaches

Pydiflumetofen residues in peaches in ranked order were (n = 12): 0.09, 0.16, 0.19, 0.20 (2), 0.21 (2), 0.22 (2), 0.25, 0.29 and 0.78 mg/kg (highest value of 0.80 mg/kg from replicate samples).

Residue levels in the field trials from Canada and the USA were reported as fruit without stone. At the 2017 Meeting, it was concluded that the weight ratio of flesh to whole fruit for peaches ranged between 0.85 and 0.96 and that correcting the residue levels for this weight/weight ratio would lead to the same maximum residue level.

Peaches are cultivated as permanent crops and are not expected to be subject to a potential uptake of pydiflumetofen from the soil. The Meeting estimated a maximum residue level of 1 mg/kg, an HR of 0.80 mg/kg and an STMR value of 0.21 mg/kg for the Subgroup of peaches.

Cherries

Pydiflumetofen residues in cherries in ranked order were (n = 14): 0.14, 0.18, 0.18, 0.21, 0.24, 0.26, 0.37, 0.42, 0.44, 0.53, 0.61, 0.72, 0.90 and 1.6 mg/kg (highest value of 1.7 mg/kg from replicate samples).

Residue levels in the field trials from Canada and the USA were reported as fruit without stone. At the 2017 Meeting, it was concluded that, based on the cherry trials, the contribution of the pit to the weight of the whole fruit is approximately 10%. Correcting the residue levels using this weight/weight ratio would lead to the same maximum residue level.

Cherries are cultivated as permanent crops and are not expected to be subject to a potential uptake of pydiflumetofen from the soil. The Meeting estimated a maximum residue level of 2.0 mg/kg, an HR of 1.7 mg/kg and an STMR of 0.395 mg/kg for the Subgroup of cherries.

Plums

Pydiflumetofen residues in plums in ranked order were (n = 9): 0.06, 0.11, 0.12, 0.13, 0.15, 0.16, 0.21, 0.32 and 0.35 mg/kg (highest value of 0.37 mg/kg from replicate samples).

Residue levels in the field trials from Canada and the USA were reported as fruit without stone. At the 2017 Meeting, it was concluded that, the weight ratio of flesh to whole fruit for plums ranged between 0.86 and 0.97 and that correcting the residue levels for this weight/weight ratio would lead to the same maximum residue level.

Plums are cultivated as permanent crops and are not expected to be subject to a potential uptake of pydiflumetofen from the soil. The Meeting estimated a maximum residue level of 0.6 mg/kg, an HR of 0.37 mg/kg and an STMR value of 0.15 mg/kg for the Subgroup of plums.

Bushberries

The critical GAP for the bushberry crop subgroup is from the USA; 2×150 g ai/ha, 7-day RTI, 0-day PHI. The Meeting received supervised residue trials on highbush blueberries conducted in Canada and the USA and approximating the critical GAP.

Pydiflumetofen residues in highbush blueberries in ranked order were (n = 10): 0.40, 0.66 (2), 0.69, 0.86, 0.90, 0.96, 1.4, 1.9 and 3.6 mg/kg (highest value of 3.9 mg/kg from replicate samples).

Bushberries are cultivated as permanent crops and are not expected to be subject to a potential uptake of pydiflumetofen from the soil. Noting that blueberries is the representative crop of the subgroup bush berries, the Meeting estimated a maximum residue level of 5 mg/kg, an HR of 3.9 mg/kg and an STMR value of 0.88 mg/kg for the Subgroup of bush berries and extrapolated these values to elderberries.

Low growing berries

In field studies on succeeding crops, reviewed by the 2019 Meeting, the scaled highest residue in fruits (strawberries) was < 0.02 mg/kg.

The current Meeting received supervised residue trials conducted in Canada and the USA on strawberries matching the critical GAP from the USA, for the low growing berry, except cranberry, subgroup; 2×150 g ai/ha, 7-day RTI, 0-day PHI.

Pydiflumetofen residues in strawberries in ranked order were (n = 10): 0.08, 0.09, 0.10, 0.16, 0.18, 0.19, 0.30, 0.44, 0.46 and 0.56 mg/kg (highest value of 0.62 mg/kg from replicate samples).

The Meeting concluded that residues from uptake of pydiflumetofen via the roots are insignificant in comparison to residue levels following direct treatment. The Meeting estimated a maximum residue level of 1 mg/kg, an HR of 0.62 mg/kg and an STMR of 0.185 mg/kg for the Subgroup of low growing berries, except cranberries.

Bulb vegetables

The current Meeting received supervised residue trials conducted in the USA on bulb vegetables matching the critical GAP from the USA for bulb vegetables; 3×125 g ai/ha, 7-day RTI, 7-day PHI.

Bulb onions

Pydiflumetofen residues in bulb onions in ranked order were (n = 8): < 0.01 (2), 0.01, 0.05 (2), 0.06 (2) and 0.12 mg/kg (highest value of 0.13 mg/kg from replicate samples).

Bulb onions may be subject to crop rotation, however, no residue data on suitable representative rotational crops of bulb or stem vegetables were provided to the 2019 Meeting. Based on the consistent range of residues across rotational crop commodities and that the edible portion of bulb onion is below ground, the Meeting agreed to use residues from a root crop to estimate the uptake of pydiflumetofen via the roots. Therefore, the Meeting decided to extrapolate the mean, median and highest residues in the succeeding crop of radish roots of 0.02, 0.02 and 0.07 mg/kg, respectively, to bulb onions.

The Meeting estimated a maximum residue level of 0.3 mg/kg, an HR of 0.20 mg/kg (scaled highest residue found in succeeding crop (radish root) of 0.07 mg/kg added to the highest residue in bulb onions of 0.13 mg/kg obtained from supervised field trials) and an STMR of 0.07 mg/kg (scaled mean residue found in succeeding crop (radish root) of 0.02 mg/kg added to the median residue in bulb onion of 0.05 mg/kg obtained from supervised field trials) for the Subgroup of bulb onions.

Green onions

Pydiflumetofen residues in green onions in ranked order were (n = 4): 0.28, 0.30, 0.36 and 1.1 mg/kg (highest value of 1.3 mg/kg from replicate samples).

Green onions may be subject to crop rotation, however, no residue data on suitable representative rotational crops of bulb or stem vegetables were provided to the 2019 Meeting. Based on the consistent range of residues across rotational crop commodities and that the edible portion of green onion includes the aerial portion of the plant, the Meeting agreed to use residues from a leafy crop to estimate the uptake of pydiflumetofen via the roots. Therefore, the Meeting decided to extrapolate the mean, median and highest residues in the succeeding crop of mature spinach of 0.03, 0.02 and 0.09 mg/kg, respectively, to green onions.

The Meeting estimated a maximum residue level of 1.5 mg/kg, an HR of 1.39 mg/kg (scaled highest residue found in succeeding crop (mature spinach) of 0.09 mg/kg added to the highest residue in green onions of 1.3 mg/kg obtained from supervised field trials) and an STMR of 0.36 mg/kg (scaled mean residue found in succeeding crop (mature spinach) of 0.03 mg/kg added to the median residue in bulb onion of 0.33 mg/kg obtained from supervised field trials) for the Subgroup of green onions.

Brassica vegetables

In field studies on succeeding crops, reviewed by the 2019 Meeting, the scaled mean, median and highest residues in leafy vegetables and Brassicas (mature spinach) were 0.03, 0.02 and 0.09 mg/kg, respectively. Using this data, the Meeting recommended a maximum residue level of 0.1 mg/kg for the Group of Brassica vegetables (except Brassica leafy vegetables).

The current Meeting received supervised residue trials conducted in the USA on Brassica vegetables matching the critical GAP for Brassica vegetables from the USA; 3×125 g ai/ha, 7-day RTI, 0-day PHI.

Flowerhead Brassicas

Pydiflumetofen residues in broccoli and cauliflower in ranked order were (n = 8): 0.04, 0.05, 0.31, 0.36, 0.42, 0.67, 0.94 and 1.4 mg/kg (highest value of 1.5 mg/kg from replicate samples).

The Meeting estimated a maximum residue level of 3.0 mg/kg, an HR of 1.5 mg/kg and an STMR of 0.39 mg/kg for the Subgroup of flowerhead Brassicas.

Head Brassicas

Pydiflumetofen residues in cabbage with wrapper leaves in ranked order were (n = 6): 0.30, 0.33, 0.36, 0.88, 0.95 and 0.99 mg/kg.

Pydiflumetofen residues in cabbage without wrapper leaves in ranked order were (n = 6): < 0.01, 0.02, 0.03, 0.04, 0.05 and 0.12 mg/kg (highest value of 0.13 mg/kg from replicate samples).

The Meeting concluded that for estimating a maximum residue level and for calculating the animal dietary burden, residues from uptake of pydiflumetofen via the roots of head Brassicas are insignificant in comparison to residue levels following direct treatment. However, for dietary risk assessment, the Meeting agreed to take into account the residues from rotational crops since the contribution is more significant in comparison to residues resulting from direct treatment in cabbage without wrapper leaves.

The Meeting estimated a maximum residue level of 2.0 mg/kg, an HR of 0.22 mg/kg (scaled highest residue found in succeeding crop (spinach) 0.09 mg/kg added to the highest residue in cabbage without wrapper leaves of 0.13 mg/kg obtained from supervised field trials) and an STMR of 0.065 mg/kg (scaled

mean residue found in succeeding crop (spinach) 0.03 mg/kg added to the median residue in cabbage without wrapper leaves of 0.035 mg/kg obtained from supervised field trials) for the Subgroup of head Brassicas.

The Meeting estimated a highest residue and a median residue for animal dietary burden calculation of 0.99 and 0.62 mg/kg, respectively, for cabbage leaves.

Stem Brassicas

Although pydiflumetofen is not registered for use on the Subgroup of stem Brassicas, these crops may still be subject to crop rotation and therefore contain pydiflumetofen residues after uptake via the roots. The Meeting estimated a maximum residue level of 0.1 mg/kg for the Subgroup of stem Brassicas, an HR of 0.09 mg/kg and an STMR of 0.02 mg/kg and withdrew its previous its previous recommendation for the Group of Brassica vegetables (except Brassica leafy vegetables).

Legume vegetables

In field studies on succeeding crops, reviewed by the 2019 Meeting, the scaled mean, median and highest residues in beans fresh seeds were each < 0.02 mg/kg. Using this data, the Meeting recommended a maximum residue level of 0.02 mg/kg for the Group of legume vegetables.

The current Meeting received a critical GAP for legume vegetables from the USA; 2×200 g ai/ha, 14-day RTI, 14-day PHI. The legume vegetable field trials from Canada and the USA (2×200 g ai/ha, 7-day RTI, 14-day PHI) differed from the critical GAP with regard to the RTI. Comparison of the application scenarios of the trials with the critical GAP, showed that the expected residues are within 20% of the critical GAP. Therefore, the Meeting concluded that the supervised residue trials could be used for estimation of the maximum residue level.

Beans with pods

In trials approximating the critical GAP, pydiflumetofen residues in beans with pods in ranked order were (n = 10): 0.01 (2), 0.02 (2), 0.03, 0.06, 0.08, 0.14, 0.26 and 0.43 mg/kg (highest value of 0.47 mg/kg from replicate samples).

The Meeting concluded that residues from uptake of pydiflumetofen via the roots are insignificant in comparison to residue levels following direct treatment.

The Meeting estimated a maximum residue level of 0.7 mg/kg, an HR of 0.47 mg/kg and an STMR of 0.045 mg/kg for the Subgroup of beans with pods.

Peas with pods

In trials approximating the critical GAP, pydiflumetofen residues in peas with pods in ranked order were (n = 7): 0.01 (2), 0.05, 0.12, 0.16 and 0.64 (2) mg/kg (highest value of 0.84 mg/kg from replicate samples).

The Meeting concluded that residues from uptake of pydiflumetofen via the roots are insignificant in comparison to residue levels following direct treatment.

The Meeting estimated a maximum residue level of 1.5 mg/kg, an HR of 0.84 mg/kg and an STMR of 0.12 mg/kg for the Subgroup of peas with pods.

Succulent beans without pods

In trials approximating the critical GAP, pydiflumetofen residues in beans without pods in ranked order were (n = 9): 0.010 (2), 0.011, 0.013(2), 0.017, 0.018, 0.034 and 0.065 mg/kg (highest value of 0.079 mg/kg from replicate samples).

The Meeting concluded that residues in beans without pods may be influenced significantly by uptake of pydiflumetofen from the soil. For the estimation of a maximum residue level, HR and STMR, all scaled residues were < 0.02 mg/kg in beans fresh seeds from the succeeding crop field trials. Therefore, a value of 0.02 mg/kg was added to the median residue obtained from supervised field trials on beans without pods of 0.013 mg/kg for an overall STMR for beans without pods of 0.033 mg/kg. For the estimation of a maximum residue level, the value of 0.02 mg/kg was added to the highest residue of 0.079 mg/kg found in supervised field trials, resulting in an overall highest residue in succulent beans without pods of 0.099 mg/kg.

The Meeting estimated a maximum residue level of 0.15 mg/kg, an HR of 0.099 mg/kg and an STMR of 0.033 mg/kg for the Subgroup of succulent beans without pods.

Succulent peas without pods

In trials approximating the critical GAP, pydiflumetofen residues in peas without pods in ranked order were (n = 10): < 0.01 (5), 0.011, 0.012, 0.013 (2) and 0.018 mg/kg (highest value of 0.022 mg/kg from replicate samples).

The Meeting concluded that residues in peas without pods may be influenced significantly by uptake of pydiflumetofen from the soil. For the estimation of a maximum residue level, HR and STMR, all scaled residues were < 0.02 mg/kg in beans fresh seeds from the succeeding crop field trials. Therefore, a value of 0.02 mg/kg was added to the median residue obtained from supervised field trials on peas without pods of 0.011 mg/kg for an overall STMR for peas without pods of 0.031 mg/kg. For the estimation of a maximum residue level, the value of 0.02 mg/kg was added to the highest residue of 0.022 mg/kg found in supervised field trials, resulting in an overall highest residue in succulent peas without pods of 0.042 mg/kg.

The Meeting estimated a maximum residue level of 0.05 mg/kg, an HR of 0.042 mg/kg and an STMR of 0.031 mg/kg for the Subgroup of succulent peas without pods.

Underground immature beans and peas

Although pydiflumetofen is not registered for use on the Subgroup of underground immature beans and peas, these crops may still be subject to crop rotation and therefore contain pydiflumetofen residues after uptake via the roots. The Meeting estimated a maximum residue level of 0.02 mg/kg, an HR of 0.02 mg/kg and an STMR of 0.02 mg/kg for the Subgroup of underground immature beans and peas and withdrew its previous recommendation for the Group of legume vegetables.

Root vegetables

In field studies on succeeding crops, reviewed by the 2019 Meeting, the scaled mean, median and highest residues in root and tuber vegetables (radish) were 0.02, 0.02 and 0.07 mg/kg. Using this data, the previous Meeting estimated a maximum residue level of 0.1 mg/kg for the Subgroup of root vegetables.

The current Meeting received supervised residue trials conducted in Canada and the USA on root vegetables matching the critical GAP for root vegetables from the USA; 4×75 g ai/ha, 7-day RTI, 7-day PHI.

Pydiflumetofen residues in sugar beets in ranked order were (n = 17): 0.02 (2), 0.04 (2), 0.05, 0.06 (5), 0.08, 0.09, 0.10 (2), 0.12, 0.13 and 0.14 mg/kg (highest value of 0.15 mg/kg from replicate samples).

Pydiflumetofen residues in carrots in ranked order were (n = 6): 0.02, 0.03, 0.06, 0.07, 0.08 and 0.10 mg/kg (highest value of 0.11 mg/kg from replicate samples).

Pydiflumetofen residues in radish roots in ranked order were (n = 5): 0.01, 0.02, 0.14 and 0.17 (2) mg/kg (highest value of 0.18 mg/kg from replicate samples).

The Meeting noted that the GAP covers the Subgroup of root vegetables and that median residues of sugar beet roots, carrots and radish roots are within a 5-fold difference. The Kruskal Wallis H-test also determined that the datasets were from the same population. Therefore, the Meeting decided to combine the three datasets of sugar beet roots, carrots and radish roots.

Combined pydiflumetofen residues in sugar beet roots, carrots and radish roots were (n = 28): 0.01, 0.02 (4), 0.03, 0.04 (2), 0.05, 0.06 (6), 0.07, 0.08 (2), 0.09, 0.10 (3), 0.12, 0.13, 0.14 (2) and 0.17 (2) mg/kg.

The Meeting concluded that residues in sugar beet roots, carrots and radish roots may be influenced significantly by uptake of pydiflumetofen from the soil. The Meeting decided to add the scaled mean residue found in field studies on succeeding crops (radish) of 0.02 mg/kg to the median residue obtained from supervised field trials on sugar beet roots, carrots and radish roots of 0.06 mg/kg for an overall STMR for sugar beet roots, carrots and radish roots of 0.08 mg/kg. For the estimation of a maximum residue level, the highest residue of 0.07 mg/kg found in radish roots from the succeeding crop field trials was added to the highest residue of 0.18 mg/kg found in supervised field trials, resulting in an overall highest residue in root vegetables of 0.25 mg/kg.

The Meeting estimated a maximum residue level of 0.3 mg/kg, an HR value of 0.25 mg/kg and an STMR value of 0.08 mg/kg for the Subgroup of root vegetables to replace its previous recommendation.

Sorghum

In field studies on succeeding crops, reviewed by the 2019 Meeting, the scaled mean, median and highest residues in cereals (wheat) were < 0.03, < 0.03, < 0.03 mg/kg, respectively. Using this data, the Meeting recommended a maximum residue level of 0.03 mg/kg for the Subgroup of sorghum grain and millet.

The current Meeting received supervised residue trials conducted in the USA on sorghum matching the critical GAP for sorghum from the USA; 2×126 g ai/ha, 7-day RTI, 21-day PHI.

Pydiflumetofen residues in sorghum grain in ranked order were (n = 12): 0.11 (3), 0.30, 0.44, 0.45, 0.58, 0.64 (2), 0.70, 1.1 and 1.8 mg/kg.

The Meeting concluded that residues from uptake of pydiflumetofen via the roots are insignificant in comparison to residue levels following direct treatment.

The Meeting estimated a maximum residue level of 3 mg/kg and an STMR of 0.515 mg/kg for grain sorghum. As the registered use is only for grain sorghum, the Meeting estimated a maximum residue level of 0.03 mg/kg and STMR of 0.03 mg/kg for the Subgroup of sorghum grain and millet, except grain sorghum and withdrew its previous recommendation for the Subgroup of sorghum grain and millet.

Tree nuts

The critical GAP for tree nuts is from the USA; 3×100 g ai/ha, 7-day RTI, 14-day PHI. The Meeting received supervised residue trials conducted in the USA on almond and pecan approximating the critical GAP.

Pydiflumetofen residues in almonds in ranked order were (n = 5): < 0.01 (3) and 0.03 (2) mg/kg.

Pydiflumetofen residues in pecans in ranked order were (n = 5): < 0.01 (4) and 0.02 mg/kg.

The Meeting noted that the GAP covers the group of tree nuts and that median residues of almonds and pecans are within a 5-fold difference. Therefore, the Meeting decided to combine the two datasets of almonds and pecans.

Combined pydiflumetofen residues in almonds and pecans were (n = 10): < 0.01 (7), 0.02 and 0.03 (2) mg/kg.

Tree nuts are cultivated as permanent crops and are not expected to be subject to a potential uptake of pydiflumetofen from the soil. The Meeting estimated a maximum residue level of 0.05 mg/kg, an HR of 0.03 mg/kg and an STMR of 0.01 mg/kg for the Group of tree nuts.

Subgroups of sunflower seeds and cotton seeds

In field studies on succeeding crops, reviewed by the 2019 Meeting, the scaled mean, median and highest residues in wheat straw (worse case) were 0.10, 0.08 and 0.28 mg/kg, respectively. Using this data, the Meeting recommended a maximum residue level of 0.3 mg/kg for the Subgroups of sunflower seeds and cottonseed.

Sunflower seeds

The current Meeting received supervised residue trials conducted in Canada and the USA on sunflower seed matching the critical GAP for the sunflower seed subgroup from the USA; 2×200 g ai/ha, 14-day RTI, 30-day PHI.

Pydiflumetofen residues in sunflower seeds in ranked order were (n = 11): 0.02 (3), 0.05, 0.07 (2), 0.08, 0.12, 0.13 (2) and 0.37 mg/kg (highest value of 0.41 mg/kg from replicate samples).

The Meeting concluded that residues in sunflower seeds may be influenced by uptake of pydiflumetofen from the soil. Following a re-assessment of the field studies on succeeding crops, the Meeting concluded that the soya bean seed data was most relevant to oilseeds, where all scaled residues were < 0.02 mg/kg in soya bean seeds. Therefore, for the estimation of a maximum residue level, the value of 0.02 mg/kg was added to the highest residue of 0.41 mg/kg found in supervised field trials, resulting in an overall highest residue in sunflower seeds of 0.43 mg/kg. For the estimation of the STMR, a value of 0.02 mg/kg was added to the median residue of 0.07 mg/kg for an overall STMR for sunflower seeds of 0.09 mg/kg.

Noting that the GAP covers the sunflower seed subgroup, the Meeting estimated a maximum residue level of 0.5 mg/kg and an STMR of 0.09 mg/kg for the Subgroup of sunflower seeds to replace its previous recommendation.

Cotton seeds

The critical GAP for cotton seed is from the USA; 3×75 g ai/ha, 10-day RTI, 45-day PHI. None of the cotton seed supervised residue trials were conducted according to the US GAP.

Although the Meeting could not estimate a maximum residue level on cotton seeds to account for primary use of pydiflumetofen, cotton seeds may still be subject to crop rotation and therefore contain pydiflumetofen residues after uptake via the roots. Similar to sunflower seeds, the Meeting concluded that the secondary crop field studies on soya bean seeds were most relevant to oilseeds, where all scaled pydiflumetofen residues were < 0.02 mg/kg. Therefore, the Meeting estimated a maximum residue level of 0.02 mg/kg and an STMR of 0.02 mg/kg for cottonseed to replace its previous recommendation.

Residues in animal feeds

Leaves of root vegetables

In field studies on succeeding crops, reviewed by the 2019 JMPR, the scaled mean, median and highest residues in radish tops were 0.02, 0.02, 0.05 mg/kg, respectively. Using this data, the 2019 JMPR recommended a maximum residue level of 0.07 mg/kg for the Subgroup of Leaves of root and tuber vegetables, except leaves of tuber vegetables.

The US GAP prohibits the feeding of radish tops to livestock, therefore, only sugar beet tops were considered as animal feed. In trials matching the critical GAP, pydiflumetofen residues in sugar beet tops in ranked order were (n = 17); 0.76, 0.77, 0.92, 0.97, 1.0 (2), 1.4 (2), 1.5 (2), 1.6, 1.7, 3.4, 3.6, 5.3, 5.7 and 6.3 mg/kg (highest value of 8.0 mg/kg from replicate samples).

The Meeting concluded that residues from uptake of pydiflumetofen via the roots are insignificant in comparison to residue levels following direct treatment.

The Meeting estimated a maximum residue level of 40 mg/kg (dw; using the OECD standard dry matter content of 23%), a highest residue of 8.0 mg/kg and a median residue of 1.5 mg/kg for sugar beet tops (as received) and withdraws its previous recommendation for the subgroup.

Sorghum forage

In field studies on succeeding crops, reviewed by the 2019 Meeting, the scaled mean, median and highest residues in cereal forages (wheat forage) were 0.03, 0.03, 0.03 mg/kg, respectively.

In trials matching the critical GAP, pydiflumetofen residues in sorghum forage in ranked order were (n = 12): 0.05, 0.09 (2), 0.14 (2), 0.18, 0.28, 0.45 (2), 0.53, 0.61 and 0.70 mg/kg (highest value of 0.73 mg/kg from replicate samples).

The Meeting concluded that residues from uptake of pydiflumetofen via the roots are insignificant in comparison to residue levels following direct treatment.

The Meeting estimated a median residue value of 0.23 mg/kg and a highest residue value of 0.73 mg/kg for sorghum forage (as received).

Sorghum stover

In field studies on succeeding crops, reviewed by the 2019 Meeting, the scaled mean, median and highest residues in cereal straw (wheat straw) were 0.10, 0.08, 0.28 mg/kg, respectively. Using this data, the 2019 Meeting recommended a maximum residue level of 0.3 mg/kg (dry weight) for sorghum straw and fodder, dry.

In trials matching the critical GAP, pydiflumetofen residues in sorghum stover (dry weight) in ranked order were (n = 12): 0.07, 0.15, 0.19, 0.21, 0.25, 0.38, 0.62, 0.66, 1.2, 3.4, 5.0 and 6.0 mg/kg (highest value of 7.6 mg/kg from replicate samples).

The Meeting concluded that residues from uptake of pydiflumetofen via the roots are insignificant in comparison to residue levels following direct treatment.

The Meeting estimated a maximum residue level of 10 mg/kg (dry weight), a highest residue value of 7.6 mg/kg and a median residue value of 0.50 mg/kg for sorghum straw and fodder, dry to replace its previous recommendation.

Almond hulls

In trials matching the critical GAP, pydiflumetofen residues in almond hulls in ranked order were (n = 5): 0.37, 1.4, 1.6, 3.6 and 3.9 mg/kg.

The Meeting estimated a maximum residue level of 10 mg/kg (dw; using the OECD standard dry matter content of 90%) and a median residue value of 1.6 mg/kg (as received) for almond hulls.

Cotton gin by-products

None of the cotton seed supervised residue trials were conducted according to the critical GAP from the USA; 3×75 g ai/ha, 10-day RTI, 45-day PHI.

Therefore, the Meeting did not estimate a maximum residue level for cotton gin by-products.

Fate of residues during processing

Processing data on oranges, apples, plums, sugar beets, grain sorghum, almonds, sunflower and cotton were provided. All data relevant for an estimation of maximum residue levels in processed commodities or for dietary exposure calculations are summarized in the following table.

Table 1 Processing Factors and STMR-P/HR-P

RAC [STMR/HR]	Processed commodity	Individual processing factors	Mean or best estimate processing factor	STMR-P = $STMR_{RAC}$ × PF (mg/kg)	HR-P = HR_{RAC} × PF (mg/kg)
Citrus [0.21 mg/kg / 0.76 mg/kg]	Peel	1.78, 1.94	1.86	0.39	1.4
	Juice	< 0.02, < 0.02	< 0.02	0.004	
	Oil	33.7, 52.7	43.2	9.1	
	Dried pulp	1.45	1.45	0.30	
Apple [0.06 mg/kg / 0.13 mg/kg]	Canned apple	0.03	0.03	0.002	
	Apple juice	0.06	0.06	0.004	
	Apple sauce	0.06	0.06	0.004	
	Dried apple	0.41	0.41	0.02	0.05
	Apple wet pomace	3.77	3.77	0.22	
Pear [0.05 mg/kg / 0.13 mg/kg]	Canned fruit	0.09	0.09	0.004	
	Dried fruit	0.62	0.62	0.03	0.08
	Juice	0.11	0.11	0.006	
	Wet pomace	3.0	3.0	0.15	
Plum [0.15 mg/kg / 0.37 mg/kg]	Juice	0.01, 0.02	0.02	0.003	
	Puree	0.15, 0.27	0.21	0.03	
	Prune, dried	1.8, 2.8	2.3	0.34	0.85
Sugar beet [0.06 mg/kg / 0.15 mg/kg]	Refined sugar	< 0.07, 0.07	0.07	0.004	
	Molasses	0.50, 0.68	0.59	0.04	
	Dried pulp	1.8, 6.0	3.9	0.24	
Grain sorghum [0.515 mg/kg / 1.84 mg/kg]	Flour	0.82, 0.91	0.87	0.45	
	Aspirated grain fraction	61, 63	62	31.9	
Almond [0.01 mg/kg / 0.03 mg/kg]	Roasted	0.37	0.37	0.004	0.01
	Oil	0.34	0.34	0.003	

RAC [STMR/HR]	Processed commodity	Individual processing factors	Mean or best estimate processing factor	STMR-P = $STMR_{RAC}$ × PF (mg/kg)	HR-P = HR_{RAC} × PF (mg/kg)
Sunflower [0.09 mg/kg / 0.43 mg/kg]	Oil (refined)	0.02, 0.09	0.06	0.005	
	Meal	< 0.02, 0.06	0.04	0.004	
Cotton [0.02 mg/kg / 0.02 mg/kg]	Oil (refined)	0.02, 0.04	0.03	0.0006	
	Hulls	0.12, 0.13	0.13	0.003	
	Meal	< 0.01, < 0.01	< 0.01	0.0002	

Using the estimated maximum residue level of 0.9 mg/kg for the group of citrus fruits and applying the processing factors of 43.2 for citrus oil and 1.45 for citrus pulp, dry, the Meeting estimated maximum residue levels of 40 mg/kg for citrus oil and 1.5 mg/kg for citrus pulp, dry.

Using the estimated maximum residue level of 0.6 mg/kg for the subgroup of plums and applying the processing factor of 2.3 for dried prune plums, the Meeting estimated a maximum residue level of 1.5 mg/kg for prunes dried.

Residues in animal commodities

Farm animal feeding studies

Farm animal feeding studies (lactating dairy cow and laying hen) are reported in the evaluation of the 2018 JMPR.

Farm animal dietary burden

The dietary burdens for beef cattle, dairy cattle, broilers and laying poultry determined at the 2019 Meeting were updated with the feed items listed in the table below.

Table 2 Potential feed items

Codex Classification	Commodity	Median residue (-P) mg/kg	Highest residue (-P) mg/kg
Orange	Dried pulp	0.30	
Apple	Wet pomace	0.22	
Brassica Head Vegetables	Cabbage head with wrapper leaves	0.62	1.2
Root vegetables	Carrot culls, swede roots, turnip roots	0.08	0.24
Sugar beet	Molasses	0.04	
	Dried pulp	0.24	
Leaves of root vegetables	Sugar beet tops	1.5	8.0
Grain sorghum	Grain	0.515	
	Aspirated grain fractions	31.9	
	Forage	0.23	0.73
	Stover	0.50	7.6
Almonds	Hulls	1.6	

Codex Classification	Commodity	Median residue (-P) mg/kg	Highest residue (-P) mg/kg
Sunflower	Meal	0.004	

The dietary burdens, estimated using the most recent version of the OECD livestock dietary burden calculator, are presented in Annex 6 of the Report and summarized below.

Table 3 Estimated maximum and mean dietary burdens of farm animals

	Animal Dietary Burden: Pydiflumetofen, ppm of dry matter diet							
	US-Canada		EU		Australia		Japan	
	Max	Mean	Max	Mean	Max	Mean	Max	Mean
Beef cattle	12	5.6	31	9.0	45[a]	19[b]	1.4	1.0
Dairy cattle	28	9.5	31	10	45[c]	13[d]	13	4.7
Poultry - broiler	0.56	0.56	0.76	0.59	0.51	0.51	0.42	0.42
Poultry - layer	0.56	0.56	8.7[e]	3.0[f]	0.51	0.51	0.46	0.46

[a] Highest maximum beef cattle dietary burden suitable for maximum residue level estimates for mammalian tissues

[b] Highest mean beef cattle dietary burden suitable for STMR estimates for mammalian tissues

[c] Highest maximum dairy cattle dietary burden suitable for MRL estimate for milk

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

[e] Highest maximum poultry dietary burden suitable for MRL estimates for poultry tissues and eggs

[f] Highest mean poultry dietary burden suitable for STMR estimates for poultry tissues and eggs

Animal commodity maximum residue levels

Cattle residues in tissues and milk at the expected dietary burden for dairy cattle are shown in the Table below.

Table 4 Anticipated residues of pydiflumetofen in mammalian commodities

	Feed level (ppm) for milk residues	Total residues in milk (mg/kg)	Feed level (ppm) for tissue residues	Total residues [a] (mg/kg)			
				Muscle	Liver	Kidney	Fat
MRL Determination							
Feeding study	45	< 0.01	45	< 0.01	0.05	< 0.01	0.05
Dietary burden	45	< 0.01	45	< 0.01	0.05	< 0.01	0.05
HR Determination							
Feeding study			45	< 0.02	0.44	0.30	0.07
Dietary burden			45	< 0.02	0.44	0.30	0.07
STMR Determination							
Feeding study	15	< 0.02	15	< 0.02	0.06	0.07	0.02
			45	< 0.02	0.29	0.25	0.06
Dietary burden	13	< 0.02	19	< 0.02	0.09	0.09	0.02

[a] For MRL determination: pydiflumetofen; For HR and STMR determination for milk, muscle and fat: sum of pydiflumetofen and 2,4,6-TCP and its conjugates, expressed as pydiflumetofen; For HR and STMR determination for liver and kidney: sum of pydiflumetofen, 2,4,6-TCP and its conjugates and SYN547897 and its conjugates, expressed as pydiflumetofen

Based on pydiflumetofen residues in milk and cattle tissues, the Meeting confirmed the maximum residue levels estimated by the 2019 Meeting (0.01 (*) mg/kg in milk, 0.1 mg/kg in mammalian meat (in the fat), mammalian edible offal and mammalian fat).

Based on the highest estimated total residues of pydiflumetofen and 2,4,6-TCP expressed as pydiflumetofen in muscle and fat, the Meeting estimated HR values of 0.02 mg/kg in mammalian meat and 0.07 mg/kg in mammalian fat.

Based on the highest estimated total residues of pydiflumetofen, 2,4,6-TCP and SYN547897 expressed as pydiflumetofen in liver and kidney, the Meeting estimated an HR value of 0.44 mg/kg in liver and 0.30 mg/kg in kidney.

Based on the mean estimated total residues of pydiflumetofen and 2,4,6-TCP expressed as pydiflumetofen in milk, muscle and fat, the Meeting estimated STMR values of 0.02 mg/kg in milk, 0.02 mg/kg in mammalian meat and 0.02 mg/kg in mammalian fat.

Based on the mean estimated total residues of pydiflumetofen, 2,4,6-TCP and SYN547897 expressed as pydiflumetofen in liver and kidney, the Meeting estimated STMR values of 0.09 mg/kg for each.

Table 5 Anticipated residues of pydiflumetofen in poultry commodities

	Feed level (ppm) for egg residues	Total residues in egg (mg/kg)	Feed level (ppm) for tissue residues	Total residues (mg/kg)		
				Muscle	Liver	Fat
MRL Determination - Pydiflumetofen						
Feeding study	9	0.011	9	< 0.01	< 0.01	< 0.01
Dietary burden	8.7	0.011	8.7	< 0.01	< 0.01	< 0.01
HR Determination - Sum of pydiflumetofen and 2,4,6-TCP and its conjugates, expressed as pydiflumetofen						
Feeding study	9	0.023	9	< 0.02	< 0.02	< 0.02
Dietary burden	8.7	0.023	8.7	< 0.02	< 0.02	< 0.02
STMR Determination - Sum of pydiflumetofen and 2,4,6-TCP and its conjugates, expressed as pydiflumetofen						
Feeding study	3	< 0.02	3	< 0.02	< 0.02	< 0.02
Dietary burden	3.0	< 0.02	3.0	< 0.02	< 0.02	< 0.02

Based on pydiflumetofen residues in eggs and poultry tissues, the Meeting confirmed the maximum residue levels estimated by the 2019 Meeting (0.02 mg/kg in eggs, 0.01 (*) mg/kg in poultry meat, poultry edible offal of and poultry fat).

Based on the highest estimated total residues of pydiflumetofen and 2,4,6-TCP expressed as pydiflumetofen in eggs, muscle, liver and fat, the Meeting estimated HR values of 0.023 mg/kg in eggs and 0.02 mg/kg in poultry meat, poultry, edible offal of and poultry fat.

Based on the mean estimated total residues of pydiflumetofen and 2,4,6-TCP expressed as pydiflumetofen in eggs, muscle, liver and fat, the Meeting estimated STMR values of 0.02 mg/kg in eggs, poultry meat, poultry, edible offal of and poultry fat.

RECOMMENDATIONS

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

Definition of the residue for compliance with the MRL for plant and animal commodities and dietary risk assessment for plant commodities: pydiflumetofen.

Definition of the residue for dietary risk assessment for animal commodities other than mammalian liver and kidney: Sum of pydiflumetofen and 2,4,6-TCP and its conjugates, expressed as pydiflumetofen

Definition of the residue for dietary risk assessment for mammalian liver and kidney: Sum of pydiflumetofen, 2,4,6-TCP and its conjugates and SYN547897 and its conjugates, expressed as pydiflumetofen.

The residue is fat-soluble.

Table 6 Recommendations for residues of pydiflumetofen from the 2021 Extra JMPR

CCN	Commodity	Recommended maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
AM 0660	Almond hulls	10 (dw)		Median: 1.6 (as)	
VP 2060	Beans with pods, Subgroup of	0.7		0.045	0.47
VB 0040	Brassica vegetables (except Brassica leafy vegetables), Group of	W	0.1		
VA 2031	Bulb onions, Subgroup of	0.3		0.07	0.20
FB 2006	Bush berries, Subgroup of	5		0.88	3.9
FS 0013	Cherries, Subgroup of	2		0.395	1.7
FC 0001	Citrus Fruit, Group of	0.9		0.05 (except kumquats) 0.21 (kumquats)	0.16 (excluding kumquats) 0.76 (kumquats)
OR 0001	Citrus oil, edible	40		9.1	
AB 0001	Citrus pulp, dry	1.5		0.30	
SO 0691	Cottonseed	0.02 [a]	0.3	0.02 [a]	
MO 0105	Edible offal (mammalian)	0.1	0.1	Liver: 0.09 Kidney: 0.09	Liver: 0.44 Kidney: 0.30
FB 0267	Elderberries	5		0.88	3.9
VB 0042	Flowerhead Brassicas, Subgroup of	3		0.39	1.5
VA 2032	Green onions, Subgroup of	1.5		0.36	1.39
VB 2036	Head Brassicas, Subgroup of	2		0.065	0.22
VL 2052	Leaves of root and tuber vegetables, Subgroup of (except leaves of tuber vegetables)	W	0.07		
VP 0060	Legume vegetables, Group of	W	0.02		
FB 2009	Low growing berries, Subgroup of (except cranberries)	1		0.185	0.62
MF 0100	Mammalian fats (except milk fats)	0.1	0.1	0.02	0.07
MM 0095	Meat (from mammals other than marine mammals)	0.1 (fat)	0.1 (fat)	Muscle: 0.02 Fat: 0.02	Muscle: 0.02 Fat: 0.07
ML 0106	Milks	0.01(*)	0.01(*)	0.02	

CCN	Commodity	Recommended maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
FS 2001	Peaches, Subgroup of	1		0.21	0.80
VP 2061	Peas with pods, Subgroup of	1.5		0.12	0.84
FS 0014	Plums, Subgroup of	0.6		0.15	0.37
FP 0009	Pome fruit, Group of, excluding persimmon, Japanese	0.2		0.06	0.13
PO 0111	Poultry, Edible offal of	0.01(*)	0.01(*)	0.02 (liver)	0.02 (liver)
PF 0111	Poultry fats	0.01(*)	0.01(*)	0.02	0.02
PM 0110	Poultry meat	0.01(*)	0.01(*)	0.02	0.02
DF 0014	Prunes	1.5		0.34	0.85
VR 2070	Root vegetables, Subgroup of	0.3	0.1	0.08	0.25
GC 2089	Sorghum Grain and Millet, Subgroup of	W	0.03		
GC 2089	Sorghum Grain and Millet, Subgroup of, except grain sorghum	0.03		0.03	
GC 0651	Sorghum Grain	3		0.515	
AS 0651	Sorghum straw and fodder, dry	10 (dw)	0.3 (dw)	Median: 0.50 (dw)	Highest: 7.6 (dw)
VB 2016	Stem Brassicas, Subgroup of	0.1 [a]		0.02 [a]	0.09 [a]
VP 2062	Succulent beans without pods, Subgroup of	0.15		0.033	0.099
VP 2063	Succulent peas without pods, Subgroup of	0.05		0.031	0.042
AV 0596	Sugar beet leaves or tops (dry)	40 (dw)		Median:1.50 (as)	Highest: 8.0 (as)
SO 2091	Sunflower seeds, Subgroup of	0.5	0.3	0.09	
TN 0085	Tree nuts, Group of	0.05		0.01	0.03
VP 2064	Underground immature beans and peas, Subgroup of	0.02 [a]		0.02 [a]	0.02 [a]
	Almond, roasted				
	Almond, oil				
JF 0226	Apple juice			0.004	
	Apple sauce			0.004	
	Apple, canned			0.002	
DF 0226	Apple, dried			0.02	0.05
AB 1230	Apple pomace, wet			0.22	
	Cabbage leaves			Median: 0.62	Highest: 0.99
JF 0001	Citrus juice			0.004	
	Citrus peel			0.39	1.4
OR 0691	Cotton seed oil (refined)			0.0006	
AB 0691	Cotton seed hulls			0.003	

CCN	Commodity	Recommended maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
AB 1203	Cotton seed meal			0.0002	
	Pear, canned			0.004	
	Pear, dried			0.03	0.08
	Pear juice			0.006	
	Sugar beet refined sugar			0.004	
	Sorghum, aspirated grain fractions			31.9	
	Sorghum flour			0.45	
AF 0651	Sorghum forage (green)			Median: 0.23 (as)	Highest: 0.73 (as)
OR 0702	Sunflower oil, Edible			0.005	
	Sunflower meal			0.004	

[a] based on rotational crops

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for pydiflumetofen is 0–0.1 mg/kg bw. The International Estimated Daily Intakes (IEDIs) for pydiflumetofen were estimated for the 17 GEMS/Food Consumption Cluster Diets using the STMR or STMR-P values estimated by the JMPR. The results are shown in Annex 3 of the 2021 Extra JMPR Report.

The IEDIs ranged from 2–20% of the maximum ADI. The Meeting concluded that long-term dietary exposure to residues of pydiflumetofen from uses considered by the JMPR is unlikely to present a public health concern.

Acute dietary exposure

The ARfD for pydiflumetofen is 0.3 mg/kg bw. The International Estimate of Short-Term Intakes (IESTIs) for pydiflumetofen were calculated for the food commodities and their processed commodities for which HRs/HR-Ps or STMRs/STMR-Ps were estimated by the present Meeting and for which consumption data were available. The results are shown in Annex 4 of the 2021 Extra JMPR Report.

The IESTIs were 0–20% of the ARfD for the general population and 0–20% of the ARfD for children. The Meeting concluded that acute dietary exposure to residues of pydiflumetofen from uses considered by the present Meeting are unlikely to present a public health concern.

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QUINOXYFEN (222)

First draft prepared by Ms M Thomas, Pest Management Regulatory Agency, Canada

EXPLANATION

Quinoxyfen is a fungicide used for protection against powdery mildew diseases on a variety of crops.

Quinoxyfen was first evaluated for toxicology and residues by the JMPR in 2006. An ADI of 0–0.2 mg/kg bw was established while an ARfD was deemed unnecessary. The residue definition for compliance with the MRL and dietary risk assessment for plant and animal commodities is *quinoxyfen.*

The residue is fat-soluble.

The Forty-third Codex Alimentarius Commission (2020) approved the new work proposals including the priority list of pesticides for evaluation by the current Meeting. The priority list included quinoxyfen for evaluation of additional uses by the 2021 Extra JMPR.

The Meeting received information from the sponsor on use patterns, supervised residue trials on stone fruits, melons, winter squash, tomato and globe artichokes and processing studies.

RESIDUE ANALYSIS

Analytical methods

A number of methods for the quantification of quinoxyfen in plant and animal matrices were evaluated by the 2006 JMPR. Among them were the data collection methods ERC 95.26 and ERC 95.26.S1 (a modified version of ERC 95.26), used for the quantification of quinoxyfen residues in the supervised residue trials and processing studies provided to the current Meeting. The recoveries resulting from the concurrent method validation are summarized in Table 1.

Method ERC 95.26

Quinoxyfen was extracted from cherries, plum and dried plum, peach, cantaloupe, winter squash and globe artichoke using acidified acetone. An aliquot of the extract was partitioned with hexane and cleaned up using a solid phase extraction column. The eluant was evaporated, re-dissolved in 0.1% corn oil in isooctane and analysed by GC-MSD (*m/z* 237, 307 and 309). The validated limit of quantification (LOQ) of the method was reported to be 0.01 mg/kg for all matrices.

Method ERC 95.26.S1

Quinoxyfen was extracted from tomato fruit using acetonitrile and from tomato paste and puree using a mixture of acetonitrile/water (1:5, *v/v*). The paste was filtered, and the fruit and puree samples were diluted with water. Extracts were purified by solid phase extraction, eluting with acetonitrile/water (80:20, *v/v*). The eluate was diluted in acetonitrile:water (50:50, *v/v*) before analysis by LC-MS/MS (*m/z* 308 → 197). The validated LOQ of the method was reported to be 0.01 mg/kg for all tomato matrices.

Table 1 Summary of concurrent method validation data

Matrix	Fortification level (mg/kg)	Individual recoveries (%)	Range of recoveries (%)	Mean recovery (%)	RSD (%)	Reference
ERC 95.26						

Matrix	Fortification level (mg/kg)	Individual recoveries (%)	Range of recoveries (%)	Mean recovery (%)	RSD (%)	Reference
Cherry pitted fruit	0.01	92, 96, 94	92-96	94	2.1	IR-4 PR Nos. 07757/A7757
	0.05	98, 98	98	98	0.0	
	0.10	94, 97, 98, 95, 96, 98, 94, 95	94-98	96	1.7	
	0.30	94, 89, 96, 96	89-96	94	3.5	
	1.0	79, 98, 98, 93, 94, 95	79-98	93	7.6	
Plum pitted fruit	0.01	90, 91, 101, 97, 101, 91	90-101	95	5.4	IR-4 PR No. 08463
	0.1	98, 94, 99	94-99	97	2.7	
	1.0	91, 95, 94	91-95	93	2.2	
Plum dried pitted fruit	0.01	107, 110, 108, 103, 100, 102	100-110	105	3.7	
	0.1	99, 109, 104	99-109	104	4.8	
	1.0	100, 100, 108	100-108	103	4.5	
Peach pitted fruit	0.01	89, 90, 91, 97, 87, 87	87-97	90	4.1	IR-4 PR No. 08462
	0.1	89, 97, 92	89-97	93	4.4	
	1.0	92, 89, 89	89-92	90	1.9	
Cantaloupe fruit	0.01	101, 98, 111	98-111	103	6.6	IR-4 PR No. 07252
	0.5	89, 81, 85	81-89	85	4.7	
	5.0	86, 82, 81	81-86	83	3.2	
Winter squash fruit	0.01	89, 93, 95, 93, 101, 92	89-101	94	4.3	IR-4 PR No. 07653
	0.1	94*, 91, 89*	89-94	91	2.7	
	1.0	90, 94, 92	90-94	92	2.2	
Artichoke buds	0.01	104, 108, 95	95-108	102	6.5	IR-4 PR No. 08817
	0.1	93, 93, 95	93-95	94	1.2	
	1.0	88, 87, 86	86-88	87	1.1	
ERC 95.26.S1						
Tomato fruit	0.01	113, 113, 111, 114, 113, 112	111-114	113	0.9	IR-4 PR No. 09289
	0.1	93, 91, 91	91-93	92	1.3	
	3.0	94, 94, 92	92-94	93	1.2	
Tomato paste	0.01	114, 110, 108	108-114	111	2.8	
	0.1	89, 89, 91	89-91	90	1.3	
	3.0	89, 91, 89	89-91	90	1.3	
Tomato puree	0.01	125, 125, 125	125	125	0.0	
	0.1	95, 95, 97	95-97	96	1.2	
	3.0	95, 94, 94	94-95	94	0.6	

*Average of 2 determinations

Stability of residues in stored analytical samples

The stability of quinoxyfen residues in a number of commodities stored under frozen conditions was investigated by the 2006 JMPR. Of relevance to the crops presented in this submission, high water commodities (cherries, lettuce and bell pepper) were considered to be stable when stored frozen at -15 °C

for at least 320 days. This period of storage covers the maximum storage intervals in the residue trials presented to the current Meeting. Nevertheless, concurrent storage stability studies were conducted and results are summarized in the table below.

Table 2 Stability of residues of quinoxyfen following storage at <-20 ºC

Commodity	Storage Duration (days)	Fortification level (mg/kg)	Procedural recoveries (%)	% Remaining (Mean)
Pitted cherry, fruit	80	1.0	93, 94, 95	91, 93, 92 (92)
Pitted plum fruit	158	0.10	90, 91, 91	88, 90, 88 (89)
Dried plum fruit	92	0.10	95, 92, 93	91, 92, 93 (92)
Pitted peach fruit	183	0.10	92, 94, 91	89, 87, 85 (87)
Cantaloupe fruit	251	0.50	94, 95, 93	89, 95, 91 (92)
Winter squash fruit	109	0.10	91, 92, 92	88, 88, 87 (88)
Artichoke buds	83	1.0	91, 91, 93	91, 92, 89 (91)

USE PATTERN

Quinoxyfen is a protectant fungicide for the control of powdery mildew diseases in a range of crops.

Quinoxyfen does not control existing or latent powdery mildew infections and therefore, it must be applied before symptoms of the disease appear, on a protectant schedule. The product is diluted with water and applied as foliar spray or broadcast treatment using conventional spray equipment. Quinoxyfen is registered for use in a wide range of crops in several countries. The use patterns of the suspension concentrate formulations, each containing 250 g ai/L, registered in Mexico and the USA are summarised in Table 3.

Table 3 Summary of registered use patterns

Crop	Country	Method	Formulation	Application					PHI (days)
				Rate (g ai/ha)	Water (L/ha)	No.	RTI (days)	Max. (g ai/ha/ season)	
Stone fruits [a]	USA	Foliar spray (ground)	250 g/L SC	128	min. 281	4	10-14	512	7
Cherry	USA	Foliar spray (ground)	250 g/L SC	128	min. 281	4-5	7	585	7
Melons [b]	USA	Foliar spray	250 g/L SC	73-110	min. 281 (ground) min. 47 (aerial)	4	10-14	438	3
Winter squash, gourds, pumpkin	USA	Foliar spray	250 g/L SC	146	min. 281 (ground) min. 47 (aerial)	4	10-14	583	3

Crop	Country	Method	Formulation	Application					PHI (days)
				Rate (g ai/ha)	Water (L/ha)	No.	RTI (days)	Max. (g ai/ha/ season)	
Butternut squash, cucumber, melon, watermelon	Mexico	Foliar spray	Not specified	50-75	Not specified	Not specified	Not specified	Not specified	3
Tomato	USA	Foliar spray	250 g/L SC	73-110	min. 281 (ground) min. 47 (aerial)	4	10-14	438	3
Chili pepper, bell pepper, tomato, tomatillo	Mexico	Foliar spray	Not specified	25-75	Not specified	4	7	225	3
Globe artichoke	USA	Foliar spray	250 g/L SC	73-110	min. 281 (ground) min. 47 (aerial)	Not specified	10-14	583	Not specified

[a] Stone fruits including apricot, chickasaw plum, damson plum, Japanese plum, nectarine, peach, plum, plumcot, prune (fresh)

[a] Melons (subgroup 9A) including citron melon, muskmelon (cantaloupe, casaba, crenshaw melon, golden pershaw melon, honeydew melon, honey balls, mango melon, Persian melon, pineapple melon, Santa Claus melon, snake melon, true cantaloupe), watermelon

RESULTS OF SUPERVISED RESIDUE TRIALS ON CROPS

The Meeting received information on supervised field trials for quinoxyfen on the following crops or crop groups:

Crop Group	Commodity	Table No.
Stone fruits	Cherry Plum Peach	4
Fruiting vegetables, cucurbits	Cantaloupe Winter squash	5
Tomatoes	Tomato	6
Stalk and stem vegetables	Globe artichoke	7

Trials were well documented with laboratory and field reports. Laboratory reports included method validation with procedural recoveries from spiking at residue levels generally bracketing 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 as residues in all control samples were <LOQ. Residue data are recorded unadjusted for recovery.

Residue values from the trials conducted in accordance with the critical 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.

Where duplicate field samples from an un-replicated plot were taken at each sampling time and were analysed separately, the individual analytical results together with the mean of these was taken as the best estimate of the residues in the plot. Similarly, where samples were collected from replicate plots the mean result is reported.

Stone fruits

Twenty-three independent supervised trials were carried out on stone fruits (cherries: seven trials [previously reviewed by 2006 JMPR and copied below], peaches: 10 trials, plums: six trials) in the USA during the 2000/2001 (cherries) and 2003 (peaches and plums) growing seasons (Chen; 2002; IR-4 PR Nos. 07757/A7757; Carpenter, 2006, IR-4 PR No. 08463; Carpenter, 2007, IR-4 PR No. 08462).

Each trial site consisted of one untreated control plot and one treated plot. The treated cherry plots received five foliar airblast applications of a suspension concentrate (SC) formulation of quinoxyfen (nominal concentration 250 g ai/L) at 114–130 g ai/ha per application, with retreatment intervals of 6–8 days, totaling 595–633 g ai/ha. For plums and peaches, treated plots received four foliar broadcast applications of the same formulation at 142–154 g ai/ha per application, with retreatment intervals of 6–8 days, totaling 575–599 g ai/ha. One peach plot received 5 applications at 145 g ai/ha per application with retreatment intervals of 6–8 days for a total of 725 g ai/ha/season. All applications were made with either concentrated spray volumes (290–951 L/ha) or with dilute spray volumes (1102–2362 L/ha). Duplicate samples were collected 6–8 days after the last application (DALA). The minimum sample size for cherries was 1 kg and for plums and peaches the minimum sample size was 2 kg.

Residues of quinoxyfen were determined in cherries, plums and peaches using the GC-MSD method ERC 95.26. Acceptable concurrent recoveries were reported at fortification levels of 0.01 to 1.0 mg/kg for cherry, plum and peach, thus validating the method. The validated LOQ was reported as 0.01 mg/kg.

Samples of pitted cherries, plums and peaches were maintained frozen at <-20 °C for a maximum of 77, 147 and 134 days, respectively, until analysis. Concurrent storage stability data demonstrated stability of quinoxyfen in pitted stone fruits for at least 183 days, which covers the maximum storage periods in the residue trials.

Table 4 Residues of quinoxyfen in pitted stone fruits from trials conducted in the USA following application of an SC formulation.

Location; Year, Variety (Trial ID)	Application						DALA	Quinoxyfen Residues mg/kg (average)
	No.	Rate (g ai/ha)	Rate (g ai/hL)	Water (L/ha)	RTI	Total		
CHERRIES - See JMPR 2006								
US GAP	4-5	128		>281	7	585	7	
Fennville, Michigan; USA 2000 Montmorency (00-M121)[a]	5	124 126 123 123 123	13.2 13.2 13.2 13.2 13.3	944 951 932 931 929	- 8 7 8 6	619	7	0.125,0.125 (0.125)

Location; Year, Variety (Trial ID)	Application						DALA	Quinoxyfen Residues mg/kg (average)
	No.	Rate (g ai/ha)	Rate (g ai/hL)	Water (L/ha)	RTI	Total		
Fennville, Michigan; USA 2000 Montmorency (00-M122)[a]	5	124 123 123 123 124	13.2 13.0 13.2 13.2 13.3	944 946 932 936 939	- 6 8 6 7	619	7	0.077, 0.076 (0.077)
Fennville, Michigan; USA 2000 Montmorency (00-M123)[a]	5	123 126 123 124 126	13.2 13.3 13.2 13.2 13.2	931 946 933 944 948	- 7 7 7 7	622	7	0.096, 0.132 (0.114)
East Lansing, Michigan; USA 2000 Montmorency (00-M124)[b]	5	124 126 123 124 123	13.2 13.3 13.3 13.2 13.2	943 945 930 944 935	- 7 7 7 7	621	7	0.120, 0.159 (0.140)
East Lansing, Michigan; USA 2000 Emperor Francis (00-M125)[b]	5	126 126 127 127 127	22.0 22.0 22.0 22.0 22.2	571 571 575 575 574	- 7 7 7 7	631	7	0.104, 0.129 (0.117)
East Lansing, Michigan; USA 2000 Hedelfinger (00-M126)[b]	5	128 127 126 128 126	22.1 21.9 22.0 22.0 22.0	579 578 571 579 571	- 7 7 7 7	633	7	0.124, 0.125 (0.125)
Prosser, Washington; USA 2000 Bing (00-WA39)[c]	5	123 126 129 123 130	9.2 9.5 9.5 9.4 9.3	1344 1315 1357 1312 1399	- 6 7 6 6	631	7	0.146, 0.136 (0.141)
Prosser, Washington; USA 2000 Bing (00-WA40)[c]	5	121 124 124 121 126	10.7 11.3 11.1 11.0 10.8	1130 1104 1122 1102 1160	- 6 7 6 6	616	7	0.131, 0.094 (0.113)
Mabton, Washington; USA 2000 Montmorency (00-WA41)	5	124 124 124 124 124	5.7 5.3 5.3 5.3 5.5	2179 2354 2344 2362 2264	- 6 6 6 6	622	6	0.053, 0.046 (0.050)

Location; Year, Variety (Trial ID)	Application						DALA	Quinoxyfen Residues mg/kg (average)
	No.	Rate (g ai/ha)	Rate (g ai/hL)	Water (L/ha)	RTI	Total		
Hotchkiss, Colorado; USA 2000 Montmorency (00-CO01)	5	123 114 119 119 120	6.4 6.5 6.5 6.5 6.5	1914 1766 1835 1841 1850	- 7 7 7 6	595	6	0.138, 0.156 (0.147)
Guthsville, Pennsylvania; USA 2000 Montmorency (00-PA01)	5	129 124 123 127 128	11.1 11.1 11.0 11.1 11.2	1159 1124 1122 1140 1140	- 7 6 6 8	615	6	0.269, 0.265 (0.267)
Visalia, California; USA 2001 Brooks (01-CA49)[d]	5	123 122 123 123 123	10.0 9.7 9.0 9.1 9.1	1239 1257 1370 1357 1361	- 6 7 7 7	615	7	0.037, 0.030 (0.034)
Visalia, California; USA 2001 Brooks (01-CA50)[d]	5	127 126 127 126 127	8.6 8.4 7.8 7.8 7.9	1469 1492 1625 1604 1611	- 6 7 7 7	631	8	0.082, 0.068 (0.075)
PLUMS (Carpenter, 2006, IR-4 PR No. 08463)								
US GAP	4	128		>281	10-14	512	7	
Fennville, MI; USA 2003 Early Golden (03-MI15)	4	145 147 145 145	15.5 15.5 15.6 15.5	930 945 929 934	- 6 7 7	581	7	0.087, 0.095 (0.091)
Parlier, CA; USA 2003 Friar (03-CA30)[e]	4	142 147 145 146	22.6 23.0 22.5 22.6	629 638 643 644	- 7 7 7	579	7	<0.01, <0.01 (<0.01)
Parlier, CA; USA 2003 Casselman (03-CA31)[e]	4	146 149 147 143	31.4 31.1 31.4 31.6	463 479 467 455	- 6 8 7	585	7	<0.01, <0.01 (<0.01)
Porterville, CA; USA 2003 Angelina (03-CA32)	4	145 145 145 146	33.5 31.7 30.3 30.0	432 456 476 485	- 7 7 7	579	7	0.011, 0.012 (0.012)

Location; Year, Variety (Trial ID)	Application						DALA	Quinoxyfen Residues mg/kg (average)
	No.	Rate (g ai/ha)	Rate (g ai/hL)	Water (L/ha)	RTI	Total		
Madera, CA; USA 2003 Fortune (03-CA33)	4	147 143 143 145	20.7 20.7 20.7 20.7	708 694 693 698	- 7 7 7	578	7	<0.01, 0.013 (0.012)
NE Brooks, OR; USA 2003 Brooks (03-OR04)	4	146 148 147 143	26.2 26.2 26.1 26.2	556 564 562 547	- 7 7 7	584	7	<0.01, <0.01 (<0.01)
PEACHES (Carpenter, 2007, IR-4 PR No. 08462)								
US GAP	4	128		>281	10-14	512	7	
Poplar, CA; USA 2003 September Sun (03-CA26)	4	145 145 146 143	20.9 20.1 20.0 20.4	692 720 730 702	- 7 7 7	578	7	0.063, 0.080 (0.072)
Porterville, CA; USA 2003 September Sun (03-CA27)	4	145 147 146 145	49.9 50.6 50.2 49.9	290 290 290 290	- 7 7 7	582	7	0.078, 0.057 (0.068)
Madera, CA; USA 2003 Champagne (03-CA28)	4	147 146 146 147	20.7 20.5 20.8 20.7	711 711 702 711	- 7 7 7	585	7	0.16, 0.091 (0.13)
Parlier, CA; USA 2003 O'Henry (03-CA29)	4	146 148 147 147	21.6 21.7 21.2 21.2	673 683 692 692	- 6 7 7	587	7	0.095, 0.076 (0.086)
Fennville, MI; USA 2003 Elberta (03-MI14)	4	146 142 143 143	15.6 15.5 15.5 15.7	935 917 926 917	- 7 7 7	575	7	0.21, 0.14 (0.18)
Jackson Springs, NC; USA 2003 Contender (03-NC07)[f]	4	143 147 147 145	10.4 10.5 10.4 10.4	1384 1403 1412 1384	- 7 7 7	582	7	0.094, 0.13 (0.11)
Jackson Springs, NC; USA 2003 Emery (03-NC08)[f]	4	149 154 149 147	10.6 10.6 10.6 10.6	1403 1450 1403 1384	- 7 7 7	599	7	0.20, 0.22 (0.21)

Location; Year, Variety (Trial ID)	Application						DALA	Quinoxyfen Residues mg/kg (average)
	No.	Rate (g ai/ha)	Rate (g ai/hL)	Water (L/ha)	RTI	Total		
Bridgeton, NJ; USA 2003 Dixie Red (03-NJ11)[g]	4	145 145 147 143	25.3 25.3 25.7 25.1	571 571 571 571	- 7 7 7	579	8	0.090, 0.083 (0.087)
Bridgeton, NJ; USA 2003 Ernie's Choice (03-NJ12)[G]	4	147 146 146 147	20.9 20.8 20.8 21.5	702 702 702 683	- 8 6 7	585	6	0.12, 0.069 (0.095)
Lansing, NY; USA 2003 Harrow Beauty (03-NY03)	4	146 145 145 145	31.2 30.9 30.9 30.9	468 468 468 468	- 6 7 7	579	7	0.41, 0.54 (0.48)
Devine, TX; USA 2003 Tex Royal (03-TX13)	5	145 145 145 145 145	24.9 24.9 24.9 24.9 24.9	580 580 580 580 580	- 7 8 6 7	725	8	0.55, 0.43 (0.49)

[a] Applications were separated by 3-6 days, rendering the trials dependent.

[b] Applications were separated by 3-7 days, rendering the trials dependent.

[c] Applications were made on the same day, rendering the trials dependent.

[d] Applications were separated by 1 day, rendering the trials dependent.

[e] Applications were separated by 15 days, rendering the trials independent.

[f] Applications were separated by 9 days, rendering the trials dependent.

[g] Applications were separated by 28 days, rendering the trials independent.

Fruiting vegetables, cucurbits

Fifteen independent supervised trials were carried out on fruiting vegetables - cucurbits (cantaloupe: 10 trials [previously reviewed by 2006 JMPR and copied below] and winter squash: five trials) in Canada and the USA during the 2001, 2003 and 2004 growing seasons (Corley, 2004, IR-4 PR No. 07252; Carpenter, 2007, IR-4 PR No. 07653).

Each trial site consisted of one untreated control plot and one treated plot. The treated plots received four foliar ground applications of a suspension concentrate (SC) formulation of quinoxyfen (nominal concentration 250 g ai/L) at 139–169 g ai/ha per application, with retreatment intervals of 6–12 days, totaling 582–620 g ai/ha. In one cantaloupe trial, the treated plot received five spray applications of the same formulation at 148–151 g ai/ha per application at retreatment intervals of 6–7 days for a total of 748 g ai/ha per season. All applications were made with spray volumes of 281–765 L/ha. Samples were collected 2–4 DALA. For the decline trials, cantaloupe samples were collected at 0, 3, 7 and 14 DALA. After

harvest, the cantaloupes were quartered and at least one quarter of each melon was retained per sample. The minimum sample size for cantaloupe and squash was 2 kg.

Residues of quinoxyfen were determined in cantaloupe and winter squash using the GC-MSD method ERC 95.26. Acceptable concurrent recoveries in both cantaloupe and winter squash were reported at fortification levels of 0.01 to 5.0 mg/kg, thus validating the method. The validated LOQ was reported as 0.01 mg/kg.

Samples of cantaloupe quarters and winter squash were maintained frozen at <-20 ºC for a maximum of 196 and 98 days, respectively, until analysis. Concurrent storage stability data demonstrated stability of quinoxyfen in cantaloupe and winter squash for up to 251 days and 109 days, respectively, which covers the maximum storage periods in the residue trials.

Table 5 Residues of quinoxyfen in cantaloupe and winter squash from trials conducted in Canada and the USA following application of an SC formulation

Location; Year, Variety (Trial ID)	Application						DALA	Quinoxyfen Residues mg/kg (average)
	No.	Rate (g ai/ha)	Rate (g ai/hL)	Water (L/ha)	RTI	Total		
CANTALOUPE - See JMPR 2006								
US GAP	4	73-110	Not specified	min. 281 (ground) min. 47 (aerial)	10-14	438	3	
Ashcroft, BC; Canada 2001 Athena (01-BC01)	4	147 145 146 145	26.4 26.4 26.4 26.5	555 547 552 545	- 8 6 12	582	4	0.035, 0.025 (0.030)
Fresno Co., CA; USA 2001 Aclaim (01-CA60)	4	149 151 149 149	31.1 31.7 40.6 38.6	480 477 367 386	- 7 7 7	599	3	0.031, 0.037 (0.034)
Visalia, CA; USA 2001 Hale's Best (03-CA82)[a]	4	156 155 155 155	33.3 32.1 32.1 31.8	468 482 482 487	- 7 7 7	620	3	0.018, 0.024 (0.021)
Visalia, CA; USA 2001 Hearts of Gold (01-CA83)[a]	4	155 155 155 152	31.4 32.1 31.4 31.4	492 482 493 486	- 7 8 7	616	3	0.025, 0.023 (0.024)
Tifton, GA; USA 2001 Vienna (01-GA*16)	5	150 148 150 148 151	52.2 52.1 52.4 52.1 52.4	288 284 286 284 289	- 6 7 7 7	748	2	<0.01, <0.01 (<0.01)

Location; Year, Variety (Trial ID)	Application						DALA	Quinoxyfen Residues mg/kg (average)
	No.	Rate (g ai/ha)	Rate (g ai/hL)	Water (L/ha)	RTI	Total		
Mesilla, NM; USA 2001 Hale's Best Jumbo (01-NM09)	4	148 150 148 146	43.3 42.3 40.1 42.5	342 355 369 343	- 7 7 7	592	3	0.018, 0.025 (0.022)
Bridgeton, NJ; USA 2001 Ambrosia (01-NJ20)	4	149 151 150 147	38.5 38.6 38.6 38.6	387 392 389 380	- 9 8 8	597	0 3 7 14	0.029, 0.043 (0.036) 0.034, 0.034 (0.034) 0.024, 0.030 (0.027) 0.020, 0.020 (0.020)
Innerkip, ON; Canada 2001 Athena (01-ON01)	4	151 141 169 139	19.8 26.9 26.9 26.9	765 526 628 516	- 7 6 8	601	2	0.056, 0.043 (0.050)
St. Isidore, QC; Canada, 2001 Early Dawn (01-QC03)	4	147 151 149 147	31.1 31.0 30.9 31.0	473 488 482 474	- 7 7 7	594	4	0.031, 0.032 (0.032)
Weslaco, TX; USA 2001 Explorer (01-TX23)[b]	4	150 157 150 149	47.2 47.3 44.1 45.3	318 332 340 329	- 6 6 6	606	0 3 7 14	0.076, 0.060 (0.068) 0.023, 0.024 (0.024) 0.017, 0.018 (0.018) <0.01, <0.01 (<0.01)
Weslaco, TX; USA 2001 Mission (01-TX*24)[b]	4	154 149 146 149	51.1 51.6 50.5 50.7	301 289 288 294	- 6 6 6	597	2	0.049, 0.049 (0.049)
WINTER SQUASH (Carpenter, 2007, IR-4 PR No. 07653)								
USA GAP	4	73-146		min. 281 (ground) min. 47 (aerial)	10-14	583	3	
Davis, CA; USA 2003 Waltham Butternut (03-CA112)	4	149 149 145 147	44.3 44.3 44.2 44.8	337 337 327 327	- 9 6 8	590	3	0.039, 0.054 (0.047)
Salisbury, MD; USA 2003 Tuffy Green Acorn (03-MD12)	4	145 146 146 147	48.3 48.7 48.7 49.1	299 299 299 299	- 6 8 6	583	4	0.031, 0.027 (0.029)

Location; Year, Variety (Trial ID)	Application						DALA	Quinoxyfen Residues mg/kg (average)
	No.	Rate (g ai/ha)	Rate (g ai/hL)	Water (L/ha)	RTI	Total		
Bridgeton, NJ; USA 2003 Table Queen Acorn (03-NJ29)	4	141 143 148 150	50.3 51.1 51.0 50.2	281 281 290 299	- 7 8 6	583	3	0.044, 0.067 (0.056)
Fremont, OH; USA 2003 Burgees Buttercup (03-OH*12)	4	145 145 147 146	45.5 45.5 40.2 38.0	318 318 365 384	- 7 7 7	582	3	0.082, 0.064 (0.073)
Citra, FL; USA 2004 Mean Queen Acorn (03-FL52)	4	147 152 150 151	44.8 45.3 44.6 44.9	327 337 337 337	- 7 7 7	601	3	0.11, 0.11 (0.11)

[a] Applications were separated by 31 days, rendering the trials independent.

[b] Applications were made on the same day, rendering the trials dependent

Tomato

Eleven independent supervised trials were carried out on field tomato in the USA during the 2011 growing season (Homa, 2012, IR-4 PR No. 09289).

Each trial site consisted of one untreated control plot and one treated plot. The treated plots received four foliar ground applications of a suspension concentrate (SC) formulation of quinoxyfen (nominal concentration 250 g ai/L) at 143–162 g ai/ha per application, with retreatment intervals of 6–8 days, totaling 574–624 g ai/ha. In one tomato trial, the treated plot received five spray applications of the same formulation at 145–149 g ai/ha per application at retreatment intervals of 6–8 days for a total of 733 g ai/ha per season. All applications included and adjuvant and were made with spray volumes of 195–879 L/ha. Samples were collected 2–4 DALA. For the decline trials, tomato samples were collected at 0, 2/3, 6/8 and 13/14 DALA. More than 24 fruit were collected per sample.

Residues of quinoxyfen were determined in tomato using the GC-MSD method ERC 95.26. S1. Acceptable concurrent recoveries were reported at fortification levels of 0.01 to 3.0 mg/kg, thus validating the method. The validated LOQ was reported as 0.01 mg/kg.

Samples of tomato were maintained frozen at <-20 °C for a maximum of 183 days until analysis. Storage stability data demonstrated the stability of quinoxyfen in various high water content commodities for up to 320 days (JMPR 2006), which covers the maximum storage period in the residue trials.

Table 6 Residues of quinoxyfen in tomato from trials conducted in the USA following application of an SC formulation. (Homa, 2012, IR-4 PR No. 09289)

Location; Year, Variety (Trial ID)	Application						DALA	Quinoxyfen Residues mg/kg (average)
	No.	Rate (g ai/ha)	Rate (g ai/hL)	Water (L/ha)	RTI	Total		
USA GAP	4	73-110	Not specified	min. 281 (ground) min. 47 (aerial)	10-14	438	3	
Salisbury, MD: USA 2011 Tami G; small variety (11-MD-04)	4	144 145 145 145	29.9 29.9 29.9 29.9	483 485 486 484	- 6 7 7	579	3	0.31, 0.32 (0.32)
Fremont, OH; USA 2011 Primo Red; large variety (11-OH*01)	4	146 145 145 145	22.1 42.9 34.9 37.4	661 339 414 387	- 6 8 7	581	4	0.052, 0.071 (0.062)
Arlington, WI; USA 2011 Sweet Baby Girl Hybrid FFT; small variety (11-WI02)	4	146 144 144 147	73.2 73.7 72.6 72.1	200 195 198 204	- 7 7 6	582	3	0.26, 0.31 (0.29)
Citra, FL; USA 2011 Amelia; large variety (11-FL06)	4	147 145 145 146	26.3 26.3 26.3 26.3	557 551 552 555	- 7 7 7	583	3	0.058, 0.029 (0.044)
Tifton, GA; USA 2011 Amelia; large variety (11-GA*01)	4	144 144 143 143	53.3 53.4 53.4 53.4	270 269 268 268	- 8 6 7	574	2	0.028, 0.015 (0.022)
Charleston, SC; USA 2011 Sweet Baby Girl; small variety (11-SC*03)	4	157 155 156 156	17.9 18.2 18.0 18.2	879 854 867 857	- 7 6 7	624	2	0.17, 0.16 (0.17)

Location; Year, Variety (Trial ID)	Application						DALA	Quinoxyfen Residues mg/kg (average)
	No.	Rate (g ai/ha)	Rate (g ai/hL)	Water (L/ha)	RTI	Total		
Holtville, CA; USA 2011 Bijou; small variety (11-CA16)	4	146 162 146 147	31.5 32.6 29.6 30.0	462 498 494 490	- 8 6 7	601	3	0.090, 0.068 (0.079)
Irvine, CA; USA 2011 Sun 6788; large variety (11-CA17)	5	145 145 149 148 147	26.0 26.0 26.0 26.0 26.0	558 556 571 569 564	- 6 8 7 6	733	3	0.15, 0.14 (0.15)
Irvine, CA; USA 2011 Celebrity; large variety (11-CA18)	4	151 152 146 152	47.3 47.2 47.3 47.2	319 322 309 321	- 6 7 7	601	0 2 8 13	0.12, 0.087 (0.10) 0.084, 0.065 (0.075) 0.066, 0.085 (0.076) 0.062, 0.068 (0.065)
Davis, CA; USA 2011 Halley 31155; large variety (11-CA19)[a]	4	145 151 143 143	48.6 48.6 48.6 48.7	299 310 295 294	- 6 7 6	582	2	0.072, 0.064 (0.068)
Davis, CA; USA 2011 Monica; large variety (11-CA20) [a]	4	150 147 146 149	48.5 48.7 48.6 48.6	309 309 299 306	- 7 6 8	591	2	0.15, 0.13 (0.14)
Las Cruces, NM; USA 2011 Celebrity; large variety (11-NM04)[b]	4	147 147 147 147	65.4 62.8 60.4 56.2	225 234 243 261	- 7 7 7	588	0 3 6 14	0.072, 0.033 (0.053) 0.069, 0.082 (0.076) 0.043, 0.026 (0.035) 0.021, 0.033 (0.027)
Las Cruces, NM; USA 2011 Roma; small variety (11-NM05)[b]	4	143 145 145 141	30.8 30.3 29.7 27.6	464 478 487 511	- 7 7 7	574	3	0.13, 0.093 (0.11)

[a] Applications were separated by 6 days, rendering the trials dependent

[b] Difference in variety rendered the trials independent

Globe artichoke

Three independent supervised trials were carried out on globe artichoke in the USA during the 2004 growing season (Carpenter, 2007, IR-4 PR No. 08817).

Each trial site consisted of one untreated control plot and one treated plot. The treated plots received four foliar ground applications of a suspension concentrate (SC) formulation of quinoxyfen (nominal concentration 250 g ai/L) at 145–151 g ai/ha per application, with retreatment intervals of 6–7 days, totaling 583–600 g ai/ha. All applications were made with spray volumes of 94–702 L/ha. Samples were collected 0 DALA. Each sample weighed a minimum of 3 kg.

Residues of quinoxyfen were determined in globe artichoke using the GC-MSD method ERC 95.26. Acceptable concurrent recoveries were reported at fortification levels of 0.01 to 1.0 mg/kg, thus validating the method. The validated LOQ was reported as 0.01 mg/kg.

Samples of globe artichoke were maintained frozen at <-20 °C for a maximum of 83 days until analysis. Concurrent storage stability data demonstrated the stability of quinoxyfen in globe artichoke for up 83 days.

Table 7 Residues of quinoxyfen in globe artichoke from trials conducted in the USA following application of an SC formulation. (Carpenter, 2007, IR-4 PR No. 08817)

Location; Year, Variety (Trial ID)	Application						DALA	Quinoxyfen Residues mg/kg (average)
	No.	Rate (g ai/ha)	Rate (g ai/hL)	Water (L/ha)	RTI	Total		
USA GAP	Not specified	73-110	Not specified	min. 281 (ground) min. 47 (aerial)	10-14	583	0	
Moss Landing, CA; USA 2004 Green Globe (04-CA18)[a c]	4	151 150 150 149	161 160 160 159	93.5 93.5 93.5 93.5	- 7 6 6	600	0	1.09, 0.95 (1.02)
Salinas, CA; USA 2004 Green Globe (04-CA40)[a b]	4	149 148 148 147	31.2 31.0 31.0 31.4	477 477 477 468	- 7 6 6	592	0	0.94, 0.63 (0.79)
Castroville, CA; USA 2004 Green Globe (04-CA41)[b c]	4	145 146 146 146	21.0 20.8 20.8 20.8	692 702 702 702	- 7 6 7	583	0	0.87, 0.83 (0.85)

[a] Applications were separated by 14 days, rendering the trials independent

[b] Applications were separated by 19 days, rendering the trials independent

[c] Applications were separated by 5 days, rendering the trials dependent

PROCESSING

Plums

One processing trial was conducted in conjunction with the residue field trials on plums in the USA during the 2003 growing season (Carpenter, 2006, IR-4 PR No. 08463).

Quinoxyfen, formulated as an SC formulation (containing 250 g ai/L) was applied four times at application rates of 143–149 g ai/ha per application, with retreatment intervals of 6–8 days, totaling 585 g ai/ha. One untreated control and one treated sample each containing a minimum of 6.8–9.1 kg of plum fruit were harvested 7 days after the last application. Plums were then cut in half and pits removed and discarded. The two halves were then placed on drying trays lined with paper and transferred to dryers set at 60 °C as per commercial practices for drying plums. Plum samples were dried for approximately two days.

Residues of quinoxyfen were determined in fresh plums and dried plum fruits using the GC-MSD method ERC 95.26. Acceptable concurrent recoveries were reported at fortification levels of 0.01 to 1.0 mg/kg, thus validating the method. The validated LOQ was reported as 0.01 mg/kg.

Samples were maintained frozen at <-20 ºC for a maximum of 89 days until analysis. The stability of quinoxyfen residues in dried plum fruit was demonstrated for at least 92 days in the study, which is adequate to cover the maximum frozen storage period of the samples in the processing trial.

Table 8 Quinoxyfen residues in fresh plum and dried plum fruit

Trial Identification (City, State/Region, Country, Year)	Crop / Variety	Commodity or Matrix	Total Rate (g ai/ha)	DALA	Quinoxyfen Residues (Average), mg/kg	Processing Factor
Parlier, CA; USA 2003 (03-CA31)	Casselman	Fresh pitted fruit, pre-processing	585	7	<0.01, <0.01 (<0.01)	-
		Dried pitted fruit			0.03	>3

Tomato

One processing trial was conducted in conjunction with the residue field trials on tomatoes in the USA during the 2011 growing season (Homa, 2012, IR-4 PR No. 09289).

Four foliar applications of an SC formulation containing 250 g ai/L of quinoxyfen were made to tomatoes at 143–151 g ai/ha, for a total rate of 582 g ai/ha. The interval between applications was 6–7 days and samples were harvested 2 days after the last application.

Tomatoes were processed into canned tomato puree and canned tomato paste using processes that simulated commercial operations as closely as possible. Briefly, tomatoes (48 kg) were immersed in basic solution at approximately 52–60 °C for 3 minutes before being rinsed with a high pressure spray of water at approximately 68–74 °C. The fruit (37 kg) was crushed using a fruit press before heating the sample to approximately 79–85 °C in a steam jacketed kettle. The break juice was separated into pomace and juice, and the wet pomace was pressed and discarded.

The fresh juice fraction was evaporated, and the puree and paste fractions removed when the desired Brix ranges were achieved (12.0–13.0° and 24.0–33.0°, respectively), adjusting with a dilute sodium chloride solution if required. The puree and paste fractions were then heated to 82–88 °C before canning. The sealed cans of puree and paste were sterilized for 15 minutes at 96–100 °C in a water bath and cooled.

Samples of tomato fruit, puree and paste were analyzed for residues of quinoxyfen using the LC-MS/MS method ERC 95.26.S1. Procedural recoveries carried out concurrently with the analyses of processing samples were within the acceptable range of 70–120%, with relative standard deviations <20%.

Samples of tomato paste and puree were maintained frozen at <-20 °C for a maximum of 104 days until analysis. This storage period is within the demonstrated storage stability duration of high-water-content commodities.

Table 9 Quinoxyfen residues in fresh tomato and tomato processed commodities

Trial Identification (City, State/Region, Country, Year)	Crop / Variety	Commodity or Matrix	Total Rate (g ai/ha)	DALA	Quinoxyfen Residues, mg/kg	Processing Factor
Davis, CA; USA 2011 (11-CA19)	Halley 31155	Fresh tomato, pre-processing	582	2	0.11	-
		Tomato paste			0.089	0.81
		Tomato puree			0.035	0.32

APPRAISAL

Quinoxyfen is a fungicide used for protection against powdery mildew diseases on a variety of crops.

Quinoxyfen was first evaluated for toxicology and residues by JMPR in 2006. An ADI of 0–0.2 mg/kg bw was established while an ARfD was unnecessary. The residue definition for compliance with the MRL and dietary risk assessment for plant and animal commodities is quinoxyfen. The residue is fat-soluble.

The Forty-third Codex Alimentarius Commission (2020) approved the new work proposals including the priority list of pesticides for evaluation by the current Meeting. The priority list included quinoxyfen for evaluation of additional uses by the 2021 Extra JMPR.

The Meeting received information from the manufacturer on use patterns, supervised residue trials on stone fruits, melons, winter squash, tomato and globe artichokes and processing studies.

Methods of analysis

The GC-MSD analytical method ERC 95.26 and the LC-MS/MS analytical method ERC 95.26.S1 used for analysis of residues of quinoxyfen in plant commodities, with a LOQ of 0.01 mg/kg, were reviewed by the 2006 JMPR. All of the samples collected in the supervised residue trials and processing studies provided to the current Meeting were analysed using the same methods. The methods are fit for purpose based on the provided results from concurrent recovery samples.

Stability of residues in stored analytical samples

The stability of residues of quinoxyfen during frozen storage was evaluated by the 2006 JMPR. Quinoxyfen was determined to be stable when stored frozen for at least 320 days at -15 °C in commodities representative of the high-water content commodity group. Together with the concurrent storage stability data, the Meeting concluded that quinoxyfen is stable in the test crops and processed commodities under frozen storage conditions.

Results of supervised residue trials on crops

Supervised trials were available for the use of quinoxyfen on cherries, plums, peaches, melons, winter squash, tomato and globe artichoke.

Stone fruits

Cherries

The Meeting received a new GAP for cherries from the USA allowing 4–5 foliar applications at a rate of 128 g ai/ha with a minimum 7-day RTI and a 7-day PHI and seasonal application rates not exceeding 585 g ai/ha.

In the supervised residue trials from the USA on cherries, previously reviewed by the 2006 JMPR and approximating the critical GAP, quinoxyfen residues in cherries in ranked order were (n = 7): 0.05, 0.08, 0.12, 0.14 (2), 0.15 and 0.27 mg/kg.

Residues in the field trials were reported as fruit without stone. At the 2017 JMPR, it was concluded that, based on the trials on cherries, the contribution of the pit to the weight of the whole fruit was approximately 10%. Correcting the residue levels using this weight/weight ratio would lead to the same maximum residue level.

Noting that the USA GAP is for the cherries group, the Meeting estimated a maximum residue level of 0.5 mg/kg and a STMR of 0.14 mg/kg for the Subgroup of cherries, except choke cherries and withdrew its previous recommendation.

Stone fruits except cherries

The critical GAP for stone fruits except cherries in the USA includes apricot, chickasaw plum, damson plum, Japanese plum, nectarine, peach, plum, plumcot, prune (fresh). The critical GAP allows 4 foliar spray applications at 128 g ai/ha, a 10-day RTI and a 7-day PHI, with a seasonal rate not exceeding 512 g ai/ha. The Meeting received trials conducted in the USA approximating the critical GAP with the exception of the RTIs which were 6–8 days.

Supervised field trials were on average conducted at rates 15% greater than the critical GAP and the RTIs were significantly shorter. Therefore, without crop-specific residue decline data to estimate half-lives, the Meeting concluded that the overall impact of these parameters on the residues was >25%, and that there were insufficient data to estimate a maximum residue level.

Fruiting vegetables – Cucurbits

Melons

The 2006 JMPR recommended a maximum residue level of 0.1 mg/kg for melons. The Meeting considered if the previously evaluated data would support extrapolation to the subgroup of melons.

The critical GAP for the melons subgroup (including citron melon, muskmelon (cantaloupe, casaba, crenshaw melon, golden pershaw melon, honeydew melon, honey balls, mango melon, Persian melon, pineapple melon, Santa Claus melon, snake melon, true cantaloupe, watermelon) is from the USA which allows 4 foliar spray applications at 110 g ai/ha, a 10-day RTI and a 3-day PHI, with a seasonal rate not exceeding 438 g ai/ha.

The Meeting received the same trials conducted in Canada and the USA as those reviewed by the 2006 Meeting, where 4–5 foliar spray applications were made at 139–169 g ai/ha, 6–8-day RTIs, seasonal application rates of 582–748 g ai/ha and 2–4-day PHIs. The 2006 Meeting also reviewed trials conducted in Europe involving 3 foliar spray applications at 0.005–0.0075 kg ai/hL and PHIs of 7–8-days, however, these trials do not match the USA GAP and were not considered by the current Meeting.

As supervised field trials were conducted at rates 33–71% greater than the critical GAP and the RTIs were shorter, the Meeting concluded that the overall impact of these parameters on the residues was > 25%. Therefore, using the latest available tools and following the most current assessment practices, the Meeting decided that a maximum residue level could not be extended to the subgroup.

Winter squashes

The critical GAP for the winter squash, gourds and pumpkins is from the USA which allows 4 foliar spray applications at 146 g ai/ha, 10-day RTI, and a 3-day PHI, with a seasonal rate not exceeding 583 g ai/ha. The Meeting received trials from the USA on winter squashes approximating the critical GAP with the exception of the RTIs, the majority of which ranged from 6–8 days.

While supervised field trials were conducted according to the critical GAP in most respects, the RTIs were significantly shorter. Without crop-specific residue decline data to estimate half-lives, the Meeting concluded that the overall impact of these parameters on the residues was > 25%, and that there were insufficient data to estimate a maximum residue level for the subgroup.

Tomato

The critical GAP for tomatoes is from the USA which allows 4 foliar spray applications at 110 g ai/ha, 10-day RTIs and a 3-day PHI, with a seasonal rate not exceeding 438 g ai/ha. The Meeting received trials from the USA on tomatoes where 4 foliar spray applications were made at 143–162 g ai/ha, 6–8-day RTIs, seasonal application rates of 574–733 g ai/ha and 2–4-day PHIs.

As supervised field trials were conducted at rates 31–67% greater than the critical GAP and the RTIs were significantly shorter, the Meeting concluded that the overall impact of these parameters on the residues was >25%. Therefore, the Meeting decided that a maximum residue level could not be estimated.

Globe artichokes

The critical GAP for globe artichokes is from the USA where the number of spray applications at 110 g ai/ha is unspecified, however the seasonal rate must not exceed 583 g ai/ha, the RTI is 10 days and the PHI is 0 days. The Meeting received trials from the USA on globe artichokes where 4 foliar spray applications were made at 145–151 g ai/ha, 6–7-day RTIs, seasonal application rates of 583–600 g ai/ha and a 0-day PHI.

The number of trials were insufficient and the use pattern did not match the critical GAP with respect to the RTIs (6–7 days vs 10 days). Therefore, the Meeting could not recommend a maximum residue level.

Residues in processed commodities

At the current Meeting, processing studies were reviewed for plums and tomato. As the Meeting could not recommend maximum residue levels for plums and tomatoes, residues in processed commodities were not estimated.

Residues in animal commodities

Farm animal dietary burden

Dietary burdens were calculated for beef cattle, dairy cattle, broilers and laying poultry at the 2006 JMPR. The dietary burdens, were estimated using the diets listed in Appendix IX of the FAO Manual, included barley (grain and straw) and sugar beet leaves. None of the crops assessed at the current Meeting are fed to

livestock, therefore no updates to the livestock dietary burdens were warranted. The Meeting confirms its previous recommendations for residues in animal commodities made by the 2006 JMPR.

RECOMMENDATIONS

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

Definition of the residue for compliance with the MRL and dietary risk assessment for plant and animal commodities: *quinoxyfen*.

The residue is fat-soluble.

Table 2 Recommendations for residues of quinoxyfen from the 2021 Extra JMPR

CCN	Commodity	Recommended maximum residue level (mg/kg)		STMR or STMR-P mg/kg
		New	Previous	
FS 0013	Cherries	W	0.4	
FS 0013	Cherries, Subgroup of (except Choke cherries)	0.5		0.14

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for quinoxyfen is 0–0.2 mg/kg bw. The International Estimated Daily Intakes (IEDIs) for quinoxyfen were estimated for the 17 GEMS/Food Consumption Cluster Diets using the STMR or STMR-P values estimated by the JMPR. The results are shown in Annex 3 of the 2021 Extra JMPR Report.

The IEDIs ranged from 0–1% of the maximum ADI. The Meeting concluded that long-term dietary exposure to residues of quinoxyfen from uses considered by the JMPR is unlikely to present a public health concern.

Acute dietary exposure

The 2006 JMPR decided that an ARfD for quinoxyfen was unnecessary. The Meeting therefore concluded that the acute dietary exposure to residues of quinoxyfen from the uses considered is unlikely to present a public health concern.

REFERENCES

Reference Number	Author(s)	Year	Study Title
07757	Chen, H.	2002	Quinoxyfen: Magnitude of the residue on cherry, volume 2 IR-4 Study No. 07757 GLP, Unpublished 18 Jun 2002
A7757	Chen, H.	2002a	Quinoxyfen: Magnitude of the residue on cherry, volume 3 IR-4 Study No. A7757 GLP, Unpublished 18 Jun 2002

08463	Carpenter, D. H.	2006	Quinoxyfen: Magnitude of the residue on plum IR-4 Project No. 08463 GLP, Unpublished 08 Dec 2006
08462	Carpenter, D. H.	2007	Quinoxyfen: Magnitude of the residue on peach IR-4 Project No. 08462 GLP, Unpublished 02 Aug 2007
07252	Corley, J.	2004	Quinoxyfen: Magnitude of the residue on cantaloupe IR-4 Project No. 07252 GLP, Unpublished 12 Oct 2004
07653	Carpenter, D. H.	2007a	Quinoxyfen: Magnitude of the residue on winter squash IR-4 Project No. 07653 GLP, Unpublished 04 Oct 2007
09289	Homa, K.	2012	Quinoxyfen: Magnitude of the residue on tomato IR-4 Project No. 09289 GLP, Unpublished 05 Jul 2012
08817	Carpenter, D. H.	2007b	Quinoxyfen: Magnitude of the residue on artichoke IR-4 Project No. 08817 GLP, Unpublished 26 Oct 2007

SPINETORAM (233)

First draft prepared by Dr G Ye, Ministry of Agriculture and Rural Affairs, China

EXPLANATION

Spinetoram is a multicomponent tetracyclic macrolide in the class of spinosyn insecticide obtained from chemical modification of a fermentation product of *Saccharopolyspora spinose*. It consists of two closely related active ingredients, spinetoram-J and spinetoram-L, present in approximately a three to one ratio. It controls Lepidopterous larvae, leafminers, and thrips on a variety of crops by disruption of nicotinic/gamma amino butyric acid-gated chloride channels.

It was first evaluated by the 2008 JMPR (T, R), which established an ADI of 0–0.05 mg/kg bw and an ARfD was unnecessary. The JMPR 2008 also concluded the residue definition of spinetoram for plant and animal commodities:

Definition of the residue for compliance with the MRL for plant and animal commodities: *Spinetoram*.

Definition of the residue for dietary risk assessment for plant and animal commodities: *Spinetoram and N-demethyl and N-formyl metabolites of the major spinetoram component*.

The residue is fat-soluble.

Spinetoram was subsequently reviewed by 2012 and 2017 JMPR for additional uses. Codex MRLs have been established for a wide range of crops and products of animal origin.

The Forty-third Codex Alimentarius Commission (2020) approved the new work proposals including the priority list of pesticides for evaluation by the current Meeting. The priority list included spinetoram for evaluation of additional uses on pitaya (dragon fruit) and tea.

The current Meeting received GAP information and supervised residue trials on pitaya (dragon fruit) and tea.

RESIDUE ANALYSIS

Analytical methods

The Meeting received descriptions and validation data for analytical methods for residues of spinetoram J and L in dry tea and tea infusion along with the supervised trials (Nanba, T., 2012, JP2011C289/RLR-0411).

Method for tea

The dry pulverised tea sample was first swollen by water for 2 hours, then extracted with methanol/water (8:2, v/v) for 30 minutes. The extract was purified using polymer-based cartridges which were conditioned and then eluted using acetonitrile. The extract was dissolved in hexane and further purified using an NH_2 cartridge column eluted with hexane/acetone. The eluants were evaporated to dryness and then dissolved in acetonitrile/water. Quantification was conducted by LC-MS.

Tea infusions were obtained by adding boiling water to dry tea samples for 5 minutes, and were extracted with acetonitrile/water (1:1, v/v). The extract was mixed with acetic acid and purified on an Oasis MCX column which was washed and eluted with acetonitrile/triethanolamine. The eluate was evaporated to dryness, dissolved in acetonitrile/water and filtered. The extract was analysed by LC-MS/MS.

The limit of quantitation of 0.01 mg/kg was validated for spinetoram-J and spinetoram-L. The recovery data for spinetoram J and L in tea and tea infusions fortified at various levels are summarised in Table 1. The residue in control samples were below the LOQ and no interference peaks were detected at the retention time of either isomer. Average procedural recoveries were all within the acceptable range of 70–120%, with relative standard deviations (RSD) below 20%. The calibration curves over the range 0.01–25 mg/kg for each analyte demonstrated linearity with r≥0.999.

The methods are suitable for analysis of spinetoram J and L in dry green tea and tea infusions.

Table 1 Summary of procedural recovery data for dry tea and tea infusions

Crop	Analyte	Fortification mg/kg	n	Range Recovery (%)	Mean recovery (%)	% RSD	Reference
Tea, dry	Spinetoram-J	0.01	6	97-103	99	2.3	RLR-0411
		1	6	76-81	79	2.5	
		25	6	84-91	87	3.5	
	Spinetoram-L	0.01	6	101-105	103	1.6	
		1	6	74-78	76	1.7	
		10	6	82- 86	84	1.9	
Tea, dry	Spinetoram-J	0.01	5	79-87	82	3.8	RLR-0454
		1	5	79-84	82	2.6	
		50	5	79-81	80	1.0	
	Spinetoram-L	0.01	5	72-84	77	5.8	
		1	5	78-83	81	2.8	
		50	5	77-79	78	1.1	
Tea, infusion	Spinetoram-J	0.01	6	115-120	118	1.8	RLR-0411
		1	6	92-95	93	1.3	
	Spinetoram-L	0.01	6	114-119	117	1.7	
		1	6	88-91	89	1.2	

Table 2 GRM-05.03/05.04Table 2 Summary of procedural recovery data for dragon fruit

Crop	Analyte	Fortification mg/kg	n	Range Recovery (%)	Mean recovery (%)	% RSD	Reference
Dragon fruit (pitaya)	Spinetoram -J	0.01	6	84-104	93	8	GRM-05.03/05.04
		0.1	6	67-89	78	11	
		1	5	95-97	96	1	
	Spinetoram -L	0.01	6	74-95	84	9	
		0.1	6	77-84	80	3	
		1	5	94-99	95	2	
	N-Demethyl-Spinetoram-J	0.01	6	76-101	84	10	
		0.1	6	69-81	75	6	
		1	5	93-99	95	3	
	N-Formyl-Spinetoram-J	0.01	6	82-102	90	8	

Crop	Analyte	Fortification mg/kg	n	Range Recovery (%)	Mean recovery (%)	% RSD	Reference
		0.1 10	6 5	83-88 89-101	86 95	2 4	

Stability of residues in stored analytical samples

The studies of the stability of spinetoram and its metabolites in dragon fruit and tea following freezer storage of samples were made available to the Meeting.

Dragon fruit

Dragon fruit samples spiked at level of ca. 0.1 mg/kg for each compound were stored at -1 to -37 ºC, and were then analysed after 342 days. The results are shown in Table 2. Storage stability was demonstrated for dragon fruit at least 342 days.

Table 3 Storage stability of spinetoram and its metabolites in dragon fruit stored at -1 to -37 ºC for 342 days (IR-4 PR No. 11514)

	Lab sample ID	**Spinetoram-J**	**Spinetoram-L**	**Spinetoram-N-demethyl-J**	**Spinetoram-N-formyl-J**
Storage stability Remaining (%)	150124A	83	82	73	93
	150124B	88	81	70	97
	150124C	88	84	73	91
Procedural recovery (%)		96	94	97	95

Tea

The tea samples spiked at level of 1 mg/kg with either spinetoram J or spinetoram L were stored frozen at -20 °C, and were then analysed after 10–166 days of storage. The results are shown in Table 3. Storage stability of spinetoram in tea was demonstrated for at least 166 days.

Table 4 Storage stability of spinetoram in dried green tea stored at -20 °C

Sample site	Analytical substance	Spiked level (mg/kg)	Storage period (days)	Mean Remaining (%)	Procedural Recovery (%)	Reference
Saitama-Tea	Spinetoram-J isomer	1	104	80	80-82	RLR-0454
JPPA Ibaraki			159	88		
JPPA Chiba			100	82		
JPPA Kochi			166	77		
JPPA Miyazaki			161	83		
Kagoshima-Tea			166	84		
Saitama-Tea	Spinetoram-L isomer	1	104	86	77-81	
JPPA Ibaraki			159	84		
JPPA Chiba			100	78		
JPPA Kochi			166	76		
JPPA Miyazaki			161	74		
Kagoshima-Tea			166	79		

Sample site	Analytical substance	Spiked level (mg/kg)	Storage period (days)	Mean Remaining (%)	Procedural Recovery (%)	Reference
JPPA Kochi	Spinetoram-J isomer	1	104	84	80-81	RLR-0411
Kagoshima-Tea			104	82	76-79	
JPPA Kochi	Spinetoram-L isomer	1	104	77	75-78	
Kagoshima-Tea			104	78	74-76	

The demonstrated stability intervals on frozen storage encompass the duration of storage in the residue trials evaluated by the Meeting, except one sample from the dragon fruit (pitaya trials) stored for 362 days, which is less than 10% longer than the period covered by the study of 342 days.

USE PATTERNS

Spinetoram is intended for use as an insecticide on a variety of crops. Spinetoram is registered for use in tea in Japan and dragon fruit (Tropical Tree Fruits) in the USA. Registered use patterns are summarised in following table.

Table 5 Use patterns

Crop	Country	Formulation		Application					PHI (days)
		% ai	type	Method	Rate g ai/ha	Rate g ai/hL	Interval (days)	Max no. or seasonal total (g ai/season)	
Dragon fruit (pitaya)*	USA	11.7	SC	Foliar	70-123	-	4	4 (420)	1
Tea	Japan	11.7	SC	Foliar		4.68-	-	1	1

* covered as part of Tropical Tree Fruits on label. Tropical tree fruits including acerola, atemoya, avocado, biriba, black sapote, canistel, cherimoya, custard apple, feijoa, guava, ilama, jaboticaba, longan, lychee, mamey sapote, mango, papaya, passionfruit, pitaya (dragon fruit), pulasan, rambutan, sapodilla, soursop, Spanish lime, star apple, starfruit, sugar apple, ti leaves, wax jambu, white sapote

RESULTS OF SUPERVISED RESIDUE TRIALS ON CROPS

The Meeting received information on supervised field trials for spinetoram on dragon fruit and tea.

Crop	Table No.
Dragon fruit	6
Tea	7

Trial documentation included laboratory and field reports, with laboratory reports containing 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 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.

In trials where replicate field samples were taken from a single plot and analysed separately, or where duplicate analyses of the same sample were made, the average of residue values from the trials conducted according to the ± 25% of maximum total seasonal application rate according to the GAP has been used for the estimation of maximum residue levels. Those results included in the tables are underlined.

Following the residue definition for spinetoram, the total residues were calculated following the same approach of the 2008 JMPR. Total residues for estimation of the maximum residue levels were calculated by summing up the concentrations of spinetoram-J and spinetoram-L, as illustrated below.

Spinetoram-J (mg/kg)	Spinetoram-L (mg/kg)	Total (mg/kg)
<0.01	<0.01	<0.01
0.05	<0.01	0.05
0.06	0.02	0.08

Total residues for estimation of the STMRs were calculated by summing up the concentrations of spinetoram (spinetoram-J + spinetoram-L), N-demethyl-J and N-formyl-J, for each combination of parent and metabolite, as illustrated below.

Spinetoram-J	Spinetoram-L	Spinetoram-*N*-demethyl-J	Spinetoram-*N*-formyl-J	Total
<0.01	<0.01	<0.01	<0.01	<0.02
0.05	<0.01	<0.01	<0.01	0.06
<0.01	<0.01	<0.01	0.05	0.06
0.05	<0.01	<0.01	0.05	0.10
0.06	0.02	0.02	0.06	0.16

Assorted (sub) tropical fruits – inedible peel - cactus

Dragon fruit

Four supervised trials on dragon fruit were conducted in the USA during 2015–2016 following the GAP in the USA (Barney, W.P, 2017, IR-4 PR No. 11514). Plots were treated with 4 foliar applications of a DW formulation at rates approximating 105 g ai/ha; re-treatment intervals were between 3–6 days. Adjuvants were used in the application. Commercially mature dragon fruit were collected 1 day following the last application in three trials. Samples were collected in a decline trial at 0, 1, 3, 7, 10 and 14 days after the last application. Samples comprised of 12 fruits and duplicate samples were taken. Samples were stored for maximum period of 318 days prior to analysis, covered by storage stability for 342 days, with exception of one sample which was stored for 362 days.

Residues of spinetoram and its metabolites were determined using a method equivalent to GRM 05.03 and 05.04, which had been validated with a LOQ of 0.01 mg/kg for each analyte. Results of the trials are summarized in Table 5.

Table 6 Residues of spinetoram from supervised trials on dragon fruit treated with a WG formulation in the USA (IR-4 PR 11514)

Location Country, year (Variety) Trial no.	Application			Total/ season g ai/ha	DALA, days	Residue, mg/kg					
	g ai/ha	L water/ha	No			Spinetoram-J	Spinetoram-L	*N*-demethyl-J	*N*-formyl-J	Total for MRL [a*]	Total diet [b*]
GAP, USA	70-123	-	4	420	1						
Homestead FL, USA 2015 (Rosa) (FL166)	110 110 112 110	1225 1216 1244 1216	4	442	0	0.058 0.057	0.022 0.021	0.022 0.022	0.029 0.028	0.080 0.078	0.131 0.127
					1	0.029 0.034	0.012 0.013	0.017 0.019	0.033 0.034	0.041 0.047 (0.044)	0.090 0.100 (0.095)
					3	0.028 0.021	0.011 <0.01	0.019 0.014	0.036 0.032	0.039 0.021	0.094 0.067
					7	0.017 0.012	<0.01 <0.01	0.012 <0.01	0.019 0.013	0.017 0.012	0.048 0.025
					10	0.011 0.015	<0.01 <0.01	<0.01 0.010	0.010 0.017	0.011 0.015	0.021 0.042
					14	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	0.013 <0.01	<0.01 <0.01	0.023 <0.02
Homestead FL, USA 2015 (Rosa) (FL167)	107 106 107 106	1187 1169 1187 1178	4	427	1	0.037 0.033	0.012 0.012	0.015 0.014	0.026 0.027	0.049 0.045 (0.047)	0.090 0.086 (0.088)
Homestead FL, USA 2016 (Rosa) (FL518)	114 112 108 107	1216 1197 1159 1150	4	441	1	0.015 0.014	<0.01 <0.01	<0.01 <0.01	0.050 0.063	0.015 0.014 (0.015)	0.065 0.077 (0.071)
Waialua, HI, USA 2015 (Kona Brazil) (HI204)	107 108 108 106	430 439 430 421	4	429	1	0.161 0.160	0.043 0.043	0.099 0.087	0.373 0.319	0.204 0.203 (0.20)	0.676 0.609 (0.64)

[a] Total MRL = spinetoram-J +spinetoram-L

[b] Total diet = spinetoram-J +spinetoram-L +N-demethyl-J + N-formyl-J

* Residues in parenthesis are the mean of two samples

Teas

Eight supervised trials on tea were conducted in Japan during 2011 and 2015 (Nanba, T., 2012, JP2011C289 / RLR-0411 and Ishizuka, H., 2016, JP2015C267 / RLR-0454). Plots were treated with 1 application of SC formulation at a rate approximating 180 g ai/ha. All trials were decline trials. Samples comprised of a

minimum of ca. 1000–2000g of fresh tea which was subsequently steamed (45 or 50 seconds with a conveyor belt-type steamer) and dried (80 or 90 minutes at target temperature of 80 °C using a shelf dryer) to form dried green tea. Samples were stored under frozen conditions for a maximum period of 139 days prior to analysis for spinetoram J and L which is within the demonstrated storage stability period of 166 days.

Table 7 Residues of spinetoram from supervised trials on dried green tea in Japan (for estimation of MRL)

Location, Country, year (Variety)	Form	Application/ treatment			DALA	Mean residue, mg/kg [b]			Report No.
		g ai/hL	L water/ha	No		Spinetoram-J	Spinetoram-L	Total [a]	
GAP, Japan	SC	4.68	-	1	1				
JPPA Kochi. Japan 2011 (Yabukita)	SC	4.68	3800	1	1	23.4	7.68	31.1	RLR-0411
					3	1.38	0.34	1.72	
					7	0.75	0.18	0.93	
					14	0.05	<0.01	0.05	
Kagoshima tea, Japan 2011 (Yamamidori)	SC	4.68	3846	1	1	9.66	2.86	12.5	
					3	3.36	0.91	4.27	
					7	0.29	0.07	0.36	
					14	0.02	<0.01	0.02	
Saitama-Tea, Japan 2015 (Fukumidori)	SC	4.68	3330	1	1	12.6	3.12	15.7	RLR-0454
					3	2.22	0.48	2.70	
					7	0.46	0.12	0.58	
JPPA Ibaraki, Japan 2015 (Yabukita)	SC	4.68	3100	1	1	16.0	3.96	20.0	
					3	1.52	0.34	1.86	
					7	0.96	0.18	1.14	
JPPA Chiba, Japan 2015 (Yabukita)	SC	4.68	3920	1	1	16.4	3.58	20.0	
					3	9.20	1.84	11.0	
					7	0.25	0.05	0.30	
JPPA Kochi. Japan, 2015 (Yabukita)	SC	4.68	3780	1	1	26.0	6.43	32.4	
					3	2.99	0.64	3.63	
					7	1.66	0.32	1.98	
JPPA Miyazaki, Japan 2015 (Yabukita)	SC	4.68	3330	1	1	32.3	8.13	40.4	
					3	2.97	0.66	3.63	
					7	0.50	0.10	0.60	
JPPA Kagoshima-Tea, Japan 2015 (Yamamidori)	SC	4.68	3850	1	1	4.94	1.14	6.08	
					3	0.28	0.06	0.34	
					7	0.42	0.06	0.48	

[a] Total residue = spinetoram-J + spinetoram-L

[b] duplicate analyses therefore mean results reported

Fate on residues during processing

The Meeting received trials on the processing of tea.

Tea

Nine grams of dry green tea without pulverization from above supervised field trials (RLR-0411) were weighed into an Erlenmeyer flask and 540 mL of boiling water was added. After 5 minutes, the filtrate was obtained as the infusion. Residues of spinetoram (L and J) in dry tea and tea infusions were determined using methods which are described above and have been validated with a LOQ of 0.01 mg/kg.

Table 7 Transfer of residues in green tea infusion from dry green tea (RLR-0411)

Country, year (Variety)	Application/ treatment			DALA	Mean residue (dried tea), mg/kg [b]			Mean residue (tea infusion), mg/kg		
	g ai/hL	L water/ha	No		Spinetoram-J	Spinetoram-L	Total [a]	Spinetoram-J	Spinetoram-L	Total [a]
GAP, Japan	4.68	-	1	1						
JPPA Kochi. Japan 2011 (Yabukita)	4.68	3800	1	1	23.4	7.68	31.08	0.32	0.08	0.40
				3	1.38	0.34	1.72	0.02	<0.01	<0.03
				7	0.75	0.18	0.93	<0.01	<0.01	<0.02
				14	0.05	<0.01	0.06	<0.01	<0.01	<0.02
Kagoshima tea, Japan 2011 (Yamamidori)	4.68	3846	1	1	9.66	2.86	12.52	0.20	0.05	0.25
				3	3.36	0.91	4.27	0.08	0.02	0.10
				7	0.29	0.07	0.36	0.02	<0.01	<0.03
				14	0.02	<0.01	0.03	<0.01	<0.01	<0.02

[a] Total residue = spinetoram-J + spinetoram-L

[b] duplicate analyses therefore mean results reported

Residues of spinetoram J and L are not concentrated in tea infusion.

APPRAISAL

Spinetoram is a spinosyn insecticide obtained by chemical modification of a fermentation product of Saccharopolyspora spinose. It consists of two closely related active ingredients, spinetoram-J and spinetoram-L, present in approximately a three to one ratio.

It was first evaluated by the 2008 JMPR (T, R), which established an ADI of 0–0.05 mg/kg bw and an ARfD was unnecessary. The 2008 JMPR also concluded the residue definition of spinetoram for plant and animal commodities:

Definition of the residue for compliance with the MRL for plant and animal commodities: *Spinetoram*.

Definition of the residue for dietary risk assessment for plant and animal commodities: *Spinetoram and N-demethyl and N-formyl metabolites of the major spinetoram component*.

The residue is fat-soluble.

Spinetoram was subsequently reviewed by 2012 and 2017 JMPR for additional uses. Codex MRLs have been established for a wide range of crops and products of animal origin.

The Forty-third Codex Alimentarius Commission (2020) approved the new work proposals including the priority list of pesticides for evaluation by the current Meeting. The priority list included spinetoram for evaluation of additional uses on pitaya (dragon fruit) and tea.

The current Meeting received GAP information and supervised residue trials on pitaya (dragon fruit) and tea.

Methods of analysis

The Meeting received descriptions and validation data for analytical methods for residues of spinetoram J and L in dried tea leaves and tea infusion along with the supervised trials.

The dry tea was first swollen by water, extracted with methanol/water (8:2, v/v), purified using polymer-based cartridges and an NH2 cartridge column, and quantified by LC/MS/MS. Tea infusions were extracted with acetonitrile/water (1:1, v/v), purified on an SPE column, and analysed by LC-MS/MS. The LOQ of 0.01 mg/kg was successfully validated for spinetoram-J and spinetoram-L.

The methods are considered suitable for analysis of spinetoram J and L in dry green tea and tea infusions.

GRM 05.03 and GRM 05.04, already reviewed by the 2008 JMPR, were satisfactorily validated to determine spinetoram-J and spinetoram-L and the metabolites N-demethyl-spinetoram-J and N-formyl-spinetoram-J in pitaya, with a LOQ of 0.01 mg/kg.

Stability of residues in stored analytical samples

The Meeting received frozen storage stability data for spinetoram and its N-demethyl and N-formyl metabolites in pitaya and dry green tea included in supervised trials. The Meeting concluded that spinetoram and its N-demethyl-spinetoram-J and N-formyl-spinetoram-J metabolites were stable for at least 11 months (342 days) in pitaya, and spinetoram was stable for at least 5 months (166 days) in dry green tea samples stored frozen at -20 °C.

The demonstrated storage stability intervals covered the duration of storage in the residue trials evaluated by the current Meeting, except for one sample in the pitaya trials stored for 362 days, which is less than 10% beyond the covered period of 342 days and was considered unlikely to have significant impact on the residues.

Results of supervised residue trials on crops

The Meeting received information on supervised trials of spinetoram on pitaya (dragon fruits) and tea.

Pitaya

The critical GAP for spinetoram on pitaya in the USA is 4 applications at 123 g ai/ha with an interval of 4 days and a PHI of 1 day.

Four trials were conducted on pitaya in the USA with four applications at rates approximating 110 g ai/ha, and re-treatment intervals of 3–6 days and a PHI of 1 day. The residues of spinetoram (J and L) in fruits were (n = 4): 0.015, 0.044, 0.047 and 0.20 mg/kg. The residues of spinetoram-J, spinetoram-L, N-demethyl-spinetoram-J and N-formyl spinetoram-J were (n = 4): 0.071, 0.088, 0.095 and 0.64 mg/kg.

The Meeting estimated a maximum residue level of 0.5 mg/kg and a STMR of 0.0915 mg/kg for spinetoram in pitaya.

Tea, Green, Black (black, fermented and dried)

The critical GAP for spinetoram on tea in Japan is 1 application at 4.68 g ai/hL (2500 times dilution with spray volume of 4000 L/ha) and a PHI of 1 day.

Eight trials were conducted on tea in Japan matching the critical GAP. The residues of spinetoram (J and L) in dry green tea were (n = 8): 6.1, 12.5, 16, 20(2), 31, 32, and 40 mg/kg. The residues of N-demethyl-spinetoram-J and N-formyl spinetoram-J were not analysed.

The Meeting estimated a maximum residue level of 70 mg/kg for spinetoram in tea, green, black (black, fermented and dried).

In an apple metabolism study evaluated by the 2008 JMPR (one foliar application at a rate of 1.8 kg ai/ha), the residues of spinetoram-J in leaves at 1 day after application were 78.6% TRR, while the residues of N-demethyl and N-formyl spinetoram-J were 12.8% TRR, the ratio of the two metabolites to spinetoram-J was 0.16.

The Meeting noted that tea infusions represent a small part of the diet and their contribution to the overall exposure is negligible. In trials on leek, spring onion and spinach reported previously by the JMPR (2012 and 2017) the ratio of N-demethyl-spinetoram-J to spinetoram J ranged from 0.07 to 0.63 and for N-formyl-spinetoram J to spinetoram J from 0.31 to 4.5. If the metabolites are assumed to be present at 1× and 5× the level of spinetoram J, a conservative assumption, the STMR would be 20+16.2+5*16.2 = 118 mg/kg. The latter could be used in estimating exposure in the absence of measured residues.

The Meeting estimated a STMR of 118 mg/kg for spinetoram in tea, green, black (black, fermented and dried).

Fate of residues during processing

The Meeting received processing studies for tea infusion. However, as not all the metabolites included in the residue definition for risk assessment were measured, the data could not be used to estimate processing factors for tea infusion.

RECOMMENDATIONS

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

Definition of the residue for compliance with the MRL for plant and animal commodities: *Spinetoram*.

Definition of the residue for dietary risk assessment for plant and animal commodities: *Spinetoram and N-demethyl and N-formyl metabolites of the major spinetoram component*.

The residue is fat-soluble.

Table 1 Recommendations for residues of spinetoram from the 2021 Extra JMPR

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg
		New	Previous	
FI 2540	Pitaya	0.5		0.0915
DT1114	Tea, Green, Black(black, fermented and dried)	70		118

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for spinetoram is 0–0.05 mg/kg bw. The International Estimated Daily Intakes (IEDIs) for spinetoram were estimated for the 17 GEMS/Food Consumption Cluster Diets using the STMR or STMR-P values estimated by the JMPR. The results are shown in Annex 3 of the 2021 Extra JMPR Report. The IEDIs ranged from 2–20% of maximum ADI of 0.05 mg/kg bw. The Meeting concluded that the long-term dietary exposure to residues of spinetoram from uses considered by the JMPR is unlikely to present a public health concern.

Acute dietary exposure

The 2008 JMPR decided that an ARfD for spinetoram was unnecessary. The Meeting therefore concluded that the acute dietary exposure to residues of spinetoram from uses considered by the current Meeting is unlikely to present a public health concern.

REFERENCES

Reference Number	Author(s)	Year	Study Title
IR-4 PR No. 11514	Barney, W.P.	2017	Spinetoram: Magnitude of the Residue on Dragon Fruit (Pitaya) IR-4 Project Headquarters, USA GLP, unpublished 17 July 2017
JP2011C289 / RLR-0411	Nanba, T.	2012	Study on Magnitude of the Residue of Spinetoram (DIANA®) Flowable in Crop, Tea Japan Plant Protection Association GLP, unpublished 24 January 2012 (Japanese with English translation)
JP2015C267 / RLR-0454	Ishizuka, H.	2016	Spinetoram (DIANA®) SC Tea, Magnitude of Residue Study Japan Plant Protection Association GLP, unpublished 12 February 2016 (Japanese with English translation)

SPIROMESIFEN (294)

First draft prepared by Dr M Lee, Andong National University, Republic of Korea

EXPLANATION

Spiromesifen is a contact insecticide-acaricide belonging to the titronic acid class of compounds. The mode of action is inhibition of lipid biosynthesis, especially triglycerides and free fatty acids.

Spiromesifen was first evaluated by the 2016 JMPR where an ADI of 0–0.03 mg/kg bw was established and an ARfD was determined to be unnecessary. The residue definition for compliance with the MRL for plant and animal commodities and for dietary risk assessment for animal commodities is the *sum of spiromesifen and spiromesifen-enol, expressed as spiromesifen*. For dietary risk assessment for plant commodities, the residue definition is the *sum of spiromesifen, spiromesifen-enol and 4-hydroxymethyl-spiromesifen-enol (free and conjugated), expressed as spiromesifen. The residue is fat-soluble.*

Spiromesifen was scheduled at the Fifty-first Session of the CCPR for evaluation of additional uses by the 2020 JMPR, which was postponed to the 2021 Extra JMPR. The Meeting received information on GAP and residue trials on chili pepper and okra.

RESIDUE ANALYSIS

Analytical methods

Spiromesifen residues (parent only) were analysed. Chili pepper and okra samples were homogenized at 3,000 rpm for 2 minutes. Representative 15 g homogenized samples were subjected to QuEChERS sample preparation method: add acetonitrile (containing 1% acetic acid) and salts (magnesium sulphate, sodium acetate), shake, centrifuge, and then clean-up of the organic layer by dispersive solid-phase extraction. Determination of the analyte was conducted by LC-MS/MS (mass transition: m/z 371>273 for quantification; m/z 371>255 for confirmation) or GC-MS (SIM mode). Matrix-matched standards at seven different concentrations, showing linearity $r^2 \geq 0.99$, were used. Recovery tests were performed at three fortification levels (n=3 at 0.05, 0.25 and 0.5 mg/kg). Mean recovery value at each fortification level/test ranged from 70–100% (RSDs, ≤16%) in chili pepper and 83–114% (RSDs, ≤9%) in okra samples. The LOQ values (LC-MS/MS and GC-MS) for spiromesifen were 0.05 mg/kg in the two matrices. Table 1 shows the results of recovery test.

Table 1 Recovery test results for spiromesifen in chili pepper and okra

Sample material	Trial location	Fortification level, mg/kg	Individual values, %	Mean value, %	RSD, %	Instrument used
Chili pepper, green	Anand	0.05	110.0, 98.0, 92.0	100	9	LC-MS/MS
		0.25	93.6, 86.4, 103	94	9	
		0.5	87.8, 90.8, 102	93	8	
	Solan	0.05	80.0, 100, 100	93	12	LC-MS/MS
		0.25	112, 88.0, 84.0	95	16	
		0.5	86.0, 82.0, 102	90	12	
	New Delhi	0.05	73.5, 71.3, 74.1	73	2	GC-MS
		0.25	85.9, 88.1, 89.3	88	2	
		0.5	94.2, 94.6, 97.5	95	2	
	Vellayani	0.05	72.4, 70.9, 72.6	72	1	LC-MS/MS
		0.25	69.7, 71.2, 69.1	70	2	

Sample material	Trial location	Fortification level, mg/kg	Individual values, %	Mean value, %	RSD, %	Instrument used
		0.5	81.9, 78.8, 79.4	80	2	
	Ludhiana	0.05	90.2, 86.9, 85.7	88	3	GC-MS
		0.25	85.2, 87.1, 87.3	87	1	
		0.5	91.6, 92.4, 93.5	93	1	
	Hisar	0.05	88.7, 93.1, 89.4	90	3	GC-MS
		0.25	94.5, 92.1, 90.4	92	2	
		0.5	94.2, 95. 6, 97.5	96	2	
Okra	Anand	0.05	101, 107, 101	103	3	LC-MS/MS
		0.25	102, 120, 118	114	9	
		0.5	112, 107, 120	113	6	
	Solan	0.05	94.6, 89.2, 92.1	92	3	GC-MS
		0.25	102, 98.6, 102	101	2	
		0.5	100, 98.1, 102	100	2	
	New Delhi	0.05	82.6, 81.7, 83.9	83	1	LC-MS/MS
		0.25	86.2, 87.2, 89.3	88	2	
		0.5	95.6, 96.2, 99.2	97	2	
	Vellayani	0.05	102, 110, 106	106	4	LC-MS/MS
		0.25	90.0, 92.0,94.0	92	2	
		0.5	89.0, 86.0, 83.0	86	3	
	Ludhiana	0.05	91.1, 90.3, 86.2	89	3	GC-MS
		0.25	96.4, 92.0, 94.6	94	2	
		0.5	89.0, 86.4, 87.9	88	1	
	Hyderabad	0.05	96.0, 88.0, 104	96	8	
		0.25	94.4, 88.4, 92.0	92	3	
		0.5	85.0, 92.8, 86.2	88	5	
	Coimbatore	0.05	96.4, 97.8, 95.8	97	1	LC-MS/MS
		0.25	102, 99.2, 101	100	1	
		0.5	105, 101, 100	102	2	

All LOQ values, <0.05 mg/kg

USE PATTERNS

The Meeting received the GAP information on chili pepper and okra from India. The information is summarised in Table 2.

Table 2 Registered uses of spiromesifen on chili pepper and okra in India

Crop	Formulation	Application			PHI (days)
		Method	kg ai/ha	Dilution in water, L/ha	
Chili pepper	22.9% w/w SC	Foliar spray	0.096	500-750	7
Okra	22.9% w/w SC	Foliar spray	0.096-0.12	500	3
Eggplant	22.9% w/w SC	Foliar spray	0.096	500	5

Number of sprays and an application interval: not specified

RESULTS OF SUPERVISED RESIDUE TRIALS ON CROPS

The Meeting received residue trials on chili pepper and okra conducted in India. The detailed information are summarised in Tables 3 and 4 below.

CODEX Group	Commodity	Table No.
Group 012 Fruiting vegetables, other than Cucurbits	Subgroup 012B Pepper and pepper-like commodities	
	VO 0444 Peppers, chili	Table 3
	VO 0442 Okra	Table 4

Fruiting vegetables, other than Cucurbits

Supervised field trials on chili pepper and okra were conducted in India under All India Network Project on Pesticide Residues. Each trial comprised three plots per treatment and one control plot. Samples, separately collected from each plot, were extracted immediately (within 24 hours) after sampling and analysed. In all control samples, spiromesifen was not detected.

Peppers, chili

Residue trials were conducted at six locations during 2015–2017 [Sharma, K.K., 2019*]. Foliar spray application (500 L/ha) of the SC formulation (22.9%, w/w) was made at a rate of 0.096 kg ai/ha in 2 applications (first spray at fruit initiation stage followed by second spray after 10 days). Green chili fruit samples (400-600 g) were taken at 0, 1, 3, 5, 7, 10, (15), (20) days after the last application.

Okra

Residue trials were conducted at seven locations during 2015–2018 [Sharma, K.K., 2019†]. Foliar spray application (500 L/ha) of the SC formulation (22.9%, w/w) was made at a rate of 0.096–0.12 kg ai/ha in 2 applications (first spray at fruit initiation stage followed by second spray after 10 days). Samples of okra (1 kg) were taken at 0, 1, 3, (5), (7), (10), (15), (20) days after the last application.

Table 3 Residue concentrations of spiromesifen from residue trials on green chili pepper in India (replicate plot)

Location, Year (Variety)	Application			DALA	Spiromesifen, mg/kg			
	n	kg ai/ha	Int. days		Individual value			Mean
GAP: India		0.096		PHI, 7 days				
Anand, 2016 (Sitara)	2	0.096	10	0	1.6	2.0	1.8	1.8
				1	1.3	1.3	1.1	1.2
				3	1.1	1.2	1.00	1.1
				5	0.60	0.70	0.62	0.64
				7	0.57	0.68	0.52	0.59
				10	0.42	0.35	0.44	0.40
				15	0.27	0.25	0.25	0.26
				20	<0.05	<0.05	<0.05	<0.05
Solan, 2015 (DKC-8)				0	0.73	0.72	0.73	0.73
				1	0.39	0.38	0.39	0.39
				3	0.21	0.20	0.20	0.20
				5	0.11	0.11	0.10	0.11
				7	0.05	0.07	0.06	0.060
				10	<0.05	<0.05	<0.05	<0.05
New Delhi, 2017 (Pusa Sadabahar)	2	0.096	10	0	0.44	0.41	0.39	0.41
				1	0.32	0.26	0.28	0.29

Location, Year (Variety)	Application			DALA	Spiromesifen, mg/kg			
	n	kg ai/ha	Int. days		Individual value			Mean
				3	0.19	0.16	0.13	0.16
				5	0.05	0.05	0.05	0.050
				7	<0.05	<0.05	<0.05	<0.05
				10	<0.05	<0.05	<0.05	<0.05
				15	<0.05	<0.05	<0.05	<0.05
				20	<0.05	<0.05	<0.05	<0.05
Vellayani, 2016 (Vellayani Athulya)	2	0.096	10	0	0.91	0.92	0.92	0.92
				1	0.64	0.70	0.71	0.68
				3	0.58	0.59	0.57	0.58
				5	0.53	0.52	0.54	0.53
				7	0.21	0.22	0.22	0.22
				10	0.20	0.21	0.21	0.21
				15	<0.05	<0.05	<0.05	<0.05
Ludhiana, 2015 (CH-3)	2	0.096	10	0	1.3	1.3	1.2	1.3
				1	0.48	0.43	0.55	0.49
				3	0.25	0.27	0.31	0.28
				5	0.19	0.18	0.16	0.18
				7	0.05	0.06	0.07	0.060
				10	<0.05	<0.05	<0.05	<0.05
				15	<0.05	<0.05	<0.05	<0.05
				20	<0.05	<0.05	<0.05	<0.05
Hisar, 2015 (Kanshi Anmol)	2	0.096	10	0	2.0	2.0	2.0	2.0
				1	1.5	1.1	0.72	1.1
				3	0.78	0.41	0.58	0.59
				5	0.61	0.34	0.47	0.47
				7	0.41	0.32	0.36	0.36
				10	0.21	0.20	0.19	0.20
				15	0.06	0.05	0.05	0.050
				20	<0.05	<0.05	<0.05	<0.05

SC 22.9% formulation was applied.

Table 4 Residue concentration of spiromesifen from residue trials on okra in India (replicate plot)

Location, Year (Variety)	Application			DALA	Spiromesifen, mg/kg			
	n	kg ai/ha	Int. days		Individual value			Mean
GAP: India		0.096-0.12		PHI, 3 days				
Anand, 2016 (GAO-5)	2	0.096	10	0	0.64	0.52	0.53	0.56
				1	0.20	0.18	0.15	0.18
				3	<0.05	<0.05	<0.05	<0.05
				5	<0.05	<0.05	<0.05	<0.05
				7	<0.05	<0.05	<0.05	<0.05
				10	<0.05	<0.05	<0.05	<0.05
Solan, 2015 (Punjab-8)	2	0.12	10	0	1.3	1.4	1.3	1.3

Location, Year (Variety)	Application			DALA	Spiromesifen, mg/kg			
	n	kg ai/ha	Int. days		Individual value			Mean
				1	0.72	0.83	0.71	0.75
				3	0.45	0.45	0.48	0.46
				5	0.23	0.24	0.24	0.23
				7	0.04	0.07	0.05	0.050
				10	<0.05	<0.05	<0.05	<0.05
New Delhi, 2016 (Nitya)	2	0.12	10	0	0.42	0.46	0.42	0.43
				1	0.30	0.28	0.26	0.28
				3	0.17	0.15	0.14	0.15
				5	0.06	0.05	0.05	0.05
				7	<0.05	<0.05	<0.05	<0.05
				10	<0.05	<0.05	<0.05	<0.05
				15	<0.05	<0.05	<0.05	<0.05
				20	<0.05	<0.05	<0.05	<0.05
Vellayani, 2016 (-)	2	0.12	10	0	0.22	0.26	0.22	0.23
				1	0.13	0.13	0.13	0.13
				3	<0.05	<0.05	<0.05	<0.05
Ludhiana, 2015 (Punjab-8)	2	0.12	10	0	0.45	0.51	0.41	0.46
				1	0.20	0.25	0.27	0.24
				3	0.12	0.14	0.17	0.14
				5	0.06	0.08	0.09	0.080
				7	<0.05	<0.05	<0.05	<0.05
				10	<0.05	<0.05	<0.05	<0.05
				15	<0.05	<0.05	<0.05	<0.05
				20	<0.05	<0.05	<0.05	<0.05
Hyderabad, 2015 (Arka Anamika)	2	0.12	10	0	1.8	1.6	1.8	1.7
				1	0.90	1.2	1.1	1.1
				3	0.80	0.74	0.77	0.77
				5	0.30	0.21	0.21	0.24
				7	0.06	0.07	0.06	0.060
				10	<0.05	<0.05	<0.05	<0.05
				15	<0.05	<0.05	<0.05	<0.05
				20	<0.05	<0.05	<0.05	<0.05
Coimbatore, 2018 (Samrat)	2	0.12	10	0	1.2	1.2	1.2	1.2
				1	0.52	0.37	0.55	0.48
				3	<0.05	<0.05	<0.05	<0.05

SC 22.9% formulation was applied.

APPRAISAL

Spiromesifen is a contact insecticide-acaricide belonging to the titronic acid class of compounds. The mode of action is inhibition of lipid biosynthesis, especially triglycerides and free fatty acids.

Spiromesifen was first evaluated by the 2016 JMPR where an ADI of 0–0.03 mg/kg bw was established and an ARfD was determined to be unnecessary. The residue definition for compliance with the MRL for plant and animal commodities and for dietary risk assessment for animal commodities is sum of spiromesifen and spiromesifen-enol, expressed as spiromesifen. For dietary risk assessment for plant commodities, the residue definition is sum of spiromesifen, spiromesifen-enol and 4-hydroxymethyl-spiromesifen-enol (free and conjugated), expressed as spiromesifen. The residue is fat-soluble.

Spiromesifen was scheduled at the Fifty-first Session of the CCPR for the evaluation of additional uses by the 2020 JMPR, which was postponed to the 2021 Extra JMPR. The Meeting received information on GAP and residue trials on chili pepper and okra.

Methods of analysis

Spiromesifen residues (parent only) in green chili pepper and okra were analysed based on the method previously evaluated by the JMPR. The method involved QuEChERS sample preparation method and determination of spiromesifen by LC-MS/MS or GC-MS. Recovery tests for the method validation showed mean recoveries of 70–100% in chili pepper and 83–114% in okra. The LOQ values (LC-MS/MS and GC-MS) for spiromesifen were 0.05 mg/kg in chili pepper and okra. The analytical method used in the chili pepper and okra residue trials was considered sufficiently validated.

Stability of residues in stored analytical samples

Residue analysis was performed within 24 hours after sample collection.

Results of supervised residue trials on crops

Fruiting vegetables, other than Cucurbits

Chili pepper

The critical GAP for the use of spiromesifen on chili pepper in India involves foliar sprays at 0.096 kg ai/ha and a 7-day PHI (the maximum number of sprays and minimum re-treatment intervals are not specified).

Six trials on field-grown chili peppers (decline trials) were conducted in India during 2015–2017 at a rate of 0.096 kg ai/ha with 2 applications at a 10-day interval and a PHI of 7 days. The residue concentrations of spiromesifen in green chili peppers were (n = 6): < 0.05, 0.060, 0.060, 0.22, 0.36 and 0.59 mg/kg.

Communication by the sponsor indicated that the local agricultural practice involves re-treatment intervals of 2–4 days. Residues declined with a median half-life of 4.07 days (1st order) and the modelled residue at a spray interval of 2 days and 2 applications differed by more than 25% compared with the supervised residue field trials. The Meeting concluded that the supervised field trials were conducted at a significantly longer re-treatment interval and cannot be used for the estimation of a maximum residue level.

Okra

The critical GAP for the use of spiromesifen on okra in India involves foliar sprays at 0.12 kg ai/ha and a 3-day PHI (the maximum number of sprays and minimum re-treatment intervals are not specified).

Seven trials on field-grown okra (decline trials) were conducted in India during 2015–2018 at a rate of 0.096–0.12 kg ai/ha with 2 applications at a 10-day interval and a PHI of 3 days. The residue concentrations of spiromesifen in okra were (n = 7): < 0.05 (3), 0.14, 0.15, 0.46 and 0.77 mg/kg.

Communication by the sponsor indicated that the local agricultural practice involves re-treatment intervals of 2–4 days. Residues declined with a median half-life of 2.41 days (1st order) and the modelled residue at a spray interval of 2 days and 2 applications differed by more than 25% compared with the supervised residue field trials. The Meeting concluded that the supervised field trials were conducted at a significantly longer re-treatment interval and cannot be used for the estimation of a maximum residue level.

REFERENCES

Author	Year	Study title, Institute
*K.K. Sharma	2019	Data/Information for Fixation of MRL of Spiromesifen on Green Chili. All India Network Project on Pesticide Residues, ICAR-Indian Agricultural Research Institute, New Delhi-110012, India
+K.K. Sharma	2019	Data/Information for Fixation of MRL of Spiromesifen on Green Chili. All India Network Project on Pesticide Residues, ICAR-Indian Agricultural Research Institute, New Delhi-110012, India

SULFOXAFLOR (252)

First draft prepared by Dr M Doherty, the Environmental Protection Agency, United States of America

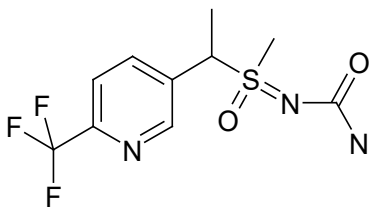
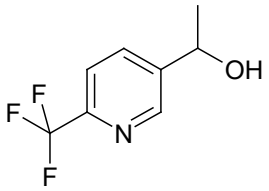
EXPLANATION

Sulfoxaflor (ISO common name) is a broad-spectrum, sulfoximine insecticide with registered uses on multiple crops. It was evaluated for the first time by the 2011 JMPR, which established an acceptable daily intake (ADI) of 0–0.05 mg/kg bw and an acute reference dose (ARfD) of 0.3 mg/kg bw. Sulfoxaflor underwent subsequent evaluations by the JMPR in 2014 and 2016.

The definition of the residue for compliance with the MRL and dietary risk assessment is *sulfoxaflor*. *The residue is not fat-soluble*.

Sulfoxaflor was scheduled at the Fifty-first Session of the CCPR for evaluation of additional uses by the 2020 JMPR, which was postponed to the 2021 Extra JMPR. The current Meeting received information on analytical methods, field trials, and processing studies to support estimation of new maximum residue levels in commodities of avocado, mango, bush berries, cane berries, globe artichoke, asparagus, sunflower, and coffee.

Table 1 Metabolites of sulfoxaflor referenced in this document

Common or code name	Chemical name	Structure
X11719474	1-[methyl(oxido){1-[6-(trifluoromethyl)pyridin-3-yl]ethyl}- λ^6-sulfanylidene]urea	
X11721061	1-[6-(trifluoromethyl)pyridin-3-yl]ethanol	

RESIDUE ANALYSIS

Analytical methods

Method 091116S1

This method was used for the determination of sulfoxaflor and its main metabolite residues in all submitted residue studies except for studies in mango and coffee. The method was evaluated previously by the 2011 JMPR and found to be acceptable.

QuEChERS

Field trial samples of mango and coffee were analysed for residues of sulfoxaflor using a standard QuEChERS method (Rawle, N. 2016. Report DAS 150108).

Concurrent recovery data for both methods are summarized below.

Table 2 Summary of validation (V) and concurrent (C) recovery of sulfoxaflor and its main metabolites from avocado, blueberry, caneberry, globe artichoke, asparagus, and sunflower commodities

Matrix	Analyte	Fortification level [mg/kg]	Individual recoveries [%]	Range of recoveries [%]	Mean recovery [%]	RSD [%]	Reference
Method 091116S1							
Avocado	Sulfoxaflor	0.01	V: 74, 92, 93 C: 86, 109, 109, 114, 118	74-93 86-118	86 107	12.4 11.6	DAS 30161
		5	C: 79, 90, 93, 96, 101	79-101	92	9.0	
		10	V: 84, 93, 94	84-94	90	6.1	
	X11719474	0.01	V: 80, 86, 92 C: 92, 100, 100, 103, 107	80-92 92-107	86 100	7.0 5.5	
		5	C: 77, 85, 89	77-89	84	7.3	
		10	V: 90, 94, 95	90-95	93	2.8	
	X11721061	0.01	V: 87, 88, 99 C: 92, 94, 103, 112, 113	87-99 92-113	91 103	7.3 9.5	
		5	C: 89, 92, 93, 110, 116	89-116	100	12.1	
		10	V: 89, 92, 95	89-95	92	3.3	
Blueberry	Sulfoxaflor	0.01	V: 107, 107, 107 C: 103, 104, 105, 105, 105, 113, 114	107-107 103-114	107 107	0.0 4.2	PR-11296
		0.1	V: 82, 83, 84 C: 82, 84, 85	82-84 82-85	83 84	1.2 1.8	
		1	V: 82, 83, 84 C: 82	82-84 82-82	83 82	1.2 --	
		2	V: 102, 102, 102	102-102	102	0.0	
	X11719474	0.01	V: 96, 97, 99 C: 89, 90, 94, 97, 99, 100, 101, 103, 103	96-99 89-103	97 97	1.6 5.4	
		0.1	V: 83, 85, 86 C: 82, 82, 85	83-86 82-85	85 83	1.8 2.1	
		1	V: 83, 83, 84 C: 81	83-84 81-81	83 81	0.7 --	
	X11721061	0.01	V: 100, 100, 102 C: 89, 95, 95, 97, 98, 98, 98, 99, 99	100-102 89-99	101 96	1.1 3.3	
		0.1	V: 83, 85, 85 C: 82, 82, 83	83-85 82-83	84 82	1.4 0.7	
		1	V: 84, 84, 85 C: 83	84-85 83-83	84 83	0.7 --	
Caneberry	Sulfoxaflor	0.01	V: 101, 109, 109 C: 98, 101, 106, 113	101-109 98-113	106 105	4.3 6.3	PR-11279
		0.1	V: 90, 93, 99 C: 86, 88	90-99 86-88	94 87	4.9 1.6	
		0.5	C: 90, 90, 93	90-93	91	1.9	
		1	V: 101, 101, 103 C: 100	101-103 100-100	102 100	1.1 --	

Matrix	Analyte	Fortification level [mg/kg]	Individual recoveries [%]	Range of recoveries [%]	Mean recovery [%]	RSD [%]	Reference
	X11719474	0.01	V: 90, 92, 94 C: 91, 93, 95, 95	90-94 91-95	92 94	2.2 2.0	
		0.1	V: 83, 84, 90 C: 80, 83	83-90 80-83	86 82	4.4 2.6	
		0.5	C: 88, 88, 89	88-89	88	0.7	
		1	V: 95, 96, 96 C: 94	95-96 94-94	96 94	0.6 --	
	X11721061	0.01	V: 96, 96, 100 C: 96, 99, 105, 107	96-100 96-107	97 102	2.4 5.0	
		0.1	V: 81, 86, 92 C: 82, 84	81-92 82-84	86 83	6.4 1.7	
		0.5	C: 88, 89, 89	88-89	89	0.7	
		1	V: 95, 95, 97 C: 95	95-97 95-95	96 95	1.2 --	
Globe artichoke	Sulfoxaflor	0.01	V: 106, 106, 107 C: 105, 111, 113	106-107 105-113	106 110	0.5 3.8	PR-10858
		0.1	V: 83, 84, 86 C: 84	83-86 84-84	84 84	1.8 --	
		2	V: 84, 84, 86 C: 89	84-86 89-89	85 89	1.4 --	
	X11719474	0.01	V: 96, 96, 98 C: 96, 100, 101	96-98 96-101	97 99	1.2 2.7	
		0.1	V: 81, 82, 83 C: 83	81-83 83-83	82 83	1.2 --	
		2	V: 82, 83, 83 C: 86	82-83 86-86	83 86	0.7 --	
	X11721061	0.01	V: 91, 92, 93 C: 87, 93, 94	91-93 87-94	92 91	1.1 4.1	
		0.1	V: 81, 82, 85 C: 83	81-85 83-83	83 83	2.5 --	
		2	V: 84, 84, 86 C: 92	84-86 92-92	85 92	1.4 --	
Asparagus	Sulfoxaflor	0.01	V: 107, 110, 110 C: 94, 95, 103, 103, 109, 109, 110, 111	107-110 94-111	109 104	1.6 6.5	PR-11321
		0.1	V: 88, 91, 92 C: 95	88-92 95-95	90 95	2.3 --	
	X11719474	0.01	V: 89, 93, 97 C: 87, 94, 95, 97, 98, 102, 104, 104	89-97 87-104	93 98	4.3 5.9	
		0.1	V: 80, 83, 83 C: 85	80-83 85-85	82 85	2.1 --	
	X11721061	0.01	V: 86, 92, 100	86-100	93	7.6	

Matrix	Analyte	Fortification level [mg/kg]	Individual recoveries [%]	Range of recoveries [%]	Mean recovery [%]	RSD [%]	Reference
			C: 100, 102, 102, 103, 103, 105, 107, 109	100-109	104	2.8	
		0.1	V: 79, 82, 85 C: 93	79-85 93-93	82 93	3.7 --	
Sunflower seed	Sulfoxaflor	0.01	V: 97, 97, 102 C: 96, 99, 100, 102, 102, 103, 111	97-102 96-111	99 102	2.9 4.6	PR-11095
		0.1	C: 84, 96, 95	84-96	92	7.3	
		0.5	V: 83, 85, 86 C: 85, 92	83-86 85-92	85 89	1.8 5.6	
		5	V: 93, 94, 96	93-96	94	1.6	
	X11719474	0.01	V: 86, 86, 86 C: 84, 86, 88, 91, 91, 93	86-86 84-93	86 89	0.0 3.9	
		0.1	C: 78, 85, 93	78-93	85	8.8	
		0.5	V: 84, 86, 87 C: 78, 88	84-87 78-88	86 83	1.8 8.5	
		5	V: 95, 95, 97	95-97	96	1.2	
	X11721061	0.01	V: 87, 88, 90 C: 82, 90, 91, 94, 94, 95, 101	87-90 82-101	88 92	1.7 6.3	
		0.1	C: 82, 89, 93	82-93	88	6.3	
		0.5	V: 85, 87, 88 C: 89, 90	85-88 89-90	87 90	1.8 0.8	
		5	V: 97, 97, 99	97-99	98	1.2	
Sunflower meal	Sulfoxaflor	0.01	V: 99, 102, 104 C: 90, 91, 91	99-104 90-91	102 91	2.5 0.6	
		0.1	C: 88, 90, 90	88-90	89	1.3	
		0.5	V: 86, 87, 89	86-89	87	1.7	
		5	V: 94, 94, 97	94-97	95	1.8	
	X11719474	0.01	V: 87, 87, 87 C: 86, 86, 87	87-87 86-87	87 86	0.0 0.7	
		0.1	C: 77, 85, 87	77-87	83	6.4	
		0.5	V: 86, 87, 89	86-89	87	1.7	
		5	V: 93, 95, 96	93-96	95	1.6	
	X11721061	0.01	V: 88, 89, 91 C: 85, 85, 86	88-91 85-86	89 85	1.7 0.7	
		0.1	C: 87, 87, 88	87-88	87	0.7	
		0.5	V: 87, 87, 88	87-88	87	0.7	
		5	96: 97, 98	97-98	98	0.7	
Sunflower refined oil	Sulfoxaflor	0.01	V: 95, 99, 99 C: 91, 93, 95	95-99 91-95	98 93	2.4 2.2	
		0.1	C: 88, 89, 95	88-95	91	4.2	
		0.5	V: 85, 85, 85	85-85	85	0.0	
		5	V: 94, 95, 98	94-98	96	2.2	

Matrix	Analyte	Fortification level [mg/kg]	Individual recoveries [%]	Range of recoveries [%]	Mean recovery [%]	RSD [%]	Reference
	X11719474	0.01	V: 79, 80, 80 C: 85, 86, 88	79-80 85-88	80 86	0.7 1.8	
		0.1	C: 76, 82, 87	76-87	82	6.7	
		0.5	V: 83, 83, 83	83-83	83	0.0	
		5	V: 91, 92, 96	91-96	93	2.8	
	X11721061	0.01	V: 83, 84, 84 C: 84, 85, 89	83-84 84-89	84 86	0.7 3.1	
		0.1	C: 87, 89, 91	87-91	89	2.2	
		0.5	V: 86, 86, 87	86-87	86	0.7	
		5	V: 94, 95, 98	94-98	96	2.2	
QuEChERS							
Mango (whole fruit)	Sulfoxaflor	0.01	V: 74, 83, 84, 87, 87, 91, 92, 92, 96 C: 77, 82, 91, 94, 94, 97	74-96 77-97	87 89	7.5 8.8	PR-11822
		0.02	V: 99, 101, 102 C: 82, 90, 90	99-102 82-90	101 87	1.5 5.3	
		0.1	V: 98, 100, 102, 102, 102, 107, 107, 110, 112 C: 87, 90, 90, 91, 91, 97, 98, 101	98-112 87-101	104 93	4.5 5.2	
		1	V: 92, 95, 96, 98, 101, 108, 109, 109, 110 C: 82, 83, 86, 93, 94, 94	92-110 82-94	102 89	6.9 6.4	
	X11719474	0.01	V: 75, 82, 90, 106, 110, 111, 127, 128, 131 C: 65, 72, 74, 90, 99, 99	75-131 65-99	107 83	19.3 17.7	
		0.02	V: 108, 108, 114 C: 84, 86, 87	108-114 84-87	110 86	3.1 1.8	
		0.1	V: 91, 94, 95, 97, 106, 107, 108, 112, 113 C: 95, 95, 96, 97, 97, 109	91-113 95-109	103 98	8.1 5.5	
		1	V: 86, 95, 96, 98, 98, 99, 101, 102, 107 C: 88, 91, 94, 96, 98, 102	86-107 88-102	98 95	5.9 5.3	
Mango (peel)	Sulfoxaflor	0.01	V: 69, 78, 79, 87, 95, 97 C: 84, 86, 87, 103	69-97 84-103	84 90	12.8 9.7	
		0.1	V: 97, 97, 98, 99, 99, 113 C: 84, 87, 91, 97, 99	97-113 84-99	101 92	6.2 7.0	
		1	V: 98, 101, 101, 108, 110, 111 C: 76, 88, 92	98-111 76-92	105 85	5.2 9.8	
	X11719474	0.01	V: 91, 92, 97, 105, 113, 115 C: 82, 87, 106	91-115 82-106	102 92	10.2 13.8	
		0.1	V: 73, 90, 94, 95, 107, 145 C: 83, 84, 89	73-145 83-89	101 85	24.2 3.8	
		1	V: 91, 93, 99, 109, 117, 120	91-120	105	11.8	

Matrix	Analyte	Fortification level [mg/kg]	Individual recoveries [%]	Range of recoveries [%]	Mean recovery [%]	RSD [%]	Reference
			C: 78, 85, 90	78-90	84	7.1	
Mango (flesh)	Sulfoxaflor	0.01	V: 75, 82, 89, 93, 104, 108, 109, 112 C: 92, 92, 94	75-112 92-94	97 93	14.3 1.2	
		0.1	V: 99, 102, 103, 106, 106, 108 C: 93, 101, 104	99-108 93-104	104 99	3.2 5.7	
		1	V: 102, 103, 104, 105, 110, 114 C: 99, 108, 114	102-114 99-114	106 107	4.4 7.1	
	X11719474	0.01	V: 98, 102, 103, 116, 119, 126 C: 86, 91, 95	98-126 86-95	111 91	10.1 5.0	
		0.1	V: 96, 98, 98, 100, 100, 101 C: 88, 100, 100	96-101 88-100	99 96	1.9 7.2	
		1	V: 90, 98, 99, 100, 100, 103	90-103	98	4.5	
			C: 96, 106, 108	96-108	103	6.2	
Coffee (dried beans w/ shell)	Sulfoxaflor	0.01	V: 99, 100, 101, 104, 106 C: 98, 103, 103, 104, 104, 105, 111, 126	99-106 98-126	103 107	3 8	109532
		0.1	V: 95, 96, 100, 100, 101 C: 103, 105, 109, 110, 111, 112	95-101 103-112	98 108	3 3	
		1.0	C: 100, 100, 101, 102, 102	100-102	101	1	
	X11719474	0.01	V: 96, 99, 100, 102, 103 C: 94, 98, 98, 99, 100, 100, 101, 101	96-103 94-101	100 99	3 2	
		0.1	V: 98, 100, 102, 104, 106 C: 94, 95, 100, 102, 103, 103	98-106 94-103	102 100	3 4	
		1.0	C: 96, 99, 100, 101, 102	96-102	100	2	
Coffee (roasted beans)	Sulfoxaflor	0.01	V: 98, 100, 103, 106, 111 C: 82, 87, 88, 91, 92, 94, 95, 95	98-111 82-95	104 91	5 5	
		0.1	V: 93, 94, 94, 96, 99 C: 97, 102, 104	93-99 97-104	95 101	3 4	
		5.0	97, 99, 100, 100, 100	97-100	99	1	
	X11719474	0.01	V: 93, 94, 96, 96, 98 C: 81, 84, 85, 88, 93	93-98 81-93	95 86	2 5	
		0.1	V: 89, 89, 89, 90, 96	89-96	91	3	
		5.0	C: 100, 100, 101, 101, 102	100-102	100	1	
Coffee (instant)	Sulfoxaflor	0.01	V: 83, 91, 92, 93, 95 C: 81, 90, 92, 96, 100, 101, 106, 109, 109, 110	83-95 81-110	91 99	5 10	
		0.1	V: 90, 91, 91, 93, 95 C: 91, 92, 95, 100, 100, 101, 105, 105	90-95 91-105	92 99	2 5	
		5.0	C: 91, 94, 96, 96, 101	91-101	96	4	
	X11719474	0.01	V: 82, 82, 82, 83, 86 C:76, 81, 90, 90, 91, 94, 96, 96, 98	82-86 76-98	83 90	2 8	
		0.1	V: 85, 85, 86, 87, 88	85-88	86	2	

Matrix	Analyte	Fortification level [mg/kg]	Individual recoveries [%]	Range of recoveries [%]	Mean recovery [%]	RSD [%]	Reference
			C: 80, 82, 89, 90, 90, 91, 93	80-93	88	6	
		5.0	C: 91, 91, 94, 94, 95	91-95	93	2	

Stability of residues in stored analytical samples

Data on the stability of sulfoxaflor and its two main metabolites was provided for blueberry, cane berry, artichoke, asparagus, and sunflower commodities. Data were generated concurrently with the field trials cited below. For all commodities, samples of control matrix were fortified with sulfoxaflor, X11719474, and X11721061, each at ca. 0.1 mg/kg (X11719474 at ca 0.2 mg/kg in some samples of sunflower seed, meal, and refined oil). All samples were analysed using Method 091116S1.

The results (Table 3) indicate that residues are stable in high water, high acid, and high oil content commodities during frozen storage for at least the storage durations examined (0.8 to 2 years, depending on the commodity).

Table 3 Summary of stability data for residues of sulfoxaflor under frozen conditions in various commodities

Matrix	Analyte	Storage time, days (years)	Procedural recovery, %	Fortification level, mg/kg	Residue remaining, mg/kg [mean]	Mean % remaining
Blueberry	Sulfoxaflor	756 (2.07)	83	0.0994	0.0895, 0.0905, 0.0914 [0.0905]	91
	X11719474		82	0.1	0.0840, 0.0850, 0.0850 [0.0847]	85
	X11721061		83	0.0994	0.130, 0.131, 0.131 [0.131]	130
Cane berry	Sulfoxaflor	549 (1.50)	88	0.117	0.0991, 0.106, 0.103 [0.103]	88
	X11719474		80	0.111	0.0910, 0.0946, 0.0942 [0.0933]	84
	X11721061		82	0.097	0.0756, 0.0788, 0.0791 [0.0778]	80
Artichoke	Sulfoxaflor	735 (2.01)	84	0.0994	0.0895, 0.0885, 0.0905 [0.0895]	90
	X11719474		83	0.1	0.0800, 0.0820, 0.0840 [0.0820]	82
	X11721061		83	0.0994	0.133, 0.133, 0.137 [0.135]	140
Asparagus	Sulfoxaflor	304 (0.83)	95	0.116	0.104, 0.106, 0.105 [0.105]	91
	X11719474		85	0.111	0.101, 0.103, 0.0984 [0.101]	91
	X11721061		93	0.0972	0.0895, 0.0862, 0.0845 [0.0868]	89
Sunflower seed	Sulfoxaflor	575 (1.58)	95	0.1	0.108, 0.110, 0.108 [0.109]	110
		597 (1.64)	93		0.110, 0.113, 0.112 [0.112]	110
		736 (2.02)	84		0.110, 0.110, 0.110 [0.110]	110
	X11719474	575 (1.58)	93	0.2	0.206, 0.210, 0.210 [0.209]	100
		597 (1.64)	85		0.204, 0.208, 0.206 [0.206]	100
		736 (2.02)	78		0.198, 0.200, 0.198 [0.199]	99
	X11721061	575 (1.58)	93	0.0982	0.117, 0.117, 0.116 [0.117]	120

Matrix	Analyte	Storage time, days (years)	Procedural recovery, %	Fortification level, mg/kg	Residue remaining, mg/kg [mean]	Mean % remaining
		597 (1.64)	89		0.117, 0.117, 0.120 [0.118]	120
		736 (2.02)	82		0.117, 0.117, 0.118 [0.117]	120
Sunflower meal	Sulfoxaflor	535 (1.47)	90	0.1	0.113, 0.110, 0.117 [0.113]	110
		559 (1.53)	90		0.113, 0.114, 0.115 [0.114]	110
		685 (1.88)	88		0.136, 0.113, 0.110 [0.120]	120
	X11719474	535 (1.47)	77	0.202	0.206, 0.198, 0.214 [0.206]	100
		559 (1.53)	85		0.212, 0.208, 0.204 [0.208]	100
		685 (1.88)	87		0.265, 0.216, 0.210 [0.230]	110
	X11721061	535 (1.47)	87	0.0982	0.124, 0.128, 0.131 [0.127]	130
		559 (1.53)	87		0.126, 0.124, 0.121 [0.123]	130
		685 (1.88)	88		0.153, 0.123, 0.122 [0.133]	140
Sunflower refined oil	Sulfoxaflor	535 (1.47)	88	0.1	0.113, 0.112, 0.102 [0.109]	110
		565 (1.55)	95		0.112, 0.108, 0.115 [0.112]	110
		696 (1.91)	89		0.110, 0.116, 0.107 [0.111]	110
	X11719474	535 (1.47)	76	0.202	0.202, 0.202, 0.194 [0.199]	99
		565 (1.55)	87		0.204, 0.202, 0.210 [0.205]	100
		696 (1.91)	82		0.200, 0.208, 0.196 [0.201]	100
	X11721061	535 (1.47)	87	0.0982	0.114, 0.114, 0.112 [0.113]	120
		565 (1.55)	91		0.111, 0.109, 0.117 [0.112]	110
		696 (1.91)	89		0.114, 0.114, 0.106 [0.111]	110

USE PATTERN

Registered labels describing the use of sulfoxaflor were provided to the current Meeting for avocado, mango, bushberry, caneberry, artichoke, asparagus, sunflower, and coffee (Table 4). For all uses, the timing of application is triggered by pest pressure, and all uses are a foliar broadcast spray.

Table 4 Registered uses of sulfoxaflor provided to the 2021 Extra JMPR

Use site	Country	Formulation		Application					PHI, days
		Conc.	Type	Rate, g/ha/applic	Rate, g/ha/year	Water, L/ha	Max No.	Interval, days	
Avocado *	USA	50%	WG	96	298	ns	4	7	7
Mango	Kenya	240 g/L	SC	96	ns	2000	3	14	3
Bush berry subgroup [a]	USA	50%	WG	96	298	ns	4	7	1
Cane berry subgroup [b]	USA	50%	WG	96	298	ns	4	7	1
Artichoke (globe)	USA	50%	WG	96	298	ns	4	14	3
Asparagus [Post harvest of spears]	USA	50%	WG	96	298	ns	4	7	--
Sunflower subgroup [c]	USA	50%	WG	96	193	ns	2	7	14
Coffee	Vietnam	50%	WG	0.125 g/L dilution†	ns	ns	ns	ns	3

* Do not apply any time between 3 days prior to bloom and until after petal fall.

[a] Includes aronia berry, highbush and lowbush blueberry, buffalo currant, Chilean guava, highbush cranberry, black and red currant, elderberry, European barberry, gooseberry, honeysuckle, huckleberry, jostaberry, Juneberry (Saskatoon berry), lingonberry, native currant, salal, and sea buckthorn.

[b] Blackberry, loganberry, black and red raspberry, and wild raspberry.

[c] Calendula, castor oil plant, Chinese tallowtree, euphorbia, evening primrose, jojoba, niger seed, rose hip, safflower, stokes aster, sunflower, tallowwood, tea oil plant, vernonia.

† Equivalent to 75 g ai/ha

RESULTS OF SUPERVISED RESIDUE TRIALS ON CROPS

The Meeting received data from supervised residue trials conducted on avocado, mango, blueberry, black berry, raspberry, globe artichoke, asparagus, sunflower, and coffee.

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 method described above for plant commodities.

The field trial study designs included control plots. Measured residues from control plots were <LOQ and are not included in the summary tables in this evaluation.

For mango, mango, and coffee, the sponsor is relying on storage stability data that were reviewed by the 2011 JMPR. That Meeting concluded that residues of sulfoxaflor are stable in oranges, peaches, wheat grain, and soya bean seed stored under frozen conditions for at least 680 days (1.9 years). For other commodities considered by the current Meeting, concurrent storage stability data adequately cover the periods of frozen storage.

When calculating average residues, values below the LOQ were assumed to be at the LOQ. In the summary tables, residue values leading to maximum residue estimations and used for long-term dietary risk assessment are underlined. The highest individual values selected for estimating acute dietary risks are bolded.

Although the provided study reports include analysis of X11719474 and X11721061, neither compound is part of the residue definitions for sulfoxaflor. Therefore, results for these compounds are not included in this evaluation.

Supervised trials for sulfoxaflor:

Category	Crop	Table
Assorted sub-tropical fruit, inedible peel	Avocado	Table 5
	Mango	Table 6
Berries and other small fruits	Blueberries	Table 7
	Cane berries	Table 8
Stalk and stem vegetables	Globe artichoke	Table 9
	Asparagus	Table 10

Oilseeds	Sunflower	Table 11
Seed for beverages and sweets	Coffee	Table 12

Assorted sub-tropical fruit, inedible peel

Avocado

Five field trials were conducted in the USA during the 2013 season (Cenni, M. 2014, Report 130161) using an SC formulation. Treatment consisted of three foliar airblast applications of ca. 96 g ai/ha, on a 7-day interval. Harvest occurred 7 days after the last application.

Following harvest, samples (~24 marketable sized fruits) were quartered with a knife and the pits were removed. Fruits and pits were weighed separately. For pulp samples, the peel was carefully removed with a knife. Samples of pitted avocados and avocado pulp were placed into coolers with dry ice, 'blue ice', or bags of wet ice prior to being transferred to a freezer in preparation for transport to the analytical facility. Upon arrival at the facility, samples were put into frozen storage. Prior to analysis, samples were homogenized in the presence of dry ice to create a homogeneous fine powder, and then returned to frozen storage. Samples were stored for a maximum of 283 days prior to analysis.

Samples were analysed for residues of sulfoxaflor, X11719474 and X11721061 using method 091031, which is essentially identical to method 091116S1 described above. Concurrent recovery data indicate that the method is suitable.

Table 5 Results of sulfoxaflor residue trials in **avocado** conducted in the USA

Location; year (Trial ID)	Crop Variety	Application				Portion	DALA	Residues (mg/kg) [Mean]	Study report
		No. [interval, days]	Conc., g ai/hL	Rate, g ai/ha	L/ha			Sulfoxaflor	
Critical GAP (US)	--	4 [7]	ns	96	ns	--	7	--	
Canal Point, Florida; 2013 (A)	Tonnage	1 [--] 2 [7] 3 [7]	5.4 5.5 5.2	96 96 96	1786 1761 1853	Whole fruit, calculated	7	0.058, 0.040 [0.049]	130161
						Fruit, pitted	7	0.068, 0.049 [0.059]	
						Pulp	7	0.032, **0.036** [0.034]	
Camarillo, CA; 2013 (B)	Haas	1 [--] 2 [7] 3 [7]	4.7 4.7 4.7	96 96 97	2055 2059 2073	Whole fruit, calculated	7	0.053, 0.047 [0.050]	
						Fruit, pitted	7	0.062, 0.054 [0.058]	
						Pulp	7	0.010, 0.012 [0.011]	
Riverside, CA; 2013 (C)	Gwen	1 [--] 2 [7] 3 [7]	4.7 4.7 4.7	95 96 95	2039 2061 2042	Whole fruit, calculated	0	0.10, 0.085 [0.094]	

Location; year (Trial ID)	Crop Variety	Application				Portion	DALA	Residues (mg/kg) [Mean]	Study report
		No. [interval, days]	Conc., g ai/hL	Rate, g ai/ha	L/ha			Sulfoxaflor	
							7	0.064, 0.060 [0.062]	
							14	0.068, 0.063 [0.066]	
							21	0.051, 0.067 [0.059]	
							28	0.048, 0.046 [0.047]	
						Fruit, pitted	0	0.12, 0.097 [0.109]	
							7	0.074, 0.071 [0.072]	
							14	0.079, 0.074 [0.075]	
							21	0.059, 0.078 [0.069]	
							28	0.056, 0.053 [0.054]	
						Pulp	7	0.028, 0.022 [0.025]	
San Luis Obispo, CA; 2013 (D)	Haas	1 [--] 2 [7] 3 [7]	4.1 5.0 4.3	96 127 91	2333 2557 2129	Whole fruit	7	0.015, ND [<0.013]	
						Fruit, pitted	7	0.017, ND [<0.014]	
						Pulp	7	<0.01, <0.01 [<0.010]	
Nipomo, CA; 2013 (E)	Haas	1 [--] 2 [8] 3 [7]	4.5 4.3 4.7	98 98 102	2192 2268 2150	Whole fruit, calculated	0	0.014, 0.044 [0.029]	
							7	0.021, 0.016 [0.019]	
							14	0.023, 0.014 [0.018]	
							21	0.019, <0.01 [<0.014]	
							28	<0.01, 0.022 [<0.016]	
						Fruit, pitted	0	0.035, 0.049 [0.042]	
							7	0.024, 0.018 [0.021]	
							14	0.026, 0.016 [0.021]	

Location; year (Trial ID)	Crop Variety	Application				Portion	DALA	Residues (mg/kg) [Mean]	Study report
		No. [interval, days]	Conc., g ai/hL	Rate, g ai/ha	L/ha			Sulfoxaflor	
							21	0.021, 0.010 [0.016]	
							28	<0.01, 0.025 [<0.017]	
						Pulp	7	<0.01, 0.014 [<0.012]	

Mango

Eight field trials were conducted in Ghana, Kenya, Senegal, Tanzania, and Uganda during the 2016, 2017, or 2018 season (DeFrancesco, J. 2019, Report PR 11822). Treatment consisted of two foliar applications of sulfoxaflor (SC formulation) at one of three rates: ca 91 to 104 g ai/ha, 107 to 131 g ai/ha, or 127 to 155 g ai/ha. The retreatment interval was 14–15 days. Harvest occurred 3 days after the last application.

Following harvest, samples (≥ 24 fruits) were put into coolers with blue ice and transported to the laboratory. In the laboratory, the fruits were pitted and cut into quarters to reduce sample weight. At two sites (UG02 and KE02), additional fruits were harvested to provide pulp-only and peel-only samples. Following pitting, quartering, and peeling, all samples were frozen and were stored/shipped frozen prior to analysis. In preparation for analysis, samples were homogenized in the presence of dry ice and returned to frozen storage. Sample storage durations were not reported other than a statement that they were stored for less than 2 years.

Samples were analysed for residues of sulfoxaflor and X11719474 using a method similar to the reference method. Concurrent recovery data indicate that the method is suitable.

Minor deviations from GLP were noted. These are not expected to have a significant impact on the results reported in the trials. Samples from Tanzania (11822.16-TZ01) and Senegal (12502.18-SN01) thawed in transit to the analytical laboratory, and results for those samples are not reported.

Table 6 Results of sulfoxaflor residue trials in **mango**

Location; year (Trial ID)	Crop Variety	Application				DALA	Matrix	Residues (mg/kg) [Mean]	Study report
		Trtmt ID	No. [interval, days]	Rate, g ai/ha	L/ha			Sulfoxaflor	Remarks
Critical GAP (KE)	--	--	3 [14]	96	2000	3	--	--	
Sebikotane, Senegal 2016 11822.16-SN01	Kent	02	1 2 [15]	103.5 97.9	978 932	3	Whole fruit	0.074, 0.034 [0.054]	PR 11822
		03	1 2 [15]	117.2 123.7	886 936	3	Whole fruit	0.043, 0.066 [0.054]	
		04	1 2 [15]	144.0 154.6	907 975	3	Whole fruit	0.053, 0.073 [0.063]	

Location; year (Trial ID)	Crop Variety	Application				DALA	Matrix	Residues (mg/kg) [Mean]	Study report
		Trtmt ID	No. [interval, days]	Rate, g ai/ha	L/ha			Sulfoxaflor	Remarks
Kiuntu, Uganda 2017 11822.16-UG02	Kent	02	1 2 [14]	90.6 96.2	775 803	3	Whole fruit	0.028, 0.025 [0.026]	
		03	1 2 [14]	106.9 120.4	712 802	3	Whole fruit	0.067, 0.066 [0.066]	
		04	1 2 [14]	126.9 144.6	706 804	3	Whole fruit	0.076, 0.046 [0.061]	
						3	Peel	0.572, 0.235 [0.403]	
						3	Pulp	0.034, 0.022 [0.028]	
Anum, Ghana 2018 12502.18-GH01	Kent	02	1 2 [14]	99.7 100.9	832 842	3	Whole fruit	0.045, 0.033 [0.039]	
		03	1 2 [14]	23.9 124.9	827 834	3	Whole fruit	0.074, 0.064 [0.069]	
		04	1 2 [14]	149.2 147.3	830 820	3	Whole fruit	0.045, 0.062 [0.054]	
Somanya, Ghana 2018 12502.18-GH02	Kent	02	1 2 [14]	93.6 95.4	781 794	3	Whole fruit	0.036, 0.037 [0.036]	
		03	1 2 [14]	102.4 119.5	804 798	3	Whole fruit	0.027, 0.029 [0.028]	
		04	1 2 [14]	141.5 142.8	787 794	0	Whole fruit	0.101, 0.084 [0.092]	
						3		0.051, 0.041 [0.046]	
						7		0.046, 0.040 [0.043]	
						10		0.022, 0.013 [0.018]	
						14		ND, ND [ND]	
Masii, Kenya 2018 12503.18-KE01	Apple	02	1 2 [14]	95.8 91.8	1491 1421	3	Whole fruit	0.091, 0.114 [0.102]	
		03	1 2 [14]	131.0 117.9	1632 1459	3	Whole fruit	0.112, 0.132 [0.122]	
		04	1 2 [14]	143.7 145.1	1321 1347	0	Whole fruit	0.081, 0.099 [0.090]	
						3		0.096, 0.072 [0.084]	
						7		0.049, 0.090 [0.069]	
						10		0.092, 0.102 [0.097]	
						14		0.029, 0.042 [0.036]	
Wote, Kenya 2018 12503.18-KE02	Apple	02	1 2 [14]	90.8 100.9	1304 1372	3	Whole fruit	**0.133**, 0.094 [0.114]	

Location; year (Trial ID)	Crop Variety	Application				DALA	Matrix	Residues (mg/kg) [Mean]	Study report
		Trtmt ID	No. [interval, days]	Rate, g ai/ha	L/ha			Sulfoxaflor	Remarks
		03	1 2 [14]	131.7 117.0	1433 1381	3	Whole fruit	0.129, 0.149 [0.139]	
		04	1 2 [14]	139.2 141.9	1268 1396	3	Whole fruit	0.221, 0.200 [0.210]	
							Peel	1.079, 0.849 [0.964]	
							Pulp	<0.01, ND [<0.01]	

Berries and other small fruits

Blueberry

Twelve field trials were conducted in Canada and the USA during the 2014 season (Dorschner, K., 2017, Report PR 11296). Treatment consisted of three foliar applications of ca. 100 g sulfoxaflor/ha, on a 7-day interval. A non-ionic surfactant or crop oil concentrate was used at all trial locations. Samples of blueberries were taken 1 day after the last application.

Following harvest, samples (*ca* 1 to 1.4 kg) were bagged and put into put into frozen storage prior to transport to the analytical facility. Samples were shipped frozen to the facility and were immediately placed into frozen storage. Prior to analysis, samples were cryogenically homogenized and returned to frozen storage. Samples were stored for a maximum of 23.7 months prior to analysis.

Samples were analysed for residues of sulfoxaflor using the method Method 091116S1. Concurrent recovery data indicate that the method is suitable.

Table 7 Residues of sulfoxaflor in **blueberries** from residue trials conducted in Canada and the USA

Location; year (Trial ID)	Crop Variety	Application				DALA	Residues (mg/kg) [Mean]	Study report
		No. [interval, days]	Rate, g ai/ha	L/ha	Formulation/ Adjuvant [a]		Sulfoxaflor	Remarks
Critical GAP (US)	--	4 [7]	96	ns	--	1	--	
Langley, BC, CA; 2014 (BC23)	Brigita highbush	1 [--] 2 [7] 3 [7]	102.8 101.8 103.0	503 498 505	SC / NIS	1	0.184, 0.126 [0.155]	PR 11296
Alapha, GA, US; 2014 (GA*187)	TH653	1 [--] 2 [7] 3 [8]	98.6 101.1 99.2	274 281 276	SC / NIS	1	0.402, 0.434 [0.418]	
Jonesboro, ME, US; 2014 (ME242)	Lowbush	1 [--] 2 [8] 3 [6]	102.2 100.8 100.7	238 235 235	SC / veg. oil + silicone	1	0.306, 0.543 [0.424]	
Fennville, MI, US; 2014 (MI276) [b]	Rubel	1 [--] 2 [7] 3 [7]	103.2 104.4 105.7	339 343 348	SC / NIS	1	**1.39**, 1.14 [1.26]	

Location; year (Trial ID)	Crop Variety	Application				DALA	Residues (mg/kg) [Mean]	Study report
		No. [interval, days]	Rate, g ai/ha	L/ha	Formulation/ Adjuvant [a]		Sulfoxaflor	Remarks
Fennville, MI, US; 2014 (MI277) [b]	Bluecrop	1 [--] 2 [6] 3 [7]	101.4 100.3 100.7	692 684 705	SC / NIS	1	0.751, 0.914 [0.832]	
Fennville, MI, US; 2014 (MI278) [b]	Jersey	1 [--] 2 [7] 3 [7]	101.0 100.8 100.6	513 518 527	SC / NIS	1	0.642, 0.583 [0.612]	
Cream Ridge, NJ, US; 2014 (NJ306)	Bluecrop	1 [--] 2 [7] 3 [7]	101.6 101.4 99.7	283 283 275	SC / MSO	1	0.365, 0.419 [0.392]	
Chatsworth, NJ, US; 2014 (NJ307)	Duke	1 [--] 2 [7] 3 [7]	104.9 101.3 103.5	288 291 297	SC / NIS	1	0.171, 0.168 [0.170]	
Sheffield Mills, NS, CA; 2014 (NS321)	Wild clone lowbush	1 [--] 2 [7] 3 [7]	103.0 101.4 102.7	356 351 356	SC / NIS	0	0.346, 0.356 [0.351]	
						1	0.312, 0.283 [0.298]	
						8	0.203, 0.190 [0.196]	
						14	0.168, 0.187 [0.178]	
						21	0.163, 0.170 [0.166]	
Milford, NS, CA; 2014 (NS322)	Wild clone lowbush	1 [--] 2 [7] 3 [7]	101.2 100.4 101.3	351 348 351	SC / NIS	1	0.374, 0.399 [0.386]	
Delbert, NS, CA; 2014 (NS323)	Wild clone lowbush	1 [--] 2 [6] 3 [6]	101.2 100.1 100.9	351 347 350	SC / NIS	1	0.271, 0.265 [0.268]	
Aurora, OR, US; 2014 (OR399)	Bluecrop	1 [--] 2 [7] 3 [7]	100.1 100.6 100.3	373 375 374	SC / NIS	0	0.301, 0.413 [0.357]	
						1	0.292, 0.327 [0.310]	
						7	0.252, 0.211 [0.231]	
						14	0.226, 0.168 [0.197]	

[a] NIS = Non-ionic surfactant, MSO = methylated seed oil

[b] Fennville Trials: 277 and 278 are the same location. 276 and 277/278 are at a similar location and trials were conducted within 4 days of each other. 276 and 277 used highbush varieties and 278 used a lowbush variety. 276 and 277 are considered to be dependent; 278 is independent.

Cane berries

Seven field trials were conducted in Canada and the USA during the 2014 season using an SC formulation of sulfoxaflor (Dorschner, K., 2017, Report PR 11279). Treatment consisted of three foliar applications of ca. 100 g sulfoxaflor/ha, on a 7-day interval. A non-ionic surfactant or methylated seed oil was used at all trial locations. Samples of cane berries were taken 1 day after the last application.

Following harvest, samples (ca 1 to 1.4 kg) were bagged and put into put into frozen storage prior to transport to the analytical facility. Samples were shipped frozen to the facility and were immediately placed into frozen storage. Prior to analysis, samples were cryogenically homogenized and returned to frozen storage. Samples were stored for a maximum of 19 months prior to analysis.

Samples were analysed for residues of sulfoxaflor using the method Method 091116S1. Concurrent recovery data indicate that the method is suitable.

Table 8 Residues of sulfoxaflor in **cane berries** from residue trials conducted in Canada and the USA

Location; year (Trial ID)	Crop Variety	Application			DALA	Residues (mg/kg) [Mean]	Study report
		No. [interval, days]	Rate, g ai/ha	L/ha		Sulfoxaflor	
Critical GAP (US)	--	4 [7]	96	ns	1		
Agassiz, BC, CA; 2014 (BC21)	Raspberry Rudi	1 [--] 2 [6] 3 [7]	103.1 101.9 102.7	507 501 505	1	0.390, 0.420 [0.40]	PR 11279
Langley, BC, CA; 2014 (BC22)	Raspberry Cascade Delight	1 [--] 2 [6] 3 [6]	105.0 102.7 104.6	516 505 515	1	0.205, 0.266 [0.24]	
Parlier, CA, US; 2014 (CA49)	Blackberry Oauchita	1 [--] 2 [7] 3 [6]	101.9 100.8 99.0	445 480 472	1	0.314, 0.239 [0.28]	
Holt, MI, US; 2014 (MI273)	Raspberry Heritage Fall	1 [--] 2 [7] 3 [8]	100.8 102.9 101.5	398 406 401	0	0.767, 0.694 [0.73]	
					1	0.645, 0.426 [0.54]	
					6	0.383, 0.368 [0.38]	
					13	0.201, 0.215 [0.21]	
					20	0.142, 0.104 [0.12]	
Jackson Springs, NC, US; 2014 (NC280)	Blackberry Kiowa	1 [--] 2 [6] 3 [6]	99.9 102.4 102.2	223 230 229	1	0.511, 0.468 [0.49]	
Aurora, OR, US; 2014 (OR379)	Raspberry Meeker	1 [--] 2 [7] 3 [7]	102.8 101.2 98.7	381 376 366	0	0.577, 0.543 [0.56]	
					1	0.474, 0.411 [0.44]	

Location; year (Trial ID)	Crop Variety	Application			DALA	Residues (mg/kg) [Mean]	Study report
		No. [interval, days]	Rate, g ai/ha	L/ha		Sulfoxaflor	
					7	0.253, 0.207 [0.23]	
					14	0.168, 0.161 [0.16]	
					21	0.103, 0.113 [0.11]	
Aurora, OR, US; 2014 (OR380)	Blackberry Marion	1 [--] 2 [7] 3 [7]	103.3 101.5 99.0	577 567 553	1	0.709, **0.778** [0.74]	

Stalk and stem vegetables

Globe artichoke

Six field trials were conducted in Canada and the USA during the 2014 season (Dorschner, K., 2017, Report PR 10858). Treatment consisted of three foliar applications of ca. 100 g sulfoxaflor/ha, on a 7-day interval. A non-ionic surfactant was used at all trial locations. Samples of globe artichoke flowers were taken 1 day after the last application. Samples were not taken from the two trials conducted in BC, Canada, due to poor flowering.

Following harvest, samples (ca 1 to 1.4 kg) were bagged and put into put into frozen storage prior to transport to the analytical facility. Samples were shipped frozen to the facility and were immediately placed into frozen storage. Prior to analysis, samples were cryogenically homogenized and returned to frozen storage. Samples were stored for a maximum of 19 months prior to analysis.

Samples were analysed for residues of sulfoxaflor using the method Method 091116S1. Concurrent recovery data indicate that the method is suitable.

Table 9 Residues of sulfoxaflor in **globe artichoke** from residue trials conducted in Canada the USA

Location; year (Trial ID)	Crop Variety	Application				DALA	Residues (mg/kg) [Mean]	Study report
		No. [interval, days]	Rate, g ai/ha	L/ha	Formulation/ Adjuvant [a]		Sulfoxaflor	
Critical GAP (US)	--	4 [14]	96	ns	--	3	--	
Salinas, CA, US; 2014 (CA64)[b]	$F_1$41 annual	1 [--] 2 [7] 3 [6]	101.7 101.6 101.2	94 94 94	SC / NIS	1	0.291, 0.251 [0.271]	PR 10858
						3	0.254, 0.197 [0.226]	
						7	0.127, 0.258 [0.192]	
						14	0.0421, 0.0628 [0.0524]	

Location; year (Trial ID)	Crop Variety	Application				DALA	Residues (mg/kg) [Mean]	Study report
		No. [interval, days]	Rate, g ai/ha	L/ha	Formulation/ Adjuvant [a]		Sulfoxaflor	
						20	0.0179, 0.0128 [0.0154]	
Salinas, CA, US; 2014 (CA66) [b]	$F_1$41 annual	1 [--] 2 [7] 3 [9]	100.5 100.4 100.3	931 930 929	SC / NIS	3	0.149, 0.114 [0.132]	
Castroville, CA, US; 2014 (CA65)	Green globe perennial	1 [--] 2 [9] 3 [8]	101.4 101.3 103.7	705 704 721	SC / NIS	3	0.293, 0.227 [0.260]	
L'Acadie, QC, CA; 2014 (QC419)	Imperial star	1 [--] 2 [8] 3 [7]	101.6 98.2 102.6	493 477 498	SC / NIS	3	0.234, 0.199 [0.216]	

[a] NIS = Non-ionic surfactant

[b] Side-by-side trials using different spray volume/ha. Trials are not independent.

Asparagus

Eight field trials were conducted in the USA during the 2014 season using an SC formulation of sulfoxaflor (Dorschner, K., 2017, Report PR 11321). Treatment consisted of three foliar applications of ca. 100 g sulfoxaflor/ha, on a 7-day interval. A non-ionic surfactant was used at all trial locations. Samples of asparagus spears were taken at first commercial maturity the following season (126 to 302 days after the last application).

Following harvest, samples (ca 2 to 2.7 kg) were bagged and put into put into frozen storage prior to transport to the analytical facility. Samples were shipped frozen to the facility and were immediately placed into frozen storage. Prior to analysis, samples were cryogenically homogenized and returned to frozen storage. Samples were stored for a maximum of 11 months prior to analysis.

Samples were analysed for residues of sulfoxaflor using the method Method 091116S1. Concurrent recovery data indicate that the method is suitable.

Table 10 Residues of sulfoxaflor in **asparagus** from residue trials conducted in the USA

Location; year (Trial ID)	Crop Variety	Application			DALA	Residues (mg/kg) [Mean]	Study report
		No. [interval, days]	Rate, g ai/ha	L/ha		Sulfoxaflor	
Critical GAP (US)	--	4 [7]	96	ns	--		Apply to fern post-harvest of spears
Davis, CA, US; 2014 (CA67)	UC 157	1 [--] 2 [7] 3 [8]	102 102 105	281 281 290	126	<0.01, <0.01 [<0.01]	PR 11321
Parlier, CA, US; 2014 (CA68)	UC 115	1 [--] 2 [7]	103 103	571 571	217	<0.01, <0.01 [<0.01]	

Location; year (Trial ID)	Crop Variety	Application			DALA	Residues (mg/kg) [Mean]	Study report
		No. [interval, days]	Rate, g ai/ha	L/ha		Sulfoxaflor	
		3 [7]	102	552			
Salisbury, MD, US; 2014 (MD227)	Jersey Knight	1 [--] 2 [7] 3 [7]	101 101 103	271 271 281	289	<0.01, <0.01 [<0.01]	
East Lansing, MI, US; 2014 (MI261) [a]	Jersey Giant	1 [--] 2 [7] 3 [7]	102 105 103	374 393 384	302	<0.01, <0.01 [<0.01]	
East Lansing, MI, US; 2014 (MI262) [a]	Millennium	1 [--] 2 [7] 3 [7]	102 102 105	337 337 346	281	<0.01, **0.0113** [0.011]	
Moxee, WA, US; 2014 (WA*450)	Jersey Giant	1 [--] 2 [6] 3 [7]	101 101 100	580 580 580	203	<0.01, <0.01 [<0.01]	
Adams, WI, US; 2014 (WI518) [b]	Jersey Giant	1 [--] 2 [7] 3 [6]	102 103 103	215 224 215	278	<0.01, <0.01 [<0.01]	
Adams, WI, US; 2014 (WI519) [b]	Jersey Giant	1 [--] 2 [7] 3 [6]	103 103 104	393 412 412	278	<0.01, <0.01 [<0.01]	

[a] Trials are at a similar location and conducted at a similar time. Trials are not independent.

[b] Side-by-side trials with different spray volume/ha. These trials are not independent.

Sunflower seed

Eight field trials were conducted in the USA during the 2013 season (Dorschner, K., 2017, Report PR 11095). Treatment consisted of two foliar applications of ca. 100 g sulfoxaflor/ha, on a 7-day interval. A non-ionic surfactant, methylated seed oil, or crop oil concentrate was used at all trial locations. Samples of sunflower seed were taken 14 days after the last application.

Following harvest, samples (*ca* 1 to 1.8 kg) were bagged and put into put into frozen storage prior to transport to the analytical facility. Samples were shipped frozen to the facility and were immediately placed into frozen storage. Prior to analysis, samples were cryogenically homogenized and returned to frozen storage. Samples were stored for a maximum of 25 months prior to analysis.

Samples were analysed for residues of sulfoxaflor using the method Method 091116S1. Concurrent recovery data indicate that the method is suitable.

Table 11 Residues of sulfoxaflor in **sunflower seed** from residue trials conducted in the USA

Location; year (Trial ID)	Crop Variety	Application				DALA	Residues (mg/kg) [Mean]	Study report
		No. [interval days]	Rate, g ai/ha	L/ha	Formulation/ Adjuvant [a]		Sulfoxaflor	
Critical GAP (US)	--	2 [7]	96	ns	--	14	--	

Location; year (Trial ID)	Crop Variety	Application				DALA	Residues (mg/kg) [Mean]	Study report
		No. [interval days]	Rate, g ai/ha	L/ha	Formulation/ Adjuvant [a]		Sulfoxaflor	
Fargo, ND, US; 2013 (ND13) [b]	8H449CLDM	1 [-] 2 [7]	101 99	196 196	WG / NIS	14	0.0132, 0.0123 [0.013]	PR 11095
Fargo, ND, US; 2013 (ND14) [b]	8N270CLDM	1 [-] 2 [6]	100 102	309 318	WG / NIS	16	0.0132, 0.0131 [0.013]	
Minot, ND, US; 2013 (ND15) [c]	8N270CLDM	1 [-] 2 [7]	102 99	215 206	WG / NIS	15	0.0164, 0.0205 [0.018]	
Minot, ND, US; 2013 (ND16) [c]	INT 735 NS/CL	1 [-] 2 [7]	101 101	309 309	WG / MSO	0	0.0268, 0.0260 [0.026]	
						3	<0.01, <0.01 [<0.01]	
						8	<0.01, <0.01 [<0.01]	
						15	<0.01, <0.01 [<0.01]	
						21	<0.01, <0.01 [<0.01]	
Las Cruces, NM, US; 2013 (NM12)	S678	1 [-] 2 [7]	108 102	299 290	WG / NIS	14	<0.01, <0.01 [<0.01]	
Aurora, SD, US; 2013 (SD06) [d]	Badger DMR	1 [-] 2 [6]	108 105	243 234	WG / MSO	14	<0.01, <0.01 [<0.01]	
Aurora, SD, US; 2013 (SD07) [d]	Panther DMR L-10	1 [-] 2 [9]	105 105	543 543	WG / NIS	13	0.0368, 0.0627 [0.050]	
Aurora, SD, US; 2013 (SD08) [d]	Durango	1 [-] 2 [6]	105 100	421 402	WG / COC	15	0.146, 0.151 [0.150]	

[a] NIS = Non-ionic surfactant, MSO = methylated seed oil, COC = crop oil concentrate

[b], [c], [d] Trials with the same footnote designation were conducted at the same location and time; they are not independent.

Coffee

Ten field trials were conducted in Vietnam during the 2019 season (Petrova, D. and Kühnel, S., 2020, Report 190532). Treatment consisted of a single foliar applications of ca. 75 g sulfoxaflor/ha. Samples of coffee beans were harvested 3 days after application.

Following harvest, samples (generally at least 1kg) of coffee berries were peeled in the field or using a peeling machine. Within 1 day of harvest, beans with shells from all samples were separated from pulp by wet sieving and sun drying, were bagged, and put into put into frozen storage prior to transport to

the analytical facility. Samples were shipped frozen to the facility and were immediately placed into frozen storage. Prior to analysis, samples were cryogenically homogenized and returned to frozen storage. Samples were stored for a maximum of 193 days prior to analysis.

Samples were analysed for residues of sulfoxaflor using the QuEChERS method. Concurrent recovery data indicate that the method is suitable.

Table 12 Residues of sulfoxaflor in **coffee** from residue trials conducted in Vietnam

Location; year (Trial ID)	Crop Variety	Application				DALA	Residues (mg/kg) [Mean]	Study report
		No. [interval, days]	Rate, g ai/ha	L/ha	Formulation		Sulfoxaflor	
Critical GAP (Vietnam)	--	ns	0.125 g/L dilution	600	--	3	--	Equivalent to 75 g ai/ha
Lien Ha, Lam Dong, Vietnam; 2019 (S19-00420-01)	Arabica	1 [--]	75.5	1509	500 WG	3	0.15	109532
Dam Rong, Lam Dong, Vietnam; 2019 (S19-00420-02)	Robusta	1 [--]	73.7	1473	500 WG	3	0.01	
Bao Loc, Lam Dong, Vietnam; 2019 (S19-00420-03)	Robusta	1 [--]	76.1	1217	500 WG	3	<0.01	
Da Loan, Binh Thuan, Vietnam; 2019 (S19-00420-04)	Robusta	1 [--]	75.7	1515	500 WG	3	0.01	
Due Trong, Lam Dong, Vietnam; 2019 (S19-00420-05)	Arabica	1 [--]	73.5	1470	500 WG	3	0.05	
Lam Ha, Lam Dong, Vietnam; 2019 (S19-00420-06)	Robusta	1 [--]	76.2	1219	500 WG	0	0.02	
						3	<0.01	
						6	<0.01	
						15	0.01	
						19	0.01	
Loc Ngai, Lam Dong, Vietnam; 2019 (S19-00420-07)	Robusta	1 [--]	75.7	1514	500 WG	0	<0.01	
						3	0.01	
						7	0.02	
						15	0.03	
						19	0.02	
Lam Ha, Cau Dot, Vietnam; 2019 (S19-00420-08)	Arabica	1 [--]	75.2	1504	500 WG	0	0.06	
						3	0.02	

Location; year (Trial ID)	Crop Variety	Application				DALA	Residues (mg/kg) [Mean]	Study report
		No. [interval, days]	Rate, g ai/ha	L/ha	Formulation		Sulfoxaflor	
						7	0.07	
						12	0.08	
						21	0.09	
Lac Doong, Lam Dong, Vietnam; 2019 (S19-00420-09)	Arabica	1 [--]	73.2	1464	500 WG	0	0.13	
						3	0.02	
						7	0.04	
						13	0.02	
						20	0.02	
Di Linh, Gung Re, Vietnam; 2019 (S19-00420-10)	Robusta	1 [--]	74.1	1483	500 WG	0	0.06	
						3	0.06	
						7	0.04	
						15	0.02	
						19	0.01	

FATE OF RESIUDES DURING PROCESSING

Sunflower seed

In a study investigating sulfoxaflor residues in processed sunflower seed commodities (Dorschner, K., 2017, Report PR 11095), seed samples from the ND13 field trial site described above were processed into meal and refined oil using simulated commercial practices. Upon receipt at the processing facility, the sunflower seed samples were placed into frozen (ca. -25 °C) storage, where they remained prior to processing. Residues of sulfoxaflor were analysed using the method Method 091116S1.

For processing, seed samples were tempered at ca. 60 °C and then screened to remove debris. The screened seed was scarified, screened, and aspirated to separate hulls from the seed. The dehulled seeds were then heated (70–83 °C, 30 min) and flaked; flaked seed was pressed in an expeller to separate crude oil from presscake.

Meal. The presscake was placed in hexane for ca. 5 min. and the miscella was then separated from the meal by vacuum filtration. The meal was heated (ca. 90 °C) to remove solvent and then dried to produce toasted meal.

Oil. The crude oil was degummed by heating and treating with phosphoric acid and distilled water. The degummed oil was isolated by centrifugation and then treated with sodium hydroxide to separate the oil from the soapstock. The resulting refined oil was heated to remove trace moisture prior to analysis.

Table 13 Residues of sulfoxaflor in **sunflower seed processed commodities** from residue trials in sunflower conducted in the USA

Trial ID	Commodity	Residues (mg/kg) [Mean]	Processing factor [a]	Study report
		Sulfoxaflor		Remarks
ND13	Seed (pre-processing)	0.015, 0.013 [0.014]	--	PR 11095
	Meal	<0.01	<0.71	
	Refined oil	<0.01	<0.71	

[a] Values reported as <0.01 mg/kg were assumed to be 0.01 mg/kg for purposes of calculating processing factors.

Coffee

In the study investigating sulfoxaflor residues in processed coffee commodities (Petrova, D. and Kühnel, S., 2020, Report 190532), bean samples (ca. 10 kg) from the S19-00420-01 and -02 field trial sites described above were harvested from plots treated with sulfoxaflor at an exaggerated application rate (ca. 225 g ai/ha; 3× nominal GAP rate). Beans with shells were placed in an air convection oven (180 °C, 25 minutes) to produce roasted coffee. Roasted beans were ground and brewed to make a concentrated coffee solution, which was separated from the grounds and freeze dried to produce instant coffee. Residues of sulfoxaflor in roasted beans and instant coffee were analysed using the QuEChERS method.

Table 14 Residues of sulfoxaflor in **coffee processed commodities** from residue trials in coffee conducted in the USA

Trial ID	Commodity	Residues (mg/kg) [Mean]	Processing factor	Study report
		Sulfoxaflor		Remarks
S19-00420-01	Dried beans with shell	0.35, 0.37, 0.42 [0.38]	--	190532
	Roasted beans	(0.18, 0.18), 0.16 [0.17 [a]]	0.45	
	Instant coffee	(1.03, 1.44), 0.98 [1.11 [a]]	2.9	
S19-00420-02	Dried beans with shell	0.15, 0.18, 0.20 [0.177]	--	
	Roasted beans	(0.12, 0.13), 0.05 [0.088 [a]]	0.50	
	Instant coffee	(nd, 0.70), 0.32 [0.34 [a]]	1.9	

[a] Average of 2 replicate analysis averaged with a single retain-sample analysis. The nd result was assumed to be 0.01 mg/kg for purposes of calculating the average.

APPRAISAL

Sulfoxaflor (ISO common name) is a broad-spectrum, sulfoximine insecticide with registered uses on multiple crops. It was evaluated for the first time by the 2011 JMPR, which established an acceptable daily intake (ADI) of 0–0.05 mg/kg bw and an acute reference dose (ARfD) of 0.3 mg/kg bw. Sulfoxaflor underwent subsequent evaluations by the JMPR in 2014 and 2016.

The definition of the residue for compliance with the MRL and dietary risk assessment for plant and animal commodities is sulfoxaflor. The residue is not fat-soluble.

Sulfoxaflor was scheduled at the Fifty-first Session of the CCPR for evaluation of additional uses by the 2020 JMPR, which was postponed to the 2021 Extra JMPR. The current Meeting received information

on analytical methods, field trials, and processing studies to support estimation of new maximum residue levels in commodities of avocado, mango, bush berries, cane berries, globe artichoke, asparagus, sunflower, and coffee.

Methods of residue analysis

The Meeting received method validation and concurrent recovery data for use of Method 091116S1 (reviewed by the 2011 JMPR) and the QuEChERS multiresidue method. All methods were demonstrated to have adequate performance for recovery of sulfoxaflor, with a LOQ of 0.01 mg/kg based on recoveries from method-validation and concurrent-recovery samples.

Stability of pesticide residues in stored analytical samples

Storage stability data were provided for blueberry, cane berry, artichoke, asparagus, and sunflower commodities. Little, if any, dissipation during frozen storage was observed in any of the commodities tested. The Meeting concluded that residues of sulfoxaflor are stable as follows:

Blueberry: at least 2.1 years,

Cane berry: at least 1.5 years,

Artichoke: at least 2.0 years,

Asparagus: at least 10 months,

Sunflower seed: at least 2 years,

Sunflower meal: at least 22 months, and

Sunflower refined oil: at least 23 months.

Results of supervised residue trials on crops

The Meeting received data from supervised residue trials and GAP information on avocado, mango, bush berries, cane berries, artichoke, asparagus, sunflower, and coffee.

Avocado

The critical GAP for avocado is from the USA. The label provides for up to four applications, each at 96 g ai/ha, on a 7-day interval, with a 7-day PHI. The label also specifies an annual limit of 298 g ai/ha; thus, the critical GAP is, essentially, three applications at the maximum rate.

Residues of sulfoxaflor in avocado (whole fruit) from independent trials approximating the critical GAP were (n = 5): < 0.013, 0.019, 0.049, 0.050, and 0.066 mg/kg.

The Meeting estimated a maximum residue level of 0.15 mg/kg for sulfoxaflor in avocado.

Residues of sulfoxaflor in avocado (flesh) from independent trials approximating the critical GAP were (n = 5): < 0.01, < 0.012, <u>0.011</u>, 0.025, and 0.034 mg/kg.

The Meeting estimated a STMR of 0.011 mg/kg and a HR of 0.036 mg/kg (from a single sample) for residues of sulfoxaflor in avocado flesh.

Mango

The critical GAP for mango is from Kenya and consists of three applications, on a 14-day interval, each at 96 g ai/ha, with a 3-day PHI.

Field trials from Ghana, Kenya, Senegal, Tanzania, and Uganda were conducted in accordance with the cGAP for application rate, re-treatment interval, and PHI; however, two applications were made rather than three. Residue decline data for mango indicate a half-life of sulfoxaflor of approximately 3 days. Based on the half-life, the Meeting decided that a first application (31 days before harvest) would not contribute significantly to residues at harvest. Therefore, the Meeting determined that the trials sufficiently approximate the cGAP and are suitable for making residue recommendations.

Residues of sulfoxaflor in mango from independent trials approximating the cGAP were (n = 6): 0.026, 0.036, 0.039, 0.054, 0.10, and 0.11 mg/kg.

The Meeting estimated a maximum residue level of 0.3 mg/kg for sulfoxaflor in mango. The median residue and highest residue were 0.047 and 0.133 (from a single whole fruit sample) mg/kg, respectively.

Samples from two exaggerated rate trials were separated into peel and flesh. In comparison to whole fruit, residues in flesh were lower by a factor of 0.46 (0.028 mg/kg ÷ 0.061 mg/kg) and < 0.048 (< 0.01 mg/kg ÷ 0.21 mg/kg). Applying the factor of 0.46 derived from finite residues to the median and highest whole fruit residues gives a STMR of 0.022 mg/kg and a HR of 0.061 mg/kg in mango flesh for dietary risk assessment.

Subgroup of Bush berries

Blueberry

The critical GAP for blueberry is from the USA. The label provides for up to four applications, each at 96 g ai/ha, on a 7-day interval, with a 1-day PHI. The label also specifies an annual limit of 298 g ai/ha; thus, the critical GAP is, essentially, three applications at the maximum rate.

Residues of sulfoxaflor in blueberry from independent trials approximating the critical GAP were (n = 11): 0.16, 0.17, 0.27, 0.30, 0.31, 0.39 (2), 0.42 (2), 0.61, and 1.3 mg/kg.

Noting that the registered use is for the Bush berry subgroup, the Meeting estimated a maximum residue level of 2 mg/kg, a STMR of 0.39 mg/kg, and a HR of 1.4 mg/kg (from a single sample) for sulfoxaflor in the Subgroup of Bush berries. Furthermore, as the US crop subgroup contains elderberry, the Meeting decided to extrapolate the recommendation to elderberry.

Subgroup of Cane berries

The critical GAP for cane berry is from the USA. The label provides for up to four applications, each at 96 g ai/ha, on a 7-day interval, with a 1-day PHI. The label also specifies an annual limit of 298 g ai/ha; thus, the critical GAP is, essentially, three applications at the maximum rate.

Residues of sulfoxaflor in blackberries and raspberries from independent trials approximating the critical GAP were (n = 7): 0.24, 0.28, 0.40, 0.44, 0.49, 0.54, and 0.74 mg/kg.

Noting that the registered use is for the Cane berry Subgroup, the Meeting estimated a maximum residue level of 1.5 mg/kg, a STMR of 0.44 mg/kg, and a HR of 0.78 mg/kg (from a single sample) for sulfoxaflor in the Subgroup of Cane berries.

Globe artichoke

The critical GAP for globe artichoke is from the USA. The label provides for up to four applications, each at 96 g ai/ha, on a 14-day interval, with a 3-day PHI. The label also specifies an annual limit of 298 g ai/ha; thus, the critical GAP is, essentially, three applications at the maximum rate.

Residues of sulfoxaflor in globe artichoke from independent trials approximating the critical GAP were (n = 3): 0.22, 0.23, and 0.26 mg/kg.

The Meeting agreed that three trials in globe artichoke were not sufficient to estimate a maximum residue level.

Asparagus

The critical GAP for asparagus is from the USA. The label provides for up to four applications, each at 96 g ai/ha, on a 14-day interval. Application is to the ferns after harvest; as such, no PHI is specified on the label. The label specifies an annual limit of 298 g ai/ha; thus, the critical GAP is, essentially, three applications at the maximum rate.

Residues of sulfoxaflor in asparagus spears from independent trials approximating the critical GAP (harvested at least 4 months after the last treatment to the ferns) were (n = 6): < 0.01 (5) and 0.011 mg/kg.

The Meeting estimated a maximum residue level of 0.015 mg/kg, a STMR of 0.01 mg/kg, and a HR of 0.011 mg/kg for sulfoxaflor in asparagus.

Sunflower seeds

The critical GAP for sunflower is from the USA and consists of two applications, on a 7-day interval, each at 96 g ai/ha, with a 14-day PHI.

Residues of sulfoxaflor in sunflower seed from independent trials approximating the critical GAP were (n = 4): < 0.01, 0.013, 0.018, and 0.15 mg/kg.

The Meeting agreed that four trials in sunflower seed are not sufficient for estimating a maximum residue level.

Coffee

The critical GAP for coffee is from Vietnam and consists of application of a 0.125 g/L dilution of the product sprayed thoroughly to the whole plant. The number of treatments, the re-treatment interval, and the spray volume are not specified, and the PHI is 3 days.

Residues of sulfoxaflor in coffee berries (dried beans with shell) from independent trials approximating the critical GAP were (n = 10): < 0.01, 0.01 (3), 0.03, 0.04, 0.05, 0.06, 0.09, and 0.15 mg/kg.

The Meeting estimated a maximum residue level of 0.3 mg/kg and a STMR of 0.035 mg/kg.

Fate of residues during processing

The Meeting received data showing the effect of processing sunflower seed into meal and refined oil, and coffee into roasted beans and instant coffee. Processing factors and residue estimates are summarized below. The Meeting could not estimate a maximum residue level for sunflower seed and did not make residue estimates for processed sunflower seed commodities.

The estimated STMR-Ps for the processed coffee commodities considered by the Meeting are shown in Table 1.

Table 1 Processing factors and residue estimates for sulfoxaflor

Raw commodity	Residue in RAC, mg/kg			Processed commodity	Processing Factors		Residue in processed commodity, mg/kg		
	Max	STMR	HR		Individual	Best estimate	Max-P	STMR-P	HR-P
Coffee (dried beans with shell)	0.3	0.035	--	Roasted beans	0.45, 0.50	0.475	--	0.0167	--
				Instant coffee	2.9, 1.9	2.4	--	0.084	--

Residues in animal commodities

The recommendations made by the current Meeting did not include animal feed items; therefore, the Meeting confirmed is previous recommendations for residues in animal commodities.

RECOMMENDATIONS

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

Definition of the residue for compliance with the MRL and dietary risk assessment for plant and animal commodities: *sulfoxaflor.*

The residue is not fat-soluble.

Table 2 Recommendations for residues of sulfoxaflor from the 2021 Extra JMPR.

CCN	Crop/Commodity	MRL, mg/kg		STMR or STMR-P, mg/kg	HR or HR-P, mg/kg
		New	Previous		
VS 0621	Asparagus	0.015		0.01	0.011
FI 0326	Avocado	0.15		0.011	0.036
FB 2006	Bush berries, Subgroup of	2		0.39	1.4
FB 2005	Cane berries, Subgroup of	1.5		0.44	0.78
SB 0716	Coffee beans	0.3		0.035	
FB 0267	Elderberry	2		0.39	1.4
FI 0345	Mango	0.3		0.022	0.061
For dietary risk assessment and/or dietary burden calculations					
	Coffee (instant)	--	--	0.084	
SM 0716	Coffee beans, Roasted	--	--	0.0167	

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for sulfoxaflor is 0–0.05 mg/kg bw. The International Estimated Daily Intakes (IEDIs) for sulfoxaflor were estimated for the 17 GEMS/Food Consumption Cluster Diets using the STMR or STMR-P values estimated by the JMPR. The results are shown in Annex 3 of the 2021 Extra JMPR Report.

The IEDIs ranged from 0–7% of the maximum ADI. The Meeting concluded that long-term dietary exposure to residues of sulfoxaflor from uses considered by the JMPR is unlikely to present a public health concern.

Acute dietary exposure

The ARfD for sulfoxaflor is 0.3 mg/kg bw. The International Estimate of Short-Term Intakes (IESTIs) for sulfoxaflor were calculated for the food commodities and their processed commodities for which HRs/HR-Ps or STMRs/STMR-Ps were estimated by the present Meeting and for which consumption data were available. The results are shown in Annex 4 of the 2021 Extra JMPR Report.

The IESTIs varied from 0–5% of the ARfD for children and 0–6% of the ARfD for the general population. The Meeting concluded that acute dietary exposure to residues of sulfoxaflor from uses considered by the present Meeting is unlikely to present a public health concern.

REFERENCES

Code	Author(s)	Year	Study Title
DAS 150108	Rawle, N. W.	2016	Validation of a Multi-Residue (QuEChERS) Analytical Method for the Determination of Sulfoxaflor and X11719474 in Crops, Dow AgroSciences Method 150108 GLP, Unpublished 06 January 2016
PR No. 11296	Dorschner, K.	2017	Sulfoxaflor: Magnitude of the Residue on Blueberry IR-4 Project PR No. 11296 GLP; Unpublished September 2017
PR No. 11279	Dorschner, K.	2017	Sulfoxaflor: Magnitude of the Residue on Caneberry IR-4 Project PR No. 11279 GLP; Unpublished May 2017
DAS 130161	Cenni, M.	2014	Residues of Sulfoxaflor in Avocados SynTech Research Laboratory Services, LLC Project No: 14SRLS13R-1 Dow AgroSciences LLC DAS Study ID 130161 GLP, Unpublished 12 May 2014
PR No. 11822	DeFrancesco, J.	2019	Sulfoxaflor: Magnitude of the Residue on Mango USDA/IR-4 Global Tropical Fruits Project PR Nos. 11822, 12502, 12503 GLP; Unpublished 22 November 2019

Code	Author(s)	Year	Study Title
PR No. 10858	Dorschner, K.	2017	Sulfoxaflor: Magnitude of the Residue on Artichoke (Globe) IR-4 Project PR No. 10858 GLP; Unpublished April 2017
PR No. 11321	Dorschner, K.	2017	Sulfoxaflor: Magnitude of the Residue on Asparagus IR-4 Project PR No. 11321 GLP; Unpublished August 2017
PR No. 11095	Dorschner, K.	2017	Sulfoxaflor: Magnitude of the Residue on Sunflower IR-4 Project PR No. 11095 GLP; Unpublished August 2017

TEBUCONAZOLE (189)

First draft prepared by Mr D Lunn, Ministry for Primary Industries, New Zealand

EXPLANATION

Tebuconazole is a triazole broad spectrum fungicide, acting by disrupting membrane function and inhibiting sterol biosynthesis. It is a systemic fungicide (primarily translocated acropetally), with protectant, curative and eradicant activity against wide range of diseases including rusts, smut, bunt, powdery mildew, leaf spots, and blights.

Tebuconazole was evaluated for toxicology and residues by the JMPR in 1994 and periodic reviews were conducted by the 2010 JMPR (toxicology) and the 2011 JMPR (residues). FAO Specifications for tebuconazole (technical material and related formulations) were published in 2000.

An ADI of 0–0.03 mg/kg bw and an ARfD of 0.3 mg/kg bw were established by the 2010 JMPR.

The definition of the residue for compliance with the MRL and dietary risk assessment for plant and animal commodities is: *tebuconazole. The residue is not fat-soluble*.

Tebuconazole was scheduled at the Fifty-first Session of the CCPR for evaluation of additional uses by the 2020 JMPR, which was postponed to the 2021 Extra JMPR. The Meeting received new (revised) GAP information and new supporting residue information for coffee.The Meeting also considered relevant information submitted to the 2011 JMPR.

METHODS OF RESIDUE ANALYSIS

Analytical methods

The 2011 JMPR reviewed and summarised analytical method descriptions and validation data for tebuconazole on a wide range of commodities. These included Method 01013 which was used to measure residues of tebuconazole in the new supervised residue trials on coffee beans and Method 00007/M035, used in the coffee bean processing study evaluated by the 2011 JMPR.

In Method 01013, tebuconazole residues were extracted in acetonitrile/water (4:1 v/v) and after filtration, and the addition of stable isotopically labelled tebuconazole, residues were quantified by reversed-phase HPLC-MS/MS without further clean-up. Detection was by electrospray ionisation in the positive ion mode. The 2011 JMPR reviewed the method validation studies for citrus fruit, green peas (seed), rape seed, wheat grain and corn (green material). The LOQ was 0.01 mg/kg in all matrices tested.

New validation studies for Method 01013 in coffee beans were provided to the current meeting and validation studies for Method 00007/M035 for processed coffee beans were considered by the 2011 JMPR.

Table 1 Method validation data for tebuconazole in coffee beans (fresh, roast and freeze-dried)

Matrix	Tebuconazole Recovery						
	Fortification (mg/kg)	n	Range (%)	Mean (%)	RSD (%)	Analytical method	Reference
Coffee bean							
Beans (dry)	0.01 1.0	7 7	95-104 96-111	100 102	3.5 5.5	01013	F12-026
	0.01 1.0	3 3	107-112 116-120	110 118	2.3 1.7	01013	F13-018

Matrix	Tebuconazole Recovery						
	Fortification (mg/kg)	n	Range (%)	Mean (%)	RSD (%)	Analytical method	Reference
Beans (dry)	0.01 0.1	3 8	90-90 74-113	90 98	0.0 13.4	00007/M035	JMPR 2011 (108948)
Beans (roasted)	0.08 0.1	3 3	71-83 70-95	77 80	7.9 16.9	00007/M035	JMPR 2011 (108948)
Instant coffee (freeze-dried)	0.04 0.1	3 3	73-85 68-79	77 75	9.0 7.9	00007/M035	JMPR 2011 (108948)

USE PATTERNS

Tebuconazole is produced by a number of manufacturers and is registered in many countries in the world. Predominant uses are in cereals and in oil and protein crops but it is also widely used in many fruit and vegetable crops and on tree nuts and coffee.

New information on GAP in Brazil was provided to the Meeting, including a revised use on coffee for a 300 SC combination product containing tebuconazole and trifloxystrobin. The Meeting also noted that there are a number of other SC and EC formulations registered in Brazil for use on coffee. These representative uses are summarised in Table 2.

Table 2 Representative authorised uses of tebuconazole on coffee

Crop	Country	Form	Application				Max/season		PHI (days)	Remarks
			method	kg ai/ha (max)	water L/ha	RTI (days)	no	kg ai/ha		
Coffee	Brazil	SC 300 [a]	foliar	0.2	400-500	21	3		30	Label provided
		SC 360 [b]	foliar	0.24	400	30	2		30	MAPA-AgroFit
		SC 430	foliar	0.2	380	NS	2		30	MAPA-AgroFit
		SC 430	foliar	0.2	250-500	30	5		30	MAPA-AgroFit
		SC320 [b]	foliar	0.15	400	30	2		30	MAPA-AgroFit
		EC 200	foliar	0.2	250-600	30	2		30	MAPA-AgroFit
		EC 200	foliar	0.2	380	45	2		30	MAPA-AgroFit
		EC 200	foliar	0.2	400-800	NS	2		30	MAPA-AgroFit
		EC 200	foliar	0.2	250-500	30	3		30	MAPA-AgroFit
		EC 200	foliar	0.2	250-500	30d	4		30	MAPA-AgroFit
		EC 200	foliar	0.2	250-500	NS	4		30	MAPA-AgroFit
		EC 200	foliar	0.2	250-500	30	5		30	MAPA-AgroFit

RTI = Re-treatment interval

[a] co-formulated with trifloxystrobin

[b] co-formulated with azoxystrobin

RESULTS OF SUPERVISED RESIDUE TRIALS ON CROPS

The Meeting reviewed new supervised coffee field trial information and relevant data from supervised field trials on coffee that were provided to the 2011 JMPR.

Crop Group	Commodity	Region/Country	Table No.

Seeds for beverages	Coffee (2011 JMPR) Coffee (New data)	Brazil Brazil	3 4

The new 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 rate, spray concentration and spray volume were not always identical from one application to the next. If the variation was small, only the final values for application rate, concentration and spray volume were recorded. For larger variations all values were recorded.

Intervals of freezer storage between sampling and analysis were recorded for all trials and were covered by the conditions of the freezer storage stability studies reviewed by the 2011 JMPR.

Results from replicated field plots are presented without correction for concurrent method recoveries. Residues and application rates have been rounded to two significant digits. The results from trials conducted according to the maximum GAP and used for the estimation of maximum residue levels have been (underlined).

Seeds for beverages and sweets

Coffee

Results from supervised trials from Brazil and Guatemala on coffee were evaluated by the 2011 JMPR. In these trials, conducted between 1990 and 2004, three or five foliar applications of tebuconazole (EC, WP or SC formulations) were applied to coffee bushes and dried coffee beans were sampled and analysed for tebuconazole, with LOQs ranging from 0.1 mg/kg in the earlier trials and 0.01 mg/kg in the more recent studies.

Table 3 Tebuconazole residues in coffee bean from supervised trials in Brazil and Guatemala. [See 2011 JMPR Tebuconazole Evaluation, Table 85]

COFFEE BEAN Country, year Location (variety)	Application				DALA	Tebuconazole Residues (mg/kg)	Reference & Comments
	no	kg ai/ha	kg ai/hl	water (L/ha)			
GAP: Brazil	3	0.2			30		RTI: 21 days
Brazil, 1990 (Sumatra)	3	0.25	0.05		5 15 30 45	<0.1 <0.1 <0.1 <0.1	BRA-122722
	3	0.5	0.1		30	<0.1	
Brazil, 1993 (Mundo novo)	3	0.25	0.025		30	<0.1	BRA-143556
	3	0.5	0.05		30	<0.1	
Brazil, 1995 (Catuai vermelho)	3	0.2	0.05		30	< 0.1	BRA-USP 1976/95
	3	0.4	0.1		30	< 0.1	
Brazil, 1996 (Novo mundo)	5	0.25	0.05		-0 0 7	< 0.01 0.03 < 0.01	108947* BRA-FR-C01-96D

COFFEE BEAN Country, year Location (variety)	Application				DALA	Tebuconazole Residues (mg/kg)	Reference & Comments
	no	kg ai/ha	kg ai/hl	water (L/ha)			
					15 22 30 45 60	0.01 < 0.01 < 0.01 < 0.01 < 0.01	
	5	0.25	0.05		0 30	0.07 0.06	BRA-FR-C02-96H
	5	0.25	0.05		0 30	0.01 < 0.01	BRA-FR-C03-96H
Brazil, 1996 (Catuai amarelo)	5	0.25	0.05		0 30	0.02 < 0.01	BRA-FR-C04-96H
Brazil, 1998 (Mundo novo)	5	0.2	0.067		30	< 0.1	BRA-USP 2545/98
	5	0.4	0.13		30	< 0.1	
Brazil, 2004 (Catuai)	3	0.2	0.04		30	0.02	BRA-FR04BRA049-P1
	3	0.4	0.08		30	0.05	
Brazil, 2004 (Catuai)	3	0.2	0.04		30	0.02	BRA-FR04BRA049-P2
	3	0.4	0.08		30	0.05	
Guatemala, 1996 (Caturra)	3	0.25	0.042		0* 0 7 14 21 28 45 60	0.02 0.01 0.02 0.02 0.05 0.03 0.02 0.03	108947* GUA-FR-C01-96D
Guatemala, 1996	3	0.251	0.042		0 28	0.03 0.02	GUA-FR-C02-96H
Guatemala, 1996	3	0.251	0.042		0 28	0.01 < 0.01	GUA-FR-C03-96H
Guatemala, 1996 (Catimor 5269)	3	0.25	0.041		0 28	0.02 0.01	GUA-FR-C04-96H

In new trials on coffee, conducted in Brazil (2012–2013), three foliar applications of tebuconazole (300SC tebuconazole + trifloxystrobin formulation) were applied to coffee bushes, about 20 days apart from early fruit development, using single-nozzle pressurised or motorised knapsack sprayers to apply about 400 litres spray mix/ha, with added surfactant. Plot sizes were at least 60 square metres.

Samples of ripe cherries (about 3 kg) were sun-dried for up to 20 days before being processed to remove the pulp. After pulp removal, at least 1 kg samples of beans were placed into frozen storage (below -20 °C) for up to 6 months before analysis. Tebuconazole residues were measured using Method 01013, with a LOQ of 0.01 mg/kg. Mean concurrent recovery rates ranged from 100–118% in samples spiked with 0.01 mg/kg or 1.0 mg/kg.

Table 4 Tebuconazole residues in coffee beans from supervised trials in Brazil

COFFEE BEAN Country, year Location (variety)	Application				DALA	Tebuconazole Residues (mg/kg)	Reference & Comments
	no	kg ai/ha	kg ai/hl	water (L/ha)			
GAP: Brazil	3	0.2			30		RTI: 21 days
Brazil, 2012 Paulinia (Mundo novo)	3	0.2	0.05	400	0 15 30 45	0.05 0.03 0.02 0.03	F12-026 F12-026-01
Brazil, 2012 Aragurari (Mundo novo)	3	0.2	0.05	400	0 15 30 45	0.17 0.03 0.01 0.03	F12-026 F12-026-02
Brazil, 2012 Ribeirão Preto (Catuai)	3	0.2	0.05	400	0 15 30 45	0.06 0.10 0.20 0.12	F12-026 F12-026-03
Brazil, 2012 Cristais Paulista (Mundo novo)	3	0.21	0.051	400	28	0.09	F12-026 F12-026-04
Brazil, 2012 Campinas (Catuai vermelho)	3	0.2	0.05	400	30	0.05	F12-026 F12-026-05
Brazil, 2013 Ribeirao Preto (Catuai)	3	0.2	0.05	400	29 45 60	0.03 0.01 <0.01	F13-018 F13-018-01
Brazil, 2013 San Antonio da Alegria (Mundo novo)	3	0.21	0.05	410	30 45 60	0.03 0.04 0.02	F13-018 F13-018-02

FATE OF RESIDUES IN STORAGE AND IN PROCESSING

Magnitude of the residue in processing

Processing factors derived by the 2011 JMPR for processed coffee commodities are summarized below:

Table 5 Summary of coffee processing factors for tebuconazole

Raw commodity	Processed commodity	Calculated processing factors [a]	Processing factor
Beans (dried)	Roasted beans	2	2
	Freeze-dried (instant) coffee	0.8	0.8

[a] The processing factor is the ratio of the total residue in the processed item divided by the total residue in the RAC.

APPRAISAL

Tebuconazole is a triazole broad spectrum fungicide, acting by disrupting membrane function and inhibiting sterol biosynthesis. It is a systemic fungicide (primarily translocated acropetally), with protectant, curative

and eradicant activity against wide range of diseases including rusts, smut, bunt, powdery mildew, leaf spots, and blights.

Tebuconazole was evaluated for toxicology and residues by the JMPR in 1994 and periodic reviews were conducted by the 2010 JMPR (toxicology) and the 2011 JMPR (residues). FAO Specifications for tebuconazole (technical material and related formulations) were published in 2000.

An ADI of 0–0.03 mg/kg bw and an ARfD of 0.3 mg/kg bw were established by the 2010 JMPR.

The definition of the residue for compliance with the MRL and dietary risk assessment for plant and animal commodities is: *tebuconazole.* The residue is not fat-soluble.

Tebuconazole was scheduled at the Fifty-first Session of the CCPR for evaluation of additional uses by the 2020 JMPR, which was postponed to the 2021 Extra JMPR. The Meeting received new (revised) GAP information and new supporting residue information for coffee.

Results of supervised residue trials on crops

New supervised trials from Brazil were available for the use of tebuconazole on coffee to supplement those reported by the 2008 JMPR. The analytical method used in these trials was reviewed by the 2011 JMPR and additional method validation studies for coffee bean were considered by the Meeting. The demonstrated stability of residues in frozen samples (at least 30 months) covered the storage intervals in the trials considered by the Meeting.

A new product label was available from Brazil and the Meeting noted that a number of authorised tebuconazole formulations with label claims for use on coffee were listed on the MAPA AgroFit website.

Coffee bean

In 2011 the JMPR identified the critical GAP for tebuconazole on coffee as three foliar applications of 0.25 kg ai/ha, with a PHI of 30 days and estimated a maximum residue level of 0.1 mg/kg for tebuconazole in coffee beans.

The current Meeting received new Brazilian GAP information and supporting residue trials. Noting that the critical GAP identified by the 2011 JMPR was no longer authorised, the Meeting identified a new critical GAP of 3 foliar applications of 0.2 kg ai/ha, with a 21-day re-treatment interval and a PHI of 30 days.

In trials conducted in Brazil and matching the new critical GAP, residues of tebuconazole in coffee beans were (n = 7): 0.03 (3), 0.04, 0.05, 0.09 and 0.2 mg/kg.

The Meeting estimated a maximum residue level of 0.4 mg/kg and a STMR of 0.04 mg/kg for tebuconazole in coffee beans to replace the previous maximum residue level recommendation of 0.1 mg/kg.

Fate of residues during processing

The 2011 JMPR reviewed information on the fate of tebuconazole during processing of coffee beans.

The Meeting confirmed the 2011 JMPR decision that one processing study was not sufficient to recommend a maximum residue level for coffee, roasted.

Table 1 Processing factors estimated by the 2011 JMPR for dietary exposure estimation of tebuconazole residues in coffee commodities

Raw commodity [STMR]	Processed commodity	Individual processing factors	Mean or best estimate processing factor	STMR-P [$STMR_{RAC}$ × PF] (mg/kg)	Median residue [$STMR_{RAC}$ × PF] (mg/kg)
Coffee bean [0.04 mg/kg]	Roasted beans	2	2	0.08	
	Freeze dried (instant) coffee	0.8	0.8	0.032	

RECOMMENDATIONS

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

Definition of the residue for compliance with the MRL and dietary risk assessment for plant and animal commodities: T*ebuconazole*

The residue is not fat-soluble.

Table 2 Recommendations for residues of tebuconazole from the 2021 Extra JMPR

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
SB 0716	Coffee beans	0.4	0.1	0.04	
SM 0716	Coffee beans, Roasted			0.08	
	Freeze dried (instant) coffee			0.032	

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for tebuconazole is 0–0.3 mg/kg bw. The International Estimated Daily Intakes (IEDIs) for tebuconazole were estimated for the 17 GEMS/Food Consumption Cluster Diets using the STMR or STMR-P values estimated by the JMPR. The results are shown in Annex 3 of the 2021 Extra JMPR Report.

The IEDIs ranged from 1–9% of the maximum ADI. The Meeting concluded that long-term dietary exposure to residues of tebuconazole from uses considered by the JMPR is unlikely to present a public health concern.

Acute dietary exposure

The ARfD for tebuconazole is 0.3 mg/kg bw. The International Estimate of Short Term Intakes (IESTIs) for tebuconazole were calculated for the food commodities and their processed commodities for which HRs/HR-Ps or STMRs/STMR-Ps were estimated by the present Meeting and for which consumption data were available. The results are shown in Annex 4 of the 2021 Extra JMPR Report.

The IESTIs were 0% of the ARfD for children and for the general population. The Meeting concluded that acute dietary exposure to residues of tebuconazole from uses considered by the present Meeting is unlikely to present a public health concern.

REFERENCES

Reference	Author(s)	Year	Title	Edition No
F12-026	Resende, G.	2012	Determinação de resíduos de tebuconazol, trifloxystrobina e seu respectivo metabólito CGA 321113, na cultura do café após aplicações em pulverização foliar de Nativo juntamente com o adjuvante óleo metilado de soja em ensaios no Brasil. Report No. F12-026	M-443949-01-1
F12-026 (translation)	Anon.	2012	HWG 1608 & CGA 279202; SC 300; coffee; Brazil; BBA	M-445260-01-1
F13-018	Resende, G.	2014	Determinação de resíduos de tebuconazole, trifloxystrobin e seu respectivo metabólito CGA 321113, na cultura do Café após aplicações em pulverização foliar de Nativo juntamente com o adjuvante óleo metilado de soja em ensaios no Brasil. Report No. F13-018	M-487637-01-1

THIAMETHOXAM (245)

First draft prepared by Dr M Doherty, the Environmental Protection Agency, United States of America

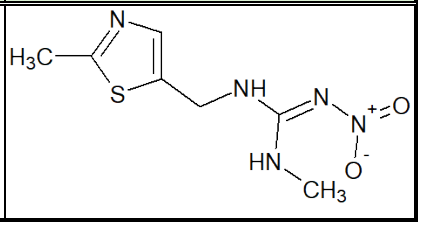
EXPLANATION

Thiamethoxam (ISO common name) is a broad-spectrum, neonicotinoid insecticide with registered uses on multiple crops. It was evaluated for the first time by the 2010 JMPR, which established an acceptable daily intake (ADI) of 0–0.08 mg/kg bw and an acute reference dose (ARfD) of 1 mg/kg bw. Thiamethoxam underwent subsequent evaluations by the JMPR in 2011, 2012, and 2014.

The definition of the residue for compliance with the MRL for animal and plant commodities is *thiamethoxam*. For dietary risk assessment, the residue definitions are thiamethoxam and clothianidin (CGA322704), assessed separately, for plant and animal commodities except poultry and *the sum of thiamethoxam, CGA 265307 (N-(2-chlorothiazol-5-ylmethyl)-N'-nitroguanidine), and MU3 (amino-([(2-chlorothiazol-5-ylmethyl)-amino]-methylene)-hydrazide), expressed as thiamethoxam*, along with *clothianidin* (assessed separately) for poultry commodities. *The residue is not fat-soluble*.

Thiamethoxam was scheduled at the Fifty-first Session of the CCPR for evaluation of additional uses by the 2020 JMPR, which was postponed to the 2021 Extra JMPR. The current Meeting received information on analytical methods, field trials, and processing studies to support estimation of maximum residue levels in persimmon, barley, rice, sorghum, sweet corn, and wheat.

Table 1 Metabolites of thiamethoxam referenced in this document

Common or code name	Chemical name	Structure
CGA-322704, clothianidin	(*E*)-1-[(2-chlorothiazol-5-yl)methyl]-3-methyl-2-nitroguanidine	

RESIDUE ANALYSIS

Analytical methods

Method AG-675

This method was reviewed by the 2010 JMPR and determined to be suitable. Method AG-675 was used for the analysis of residues in wheat and barley matrices.

Concurrent recoveries of thiamethoxam and clothianidin from barley and wheat matrices were determined at fortifications ranging from 0.01 mg/kg up to at least 5 mg/kg. Average concurrent recoveries in wheat ranged from 87 to 101% for both thiamethoxam and clothianidin across grain, forage, hay, straw, aspirated grain fractions, bran, flour, middlings, shorts, and germ, with relative standard deviations of up to 12% (except aspirated grain fractions). For wheat aspirated grain fractions, the relative standard deviation on concurrent recovery of thiamethoxam was ca. 19%. Average concurrent recoveries in barley ranged from 83 to 99% with relative standard deviations of up to 10% for both thiamethoxam and clothianidin across grain, hay, and straw. The concurrent recoveries confirmed the LOQ of 0.01 mg/kg for both analytes.

Method GRM.009.02A

This method (Perez *et al*., 2009, Study Report CGA293343_50046) is a modification of Method AG-675, but has not been reviewed by the JMPR previously. Briefly, residues are extracted in acetonitrile:water (8:2, v/v), concentrated and brought to volume in acetonitrile:water (1:9, v/v), and analysed by LC-MS/MS. Relative to Method AG-675, this method omits SPE and liquid-liquid partitioning cleanup steps and updates the analysis from HPLC-UV.

Method GRM.009.02A was used for the analysis of sweet corn, rice and sorghum samples and was validated to an LOQ of 0.01 mg/kg for both analytes.

QuEChERS

The QuEChERS multiresidue method was validated for the analysis of thiamethoxam and clothianidin in spinach, whole orange, wheat grain, and oilseed rape seed (Class and Richter, 2012, Study Report CGA293343_11576). The method was validated to a LOQ of 0.01 mg/kg for both analytes in all matrices. Significant matrix effects were noted, and the validation was performed with matrix-matched standards.

Unspecified (Japanese persimmon)

The analytical method used for Japanese persimmon in the supervised field trials from the 2000 growing season (Kato, 2000 and 2001, Reports A12182A-10165 and -10166) consisted of extraction of residues into acetone/water (8:2, v/v) by shaking for 30 minutes, filtering through filter paper, and concentration via rotary evaporation at ≤40 °C to remove acetone. The extract was then cleaned up using, sequentially, a diatomaceous earth column, a cation-exchange mini-column, and an alumina N mini-column. Quantification of residues was by HPLC-UV. This method is similar to Method MFDS201304 evaluated by the 2014 JMPR and determined to be suitable for analysis of residues in persimmon, with LOQs of 0.02 mg/kg for each of thiamethoxam and clothianidin.

Recovery data for the above methods are summarized below.

Table 2 Summary of validation (V), independent validation (I), and concurrent (C) recovery of thiamethoxam and clothianidin from barley, rice, spinach, orange, wheat, and oilseed rape commodities

Matrix	Analyte	Fortification level [mg/kg]	Individual recoveries [%]	Range of recoveries [%]	Mean recovery [%]	RSD [%]	Reference
Japanese persimmon	Thiamethoxam	0.25	C: 76, 84, 95, 97	76-97	88	11	A12182A-10165
	Clothianidin	0.25	C: 91, 92, 96, 101	91-101	95	4.8	
	Thiamethoxam	0.2	C: 87, 89, 90, 90	87-90	89	1.6	A12182A-10166
	Clothianidin	0.2	C: 94, 95, 95, 97	94-97	95	1.3	
Method AG-675							
Wheat forage	Thiamethoxam	0.01	C: 95, 116, 87, 100, 91, 91, 106, 108, 107, 88	87-116	99	10	TK0020708
		5	C: 89, 102, 85, 91, 111, 105, 113, 83, 102, 106	83-113	99	11	
		7	C: 102	--	100	--	
	Clothianidin	0.01	C: 103, 88, 103, 92, 98, 92, 91 93, 111, 96	88-111	97	7.3	

Matrix	Analyte	Fortification level [mg/kg]	Individual recoveries [%]	Range of recoveries [%]	Mean recovery [%]	RSD [%]	Reference
		5	C: 89, 103, 82, 86, 120, 117, 118, 91, 109, 105	82-120	100	14	
Wheat hay	Thiamethoxam	0.01	C: 101, 79, 88, 71, 84, 98, 113 98, 109, 84	71-113	92	15	
		5	C: 96, 83, 82, 84, 94, 94, 81, 103, 81	81-103	89	9.2	
		15	C: 85	--	85	--	
	Clothianidin	0.01	C: 94, 80, 86, 73, 87, 95, 109, 101, 101, 88	73-109	91	12	
		5	C: 99, 84, 78, 81, 102, 95, 86, 105, 85	78-105	91	11	
		15	C: 87	--	87	--	
Wheat grain	Thiamethoxam	0.01	C: 87, 77, 82, 98, 95, 89, 108, 103, 100, 104, 93	77-108	94	10	
		5	C: 116, 91, 98, 84, 91, 108, 95 96, 109, 107, 90	84-116	99	10	
	Clothianidin	0.01	C: 76, 87, 80, 84, 90, 108, 93, 86, 93, 112, 90	76-112	91	12	
		5	C: 112, 94, 105, 93, 95, 110, 96, 99, 111, 114, 86	86-114	100	9.4	
Wheat straw	Thiamethoxam	0.01	C: 110, 82, 77, 88, 71, 100, 83 82, 82	71-110	86	14	
		5	C: 76, 78, 81, 88, 98, 95, 96, 97, 96	76-98	89	10.0	
	Clothianidin	0.01	C: 84, 88, 89, 87, 70, 101, 72, 87, 78	70-101	84	11	
		5	C: 83, 83, 84, 87, 101, 86, 90, 92, 91	83-101	89	6.5	
Wheat AGF	Thiamethoxam	0.01	C: 74	--	74	--	
		5	C: 102, 108	102-108	100	--	
	Clothianidin	0.01	C: 91, 102	91-102	96	--	
		5	C: 100, 108	100-108	100	--	
Wheat bran	Thiamethoxam	0.01	C: 94, 95	94-95	94	--	
		5	C: 92, 104	92-104	98	--	
	Clothianidin	0.01	C: 89, 101	89-101	95	--	
		5	C: 88, 104	88-104	96	--	
Wheat flour	Thiamethoxam	0.01	C: 98, 100	98-100	99	--	
		5	C: 86, 102	86-102	94	--	
	Clothianidin	0.01	C: 105, 109	105-109	110	--	
		5	C: 89, 102	89-102	96	--	
Wheat middlings	Thiamethoxam	0.01	C: 90, 98	90-98	94	--	

Matrix	Analyte	Fortification level [mg/kg]	Individual recoveries [%]	Range of recoveries [%]	Mean recovery [%]	RSD [%]	Reference
		5	C: 88, 103	88-103	96	--	
	Clothianidin	0.01	C: 102, 109	102-109	110	--	
		5	C: 90, 103	90-103	96	--	
Wheat shorts	Thiamethoxam	0.01	C: 84, 88	84-88	86	--	
		5	C: 87, 98	87-98	92	--	
	Clothianidin	0.01	C: 91, 94	91-94	92	--	
		5	C: 93, 101	93-101	97	--	
Wheat germ	Thiamethoxam	0.01	C: 94, 98	94-98	96	--	
		5	C: 88, 98	88-98	93	--	
	Clothianidin	0.01	C: 84, 97	84-97	90	--	
		5	C: 95, 97	95-97	96	--	
Barley hay	Thiamethoxam	0.01	C: 104, 106, 124c, 93, 91	91-106	98	7.7	TK0011969
		5	C: 105, 81, 89, 85, 93	81-105	91	10	
	Clothianidin	0.01	C: 79, 102, 136[c)], 101, 104	79-104	96	12	
		5	C: 102, 87, 90, 93, 94	87-102	93	6.1	
Barley straw	Thiamethoxam	0.01	C: 84, 77, 88, 84	77-88	83	5.5	
		5	C: 82, 82, 85, 89	82-89	84	3.9	
	Clothianidin	0.01	C: 86, 93, 83, 92	83-93	88	5.4	
		5	C: 82, 91, 90, 95	82-95	90	6.1	
Barley grain	Thiamethoxam	0.01	C: 79, 85, 94, 99	79-99	89	10	
		5	C: 91, 96, 99, 100	91-100	96	4.2	
	Clothianidin	0.01	C: 82, 95, 106, 99	82-106	96	11	
		5	C: 99, 102, 104, 104	99-104	100	2.4	
Method GRM.009.02A							
Barley grain	Thiamethoxam	0.01	V: 91, 96 C: 116	94 --	-- 120	3.8 --	ADPEN M1104
		0.1	V: 113, 117 C: 91	120 91	-- --	2.4	
	Clothianidin	0.01	V: 106, 108 C: 128	110 130	-- --	1.3	
		0.1	V: 108, 109 C: 105	110 100	-- --	0.64	
Barley straw	Thiamethoxam	0.01	V: 82, 117 C: 104	100 100	-- --	25	
		0.1	V: 90, 102 C: 109	96 110	-- --	8.8	
	Clothianidin	0.01	V: 83, 99 C: 107	91 110	-- --	12	

Matrix	Analyte	Fortification level [mg/kg]	Individual recoveries [%]	Range of recoveries [%]	Mean recovery [%]	RSD [%]	Reference
		0.1	V: 78, 90 C: 101	84 100	-- --	10	
Barley hay	Thiamethoxam	0.01	V: 69, 105 C: 78	87 78	-- --	29	
		0.1	V: 82, 108 C: 102	95 100	-- --	19	
	Clothianidin	0.01	V: 82, 90 C: 99	86 99	-- --	6.6	
		0.1	V: 72, 86	79	--	13	
			C: 92	92	--		
Rice grain (rough)	Thiamethoxam	0.01	V: 99, 102, 104 C: 77, 79, 84, 87, 91, 94, 98	100 87	-- 8.9	2.5 8.9	
		0.05	V: 79, 79, 80	79	--	0.73	
		0.1	V: 76, 78, 78 C: 71, 73, 74, 85, 86, 86, 97	77 82	-- 11	1.5 11	
	Clothianidin	0.01	V: 99, 100, 100 C: 74, 78, 78, 83, 87, 91, 100	100 84	-- 11	0.58 11	
		0.05	V: 90, 90, 91	90	--	0.64	
		0.1	V: 83, 86, 88 C: 71, 76, 79, 87, 90, 90, 91	86 83	-- 9.7	2.9 9.7	
Rice bran	Thiamethoxam	0.01	C: 83	83	--		
		0.1	C: 70	70	--		
	Clothianidin	0.01	C: 85	85	--		
		0.1	C: 80	80	--		
Rice straw	Thiamethoxam	0.01	V: 87, 89, 93 C: 73, 79, 83, 94, 110	90 88	-- 17	3.4 17	
		0.05	V: 73, 73, 73	73	--	0.0	
		0.1	V: 63, 66, 67 C: 61, 67, 70, 72	65 68	-- 7.1	3.2 7.1	
	Clothianidin	0.01	V: 92, 94, 96 C: 72, 75, 75, 76, 77	94 75	-- 2.5	2.1 2.5	
		0.05	V: 85, 86, 88	86	--	1.8	
		0.1	V: 80, 85, 88 C: 70, 71, 72, 73	84 72	-- 1.8	4.8 1.8	
Rice hulls	Thiamethoxam	0.01	C: 90	90	--		
		0.1	C: 51	51	--		
	Clothianidin	0.01	C: 66	66	--		
		0.1	C: 77	77	--		

Matrix	Analyte	Fortification level [mg/kg]	Individual recoveries [%]	Range of recoveries [%]	Mean recovery [%]	RSD [%]	Reference
Rice grain (polished)	Thiamethoxam	0.01	C: 72, 115	94	--	32	
		0.1	C: 101	100	--		
		0.2	C: 95	95	--		
	Clothianidin	0.01	C: 77, 80	78	--	2.7	
		0.1	C: 104	100	--		
		0.2	C: 117	--	120	--	
Sweet corn forage	Thiamethoxam	0.01	V: 71, 72, 73	71-73	72	1.4	TK0014208
		5	V: 76, 101, 102	76-102	93	16	
	Clothianidin	0.01	V: 77, 77, 79	77-79	78	1.5	
		5	V: 71, 93, 96	71-96	87	16	
Sweet corn kernel + cob w/ husk removed	Thiamethoxam	0.01	V: 78, 86, 100	78-86	88	13	
		5	V: 102, 114, 116	102-116	111	6.8	
	Clothianidin	0.01	V: 76, 78, 82	76-82	79	3.9	
		5	V: 90, 93, 97	90-97	93	3.8	
Sweet corn stover	Thiamethoxam	0.01	V: 78, 79, 82	78-82	80	2.6	
		5	V: 99, 102, 102	99-102	101	1.7	
	Clothianidin	0.01	V: 72, 77, 78	72-78	75	4.2	
		5	V: 91, 93, 102	91-102	95	6.2	
QuEChERS							
Spinach	Thiamethoxam	0.01	V: 86, 88, 91, 92, 93	86-93	90	3.2	B 2661 G
			I: 94, 97, 101, 102, 103	94-103	99	3.8	
		0.1	V: 91, 92, 93, 94, 95	91-95	93	1.7	
			I: 87, 91, 91, 92, 95	87-95	91	3.1	
	Clothianidin	0.01	V: 89, 89, 89, 91, 94	89-94	90	2.4	
			I: 94, 94, 96, 101, 102	94-102	97	4.0	
		0.1	V: 89, 91, 93, 95, 96	89-96	93	3.1	
			I: 95, 97, 100, 101, 101	95-101	99	2.7	
Whole orange	Thiamethoxam	0.01	V: 88, 93, 94, 97	88-97	93	4.0	
		0.1	V: 91, 93, 93, 94, 97	91-97	94	2.3	
	Clothianidin	0.01	V: 92, 102, 103, 104, 105	92-105	100	5.3	
		0.1	V: 93, 98, 99, 100, 102	93-102	98	3.4	
Wheat grain	Thiamethoxam	0.01	V: 86, 87, 88, 89, 92	86-92	88	2.6	
			I: 92, 100, 106, 109, 109	92-109	100	7.3	
		0.1	V: 84, 85, 86, 87, 87	84-87	86	1.5	

Matrix	Analyte	Fortification level [mg/kg]	Individual recoveries [%]	Range of recoveries [%]	Mean recovery [%]	RSD [%]	Reference
			I: 103, 104, 106, 107, 110	103-110	110	2.5	
	Clothianidin	0.01	V: 89, 93, 93, 95, 96 I: 91, 99, 102, 104, 107	89-96 91-107	93 100	2.9 6.1	
		0.1	V: 87, 90, 92, 94, 95 I: 107, 107, 109, 110, 110	87-95 107-110	92 110	3.5 1.4	
Oilseed rape seed	Thiamethoxam	0.01	V: 93, 96, 96, 97, 98 I: 80, 83, 84, 86, 86	93-98 80-86	96 84	1.9 3.0	
		0.1	V: 91, 92, 94, 95, 96 I: 91, 93, 94, 98, 103	91-96 91-103	94 96	2.2 5.0	
	Clothianidin	0.01	V: 89, 92, 99, 102, 103 I: 92, 93, 95, 95, 96	89-103 92-96	97 94	6.4 1.7	
		0.1	V: 87, 90, 93, 93, 96 I: 92, 93, 96, 105, 105	87-96 92-105	92 98	3.7 6.5	

[c] Corresponding control sample had residues >1/2 the fortification level. Result considered invalid.

USE PATTERN

Registered labels describing the use of thiamethoxam were provided to the current Meeting for persimmon, wheat, barley, rice, sorghum, and sweet corn (Table 3).

Table 3 Registered uses of thiamethoxam provided to the 2021 Extra JMPR

Use site	Country	Formulation		Application					PHI, days
		Conc.	Type	Rate, g/ha/applic	Rate, g/ha/year	Water, L/ha	Max No.	Interval, days	
Persimmon	Republic of Korea	10%	WG	2000× dilution	n.s.	Apply to runoff	3	10	7
Wheat	Mexico [C]	216 g/L	SC	56	n.s.	400	2	7	14
	USA [a]	599 g/L	FS	52 g ai/100 kg seed [f]	--	Seed trtmt	1	--	--
		216 g/L	SC	96 [b]	193 [b]	94 (grnd) 19 (air)	2	5	7 (forage/hay) and 30 (grain/straw)
Barley	Mexico [c]	216 g/L	SC	56	n.s.	400	2	7	14
	USA [a]	599 g/L	FS	52 g ai/100 kg seed	--	Seed trtmt	1	--	--
		216 g/L	SC	96 [b]	193 [b]	94 (grnd) 19 (air)	2	5	7 (forage/hay) and 30 (grain/straw)
Rice	Japan	6.5	SC	1000× dilution	n.s.	1500	2	n.s.	14

Use site	Country	Formulation		Application					PHI, days
		Conc.	Type	Rate, g/ha/applic	Rate, g/ha/year	Water, L/ha	Max No.	Interval, days	
	Republic of Korea	10%	WG	1250× dilution	n.s.	Until dripping	n.s.	7	15
	USA	599 g/L	FS	52 g ai/100 kg seed	--	Seed trtmt	1	--	--
		216 g/L	SC	96 [b]	193 [b]	n.s. (grnd) 19 (air)	n.s.	5	21
Sorghum	Mexico (grain)	216 g/L	SC	56	n.s.	400	2	n.s.	30
	USA [d] (grain & sweet)	599 g/L	FS	2.97 g ai/kg seed	--	Seed trtmt	1	--	--
		40%	WG	96 [b]	193 [b]	94 (grnd) 19 (air)		7	7 (forage) and 14 (grain/stover) and 14 (stalks)
Sweet corn	USA	599 g/L	FS	1.25 mg ai/kernel [e]	--	Seed trtmt	1	--	--
		216 g/L	SC	96 [b]	193 [b]	94 (grnd) 19 (air)	2	5	1 (silage/forage/ears) and 26 (fodder/stover)

[a] Registered use pattern applies to barley, oats, rye, triticale, and wheat.

[b] Do not exceed 96 g ai/ha per application or 193 g ai/ha per year of thiamethoxam from any product.

[c] Registered use pattern applies to barley, oats, triticale, and wheat.

[d] Do not apply between heading (50% of panicles visible) and completion of pollen shed.

[e] Do not apply at a rate that will exceed 235 g/ha based on a seeding rate of 30,351 seeds/ha (75,000 seeds/A).

[f] The label also specifies 0.03 mg thiamethoxam/seed, assuming 9525 seeds/kg (21000 seeds/pound).

RESULTS OF SUPERVISED RESIDUE TRIALS ON CROPS

The Meeting received data from supervised residue trials conducted on persimmon, wheat, barley, rice, sorghum, and sweet corn.

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. For most trials, detailed information on the field site and treatment parameters were provided. Analytical reports were sufficiently detailed and included example chromatograms and example calculations. Samples were analysed by the method described above for plant commodities.

The field trial study designs included control plots. Measured residues from control plots were <LOQ and are not included in the summary tables in this evaluation.

When calculating average residues, values below the LOQ were assumed to be at the LOQ. In the summary tables, residue values leading to maximum residue estimations and used for long-term dietary risk assessment are underlined. The highest individual values selected for estimating acute dietary risks are emboldened.

Supervised trials for thiamethoxam

Category	Crop	Table
Assorted Sub-tropical fruit, edible peel	Persimmon	Table4
Grasses (cereal grains)	Wheat	Table 5
	Barley	Table 6
	Rice	Table 7
	Sorghum (grain)	Table 8
	Sweet corn	Table 9
Grasses for sugar or syrup	Sorghum (sweet)	Table 10
Animal Feeds	Wheat	Table 11
	Barley	Table 12
	Rice	Table 13
	Sorghum	Table 14
	Sweet corn	Table 15

Japanese persimmon

Two crop residue analysis reports were provided for persimmon (2000, Report A12182A-10165; 2001, Report A12182A-10166). The reports provide results for the same two sites each, with each site divided into one untreated plot and two treated plots. The trials conducted at the same locations are not considered to be independent. Each treated plot received 3 applications of thiamethoxam on a 7–day retreatment interval. Applications consisted of spraying a 2000× dilution of a 10% ai SG product at a rate of 5000 L/ha. Harvest occurred 3, 7, 14, or 21 days after the last application.

Following harvest, samples (~2 kg fruits) were shipped under refrigerated conditions (*ca.* 5 °C) to the analytical facility. Time from harvest to residue analysis was typically 1–2 days.

Samples were analysed for residues of thiamethoxam and clothianidin using a method similar to MFDS201304, which was evaluated by the 2014 JMPR and determined to be suitable for analysis of those residues in persimmon. Mean concurrent recoveries at 0.25 ppm for each analyte ranged from 80 to 99%. The LOD was 0.005 mg/kg; a LOQ was not reported.

Table 4 Results of thiamethoxam residue trials in **persimmon** in Japan. Trials conducted at the same location are not considered to be independent

Location; year (Trial ID)	Crop Variety	Application				DALA	Residues (mg/kg)Mean of duplicate analysis		Study report
		No. [interval, days]	Dilution	L/ha	Rate, g ai/ha		Thiamethoxam	Clothianidin	
Critical GAP (KR)	--	3 [10]	2000×	Until dripping		7	--		
Fukushima, JP; 2000 1	Hiratanenashi	1 [--] 2 [7] 3 [7]	2000×	5000		3	0.243	<0.02	A12182A-10165
						7	0.200	<0.02	
						14	0.172	<0.02	
2		1 [--]	2000×	5000		21	0.187	<0.02	

Location; year (Trial ID)	Crop Variety	Application				DALA	Residues (mg/kg)Mean of duplicate analysis		Study report
		No. [interval, days]	Dilution	L/ha	Rate, g ai/ha		Thiamethoxam	Clothianidin	
		2 [7] 3 [7]							
Fukushima, JP; 2000 1	Hiratanenashi	1 [--] 2 [7] 3 [7]	2000×	5000		3	0.320	<0.02	A12182A-10166
						7	0.261	<0.02	
						14	0.210	<0.02	
2		1 [--] 2 [7] 3 [7]	2000×	5000		21	0.202	<0.02	
Wakayama, JP; 2000 1	Fuyuh	1 [--] 2 [7] 3 [7]	2000×	5000		3	0.086	<0.02	A12182A-10165
						7	0.072	<0.02	
						14	0.092	<0.02	
2		1 [--] 2 [7] 3 [7]	2000×	5000		21	0.048	<0.02	
Wakayama, JP; 2000 1	Fuyuh	1 [--] 2 [7] 3 [7]	2000×	5000		3	0.164	<0.02	A12182A-10166
						7	0.081	<0.02	
						14	0.062	0.014	
2		1 [--] 2 [7] 3 [7]	2000×	5000		21	0.072	<0.02	

Wheat

Nineteen field trials were conducted in the USA during the 2010 season (Willard, T, 2013, Study Report TK0020708). Treatment consisted of combined seed treatment (FS formulation) and foliar (WG formulation) applications. The seed treatment was made at a nominal rate of 52 g ai/100 kg seed. Plants grown from treated seed received 2 foliar applications, each at a nominal rate of 96 g ai/ha 5 days apart. An adjuvant was used with each foliar application. At three locations, additional plots were established to evaluate aerial (ultra-low volume) application. Grain was harvested 14 DALA; the sample size was not reported.

Following harvest, samples were put into frozen storage at the field trial facilities and transported frozen to the laboratory. In the laboratory, the samples were placed into frozen storage. Grain samples were

stored for up to 8.8 months. The storage period is covered by previously evaluated storage stability data that showed residues are stable under frozen conditions in a wide range of commodities for 1–2 years.

Samples were analysed for residues of thiamethoxam and clothianidin using method AG-675. Concurrent recovery data indicate that the method is suitable.

Table 5 Results of thiamethoxam residue trials in **wheat (grain)** in 2010

Location; (Trial ID)	Crop Variety	Application			DALA	Residues (mg/kg) [Mean]		Study report
		No. [interval, days]	Rate, g ai/ha	L/ha		Thiamethoxam	Clothianidin	Remarks
Critical GAP (US)	--	1 [--] 2 [--] 3 [5]	52** 96† 97	n/a 94 (grnd) 19 (air)	30	--	--	
GAP (MX)		1 [--] 2 [7]	56 56	400	14			
Suffolk, VA, US; (E07-0061)	Art	1 [--] 2 [252] 3 [5]	58** 98.52 98.07	n/a 148.6 123.1	14	0.119, 0.134 [0.13]	0.033, 0.0354 [0.034]	TK0020708
Fisk, MO, US; (C23-0062) [a]	Branson	1 [--] 2 [255] 3 [5]	59** 96.84 96.84	n/a 18.8 18.8	14	0.0444, 0.0378 [0.041]	0.0136, 0.0124 [0.013]	
Fisk, MO, US; (C23-0164) [a]	Branson	1 [--] 2 [255] 3 [5]	59** 96.62 96.51	n/a 187.6 187.3	14	0.0808, 0.0586 [0.070]	0.029, 0.0226 [0.026]	
Geneva, MN, US; (C09-0163)	Fuller	1 [--] 2 [95] 3 [5]	61** 96.62 97.07	n/a 170.5 162	14	0.046, 0.0354 [0.041]	0.142, 0.112 [0.13]	
Fitchburg, WI, US; (C08-0165)	Klasic	1 [--] 2 [77] 3 [5]	57** 94.82 98.19	n/a 18.71 19.55	14	0.0193, 0.0104 [0.015]	0.014, 0.0107 [0.012]	
Richland, IA, US; (C18-0166)	Branson	1 [--] 2 [296] 3 [5]	59** 99.53 94.71	n/a 5.893 5.332	14	0.0614, 0.0446 [0.053]	<0.01, <0.01 [<0.010]	
			59** 96.28 96.73	n/a 138.1 140.3	14	0.057, 0.0618 [0.059]	<0.01, <0.01 [<0.010]	
Northwood, ND, US; (C13-0167)	Divide	1 [--] 2 [97] 3 [5]	54** 96.51 95.72	n/a 187.8 186.2	7	0.038 [0.038]	0.038 [0.038]	
					10	0.05 [0.050]	0.0438 [0.044]	
					14	0.0422, 0.045 [0.044]	0.043, 0.049 [0.046]	
					17	0.175 [0.18]	0.148 [0.15]	

Location; (Trial ID)	Crop Variety	Application			DALA	Residues (mg/kg) [Mean]		Study report
		No. [interval, days]	Rate, g ai/ha	L/ha		Thiamethoxam	Clothianidin	Remarks
					21	0.044 [0.044]	0.0428 [0.043]	
Raymondville TX, US; (W08-0168)	Fannin	1 [--] 2 [218] 3 [5]	55** 98.30 98.19	n/a 190.7 190.6	14	0.0518, 0.0522 [0.052]	<0.01, <0.01 [<0.010]	
Carrington, ND, US; (C13-0169)	Art	1 [--] 2 [303] 3 [5]	58** 94.38 95.61	n/a 185.8 188.9	14	0.0322, 0.0312 [0.032]	0.0178, 0.0167 [0.017]	
			58** 482.4 484.8	n/a 188.4 187.5	14	0.187, 0.177 [0.18]	0.164, 0.162 [0.16]	
Grand Island, NE, US; (C33-0170) ‡	NuDakota	1 [--] 2 [275] 3 [5]	61** 96.73 96.62	n/a 187.7 183	7	<0.01 [<0.010]	<0.01 [<0.010]	
					10	<0.01 [<0.010]	<0.01 [<0.010]	
					14	<0.01, <0.01 [<0.010]	<0.01, <0.01 [<0.010]	
					17	<0.01 [<0.010]	<0.01 [<0.010]	
					21	4.97 [5.0]	4.5 [4.5]	
Lake Andes, SD, US; (C16-0171)	Fuller	1 [--] 2 [99] 3 [5]	61** 94.04 97.29	n/a 185.9 187.4	14	0.127, 0.125 [0.13]	0.0181, 0.0171 [0.018]	
Jamestown, ND, US; (C12-0172)	Fuller	1 [--] 2 [94] 3 [5]	61** 98.19 97.29	n/a 143 141.7	14	0.0388, 0.0382 [0.038]	0.0236, 0.0232 [0.023]	
			61** 474.8 490.1	n/a 143.6 148.1	14	0.154, 0.079 [0.12]	0.169, 0.061 [0.12]	
Oberon, ND, US: (C13-0173)	Art	1 [--] 2 [309] 3 [5]	58** 96.51 96.95	n/a 18.71 18.8	14	0.0878, 0.095 [0.091]	0.0314, 0.0274 [0.029]	
Uvalde, TX, US; (W07-0174)	Fannin	1 [--] 2 [224] 3 [4]	55** 95.16 98.41	n/a 196.1 193.1	14	0.0958, 0.11 [0.10]	<0.01, <0.01 [<0.010]	
Levelland, TX, US; (W39-0175)	Fannin	1 [--] 2 [251] 3 [6]	55** 95.83 96.06	n/a 186.1 186.4	14	0.0666, 0.0598 [0.063]	<0.01, <0.01 [<0.010]	
Wall, TX, US; (W40-0177) [b]	Fannin	1 [--] 2 [230] 3 [5]	55** 98.19 95.16	n/a 184.2 175.7	7	0.0534 [0.053]	<0.01 [<0.010]	

Location; (Trial ID)	Crop Variety	Application			DALA	Residues (mg/kg) [Mean]		Study report
		No. [interval, days]	Rate, g ai/ha	L/ha		Thiamethoxam	Clothianidin	Remarks
					10	0.053 [0.053]	<0.01 [<0.010]	
					14	0.0386, 0.0422 [0.040]	<0.01, <0.01 [<0.010]	
					17	0.0408 [0.041]	<0.01 [<0.010]	
					21	0.0474 [0.047]	<0.01 [<0.010]	
Wall, TX, US; (W40-0178) [b]	Fannin	1 [--] 2 [240] 3 [7]	55** 96.39 95.38	n/a 180.5 175.7	14	0.0162, 0.0151 [0.016]	<0.01, <0.01 [<0.010]	
Johnstown, CO, US; (W12-0179)	Art	1 [--] 2 [285] 3 [5]	58** 97.29 97.18	n/a 6.361 6.454	14	0.0232, 0.0192 [0.021]	<0.01, <0.01 [<0.010]	
			58** 97.85 97.40	n/a 116.2 114.9	14	<0.01, <0.01 [<0.010]	<0.01, <0.01 [<0.010]	
Ephrata, WA, US; (W18-0180)	Fuller	1 [--] 2 [117] 3 [5]	61** 96.39 97.85	n/a 186.9 187.7	14	0.0206, 0.0232 [0.022]	<0.01, <0.01 [<0.010]	
			61** 97.18 97.29	n/a 4.677 4.771	14	0.0278, 0.0212 [0.024]	<0.01, <0.01 [<0.010]	

** g ai/100 kg seed

† Second and third applications are foliar, and the rate is the allowable for all formulations containing thiamethoxam

‡ The Meeting did not have confidence in this field trial based on all residues being <LOQ except at the last sampling time where residues were well above those of any other field trial for both thiamethoxam and clothianidin. The Meeting decided not to use the trial when considering recommendations.

[a] Same location, variety, and application and harvest dates.

[b] Same location and variety; application and harvest dates are offset by 10 days.

Barley

Twelve field trials were conducted in the USA during the 2010 season (Oakes, T., 2013, Report TK0011969). Treatment consisted of combined seed treatment (FS formulation) and foliar (WG formulation) applications. The seed treatment was made at a nominal rate of 52 g ai/100 kg seed. Plants grown from treated seed received 2 foliar applications, each at a nominal rate of 96 g ai/ha 5 days apart. An adjuvant was used with each foliar application. Grain was harvested 14 DALA; the sample size was not reported.

Following harvest, samples were put into frozen storage at the field trial facilities and transported frozen to the laboratory. In the laboratory, the samples were placed into frozen storage. Grain samples were stored for up to 10 months. The storage period is covered by previously evaluated storage stability data that showed residues are stable under frozen conditions in a wide range of commodities for 1–2 years.

Samples were analysed for residues of thiamethoxam and clothianidin using method AG-675. Concurrent recovery data indicate that the method is suitable.

Table 6 Results of thiamethoxam residue trials in **barley (grain)** in 2010

Location; (Trial ID)	Crop Variety	Application			DALA	Residues (mg/kg) [Mean]		Study report
		No. [interval, days]	Rate, g ai/ha	L/ha		Thiamethoxam	Clothianidin	Remarks
Critical GAP (US)	--	1 [--] 2 [--] 3 [5]	52** 96† 97	n/a 94 (grnd) 19 (air)	30	--	--	
Alternative GAP (MX)		1 [--] 2 [7]	56 56	400	14	--	--	
North Rose, NY, US; (E02-0121)	Kold	1 [--] 2 [280] 3 [5]	50** 96.17 95.61	n/a 186.3 185.2	14	0.270, 0.261 [0.27]	0.0762, 0.0684 [0.072]	TK0011969
Northwood, ND, US; (C13-0122)	Lacey	1 [--] 2 [118] 3 [4]	60** 97.40 98.75	n/a 189.5 192.1	14	0.286, 0.259 [0.27]	0.0144, 0.0158 [0.015]	
Richland, IA, US; (C18-0123)	Lacey	1 [--] 2 [102] 3 [7]	60** 97.51 97.51	n/a 19.18 18.89	14	0.0330, 0.0272 [0.030]	0.0106, <0.01 [<0.010]	
Jamestown, ND, US; (C12-0125)	Robust	1 [--] 2 [104] 3 [6]	59** 97.18 95.38	n/a 141.6 139	0	0.602 [0.60]	0.043 [0.043]	
					3	0.518 [0.52]	0.0496 [0.050]	
					7	0.463 [0.46]	0.047 [0.047]	
					14	0.255, 0.184 [0.22]	0.0412, 0.0364 [0.039]	
					21	0.181 [0.18]	0.0412 [0.041]	
Lake Andes, SD, US; (C16-0126)	Robust	1 [--] 2 [102] 3 [5]	59** 96.17 96.28	n/a 184 177.4	14	0.118, 0.122 [0.12]	0.0294, 0.0266 [0.028]	
Carrington, ND, US; (C13-0127)	Lacey	1 [--] 2 [123] 3 [5]	60** 96.06 95.61	n/a 187 186.1	14	0.119, 0.114 [0.12]	0.0428, 0.0446 [0.044]	
Grand Island, NE, US; (C33-0128)	Kold	1 [--] 2 [305] 3 [7]	50** 96.84 96.39	n/a 189.8 186.4	14	0.158, 0.165 [0.16]	0.0220, 0.0226 [0.022]	

Location; (Trial ID)	Crop Variety	Application			DALA	Residues (mg/kg) [Mean]		Study report
		No. [interval, days]	Rate, g ai/ha	L/ha		Thiamethoxam	Clothianidin	Remarks
Eaton, CO, US; (W12-0129)	Robust†	1 [--] 2 [110] 3 [5]	59** 97.18 99.64	n/a 122.1 124.5	14	0.289, 0.332 [0.31]	0.0234, 0.0260 [0.025]	
Fresno, CA, US; (E19-0130)	Kold	1 [--] 2 [259] 3 [5]	50** 99.42 84.62	n/a 289.6 246.8	14	0.0694, 0.0648 [0.067]	<0.01, <0.01 [<0.01]	
Hermiston, OR, US; (W21-0131)	Haybet†	1 [--] 2 [107] 3 [7]	57** 96.84 95.72	n/a 189.3 191.8	14	0.492, 0.563 [0.53]	0.0138, <0.01 [<0.012]	
Parkdale, OR, US; (W20-0132)	Haybet†	1 [--] 2 [116] 3 [5]	57** 96.17 97.29	n/a 185.6 188	14	0.325, 0.364 [0.34]	0.0482, 0.0496 [0.049]	
Clarence, MO, US; (TK0011969-13)	Kold	1 [--] 2 [274] 3 [6]	50** 96.84 95.94	n/a 188.7 186.9	14	0.0402, 0.0432 [0.042]	0.0500, 0.0564 [0.053]	

** g ai/100 kg seed

† Second and third applications are foliar and the rate is the allowable for all formulations containing thiamethoxam

Rice

Sixteen field trials were conducted in the USA during the 2010 season (Oakes, T., 2013, Report TK0021631). Treatment consisted of combined seed treatment (FS formulation) and foliar (WG formulation) applications. The seed treatment was made at a nominal rate of 0.04 mg ai/seed. Plants grown from treated seed received 2 foliar applications, each at a nominal rate of 96 g ai/ha 5 days apart. An adjuvant was used with each foliar application. Rice was harvested 14 DALA.

Following harvest, samples (0.68 to 1.4 kg) were bagged and put into frozen storage prior to transport to the analytical facility. Samples were shipped frozen to the facility and were immediately placed into frozen storage. Prior to analysis, samples were cleaned of inedible portions (including husks) and adhering soil, then cryogenically homogenized and returned to frozen storage. Samples were stored for a maximum of 12.2 months prior to analysis. The storage period is covered by previously evaluated storage stability data that showed residues are stable under frozen conditions in a wide range of commodities for 1–2 years.

Samples were analysed for residues of thiamethoxam and clothianidin using analytical method GRM.009.02A. Concurrent recovery data indicate that the method is suitable.

Table 7 Results of thiamethoxam residue trials in husked **rice (brown rice)** in 2010

Location; (Trial ID)	Crop Variety	Application			DALA	Residues (mg/kg) [Mean]		Study report
		No. [interval, days]	Rate, g ai/ha	L/ha		Thiamethoxam	Clothianidin	Remarks
Critical GAP (JP)	--	1 [--] 2 [n.s.]	97.5	600	14	--	--	Application to seeding box and to paddy
Cheneyville, LA, US; (E17-0191)	CL151	1 [--] 2 [111] 3 [5]	0.036** 102 94	n/a 173 158	14	1.1, 0.73 [0.92]	0.09, 0.05 [0.07]	TK0021631
Fisk , AR, US; (C23-0192)	CL151	1 [--] 2 [115] 3 [4]	0.036** 96 97.5	n/a 186 189	14	2.0, 2.0 [2.0]	0.16, 0.16 [0.16]	All trials were dry seeded except those in California
Pollard , AR, US; (C23-0193)	CL151	1 [--] 2 [109] 3 [6]	0.036** 95.7 95.5	n/a 185 185	14	1.5, 1.5 [1.5]	0.16, 0.15 [0.16]	
Proctor , AR, US; (C24-0194) [a]	CL151	1 [--] 2 [119] 3 [5]	0.036** 96 96	n/a 18.7 18.7	14	1.4, 1.5 [1.4]	0.17, 0.17 [0.17]	
Proctor , AR, US; (C24-0195) [a]	CL151	1 [--] 2 [119] 3 [5]	0.036** 96.6 96.3	n/a 181 181	0	4.1, 3.8 [3.5]	0.16, 0.15 [0.14]	
					7	2.8, 2.1 [2.4]	0.23, 0.20 [0.22]	
					14	2.0, 1.9 [2.0]	0.24, 0.23 [0.24]	
					21	2.1, 1.6 [1.8]	0.28, 0.23 [0.26]	
					28	1.7, 2.0 [1.8]	0.22, 0.26 [0.24]	
Washington , LA, US; (E18-0196) [b]	CL151	1 [--] 2 [117] 3 [5]	0.036** 99.2 99.3	n/a 154.2 132.6	14	1.3, 1.4 [1.4]	0.07, 0.06 [0.07]	
Washington , LA, US; (E18-0197) [b]	CL151	1 [--] 2 [117] 3 [5]	0.036** 99.3 99.4	n/a 154.2 132.8	14	0.79, 0.54 [0.66]	0.09, 0.07 [0.08]	
Washington , LA, US; (E18-0199) [b]	CL151	1 [--] 2 [117] 3 [5]	0.036** 96.6 98.3	n/a 150 131	14	2.1, 2.2 [2.2]	0.10, 0.12 [0.11]	
Washington, LA, US; (E18-0201) [b]	CL151	1 [--] 2 [117] 3 [5]	0.036** 99 96.8	n/a 153.8 129.4	14	2.2, 2.4 [2.3]	0.10, 0.11 [0.11]	
Bernie , MO, US; (C23-0198)	CL151	1 [--] 2 [109] 3 [6]	0.036** 96.5 96.5	n/a 187.3 187.4	14	1.7, 1.6 [1.6]	0.18, 0.18 [0.18]	

Location; (Trial ID)	Crop Variety	Application			DALA	Residues (mg/kg) [Mean]		Study report
		No. [interval, days]	Rate, g ai/ha	L/ha		Thiamethoxam	Clothianidin	Remarks
Cheneyville, LA, US; (E17-0200)	CL151	1 [--] 2 [111] 3 [5]	0.036** 103.9 96.7	n/a 176.3 161.4	0	2.3, 2.6 [2.4]	0.04, 0.04 [0.04]	
					7	2.0, 2.0 [2.0]	0.10, 0.10 [0.10]	
					14	1.9, 1.7 [1.8]	0.11, 0.11 [0.11]	
					21	1.5, 1.3 [1.4]	0.09, 0.09 [0.09]	
					28	1.2, 1.2 [1.2]	0.07, 0.07 [0.07]	
Dudley , MO, US; (C23-0202)	CL151	1 [--] 2 [115] 3 [4]	0.036** 96.8 96.2	n/a 187.8 186.7	14	1.7, 1.4 [1.5]	0.17, 0.15 [0.16]	
East Bernard , TX, US; (W41-0203) [c]	CL151	1 [--] 2 [122] 3 [4]	0.036** 96.7 96.7	n/a 173 175.5	14	1.4, 1.2 [1.3]	0.06, 0.06 [0.06]	
East Bernard , TX, US; (W41-0204) [c]	CL151	1 [--] 2 [122] 3 [4]	0.036** 96.5 97	n/a 173 176.3	14	1.1, 1.2 [1.2]	0.08, 0.09 [0.09]	
Durham, CA, US; (W23-0205)	M205	1 [--] 2 [139] 3 [5]	0.036** 96.4 98.3	n/a 18.8 19.1	14	1.5, 1.9 [1.7]	0.11, 0.13 [0.12]	
Near Willows, CA, US; (W23-0206)	M205	1 [--] 2 [139] 3 [5]	0.036** 96.4 96.5	n/a 187.7 187.9	14	2.1, 1.9 [2.0]	0.14, 0.14 [0.14]	

** mg ai/seed

† Second and third applications are foliar and the rate is the allowable for all formulations containing thiamethoxam

[a] Same location; application dates offset by 10 days.

[b] Same location and application dates.

[c] Same location and application dates.

Sorghum (grain)

Twelve field trials were conducted in the USA during the 2013 season (Salzman, F., 2016, Report TK0176048). Treatment consisted of two foliar (WG formulation) applications of *ca*. 96 g ai/ha, on a 7-day interval. A non-ionic surfactant or crop oil concentrate was used at all trial locations. Samples of sorghum grain were taken 11 to 15 days after the last application.

Following harvest, samples (*ca*. 0.9 kg) were bagged and put into put into frozen storage prior to transport to the analytical facility. Samples were shipped frozen to the facility and were immediately placed into frozen storage. Prior to analysis, samples were cryogenically homogenized and returned to frozen storage. Samples were stored for a maximum of 13 months prior to analysis. The storage period is covered by previously evaluated storage stability data that showed residues are stable under frozen conditions in a wide range of commodities for 1–2 years.

Samples were analysed for residues of thiamethoxam and clothianidin using the method AG-675. Concurrent recovery data indicate that the method is suitable.

Table 8 Results of thiamethoxam residue trials in **grain sorghum (grain)** in the USA in 2013

Location; (Trial ID)	Crop Variety	Application			DALA	Residues (mg/kg) [Mean]		Study report
		No. [interval, days]	Rate, g ai/ha	L/ha		Thiamethoxam	Clothianidin	Remarks
Critical GAP (US)	--	1 [--] 2 [n.s.] 3 [7]	2.97** 96† 97	n/a 94 (grnd) 19 (air)	14	--	--	
Seven Springs, NC, US (TK0176048-01)	5556 S	1 [--] 2 [8]	95.24 95.49	308.7 308.7	11	0.155, 0.139 [0.15]	0.043, 0.034 [0.039]	TK0176048
Proctor, AR, US (TK0176048-02)	DKS 53-67	1 [--] 2 [7]	96.02 96.39	140.3 93.54	14	0.130, 0.149 [0.14]	0.029, 0.029 [0.029]	
Lime Springs, IA, US (TK0176048-03)	-	1 [--] 2 [7]	96.15 96.70	187.1 187.1	14	0.341, 0.187 [0.26]	0.097, 0.059 [0.078]	
Stafford, KS, US (TK0176048-04)	Mycogen 627C	1 [--] 2 [7]	97.01 99.77	28.06 28.06	14	0.010, 0.016 [0.013]	<0.01, <0.01 [<0.01]	
Richland, IA, US (TK0176048-05)	85Y40	1 [--] 2 [7]	96.82 95.34	196.4 187	14	0.073, 0.080 [0.079]	0.017, 0.015 [0.015]	
York, NE, US (TK0176048-06)	A1005964	1 [--] 2 [6]	97.11 97.28	196.4 196.4	15	0.045, 0.047 [0.046]	<0.01, <0.01 [<0.01]	
			484.2 479.9	196.4 196.4	15	0.264, 0.257 [0.26]	0.024, 0.022 [0.023]	
Raymondville, TX, US (TK0176048-07)	A1020168	1 [--] 2 [7]	97.81 97.50	46.77 46.77	14	0.132, 0.136 [0.13]	0.017, 0.018 [0.018]	
Uvalde, TX, US (TK0176048-08)	Pioneer 83G19	1 [--] 2 [7]	95.17 93.57	187.1 196.4	14	0.019, 0.023 [0.021]	0.030, 0.032 [0.031]	
Grand Island, NE, US (TK0176048-09)	A1005964	1 [--] 2 [7]	96.91 96.91	196.4 196.4	14	0.050, 0.048 [0.049]	<0.01, <0.01 [<0.01]	
Hinton, OK, US (TK0176048-10) [a]	SR25835	1 [--] 2 [7]	95.54 94.78	37.4 46.77	14	0.051, 0.050 [0.051]	0.052, 0.039 [0.046]	
Hinton, OK, US (TK0176048-12) [a]	SR25835	1 [--] 2 [6]	99.13 96.54	205.8 149.7	15	0.395, 0.314 [0.35]	0.017, <0.01 [<0.013]	
			490.8 486.0	205.8 149.7	15	0.956, 1.08 [0.98]	0.077, 0.075 [0.072]	
San Angelo, TX, US (TK0176048-11)	DKS 49-45	1 [--] 2 [7]	97.70 95.99	140.3 131	13	0.043, 0.042 [0.044]	<0.01, <0.01 [<0.01]	

** g ai/kg seed

† Second and third applications are foliar and the rate is the allowable for all formulations containing thiamethoxam

[a] Same location; application dates are offset by 15 days

Sweet corn

Twelve field trials were conducted in the USA during the 2010 season (Willard, T., 2013, Report TK0014208). Treatment consisted of combined seed treatment (FS formulation) and foliar (WG formulation) applications. The seed treatment was made at a nominal rate of 1.27 mg ai/seed. Plants grown from treated seed received 2 foliar applications, each at a nominal rate of 96 g ai/ha 4 days apart. A non-ionic surfactant or crop oil concentrate was used at all trial locations. Samples of corn on the cob were harvested 1 day after the last application.

Following harvest, samples were bagged and put into put into frozen storage prior to transport to the analytical facility. Samples were shipped frozen to the facility and were immediately placed into frozen storage. Prior to analysis, samples were cryogenically homogenized and returned to frozen storage. Samples were stored for a maximum of 14.1 months prior to analysis. The storage period is covered by previously evaluated storage stability data that showed residues are stable under frozen conditions in a wide range of commodities for 1–2 years.

Samples were analysed for residues of thiamethoxam and clothianidin using the method GRM.009.02A. Concurrent recovery data indicate that the method is suitable.

Table 9 Results of thiamethoxam residue trials in **sweet corn (kernel plus cob with husk removed)** conducted in the USA in 2010

Location; (Trial ID)	Crop Variety	Application			DALA	Residues (mg/kg) [Mean]		Study report
		No. [interval days]	Rate, g ai/ha	L/ha		Thiamethoxam	Clothianidin	Remarks
Critical GAP (US)	--	1 [--] 2 [--] 3 [5]	1.25** 96† 97	n/a 94 (grnd) 19 (air)	1	--	--	
North Rose, NY, US; (E02-0131)	GH4927	1 [--] 2 [127] 3 [4]	1.27** 96 97	n/a 324.7 330	1	<0.01 , <0.01 [<0.01]	<0.01 , <0.01 [<0.01]	TK0014208
Frenchtown, PA, US; (E04-0132)	Garrison	1 [--] 2 [93] 3 [4]	1.29** 100 101	n/a 363.8 341.8	1	<0.01 , <0.01 [<0.01]	<0.01 , <0.01 [<0.01]	
Seven Springs, NC, US; (E10-0133)	GH4927	1 [--] 2 [131] 3 [4]	1.27** 96 98	n/a 278.7 282.2	1	<0.01 , <0.01 [<0.01]	<0.01 , <0.01 [<0.01]	
Clermont, FL, US; (E19-0134)	Garrison	1 [--] 2 [208] 3 [4]	1.29** 96 96	n/a 18.6 18.7	1	<0.01 , <0.01 [<0.01]	<0.01 , <0.01 [<0.01]	
Northwood, ND, US; (C13-0135)	GH4927	1 [--] 2 [91] 3 [4]	1.27** 98 97	n/a 190.3 188.8	0	<0.01 [<0.01]	<0.01 [<0.01]	
					1	<0.01 , <0.01 [<0.01]	<0.01 , <0.01 [<0.01]	
					3	<0.01 [<0.01]	<0.01 [<0.01]	
					5	<0.01 [<0.01]	<0.01 [<0.01]	

Location; (Trial ID)	Crop Variety	Application			DALA	Residues (mg/kg) [Mean]		Study report
		No. [interval days]	Rate, g ai/ha	L/ha		Thiamethoxam	Clothianidin	Remarks
					7	<0.01 [<0.01]	<0.01 [<0.01]	
Richland, IA, US; (C18-0136)	Garrison	1 [--] 2 [83] 3 [4]	1.29** 99 99	n/a 158.6 162	1	<0.01 , <0.01 [<0.01]	<0.01 , <0.01 [<0.01]	
Oregon, MO, US; (C19-0137)	GH4927	1 [--] 2 [81] 3 [4]	1.27** 100 97	n/a 147.3 140.6	1	<0.01 , <0.01 [<0.01]	<0.01 , <0.01 [<0.01]	
York, NE, US; (C33-0138)	Garrison	1 [--] 2 [85] 3 [4]	1.29** 95 96	n/a 184.9 185.8	1	<0.01 , <0.01 [<0.01]	<0.01 , <0.01 [<0.01]	
Gardner, ND, US; (C12-0139)	GH4927	1 [--] 2 [107] 3 [5]	1.27** 98 97	n/a 190.3 188.4	1	<0.01 , <0.01 [<0.01]	<0.01 , <0.01 [<0.01]	
Fresno, CA, US; (E19-0140)	Garrison	1 [--] 2 [79] 3 [4]	1.29** 95 97	n/a 402 406	1	<0.01 , <0.01 [<0.01]	<0.01 , <0.01 [<0.01]	
Rupert, ID, US; (W15-0141)	GH4927	1 [--] 2 [103] 3 [4]	1.27** 96 98	n/a 206 161.5	0	<0.01 [<0.01]	<0.01 [<0.01]	
					1	<0.01 , <0.01 [<0.01]	<0.01 , <0.01 [<0.01]	
					3	<0.01 [<0.01]	<0.01 [<0.01]	
					5	<0.01 [<0.01]	<0.01 [<0.01]	
					7	<0.01 [<0.01]	<0.01 [<0.01]	
Portland, OR, US; (W20-0142)	Garrison	1 [--] 2 [154] 3 [5]	1.29** 98 96	n/a 209.4 219.4	1	<0.01 , <0.01 [<0.01]	<0.01 , <0.01 [<0.01]	

** g ai/kernel

† Second and third applications are foliar and the rate is the allowable for all formulations containing thiamethoxam

Sorghum (sweet)

Four field trials were conducted in the USA during the 2013 season (Salzman, F., 2016, Report TK0176048). Treatment consisted of two foliar (WG formulation) applications of ca. 96 g ai/ha, on a 7-day interval. A non-ionic surfactant or crop oil concentrate was used at all trial locations. Samples of sorghum cane were taken 14 days after the last application.

Following harvest, samples (20- to 30-cm cane pieces from at least 24 plants) were bagged and put into put into frozen storage prior to transport to the analytical facility. Samples were shipped frozen to the facility and were immediately placed into frozen storage. Prior to analysis, samples were cryogenically homogenized and returned to frozen storage. Samples were stored for a maximum of 15 months prior to

analysis. The storage period is covered by previously evaluated storage stability data that showed residues are stable under frozen conditions in a wide range of commodities for 1–2 years.

Samples were analysed for residues of thiamethoxam and clothianidin using the method AG-675. Concurrent recovery data indicate that the method is suitable.

Table 10 Results of thiamethoxam residue trials in **sweet sorghum** conducted in the USA in 2013.

Location; (Trial ID)	Crop Variety	Application			DALA	Residues (mg/kg) [Mean]		Study report
		No. [interval, days]	Rate, g ai/ha	L/ha		Thiamethoxam	Clothianidin	Remarks
Critical GAP (US)	--	1 [--] 2 [n.s.] 3 [7]	2.97** 96† 97	n/a 94 (grnd) 19 (air)	14	--	--	
Hinton, OK, US; (TK0176048-13)	Della	1 [--] 2 [7]	94.14 94.53	177.7 177.7	14	<0.01, <0.01 [<0.01]	0.036, 0.042 [0.039]	TK0176048
Raymondville, TX, US; (TK0176048-14)	Super Sugar	1 [--] 2 [9]	96.39 96.39	196.4 187.1	13	<0.01, <0.01 [<0.01]	0.060, 0.064 [0.062]	
Proctor, AR, US; (TK0176048-15)	Dale	1 [--] 2 [7]	96.85 96.68	93.54 93.54	7	0.0951	0.153	
					10	0.126	0.242	
					14	0.0318, 0.0304 [0.031]	0.102, 0.0992 [0.10]	
					17	0.0228	0.0969	
					21	0.0565	0.203	
Washington, LA, US; (TK0176048-16)	SX17	1 [--] 2 [7]	98.74 98.90	149.7 243.2	15	0.131, 0.359 [0.24]	0.038, 0.096 [0.067]	
			495.1 497.4	243.2 224.5	15	0.234, 0.220, 0.236 [0.23]	0.026, 0.034, 0.653 [0.042]	

Animal Feeds

Wheat

The trials in wheat (Willard, T, 2013, Study Report TK0020708) are described above. From those trials, forage, hay, and straw were harvested 7, 7, and 14 DALA, respectively. Hay was allowed to dry in the field for several days prior to collection.

Following harvest, samples were put into frozen storage at the field trial facilities and transported frozen to the laboratory. In the laboratory, the samples were placed into frozen storage. Samples were stored for up to 10.6 months (forage), 11.3 months (hay) and 8.6 months (straw). The storage period is covered by previously evaluated storage stability data that showed residues are stable under frozen conditions in a wide range of commodities for 1–2 years.

Samples were analysed for residues of thiamethoxam and clothianidin using method AG-675. Concurrent recovery data indicate that the method is suitable.

Table 11 Results of thiamethoxam residue trials in **wheat** in 2010

Location; (Trial ID)	Crop Variety	Application			Com-modity	DALA	Residues (mg/kg; as received) [Mean]		Study report
		No. [interval, days]	Rate, g ai/ha	L/ha			Thiamethoxam	Clothianidin	Remarks
Critical GAP (US)	--	1 [--] 2 [--] 3 [5]	52** 96† 97	n/a 94 (grnd) 19 (air)	Forage Hay Straw	7 7 30	--	--	
Alternative GAP (MX)		1 [--] 2 [7]	56 56	400	--	14	--	--	
Suffolk, VA, US; (E07-0061)	Art	1 [--] 2 [211] 3 [5]	58** 97.85 98.41	n/a 124.8 126.5	Forage	7	0.446, 0.47 [0.46]	0.208, 0.216 [0.21]	TK0020708
					Hay	7	0.968, 0.845 [0.91]	0.574, 0.636 [0.60]	
		1 [--] 2 [252] 3 [5]	58** 98.52 98.07	n/a 148.6 123.1	Straw	14	2.2, 2.5 [2.4]	0.384, 0.349 [0.37]	
Fisk, MO, US; (C23-0062) [a]	Branson	1 [--] 2 [211] 3 [5]	59** 98.30 96.84	n/a 18.8 19.08	Forage	7	0.03, 0.033 [0.032]	0.0214, 0.0262 [0.024]	
					Hay	7	0.0368, 0.0294 [0.033]	0.0706, 0.0588 [0.065]	
		1 [--] 2 [255] 3 [5]	59** 96.84 96.84	n/a 18.8 18.8	Straw	14	0.701, 0.764 [0.73]	0.166, 0.156 [0.16]	
Fisk, MO, US; (C23-0164) [a]	Branson	1 [--] 2 [211] 3 [5]	59** 95.72 96.95	n/a 185.9 188.2	Forage	7	0.0318, 0.026 [0.029]	0.0242, 0.0212 [0.023]	
					Hay	7	0.0488, 0.0486 [0.049]	0.0866, 0.0726 [0.080]	
		1 [--] 2 [255] 3 [5]	59** 96.62 96.51	n/a 187.6 187.3	Straw	14	1.72, 1.59 [1.7]	0.51, 0.416 [0.46]	
Geneva, MN, US; (C09-0163)	Fuller	1 [--] 2 [51] 3 [5]	61** 96.95 97.74	n/a 164.5 169.9	Forage	7	0.0194, <0.01 [<0.015]	0.0252, <0.01 [<0.018]	
					Hay	7	0.0392, 0.033 [0.036]	0.0716, 0.057 [0.064]	
		1 [--] 2 [95] 3 [5]	61** 96.62 97.07	n/a 170.5 162	Straw	14	0.693, 0.8 [0.75]	0.252, 0.262 [0.26]	
Fitchburg, WI, US; (C08-0165)	Klasic	1 [--] 2 [50]	57** 97.85	n/a 19.92	Forage	7	0.124, 0.23 [0.18]	0.124, 0.194 [0.16]	

Location; (Trial ID)	Crop Variety	Application			Com-modity	DALA	Residues (mg/kg; as received) [Mean]		Study report
		No. [interval, days]	Rate, g ai/ha	L/ha			Thiamethoxam	Clothianidin	Remarks
		3 [6]	97.96	19.74					
					Hay	7	0.435, 0.381 [0.41]	0.386, 0.408 [0.40]	
		1 [--] 2 [77] 3 [5]	57** 94.82 98.19	n/a 18.71 19.55	Straw	14	0.156, 0.122 [0.14]	0.0308, 0.0262 [0.028]	
Richland, IA, US; (C18-0166)	Branson	1 [--] 2 [238] 3 [6]	59** 96.73 96.91	n/a 4.864 5.425	Forage	7	0.034, 0.036 [0.035]	0.0155, 0.0154 [0.015]	
			59** 96.39 97.07	n/a 184.5 187.5	Forage	7	0.126, 0.119 [0.12]	0.0608, 0.059 [0.060]	
			59** 96.73 96.91	n/a 4.864 5.425	Hay	7	0.0836, 0.107 [0.095]	0.0482, 0.055 [0.052]	
			59** 96.39 97.07	n/a 184.5 187.5	Hay	7	0.362, 0.274 [0.32]	0.222, 0.176 [0.20]	
		1 [--] 2 [296] 3 [5]	59** 99.53 94.71	n/a 5.893 5.332	Straw	14	0.881, 1.23 [1.1]	0.0368, 0.0502 [0.044]	
			59** 96.28 96.73	n/a 138.1 140.3	Straw	14	1.4, 1.82 [1.6]	0.075, 0.119 [0.097]	
Northwood, ND, US; (C13-0167)	Divide	1 [--] 2 [41] 3 [5]	54** 95.72 97.85	n/a 186.2 190.4	Forage	0	6.7	0.0444	
					Forage	3	0.531	0.043	
					Forage	7	0.025, 0.036 [0.030]	<0.01, 0.0116 [<0.011]	
					Forage	10	<0.01	<0.01	
					Forage	14	<0.01	<0.01	
					Hay	0	7.32	0.514	
					Hay	3	0.0944 [0.094]	0.105 [0.10]	
					Hay	7	0.0121, 0.0164 [0.014]	0.0139, 0.0168 [0.015]	
					Hay	10	0.0103	0.01	
					Hay	14	<0.01	<0.01	
		1 [--]	54**	n/a	Straw	7	0.284	0.147	

Location; (Trial ID)	Crop Variety	Application			Com-modity	DALA	Residues (mg/kg; as received) [Mean]		Study report
		No. [interval, days]	Rate, g ai/ha	L/ha			Thiamethoxam	Clothianidin	Remarks
		2 [97] 3 [5]	96.51 95.72	187.8 186.2					
					Straw	10	0.338	0.124	
					Straw	14	0.171, 0.266 [0.22]	0.087, 0.145 [0.12]	
					Straw	17	0.182	0.098	
					Straw	21	0.25	0.0838	
Raymondville TX, US; (W08-0168)	Fannin	1 [--] 2 [132] 3 [5]	55** 99.20 98.30	n/a 192.6 190.7	Forage	7	2.02, 2.07 [2.0]	0.191, 0.192 [0.19]	
					Hay	7	6.33, 7.00 [6.7]	0.694, 0.755 [0.72]	
		1 [--] 2 [218] 3 [5]	55** 98.30 98.19	n/a 190.7 190.6	Straw	14	3.85, 3.96 [3.9]	0.0716, 0.072 [0.072]	
Carrington, ND, US; (C13-0169)	Art	1 [--] 2 [269] 3 [5]	58** 91.91 92.02	n/a 184.1 186.3	Forage	7	0.0224, 0.0238 [0.023]	0.0536, 0.0552 [0.054]	
					Hay	7	0.0334, 0.035 [0.034]	0.0842, 0.0986 [0.091]	
		1 [--] 2 [303] 3 [5]	58** 94.38 95.61	n/a 185.8 188.9	Straw	14	0.423, 0.43 [0.43]	0.0948, 0.107 [0.10]	
Grand Island, NE, US; (C33-0170)	NuDakota	1 [--] 2 [234] 3 [5]	61** 96.51 96.39	n/a 187.5 184.8	Forage	0	0.792	0.06	
						3	0.147	0.0354	
						7	0.0886, 0.069 [0.079]	0.029, 0.023 [0.026]	
						10	0.0232	0.012	
						14	0.024	0.0111	
					Hay	0	0.125	0.295	
						3	0.31	0.105	
						7	0.0602, 0.0566 [0.058]	0.0614, 0.055	
						10	0.0244	0.0326	
						14	0.0306	0.0388	
		1 [--] 2 [275]	61** 96.73	n/a 187.7	Straw	7	0.298	0.218	

Location; (Trial ID)	Crop Variety	Application			Com-modity	DALA	Residues (mg/kg; as received) [Mean]		Study report
		No. [interval, days]	Rate, g ai/ha	L/ha			Thiamethoxam	Clothianidin	Remarks
		3 [5]	96.62	183					
						10	0.352	0.22	
						14	0.202, 0.204 [0.20]	0.119, 0.121 [0.12]	
						17	0.0822	0.0706	
						21	0.131	0.0992	
Lake Andes, SD, US; (C16-0171)	Fuller	1 [--] 2 [57] 3 [5]	61** 95.50 95.27	n/a 183.7 169.1	Forage	7	0.315, 0.341 [0.33]	0.159, 0.186 [0.17]	
					Hay	7	0.52, 0.523 [0.52]	0.425, 0.426 [0.43]	
		1 [--] 2 [99] 3 [5]	61** 94.04 97.29	n/a 185.9 187.4	Straw	14	1.83, 1.62 [1.7]	0.0318, 0.031 [0.031]	
Jamestown, ND, US; (C12-0172)	Fuller	1 [--] 2 [55] 3 [4]	61** 97.51 98.41	n/a 94.66 95.04	Forage	7	0.0338, 0.0382 [0.036]	0.0242, 0.0236 [0.024]	
					Hay	7	0.0542, 0.0536 [0.054]	0.0606, 0.0558 [0.058]	
		1 [--] 2 [94] 3 [5]	61** 98.19 97.29	n/a 143 141.7	Straw	14	0.214, 0.206 [0.21]	0.242, 0.236 [0.24]	
Oberon, ND, US: (C13-0173)	Art	1 [--] 2 [275] 3 [5]	58** 96.51 97.07	n/a 18.71 18.8	Forage	7	0.0104, 0.0119 [0.011]	0.0181, 0.0167 [0.017]	
					Hay	7	0.0159, 0.0135 [0.015]	0.0364, 0.0326 [0.034]	
		1 [--] 2 [309] 3 [5]	58** 96.51 96.95	n/a 18.71 18.8	Straw	14	0.934, 0.904 [0.92]	0.293, 0.282 [0.29]	
Uvalde, TX, US; (W07-0174)	Fannin	1 [--] 2 [168] 3 [5]	55** 96.39 96.95	n/a 168.6 192.3	Forage	7	1.07, 1.04 [1.1]	0.136, 0.127 [0.13]	
					Hay	7	2.3, 1.89 [2.1]	0.343, 0.283 [0.31]	
		1 [--] 2 [224] 3 [4]	55** 95.16 98.41	n/a 196.1 193.1	Straw	14	3.29, 3.3 [3.3]	0.0892, 0.0908 [0.090]	
Levelland, TX, US; (W39-0175)	Fannin	1 [--] 2 [204]	55** 97.29	n/a 185.3	Forage	7	0.676, 0.789 [0.73]	0.0664, 0.0766 [0.072]	

Location; (Trial ID)	Crop Variety	Application			Com-modity	DALA	Residues (mg/kg; as received) [Mean]		Study report
		No. [interval, days]	Rate, g ai/ha	L/ha			Thiamethoxam	Clothianidin	Remarks
		3 [5]	95.94	188.4					
					Hay	7	1.44, 1.47 [1.5]	0.166, 0.236 [0.20]	
		1 [--] 2 [251] 3 [6]	55** 95.83 96.06	n/a 186.1 186.4	Straw	14	0.828, 0.856 [0.84]	0.0161, 0.0158 [0.016]	
Wall, TX, US; (W40-0177) [b]	Fannin	1 [--] 2 [162] 3 [5]	55** 96.62 97.07	n/a 176.6 186	Forage	0	5.54	0.075	
						3	1.7	0.0998	
						7	1.06, 1.16 [1.1]	0.1, 0.106 [0.10]	
						10	0.936	0.0962	
						14	0.52	0.0654	
					Hay	0	12.8	0.841	
						3	3.42	0.44	
						7	2.14, 1.94 [2.0]	0.271, 0.242 [0.26]	
						10	1.75	0.249	
						14	1.35	0.204	
		1 [--] 2 [230] 3 [5]	55** 98.19 95.16	n/a 184.2 175.7	Straw	7	2.05	0.0282	
						10	1.96	0.0268	
						14	1.79, 1.7 [1.7]	0.0236, 0.0186 [0.021]	
						17	1.62	0.0202	
						21	1.23	0.016	
Wall, TX, US; (W40-0178) [b]	Fannin	1 [--] 2 [180] 3 [4]	55** 97.18 96.62	n/a 199.1 177.6	Forage	7	0.86, 0.751 [0.81]	0.06, 0.0554 [0.058]	
					Hay	7	1.52, 1.39 [1.5]	0.134, 0.131 [0.13]	
		1 [--] 2 [240] 3 [7]	55** 96.39 95.38	n/a 180.5 175.7	Straw	14	0.934, 0.878 [0.91]	0.013, 0.0127 [0.013]	
Johnstown, CO, US; (W12-0179)	Art	1 [--] 2 [240] 3 [5]	58** 96.39 96.39	n/a 114.9 114.2	Forage	7	0.301, 0.357 [0.33]	0.0704, 0.0866 [0.078]	

Location; (Trial ID)	Crop Variety	Application			Com-modity	DALA	Residues (mg/kg; as received) [Mean]		Study report
		No. [interval, days]	Rate, g ai/ha	L/ha			Thiamethoxam	Clothianidin	Remarks
			58** 97.85 96.39	n/a 6.454 6.361	Forage	7	0.143, 0.163 [0.15]	0.0402, 0.0436 [0.042]	
			58** 96.39 96.39	n/a 114.9 114.2	Hay	7	0.195, 0.212 [0.20]	0.228, 0.238 [0.23]	
			58** 97.85 96.39	n/a 6.454 6.361	Hay	7	0.105, 0.139 [0.12]	0.13, 0.159 [0.14]	
		1 [--] 2 [285] 3 [5]	58** 97.29 97.18	n/a 6.361 6.454	Straw	14	0.475, 0.507 [0.49]	0.0804, 0.0932 [0.087]	
			58** 97.85 97.40	n/a 116.2 114.9	Straw	14	0.157, 0.118 [0.14]	0.0782, 0.0528 [0.066]	
Ephrata, WA, US; (W18-0180)	Fuller	1 [--] 2 [62] 3 [5]	61** 96.39 96.39	n/a 187.2 188	Forage	7	0.0678, 0.0768 [0.072]	0.0372, 0.0482 [0.043]	
			61** 97.40 97.85	n/a 4.677 4.771	Forage	7	0.0406, 0.0476 [0.044]	0.0248, 0.0244 [0.025]	
			61** 96.39 96.39	n/a 187.2 188	Hay	7	0.157, 0.165 [0.16]	0.145, 0.154 [0.15]	
			61** 97.40 97.85	n/a 4.677 4.771	Hay	7	0.129, 0.105 [0.12]	0.113, 0.102 [0.11]	
		1 [--] 2 [117] 3 [5]	61** 96.39 97.85	n/a 186.9 187.7	Straw	14	1.31, 1.63 [1.5]	0.083, 0.0936 [0.088]	
			61** 97.18 97.29	n/a 4.677 4.771	Straw	14	2.04, 1.74 [1.9]	0.0774, 0.0654 [0.071]	

** g ai/100 kg seed

[a] Same location, variety, and application and harvest dates.

[b] Same location and variety; application and harvest dates are offset by 18 days (forage/hay) or 10 days (straw).

Barley

The trials in barley (Oakes, T., 2013, Report TK0011969) are described above. From those trials, hay and straw were harvested 7 and 14 DALA, respectively.

Following harvest, samples were put into frozen storage at the field trial facilities and transported frozen to the laboratory. In the laboratory, the samples were placed into frozen storage. Samples were stored for up to 9.9 months (straw) or 11.1 months (hay). The storage period is covered by previously evaluated storage stability data that showed residues are stable under frozen conditions in a wide range of commodities for 1–2 years.

Samples were analysed for residues of thiamethoxam and clothianidin using method AG-675. Concurrent recovery data indicate that the method is suitable.

Table 12 Results of thiamethoxam residue trials in **barley** in 2010

Location; (Trial ID)	Crop Variety	Application			Com-modity	DALA	Residues (mg/kg; as received) [Mean]		Study report
		No. [interval, days]	Rate, g ai/ha	L/ha			Thiamethoxam	Clothianidin	Remarks
Critical GAP (US)	--	1 [--] 2 [--] 3 [5]	52** 96† 97	n/a 94 (grnd) 19 (air)	Forage Hay Straw	7 7 30	--	--	
Alternative GAP (MX)		1 [--] 2 [7]	56 56	400	--	14	--	--	
North Rose, NY, US; (E02-0121)	Kold	1 [--] 2 [257] 3 [5]	48** 98.53 94.49	n/a 185.7 183	Hay	7	0.212, 0.171 [0.19]	0.109, 0.0840 [0.096]	TK0011969
		1 [--] 2 [280] 3 [5]	50** 96.17 95.61	n/a 186.3 185.2	Straw	14	0.521, 0.543 [0.53]	0.117, 0.101 [0.11]	
Northwood, ND, US; (C13-0122)	Lacey	1 [--] 2 [118] 3 [4]	59** 95.72 96.62	n/a 189.5 192.1	Hay	7	0.0232, 0.0236 [0.023]	0.0126, 0.0145 [0.014]	
		1 [--] 2 [65] 3 [5]	60** 97.40 98.75	n/a 186.2 188.1	Straw	14	0.565, 0.508 [0.54]	0.0756, 0.0716 [0.074]	
Richland, IA, US; (C18-0123)	Lacey	1 [--] 2 [82] 3 [6]	59** 97.51 96.95	n/a 18.89 18.61	Hay	7	0.0534, 0.0626 [0.058]	0.0486, 0.0480 [0.048]	
		1 [--] 2 [102] 3 [7]	60** 97.51 97.51	n/a 19.18 18.89	Straw	14	0.0135, 0.0144 [0.014]	< 0.01, < 0.01 [<0.01]	
Jamestown, ND, US; (C12-0125)	Robust	1 [--] 2 [84] 3 [5]	58** 96.84 95.61	n/a 141.1 139.2	Hay	0	6.00	0.220	
			58**			3	0.594	0.122	

Location; (Trial ID)	Crop Variety	Application			Com-modity	DALA	Residues (mg/kg; as received) [Mean]		Study report
		No. [interval, days]	Rate, g ai/ha	L/ha			Thiamethoxam	Clothianidin	Remarks
			96.84 95.61 58**						
			96.84 95.61 58**			7	0.166, 0.177 [0.17]	0.0678, 0.0662 [0.067]	
			96.84 95.61 58**			10	0.0600	0.0364	
			96.84 95.61 58**			14	0.0102	< 0.01 [<0.01]	
		1 [--] 2 [104] 3 [6]	59** 97.18 95.38	n/a 141.6 139	Straw	0	1.31	0.0822	
			59** 97.18 95.38			3	0.681	0.0906	
			59** 97.18 95.38			7	0.890	0.121	
			59** 97.18 95.38			14	0.265, 0.228 [0.25]	0.0706, 0.0626 [0.067]	
			59** 97.18 95.38			21	0.240	0.0672	
Lake Andes, SD, US; (C16-0126)	Robust	1 [--] 2 [80] 3 [5]	58** 96.73 96.62	n/a 171.5 186.1	Hay	7	0.556, 0.361 [0.46]	0.248, 0.170 [0.21]	
		1 [--] 2 [102] 3 [5]	59** 96.17 96.28	n/a 184 177.4	Straw	14	0.0832, 0.0636 [0.073]	0.0374, 0.0312 [0.034]	
Carrington, ND, US; (C13-0127)	Lacey	1 [--] 2 [89] 3 [4]	59** 94.49 95.38	n/a 183.9 185.6	Hay	7	0.120, 0.128 [0.12]	0.0676, 0.0774 [0.072]	
		1 [--] 2 [123] 3 [5]	60** 96.06 95.61	n/a 187 186.1	Straw	14	0.0550, 0.101 [0.078]	0.0362, 0.0510 [0.044]	
Grand Island, NE, US; (C33-0128)	Kold	1 [--]	48**	n/a	Hay	7	0.598, 0.432 [0.52]	0.103, 0.0748 [0.089]	

Location; (Trial ID)	Crop Variety	Application			Com-modity	DALA	Residues (mg/kg; as received) [Mean]		Study report
		No. [interval, days]	Rate, g ai/ha	L/ha			Thiamethoxam	Clothianidin	Remarks
		2 [259] 3 [5]	96.84 96.51	191.4 188.9					
		1 [--] 2 [305] 3 [7]	50** 96.84 96.39	n/a 189.8 186.4	Straw	14	0.243, 0.236 [0.24]	0.0554, 0.0628 [0.059]	
Eaton, CO, US; (W12-0129)	Robust	1 [--] 2 [79] 3 [5]	58** 96.95 97.51	n/a 121.3 122	Hay	7	0.0768, 0.0894 [0.083]	0.0270, 0.0276 [0.027]	
		1 [--] 2 [110] 3 [5]	59** 97.18 99.64	n/a 122.1 124.5	Straw	14	0.589, 0.399 [0.49]	0.0554, 0.0504 [0.053]	
Fresno, CA, US; (E19-0130)	Kold	1 [--] 2 [239] 3 [5]	48** 96.17 96.95	n/a 280.3 282.5	Hay	7	0.462, 0.386 [0.42]	0.0568, 0.0500 [0.053]	
		1 [--] 2 [259] 3 [5]	50** 99.42 84.62	n/a 289.6 246.8	Straw	14	0.276, 0.248 [0.26]	0.0226, 0.0210 [0.022]	
Hermiston, OR, US; (W21-0131)	Haybet	1 [--] 2 [93] 3 [7]	58** 95.94 92.81	n/a 187.5 186	Hay	7	0.226, 0.558 [0.39]	0.109, 0.100 [0.10]	
		1 [--] 2 [107] 3 [7]	57** 96.84 95.72	n/a 189.3 191.8	Straw	14	2.12, 2.43 [2.3]	0.0816, 0.0890 [0.085]	
Parkdale, OR, US; (W20-0132)	Haybet	1 [--] 2 [75] 3 [5]	58** 95.38 96.50	n/a 185.1 198.4	Hay	7	0.214, 0.472 [0.34]	0.0586, 0.0666 [0.063]	
		1 [--] 2 [116] 3 [5]	57** 96.17 97.29	n/a 185.6 188	Straw	14	0.277, 0.218 [0.25]	0.0456, 0.0372 [0.041]	
Clarence, MO, US; (TK0011969-13)	Kold	1 [--] 2 [259] 3 [5]	48** 96.28 96.39	n/a 187.5 187.7	Hay	7	0.259, 0.248 [0.25]	0.242, 0.210 [0.23]	
		1 [--] 2 [274] 3 [6]	50** 96.84 95.94	n/a 188.7 186.9	Straw	14	0.0166, 0.0151 [0.016]	0.0326, 0.0316 [0.032]	

** g ai/100 kg seed

Rice

The trials in rice (Oakes, T., 2013, Report TK0021631) are described above. From those trials, straw (0.57 to 1.1 kg) was harvested 14 DALA.

Following harvest, samples were bagged and put into frozen storage prior to transport to the analytical facility. Samples were shipped frozen to the facility and were immediately placed into frozen storage. Prior to analysis, samples were cryogenically homogenized and returned to frozen storage. Samples were stored for a maximum of 12.2 months prior to analysis. The storage period is covered by previously evaluated storage stability data that showed residues are stable under frozen conditions in a wide range of commodities for 1–2 years.

Samples were analysed for residues of thiamethoxam and clothianidin using analytical method GRM.009.02A. Concurrent recovery data indicate that the method is suitable.

Table 13 Results of thiamethoxam residue trials in **rice** in 2010

Location; (Trial ID)	Crop Variety	Application			Commodity	DALA	Residues (mg/kg; as received) [Mean]		Study report
		No. [interval, days]	Rate, g ai/ha	L/ha			Thiamethoxam	Clothianidin	Remarks
Critical GAP (US)	--	1 [--] 2 [n.s.] 3 [5]	0.03** 96† 97	n/a 19	Straw	21	--	--	
Alternative GAP (JP)	--	1 [--] 2 [n.s.]	97.5	600		14	--	--	
Cheneyville, LA, US; (E17-0191)	CL151	1 [--] 2 [111] 3 [5]	0.036** 102 94	n/a 173 158	Straw	14	1.1, 1.0 [1.0]	0.10, 0.10 [0.10]	TK0021631
Fisk , AR, US; (C23-0192)	CL151	1 [--] 2 [115] 3 [4]	0.036** 96 97.5	n/a 186 189	Straw	14	0.38, 0.33 [0.36]	0.04, 0.03 [0.03]	All trials were dry seeded except those in California
Pollard , AR, US; (C23-0193)	CL151	1 [--] 2 [109] 3 [6]	0.036** 95.7 95.5	n/a 185 185	Straw	14	0.47, 0.55 [0.51]	0.04, 0.05 [0.04]	
		1 [--] 2 [119] 3 [5]	0.036** 96 96	n/a 18.7 18.7	Straw	14	0.27, 0.27 [0.27]	<0.01, 0.01 [<0.01]	
Proctor , AR, US; (C24-0195)	CL151	1 [--] 2 [119] 3 [5]	0.036** 96.6 96.3	n/a 181 181	Straw	0	1.9, 1.8 [1.9]	0.02, 0.02 [0.02]	
						7	0.61, 0.71 [0.66]	0.02, 0.02 [0.02]	
						14	0.34, 0.26 [0.30]	0.01, <0.01 [0.01]	
						21	0.28, 0.32 [0.30]	0.01, 0.02 [0.01]	

Location; (Trial ID)	Crop Variety	Application			Commodity	DALA	Residues (mg/kg; as received) [Mean]		Study report
		No. [interval, days]	Rate, g ai/ha	L/ha			Thiamethoxam	Clothianidin	Remarks
						28	0.27, 0.30 [0.29]	0.01, 0.01 [0.01]	
Washington , LA, US; (E18-0196) [a]	CL151	1 [--] 2 [117] 3 [5]	0.036** 99.2 99.3	n/a 154.2 132.6	Straw	14	1.5, 0.53 [1.0]	0.08, 0.04 [0.06]	
Washington , LA, US; (E18-0197) [a]	CL151	1 [--] 2 [117] 3 [5]	0.036** 99.3 99.4	n/a 154.2 132.8	Straw	14	0.61, 0.88 [0.74]	0.12, 0.14 [0.13]	
Washington , LA, US; (E18-0199) [a]	CL151	1 [--] 2 [117] 3 [5]	0.036** 96.6 98.3	n/a 150 131	Straw	14	0.75, 0.73 [0.74]	0.07, 0.05 [0.06]	
Washington, LA, US; (E18-0201) [a]	CL151	1 [--] 2 [117] 3 [5]	0.036** 99 96.8	n/a 153.8 129.4	Straw	14	0.70, 0.85 [0.77]	0.08, 0.08 [0.08]	
Bernie , MO, US; (C23-0198)	CL151	1 [--] 2 [109] 3 [6]	0.036** 96.5 96.5	n/a 187.3 187.4	Straw	14	0.51, 0.43 [0.47]	0.05, 0.04 [0.05]	
Cheneyville, LA, US; (E17-0200)	CL151	1 [--] 2 [111] 3 [5]	0.036** 103.9 96.7	n/a 176.3 161.4	Straw	0	2.7, 2.6 [2.5]	0.02, 0.02 [0.02]	
						7	1.1, 1.0 [1.0]	0.05, 0.05 [0.05]	
						14	0.44, 0.63 [0.54]	0.03, 0.04 [0.03]	
						21	0.34, 0.38 [0.36]	0.02, 0.03 [0.03]	
						28	0.33, 0.22 [0.28]	0.02, 0.01 [0.02]	
Dudley , MO, US; (C23-0202)	CL151	1 [--] 2 [115] 3 [4]	0.036** 96.8 96.2	n/a 187.8 186.7	Straw	14	0.19, 0.21 [0.20]	0.01, 0.01 [0.01]	
East Bernard , TX, US; (W41-0203) [b]	CL151	1 [--] 2 [122] 3 [4]	0.036** 96.7 96.7	n/a 173 175.5	Straw	14	0.18, 0.21 [0.20]	0.03, 0.03 [0.03]	
East Bernard , TX, US; (W41-0204) [b]	CL151	1 [--] 2 [122] 3 [4]	0.036** 96.5 97	n/a 173 176.3	Straw	14	0.16, 0.17 [0.16]	0.03, 0.03 [0.03]	
Durham, CA, US; (W23-0205)	M205	1 [--] 2 [139] 3 [5]	0.036** 96.4 98.3	n/a 18.8 19.1	Straw	14	0.74, 0.37 [0.55]	0.03, 0.02 [0.02]	

Location; (Trial ID)	Crop Variety	Application			Commodity	DALA	Residues (mg/kg; as received) [Mean]		Study report
		No. [interval, days]	Rate, g ai/ha	L/ha			Thiamethoxam	Clothianidin	Remarks
Near Willows, CA, US; (W23-0206)	M205	1 [--] 2 [139] 3 [5]	0.036** 96.4 96.5	n/a 187.7 187.9	Straw	14	0.25, 0.48 [0.36]	0.02, 0.02 [0.02]	

** mg ai/seed

[a] Same location and application dates.

[b] Same location and application dates.

Sorghum

The trials in sorghum (Salzman, F., 2016, Report TK0176048) are described above. From those trials, samples (ca 0.9 kg) of forage and stover were harvested 7 and 14 DALA, respectively.

Following harvest, samples were bagged and put into put into frozen storage prior to transport to the analytical facility. Samples were shipped frozen to the facility and were immediately placed into frozen storage. Prior to analysis, samples were cryogenically homogenized and returned to frozen storage. Samples were stored for a maximum of 11.1 months (forage) and 10.4 months (stover) prior to analysis. The storage period is covered by previously evaluated storage stability data that showed residues are stable under frozen conditions in a wide range of commodities for 1–2 years.

Samples were analysed for residues of thiamethoxam and clothianidin using the method AG-675. Concurrent recovery data indicate that the method is suitable.

Table 14 Results of thiamethoxam residue trials in **sorghum** conducted in the USA in 2013

Location; (Trial ID)	Crop Variety	Application			Com-modity	DALA	Residues (mg/kg) [Mean]		Study report
		No. [interval, days]	Rate, g ai/ha	L/ha			Thiamethoxam	Clothianidin	Remarks
Critical GAP (US)	--	1 [--] 2 [n.s.] 3 [7]	2.97** 96† 97	n/a 94 (grnd) 19 (air)	Forage Stover	7 14	--	--	
Seven Springs, NC, US; (TK0176048-01)	5556 S	1 [--] 2 [7]	96.39 97.44	308.7 299.3	Forage	6	<0.01, <0.01 [<0.01]	0.124, 0.155 [0.14]	TK0176048
		1 [--] 2 [8]	95.24 95.49	308.7 308.7	Stover	11	0.162, 0.128 [0.15]	0.182, 0.174 [0.18]	
Proctor, AR, US; (TK0176048-02)	DKS 53-67	1 [--] 2 [7]	96.28 96.03	93.54 168.4	Forage	7	0.019, 0.017 [0.018]	0.228, 0.220 [0.22]	
		1 [--] 2 [7]	96.02 96.39	140.3 93.54	Stover	14	0.067, 0.084 [0.075]	0.074, 0.063 [0.068]	
Lime Springs, IA, US; (TK0176048-03)	-	1 [--] 2 [7]	96.66 96.67	187.1 187.1	Forage	7	0.078, 0.088 [0.083]	0.139, 0.199 [0.17]	

Location; (Trial ID)	Crop Variety	Application			Com-modity	DALA	Residues (mg/kg) [Mean]		Study report
		No. [interval, days]	Rate, g ai/ha	L/ha			Thiamethoxam	Clothianidin	Remarks
		1 [--] 2 [7]	96.15 96.70	187.1 187.1	Stover	14	0.275, 0.430 [0.35]	0.349, 0.572 [0.46]	
Stafford, KS, US; (TK0176048-04)	Mycogen 627C	1 [--] 2 [8]	98.24 97.40	28.06 28.06	Forage	6	0.019, 0.037 [0.028]	0.072, 0.127 [0.099]	
		1 [--] 2 [7]	97.01 99.77	28.06 28.06	Stover	14	0.067, 0.087 [0.077]	0.089, 0.113 [0.10]	
Richland, IA, US; (TK0176048-05)	85Y40	1 [--] 2 [7]	95.50 95.53	233.8 224.5	Forage	7	0.041, 0.015 [0.15]	0.230, 0.093 [0.26]	
		1 [--] 2 [7]	96.82 95.34	196.4 187	Stover	21	0.044, 0.099 [0.16]	0.056, 0.128 [0.17]	
York, NE, US; (TK0176048-06)	A1005964	1 [--] 2 [7]	98.53 96.82	196.4 187.1	Forage	7	0.016, 0.016 [0.016]	0.106, 0.106 [0.11]	
		1 [--] 2 [6]	97.11 97.28	196.4 196.4	Stover	15	0.090, 0.062 [0.076]	0.065, 0.025 [0.045]	
Raymondville, TX, US; (TK0176048-07)	A1020168	1 [--] 2 [8]	100.5 102.5	46.77 46.77	Forage	6	0.105, 0.138 [0.12]	0.234, 0.276 [0.26]	
		1 [--] 2 [7]	97.81 97.50	46.77 46.77	Stover	14	0.180, 0.208 [0.19]	0.060, 0.095 [0.077]	
Uvalde, TX, US; (TK0176048-08)	Pioneer 83G19	1 [--] 2 [7]	95.03 96.16	187.1 205.8	Forage	7	0.033, 0.039 [0.036]	0.101, 0.107 [0.10]	
		1 [--] 2 [7]	95.17 93.57	187.1 196.4	Stover	14	0.082, 0.091 [0.087]	0.054, 0.080 [0.067]	
Grand Island, NE, US; (TK0176048-09)	A1005964	1 [--] 2 [6]	96.55 96.66	196.4 177.7	Forage	7	0.222, 0.384 [0.30]	0.260, 0.312 [0.29]	
		1 [--] 2 [7]	96.91 96.91	196.4 196.4	Stover	14	0.124, 0.106 [0.12]	0.040, 0.036 [0.038]	
Hinton, OK, US; (TK0176048-10) [a]	SR25835	1 [--] 2 [7]	94.47 96.49	46.77 46.77	Forage	7	0.137, 0.202 [0.17]	0.095, 0.102 [0.099]	
		1 [--] 2 [7]	95.54 94.78	37.4 46.77	Stover	14	<0.01, <0.01 [<0.01]	<0.01, 0.043 [<0.026]	
Hinton, OK, US; (TK0176048-12) [a]	SR25835	1 [--] 2 [7]	97.88 95.49	271.3 271.3	Forage	7	0.119, 0.155 [0.14]	0.185, 0.236 [0.21]	
		1 [--] 2 [6]	97.70 95.99	205.8 149.7	Stover	15	0.334, 0.594 [0.46]	0.214, 0.187 [0.20]	
San Angelo, TX, US; (TK0176048-11)	DKS 49-45	1 [--] 2 [6]	96.98 96.29	131 131	Forage	3	0.361, 0.145 [0.47]	0.281, 0.123 [0.14]	
		1 [--] 2 [7]	99.13 96.54	140.3 131	Stover	20	0.115, 0.181 [0.12]	0.044, 0.186 [0.081]	

** g ai/kg seed

[a] Same location; application dates are offset by 15 days

Sweet corn

The trials in sweet corn (Willard, T., 2013, Report TK0014208) are described above. From those trials, samples (size not specified) of forage and stover were harvested 1 and 4–60 DALA, respectively.

Following harvest, samples were bagged and put into put into frozen storage prior to transport to the analytical facility. Samples were shipped frozen to the facility and were immediately placed into frozen storage. Prior to analysis, samples were cryogenically homogenized and returned to frozen storage. Samples were stored for a maximum of 13.8 months (forage) and 14.3 months (stover) prior to analysis. The storage period is covered by previously evaluated storage stability data that showed residues are stable under frozen conditions in a wide range of commodities for 1–2 years.

Samples were analysed for residues of thiamethoxam and clothianidin using the method GRM.009.02A. Concurrent recovery data indicate that the method is suitable.

Table 15 Results of thiamethoxam residue trials in **sweet corn conducted** in the USA in 2010

Location; (Trial ID)	Crop Variety	Application			Com-modity	DALA	Residues (mg/kg; as received) [Mean]		Study report
		No. [interval, days]	Rate, g ai/ha	L/ha			Thiamethoxam	Clothianidin	Remarks
Critical GAP (US)	--	1 [--] 2 [--] 3 [5]	1.25** 96† 97	n/a 94 (grnd) 19 (air)	Forage Silage Fodder Stover	1 1 26 26	--	--	
North Rose, NY, US; (E02-0131)	GH4927	1 [--] 2 [127] 3 [4]	1.27** 96 97	n/a 324.7 330	Forage	1	2.49, 3.09 [2.8]	0.192, 0.238 [0.22]	TK0014208
					Stover	8	0.135, 0.104 [0.12]	0.0683, 0.0463 [0.057]	
Frenchtown, PA, US; (E04-0132)	Garrison	1 [--] 2 [93] 3 [4]	1.29** 100 101	n/a 363.8 341.8	Forage	1	2.05, 1.83 [1.9]	0.111, 0.0907 [0.10]	
					Stover	34	0.096, 0.0607 [0.078]	0.0194, 0.0158 [0.018]	
Seven Springs, NC, US; (E10-0133)	GH4927	1 [--] 2 [131] 3 [4]	1.27** 96 98	n/a 278.7 282.2	Forage	1	1.64, 1.53 [1.6]	0.132, 0.116 [0.12]	
					Stover	28	0.0103, 0.0118 [0.011]	<0.01 , <0.01 [<0.01]	
Clermont, FL, US; (E19-0134)	Garrison	1 [--] 2 [208] 3 [4]	1.29** 96 96	n/a 18.6 18.7	Forage	1	1.11, 0.978 [1.0]	0.0279, 0.0468 [0.037]	

Location; (Trial ID)	Crop Variety	Application			Com-modity	DALA	Residues (mg/kg; as received) [Mean]		Study report
		No. [interval, days]	Rate, g ai/ha	L/ha			Thiamethoxam	Clothianidin	Remarks
					Stover	41	0.0695, 0.11 [0.090]	<0.01, <0.01 [<0.01]	
Northwood, ND, US; (C13-0135)	GH4927	1 [--] 2 [91] 3 [4]	1.27** 98 97	n/a 190.3 188.8	Forage	0	1.81	0.0331	
						1	1.46, 1.24 [1.4]	0.0389, 0.0341 [0.036]	
						3	0.262	0.0384	
						5	0.126	0.0329	
						7	0.0941	0.0354	
					Stover	0	1.91	0.0586	
						7	0.113	0.0245	
						15	0.0276, 0.0318 [0.030]	0.0111, 0.011 [0.011]	
						21	0.0218	<0.01	
						27	0.0104	<0.01	
Richland, IA, US; (C18-0136)	Garrison	1 [--] 2 [83] 3 [4]	1.29** 99 99	n/a 158.6 162	Forage	1	1.65, 1.08 [1.4]	0.115, 0.066 [0.090]	
					Stover	17	0.055, 0.0368 [0.046]	0.0133, <0.01 [<0.012]	
Oregon, MO, US; (C19-0137)	GH4927	1 [--] 2 [81] 3 [4]	1.27** 100 97	n/a 147.3 140.6	Forage	1	1.35, 1.24 [1.3]	0.121, 0.12 [0.12]	
					Stover	29	0.0211, 0.0222 [0.022]	<0.01, <0.01 [<0.01]	
York, NE, US; (C33-0138)	Garrison	1 [--] 2 [85] 3 [4]	1.29** 95 96	n/a 184.9 185.8	Forage	1	0.86, 0.597 [0.73]	0.0361, 0.0228 [0.029]	
					Stover	60	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	
Gardner, ND, US; (C12-0139)	GH4927	1 [--] 2 [107] 3 [5]	1.27** 98 97	n/a 190.3 188.4	Forage	1	2.15, 1.69 [1.9]	0.0821, 0.0744 [0.078]	
					Stover	49	0.0177, 0.0199 [0.019]	<0.01, <0.01 [<0.01]	
Fresno, CA, US; (E19-0140)	Garrison	1 [--] 2 [79] 3 [4]	1.29** 95 97	n/a 402 406	Forage	1	1.47, 1.79 [1.6]	0.0138, 0.0202 [0.017]	

Location; (Trial ID)	Crop Variety	Application			Com-modity	DALA	Residues (mg/kg; as received) [Mean]		Study report
		No. [interval, days]	Rate, g ai/ha	L/ha			Thiamethoxam	Clothianidin	Remarks
					Stover	14	0.212, 0.305 [0.26]	0.0117, 0.0123 [0.012]	
Rupert, ID, US; (W15-0141)	GH4927	1 [--] 2 [103] 3 [4]	1.27** 96 98	n/a 206 161.5	Forage	0	0.939	0.028	
						1	0.705, 0.0755 [0.39]	0.0519, 0.0265 [0.039]	
						3	0.389	0.0437	
						5	0.0976	0.0256	
						7	0.0728	0.0247	
					Stover	0	2.34	0.0731	
						7	0.282	0.112	
						14	0.0602, 0.0953 [0.078]	0.02, 0.0326 [0.026]	
						21	0.059	0.0176	
						28	0.099	0.0209	
Portland, OR, US; (W20-0142)	Garrison	1 [--] 2 [154] 3 [5]	1.29** 98 96	n/a 209.4 219.4	Forage	1	1.79, 1.51 [1.6]	0.106, 0.0748 [0.090]	
					Stover	4	2.42, 2.49 [2.5]	0.14, 0.128 [0.13]	

** mg ai/kernel

FATE OF RESIDUES DURING PROCESSING

Wheat

In the study described above investigating thiamethoxam residues in wheat (Study Report TK0020708), bulk grain samples were collected from the exaggerated rate plots at the C13-0169 and C12-0172 sites. The grain samples were processed into aspirated grain fractions, bran, flour, middlings, shorts, and germ using simulated commercial practices. Processed samples were shipped frozen to the analytical facility, where they were assayed for residues of thiamethoxam and clothianidin using Method AG-675. Processed commodities were stored no longer than 4.1 months prior to extraction and analysis.

Wheat grain samples were dried to a moisture content of 10–13%. Following drying, the grain was moved using bucket conveyors and a screw conveyor within a dust generation room. The resulting grain dust was removed by aspiration, characterized into size fractions, and reconstituted to make samples of aspirated grain fractions.

After cleaning to separate grain from foreign particles, the wheat grain was tempered to a moisture content of 16% and then milled to separate bran from germ with endosperm; the latter was milled again to separate the germ from the endosperm.

A separate batch of cleaned, tempered grain was passed through break rolls to obtain break flour (<140 µm), middlings <800 µm, and bran (>800µm). Middlings were passed through two reduction rolls to produce reduction flour (<160 µm) and shorts (>160 µm). The break flour and reduction flour were combined to provide a flour sample.

Table 16 Residues of thiamethoxam in **wheat processed commodities** from residue trials in wheat conducted in the USA

Trial ID	Commodity	Residues (mg/kg) [Mean]		Processing factor		Study report
		Thiamethoxam	Clothianidin	Thiamethoxam	Clothianidin	
C13-0169	Grain (prior to processing)	0.204, 0.206, 0.197 [0.20]	0.132, 0.159, 0.136 [0.14]	--	--	TK0020708
	Aspirated grain fractions	1.81, 1.91 [1.9]	0.524, 0.556 [0.54]	9.2	3.8	
	Bran	0.419, 0.443 [0.43]	0.266, 0.256 [0.26]	2.1	1.8	
	Flour	0.0224, 0.0198 [0.021]	0.0850, 0.0862 [0.086]	0.10	0.60	
	Middlings	0.0880, 0.0880 [0.088]	0.111, 0.121 [0.12]	0.43	0.81	
	Shorts	0.0968, 0.104 [0.10]	0.122, 0.132 [0.13]	0.50	0.89	
	Germ	0.304, 0.306 [0.30]	0.249, 0.261 [0.26]	1.5	1.8	
C12-0172	Grain (prior to processing)	0.158, 0.176, 0.169 [0.17]	0.159, 0.182, 0.173 [0.17]	--	--	
	Aspirated grain fractions	3.03, 3.81 [3.4]	1.72, 1.86 [1.8]	20	10	
	Bran	0.467, 0.455 [0.46]	0.305, 0.334 [0.32]	2.7	1.9	
	Flour	0.0191, 0.0189 [0.019]	0.124, 0.112 [0.12]	0.11	0.69	
	Middlings	0.0536, 0.0482 [0.051]	0.132, 0.134 [0.13]	0.30	0.78	
	Shorts	0.0764, 0.0536 [0.065]	0.163, 0.149 [0.16]	0.39	0.91	
	Germ	0.236, 0.244 [0.24]	0.304, 0.319 [0.31]	1.4	1.8	

Rice

In the study described above investigating thiamethoxam residues in rice (Study Report TK0021631), bulk grain samples were collected from the exaggerated rate plots at the E17-0191 and E18-0197 sites. The grain samples were processed into hulls, bran, and polished rice using simulated commercial practices. Processed samples were shipped frozen to the analytical facility, where they were assayed for residues of thiamethoxam and clothianidin using Method GRM.009.02A. Processed commodities were stored no longer than 12.5 months prior to extraction and analysis.

Rough rice samples were dried to a moisture content of 11–14%. Following drying, the grain was cleaned by aspiration and screening. Cleaned rough rice was milled to separate the hull from the seed + bran (brown rice). The brown rice was then milled to separate the bran from the polished rice.

Brown rice was not assayed for residues. To estimate residues in brown rice, the Meeting back-calculated based on residues in bran, polished rice, and the reported material balance in the processing studies. In making this estimation, the Meeting has assumed that the physical removal of the hulls in processing rice grain to husked rice would not alter the relative amounts of thiamethoxam and clothianidin in those matrices. This is supported by the results of the high-temperature hydrolysis study, which showed thiamethoxam was stable throughout the study.

	Bran			Polished Rice			Brown Rice [a]	
Trial ID	Material balance, kg	Thiamethoxam, mg/kg	Clothianidin, mg/kg	Material balance, kg	Thiamethoxam, mg/kg	Clothianidin, mg/kg	Thiamethoxam, mg/kg	Clothianidin, mg/kg
E17-0191	1.63	2.33	0.25	14.38	0.19	0.10	0.41	0.12
E18-0197	1.77	2.76	0.38	12.20	0.19	0.18	0.52	0.21

[a] Residue estimate in brown rice = [(bran material balance × bran residue) + (polished rice material balance × polished rice residue)] ÷ (bran material balance + polished rice material balance)

Table 17 Residues of thiamethoxam in **rice processed commodities** from residue trials in rice conducted in the USA

Trial ID	Commodity	Residues (mg/kg) [Mean]		Processing factor		Study report
		Thiamethoxam	Clothianidin	Thiamethoxam	Clothianidin	
E17-0191	Grain (prior to processing)	6.0, 6.4, 5.3 [5.9]	0.31, 0.35, 0.29 [0.32]	--	--	TK0021631
	Hulls	27	1.1	4.6	3.5	
	Husked rice	0.41	0.12	0.069	0.38	
	Bran	2.3	0.25	0.39	0.79	
	Polished rice	0.19	0.10	0.032	0.32	
E18-0197	Grain (prior to processing)	3.0, 2.0, 6.2 [3.7]	0.22, 0.15, 0.42 [0.26]	--	--	
	Hulls	24	1.31	6.4	5.0	
	Husked rice	0.52	0.21	0.14	0.81	
	Bran	2.8	0.38	0.75	1.4	
	Polished rice	0.19	0.18	0.051	0.68	

Grain Sorghum

In the study described above investigating thiamethoxam residues in grain sorghum (Study Report TK0176048), bulk grain samples were collected from the exaggerated rate plots at the TK0176048-06 and -12 sites. The sample from the -06 site were stored frozen and shipped frozen to the processing facility whereas the sample from the -12 site was stored at ambient temperatures and shipped frozen to the processing facility. The grain samples were processed into aspirated grain fractions and flour using simulated commercial practices. Processed samples were shipped frozen to the analytical facility, where they were assayed for residues of thiamethoxam and clothianidin using Method AG-675. Processed commodities were stored no longer than 12.9 months prior to extraction and analysis.

Sorghum grain samples were dried to a moisture content of 10–13%. Following drying, the grain was moved using bucket conveyors and a screw conveyor within a dust generation room. The resulting

grain dust was removed by aspiration, characterized into size fractions, and reconstituted to make samples of aspirated grain fractions.

A separate sample of grain sorghum was cleaned by aspiration and screening to separate the grain from foreign particles. The cleaned grain was then milled and sieved to generate sorghum flour.

Sweet sorghum

In the study described above investigating thiamethoxam residues in sweet sorghum (Study Report TK0176048), bulk stalk samples were collected from the exaggerated rate plots at the TK0176048-16 site. The stalks were thawed and processed into sorghum syrup using simulated commercial practices. Processed samples were shipped frozen to the analytical facility, where they were assayed for residues of thiamethoxam and clothianidin using Method AG-675. Processed commodities were stored no longer than 8.8 months prior to extraction and analysis.

Syrup was obtained by passing the stalks two times through a roller press to express raw sorghum juice. The juice was screened to remove suspended material and then heated to 71–82 °C and allowed to settle for 2 hours. During the settling period, material that rose to the surface was skimmed. After the 2-hour period, the juice was decanted to separate it from material that had settled to the bottom of the container. The clarified juice was then boiled (96–107 °C) until a Brix density of 73–80° was reached; again, any material that rose to the top during the boiling process was removed by skimming. After boiling, the syrup was allowed to cool and was then filtered through a 354-µm screen to produce the final sweet sorghum syrup.

Table 18 Residues of thiamethoxam in **sorghum processed commodities** from residue trials in sorghum conducted in the USA

Trial ID	Commodity	Residues (mg/kg) [Mean]		Processing factor		Study report
		Thiamethoxam	Clothianidin	Thiamethoxam	Clothianidin	
TK017048-06	Grain (prior to processing)	0.257, 0.266, 0.264 [0.26]	0.0223, 0.0226, 0.0240 [0.023]	--	--	TK0176048
	Aspirated grain fractions	3.09	0.385	12	17	
	Flour	0.197	0.0127	0.75	0.55	
TK0176048-12	Grain (prior to processing)	0.892, 0.956, 1.08 [0.98]	0.0644, 0.0767, 0.0745 [0.072]	--	--	
	Aspirated grain fractions	11.0	0.589	11	8.2	
	Flour	0.711	0.0521	0.73	0.72	
TK0176048-16	Cane	0.234, 0.220, 0.236 [0.23]	0.0262, 0.0342, 0.0653 [0.042]	--	--	
	Syrup	0.187	0.0386	0.81	0.92	

APPRAISAL

Thiamethoxam (ISO common name) is a broad-spectrum, neonicotinoid insecticide with registered uses on multiple crops. It was evaluated for the first time by the 2010 JMPR, which established an acceptable daily

intake (ADI) of 0–0.08 mg/kg bw and an acute reference dose (ARfD) of 1 mg/kg bw. Thiamethoxam underwent subsequent evaluations by the JMPR in 2011, 2012 and 2014.

The definition of the residue for compliance with the MRL for animal and plant commodities is thiamethoxam. For dietary risk assessment, the residue definitions are thiamethoxam and clothianidin (a.k.a. CGA322704), assessed separately, for plant and animal commodities except poultry and the sum of thiamethoxam, CGA 265307 (N-(2-chlorothiazol-5-ylmethyl)-N'-nitroguanidine) and MU3 (amino-([(2-chlorothiazol-5-ylmethyl)-amino]-methylene)-hydrazide), expressed as thiamethoxam, along with clothianidin (assessed separately) for poultry commodities. The residue is not fat-soluble.

Thiamethoxam was scheduled at the Fifty-first Session of the CCPR for evaluation of additional uses by the 2020 JMPR, which was postponed to the 2021 Extra JMPR. The current Meeting received information on analytical methods, field trials and processing studies to support estimation of maximum residue levels in persimmon, barley, rice, sorghum, sweet corn and wheat.

Clothianidin (238) is a metabolite of thiamethoxam and a registered active ingredient. In addition to considering residues of thiamethoxam, the Meeting also considered residues of clothianidin arising from the uses of thiamethoxam (see section 5.5 Clothianidin (238)).

Methods of analysis

The Meeting received method validation and concurrent recovery data for use of Method AG-765 (reviewed by the 2010 JMPR) and the related Method GRM.009.02A, an unnamed method for analysis of residues in Japanese persimmon, as well as the QuEChERS multiresidue method. All methods were demonstrated to have adequate performance for recovery of thiamethoxam, with an LOQ of 0.01 mg/kg (0.02 mg/kg in Japanese persimmon).

Stability of residues in stored analytical samples

No new information on the stability of thiamethoxam residues during frozen storage was provided. The 2010 JMPR had determined that residues of thiamethoxam are stable for 1–2 years under frozen (-18 °C) conditions for a large range of commodities, including the high-water and high-starch commodities considered by the current Meeting. The stability of residues in the crops under consideration by the current Meeting is considered to be adequately demonstrated for the periods that field trial samples were stored prior to analysis.

Results of supervised residue trials on crops

The Meeting received GAP information and data from supervised residue trials on persimmon, wheat, barley, rice, sorghum (grain and sweet) and sweet corn.

Persimmon, Japanese

The critical GAP for Japanese persimmon is from the Republic of Korea and consists of three applications, on a 10-day interval, of a 2000-fold dilution of the formulation (equivalent to 5 g ai/hL). Applications are made to the point of run-off and there is a 7-day PHI.

Residues of thiamethoxam in independent trials provided to the current Meeting approximating the critical GAP were (n = 2): 0.092 and 0.26 mg/kg.

The 2014 Meeting evaluated residues in Japanese persimmon in trials approximating the critical GAP. Residues of thiamethoxam in independent trials were (n = 2): 0.14 and 0.19 mg/kg.

The Meeting decided to combine the data from the two sets of trials (n = 4): 0.092, 0.14, 0.19 and 0.26 mg/kg.

The Meeting estimated a maximum residue level of 0.6 mg/kg, an STMR of 0.165 mg/kg and an HR of 0.26 mg/kg for Japanese persimmon.

Wheat

Labels were provided for registrations in Mexico (2 foliar applications at 56 g ai/ha, 7-day RTI, 14-day PHI) and the USA (seed treatment at 52 g ai/100 kg seed + 2 foliar applications at 96 g ai/ha, 5-day RTI, 30-day PHI). Residue decline data indicated that for those two GAPs, the rate is the determining factor in residues at harvest rather than the RTI or the PHI. Therefore, the Meeting decided that the GAP from the USA is the critical GAP.

Trials in the USA approximated the US GAP for application rate and timing, but with a significantly shorter interval between the last application and harvest (14 days). The Meeting decided that the data are not suitable to support recommendations based on the US GAP. The Meeting agreed to consider the GAP from Mexico.

The Meeting determined that the seed treatment application used in the trials was not likely to contribute significantly to residues at harvest and decided that the US trials can be adjusted using proportionality to approximate the GAP from Mexico.

The Meeting noted that the results from the trial in Grand Island, NE (Trial C33-0170) are inconsistent within the residue decline data (< 0.01 mg/kg in the 7, 10, 14 and 17 DALA samples and 5.0 mg/kg in the 21 DALA sample) and also in terms of the full set of available field trials (residue range = 0.015 to 0.18 mg/kg). The Meeting agreed not to consider data from that trial in any residue recommendations.

Residues (unscaled) of thiamethoxam in independent trials were (n = 16): 0.015, 0.021, 0.024, 0.032, 0.038, 0.041, 0.047, 0.052, 0.059. 0.063, 0.070, 0.091, 0.10, 0.13 (2) and 0.18 mg/kg.

When scaled (scaling factors range from 0.57 to 0.59), residues were (n = 16): < 0.01, 0.012, 0.014, 0.019, 0.022, 0.024, 0.027, 0.030, 0.034, 0.037, 0.041, 0.053, 0.058, 0.074, 0.076 and 0.10 mg/kg.

The Meeting estimated a maximum residue level of 0.15 mg/kg and an STMR of 0.032 mg/kg for wheat grain. Based on the registered use including triticale, the Meeting decided to extrapolate the residue estimates to triticale.

Barley

Labels were provided for registrations in Mexico (2 foliar applications at 56 g ai/ha, 7-day RTI, 14-day PHI) and the USA (seed treatment at 52 g ai/100 kg seed + 2 foliar applications at 96 g ai/ha, 5-day RTI, 30-day PHI). Residue decline data indicated that for those two GAPs, the rate is the determining factor in residues at harvest rather than the RTI or the PHI. Therefore, the Meeting decided that the GAP from the USA is the critical GAP.

Trials in the USA approximated the US GAP for application rate and timing, but with a significantly shorter interval between the last application and harvest (14 days). The Meeting decided that the data are not suitable to support recommendations based on the US GAP. The Meeting agreed to consider the GAP from Mexico.

The Meeting determined that the seed treatment application used in the trials was not likely to contribute significantly to residues at harvest and decided that the US trials can be adjusted using proportionality to approximate the GAP from Mexico.

Residues (unscaled) of thiamethoxam in independent trials were (n = 12): 0.030, 0.042, 0.067, 0.12 (2), 0.16, 0.22, 0.27 (2), 0.31, 0.34 and 0.53 mg/kg.

When scaled (scaling factors range from 0.57 to 0.61), residues were (n = 12): 0.017, 0.024, 0.041, 0.070 (2), 0.093, 0.13, 0.15, 0.16, 0.18, 0.20 and 0.31 mg/kg.

The Meeting estimated a maximum residue level of 0.5 mg/kg and an STMR of 0.112 mg/kg for barley grain. Based on the registered uses including oat and barley being an example representative commodity of the subgroup that includes barley and oat, the Meeting decided to extrapolate the residue estimates to oat.

Rice

The critical GAP for rice is from Japan and consists of two applications of a 1000x dilution in up to 1500 L/ha (equivalent to 97.5 g ai/ha) with a PHI of 14 days; a re-treatment interval is not specified.

Residues of thiamethoxam in husked rice from independent trials conducted in the USA and approximating the critical GAP were (n = 11): 0.92, 1.3, 1.5 (2), 1.6, 1.7, 1.8, 2.0 (3) and 2.3 mg/kg. Of those, two trials were done in paddy rice with residues of 1.7 and 2.0 mg/kg.

Since the residue populations of husked paddy rice and husked upland rice are similar, the Meeting estimated a maximum residue level of 5 mg/kg and an STMR of 1.7 mg/kg for husked rice.

Grain sorghum

The critical GAP for grain sorghum is from the USA and consists of three applications: a seed treatment at 2.97 g ai/100 kg seed followed by two in-season foliar applications, each at 96 g ai/ha, on a 7-day interval, with a PHI of 14 days.

Trials were conducted in the USA using the in-season applications only. The Meeting determined that residues of thiamethoxam coming from the seed treatment use were not likely to contribute significantly to residues at harvest. Residues of thiamethoxam in grain sorghum from independent trials approximating the critical GAP were (n = 11): 0.013, 0.021, 0.044, 0.046, 0.049, 0.079, 0.13, 0.14, 0.15, 0.26 and 0.35 mg/kg.

The Meeting estimated a maximum residue level of 0.6 mg/kg and an STMR of 0.079 mg/kg for grain sorghum.

Sweet corn

The critical GAP for sweet corn is from the USA and consists of three applications: a seed treatment at 52 g ai/100 kg seed followed by two in-season foliar applications, each at 96 g ai/ha, on a 5-day interval, with a PHI of 1 day.

Residues of thiamethoxam in sweet corn (kernel + cob with husk removed) from independent trials conducted in the USA and approximating the critical GAP were (n = 12): < 0.01 (12) mg/kg.

The Meeting noted that the only crop in the Subgroup of sweet corns is sweet corn and that the representative commodity is sweet corn (corn on the cob, kernels plus cob with husk removed) and agreed to estimate 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 residues of thiamethoxam in the Subgroup of sweet corns (Subgroup 020F).

Grasses for sugar or syrup production

Sweet sorghum

The critical GAP for sweet sorghum is from the USA and consists of three applications: a seed treatment at 2.97 g ai/100 kg seed followed by two in-season foliar applications, each at 96 g ai/ha, on a 7-day interval, with a PHI of 14 days.

Trials were conducted in the USA using the in-season applications only. The Meeting determined that residues of thiamethoxam coming from the seed treatment use were not likely to contribute significantly to residues at harvest. Residues of thiamethoxam in sweet sorghum cane from independent trials approximating the critical GAP were (n = 4): < 0.01, < 0.01, 0.056 and 0.24 mg/kg.

The Meeting estimated a maximum residue level of 0.6 mg/kg, an STMR of 0.033 mg/kg and an HR of 0.24 mg/kg for sorgo or sorghum, sweet.

Residues in animal feeds

Forages of cereal grains

Wheat

The critical GAP for forage is from the USA (seed treatment at 52 g ai/100 kg seed + 2 foliar applications at 96 g ai/ha, 5-day RTI, 7-day PHI).

Residues in forage from trials in the USA approximating the US GAP were (n = 17): < 0.015, 0.011, 0.023, 0.03, 0.032, 0.036, 0.072, 0.079, 0.12, 0.18, 0.33 (2), 0.46, 0.73, 1.1 (2) and 2.0 mg/kg.

The registration in the USA includes barley, oat, rye, triticale and wheat. Based on the residue data (as received) for wheat forage listed above, the Meeting estimated a median residue of 0.12 mg/kg and a highest residue of 2 mg/kg for wheat forage. The Meeting agreed to extrapolate the median and highest residue estimates from wheat forage to forage of barley, oat, rye and triticale.

Hay of cereal grains

Wheat

The critical GAP for hay is from the USA (seed treatment at 52 g ai/100 kg seed + 2 foliar applications at 96 g ai/ha, 5-day RTI, 7-day PHI).

Residues in wheat hay from trials in the USA approximating the US GAP were (n = 17): 0.014, 0.015, 0.034, 0.036, 0.049, 0.054, 0.058, 0.16, 0.20, 0.32, 0.41, 0.52, 0.91, 1.5, 2.0, 2.1 and 6.7 mg/kg.

Barley

The critical GAP for hay is from the USA (seed treatment at 52 g ai/100 kg seed + 2 foliar applications at 96 g ai/ha, 5-day RTI, 7-day PHI).

Residues in barley hay from trials in the USA approximating the US GAP were (n = 12): 0.023, 0.058, 0.083, 0.12, 0.17, 0.19, 0.25, 0.34, 0.39, 0.42, 0.46 and 0.52 mg/kg.

The registration in the USA includes barley, oat, rye, triticale and wheat. The Meeting determined via a Mann-Whitney U Test that the residue data listed above for wheat and barley hay are not statistically different and agreed to combine the data. Residues of thiamethoxam in wheat and barley hay following treatment approximating the US GAP were (n = 29): 0.014, 0.015, 0.023, 0.034, 0.036, 0.049, 0.054, 0.058,

0.058, 0.083, 0.12, 0.16, 0.17, 0.19, 0.20, 0.25, 0.32, 0.34, 0.39, 0.41, 0.42, 0.46, 0.52, 0.52, 0.91, 1.5, 2.0, 2.1 and 6.7 mg/kg.

Based on the registered uses and on the data for wheat and barley hay and on an OECD standard dry-matter content of 88%, the Meeting estimated a maximum residue level of 8 mg/kg (dw), a median residue of 0.2 mg/kg (as received) and a highest residue of 6.7 mg/kg (as received) in hay of wheat, barley, oat, rye and triticale.

Straw of cereal grains

Wheat

Labels were provided for registrations in Mexico (2 foliar applications at 56 g ai/ha, 7-day RTI, 14-day PHI) and the USA (seed treatment at 52 g ai/100 kg seed + 2 foliar applications at 96 g ai/ha, 5-day RTI, 30-day PHI).

For wheat straw, the trials in the USA did not match the USA GAP due to the straw being harvested 14 DALA rather than the 30 days specified for the PHI. Therefore, the Meeting decided that the data are not suitable to support recommendations based on the USA GAP. The Meeting agreed to consider the GAP from Mexico. As noted for wheat grain, the Meeting determined that residues of thiamethoxam coming from the seed treatment use were not likely to contribute significantly to residues at harvest and that residues from the USA trials can be adjusted using proportionality to approximate the GAP from Mexico.

Residues (unscaled) of thiamethoxam in wheat straw from independent trials were (n = 17): 0.14, 0.20, 0.21, 0.25, 0.43, 0.49, 0.75, 0.84, 0.92, 1.6, 1.7 (3), 1.9, 2.4, 3.3 and 3.9 mg/kg.

When scaled (scaling factors range from 0.57 to 0.59), residues in wheat straw were (n = 17): 0.081, 0.12, 0.12, 0.15, 0.25, 0.28, 0.43, 0.49, 0.53, 0.93, 0.98, 0.99, 1.0, 1.1, 1.4, 1.9 and 2.2 mg/kg.

Barley

Labels were provided for registrations in Mexico (2 foliar applications at 56 g ai/ha, 7-day RTI, 14-day PHI) and the USA (seed treatment at 52 g ai/100 kg seed + 2 foliar applications at 96 g ai/ha, 5-day RTI, 30-day PHI).

For barley straw, the trials in the USA did not match the USA GAP due to the straw being harvested 14 DALA rather than the 30 days specified for the PHI. Therefore, the Meeting decided that the data are not suitable to support recommendations based on the US GAP. The Meeting agreed to consider the GAP from Mexico. As noted for barley grain, the Meeting determined that residues of thiamethoxam coming from the seed treatment use were not likely to contribute significantly to residues at harvest and that residues from the US trials can be adjusted using proportionality to approximate the GAP from Mexico.

Residues (unscaled) of thiamethoxam in barley straw from independent trials were (n = 12): 0.014, 0.016, 0.073, 0.078, 0.24, 0.25, 0.25, 0.26, 0.49, 0.53, 0.54, 2.3 mg/kg.

When scaled (scaling factors range from 0.57 to 0.59), residues in barley straw were (n = 12): < 0.01 (2), 0.042, 0.046, 0.14 (2), 0.15, 0.16, 0.28, 0.31 (2) and 1.3 mg/kg.

The registration in Mexico includes barley, oat, triticale and wheat. Noting that residues appear to be similar between the straw data for wheat and barley, the Meeting agreed to explore a common set of residue estimates for these straw commodities. A Mann-Whitney U-Test indicated that the residue distributions from wheat and barley are significantly different. The OECD MRL calculator suggested maximum residue levels of 4 mg/kg (median = 0.53 mg/kg) for wheat straw and 2 mg/kg for barley (median = 0.145 mg/kg), all on an as-received basis.

The Meeting noted that the maximum residue level and highest residue estimates in hay are greater than those in straw and agreed to apply those estimates to straw and to use the more conservative median residue from wheat for estimating dietary burdens. Furthermore, noting that the straw commodities of different cereal crops are generally indistinguishable from each other when in trade, the Meeting agreed to make recommendations for the straw of cereals included on the Mexico label (barley, oat, triticale, wheat).

Based on OECD standard dry-matter content of 88%, the Meeting estimated a maximum residue level of 8 mg/kg (dw), a median residue of 0.53 mg/kg (as received) and a highest residue of 6.7 mg/kg (as received) for residues of thiamethoxam in straw and fodder, dry of wheat, barley, oat and triticale.

The above recommendations replace the previous recommendations, each at 2 mg/kg, for barley straw and fodder, dry and wheat straw and fodder, dry.

Rice

The critical GAP for rice is from Japan and consists of two applications of a 1000x dilution in up to 1500 L/ha (equivalent to 97.5 g ai/ha) with a PHI of 14 days; a re-treatment interval is not specified.

Residues of thiamethoxam in rice straw from independent trials approximating the critical GAP were (n = 11): 0.2 (2), 0.3, 0.36(2), 0.47, 0.51, 0.54, 0.55 and 1.0 (2) mg/kg.

Based on OECD dry-matter content of 88%, the Meeting estimated a maximum residue level of 3 mg/kg (dw), a median residue of 0.47 mg/kg (as received) and a highest residue of 1 mg/kg (as received) for residues of thiamethoxam in rice straw and fodder, dry.

Sorghum

The critical GAP for sorghum is from the USA and consists of three applications: a seed treatment at 2.97 g ai/100 kg seed followed by two in-season foliar applications, each at 96 g ai/ha, on a 7-day interval, with a PHI of 14 days.

Trials were conducted in the USA using the in-season applications only. The Meeting determined that residues of thiamethoxam coming from the seed treatment use were not likely to contribute significantly to residues at harvest.

Residues of thiamethoxam in sorghum forage from independent trials approximating the critical GAP were (n = 10): < 0.01, 0.016, 0.018, 0.028, 0.036, 0.083, 0.12, 0.15, 0.17 and 0.30 mg/kg (as received).

The Meeting estimated a median residue of 0.06 mg/kg (as received) and a highest residue of 0.3 mg/kg (as received) in sorghum forage.

Residues of thiamethoxam in sorghum stover from independent trials approximating the critical GAP were (n = 10): 0.075, 0.076, 0.077, 0.087, 0.12, 0.15, 0.16, 0.19, 0.35 and 0.46 mg/kg (as received).

Based on OECD standard dry-matter content of 88%, the Meeting estimated a maximum residue level of 0.8 mg/kg (dw), a median residue of 0.14 mg/kg (as received) and a highest residue of 0.49 mg/kg (as received) in sorghum straw and fodder (dry).

Sweet corn

The critical GAP for sweet corn is from the USA and consists of three applications: a seed treatment at 52 g ai/100 kg seed followed by two in-season foliar applications, each at 96 g ai/ha, on a 5-day interval, with a PHI of 1 day for forage and 26 days for stover.

Residues of thiamethoxam in sweet corn forage from independent trials approximating the critical GAP were (n = 12): 0.39, 0.73, 1.0, 1.3, 1.4, 1.4, 1.6, 1.6, 1.6, 1.9, 1.9 and 2.8 mg/kg (as received).

The Meeting estimated a median residue of 1.5 mg/kg (as) and a highest residue of 2.8 mg/kg (as received) in sweet corn forage.

Residues of thiamethoxam in sweet corn stover from independent trials approximating the critical GAP were (n = 4): 0.010, 0.011, 0.022 and 0.099 mg/kg (as received).

Based on OECD standard dry-matter content of 83%, the Meeting estimated a maximum residue level of 0.25 mg/kg (dw), a median residue of 0.0165 mg/kg (as received) and a highest residue of 0.099 mg/kg (as received) in sweet corn stover.

Fate of residues during processing

The Meeting received data showing the effect of processing wheat grain into aspirated grain fractions, bran, flour, middlings, shorts and germ; rice into hulls, bran and polished rice; grain sorghum into aspirated grain fractions and flour; and sweet sorghum into syrup. Processing factors and residue estimates are summarized below.

Table 1 Processing factors and residue estimates for thiamethoxam in wheat, sorghum and sweet sorghum commodities

	Residue in RAC, mg/kg				Processing Factors		Residue in processed commodity, mg/kg		
Raw commodity	Max	STMR	HR	Processed commodity	Individual	Best estimate	Max-P	STMR-P	HR-P
Wheat grain	0.15	0.032	--	Aspirated fractions	9.2, 20	14.6	--	0.467	--
				Bran	2.1, 2.7	2.4	0.36	0.0768	--
				Flour	0.10, 0.11	0.105	--	0.00336	--
				Middlings	0.30, 0.43	0.365	--	0.0117	--
				Shorts	0.39, 0.50	0.445	--	0.0142	--
				Germ	1.4, 1.5	1.45	0.218	0.0464	--
Sorghum grain	0.6	0.079	--	Aspirated fractions	11, 12	11.5	--	0.908	--
				Flour	0.73, 0.75	0.74	--	0.0548	--
Sorghum cane	0.6	0.033	--	Syrup	0.81	0.81	--	0.0267	--

Based on the maximum residue estimates above and the OECD rounding classes, the Meeting estimated maximum residue levels of 0.4 mg/kg for wheat bran and 0.3 mg/kg for wheat germ. The Meeting decided to apply the processing factor for wheat bran (2.4) to barley bran, giving a maximum residue level of 1.5 mg/kg. The estimated STMR-Ps for the processed commodities considered by the Meeting are shown in Table 1. Furthermore, the Meeting decided to apply the processing factors for wheat bran (2.4) and flour (0.105) to barley and triticale, giving STMR-Ps as follows:

Barley bran = 0.269 mg/kg,

Barley flour = 0.0118 mg/kg,

Triticale flour = 0.00336 mg/kg.

For rice commodities, the processing study provided residue analysis for rice grain, rice hulls, rice bran and polished rice, but not for husked rice. The Meeting derived a processing factor for husked rice by mathematically reconstituting the commodity based on residues in bran, residues in polished rice and their respective material balances reported in the processing study. Conversely, the residue trials in rice only reported results for husked rice. Processing factors describing residues relative to the rice RAC (grain) and from husked rice are shown in Table 2.

Table 2 Processing factors and residue estimates for thiamethoxam in rice commodities

Commodity	Residue in starting commodity, mg/kg			Processed commodity	Processing Factors		Residue in processed commodity, mg/kg		
	Max	STMR	HR		Individual	Best estimate	Max-P	STMR-P	HR-P
Rice grain	See text below			Husked rice	0.069, 0.14	0.104	No field trial data were provided for rice grain		
				Hulls	4.6, 6.4	5.5			
				Bran	0.39, 0.75	0.57			
				Polished rice	0.032, 0.051	0.0415			
Husked Rice	5 [a]	1.7 [a]		Hulls	66, 45	56	280	95.2	--
				Bran	5.6, 5.4	5.5	27.5	9.35	--
				Polished rice	0.46, 0.37	0.41	2.07	0.704	--

[a] From field trials

For rice, the Meeting estimated maximum residue levels of 300 mg/kg for rice hulls, 30 mg/kg for rice bran and 3 mg/kg for polished rice. Furthermore, the Meeting used the residue estimates and the processing factor from rice grain to husked rice to derive residue estimates for rice grain. The Meeting estimated a maximum residue level of 50 mg/kg and a median residue of 17 mg/kg for rice grain.

Residues in animal commodities

The Meeting has added feed items and their associated residues to the dietary burden calculation used by the 2014 JMPR. Dietary burden calculations are provided in Annex 6; the dietary burden estimates are summarized below.

Table 3 Estimated maximum and mean dietary burdens of farm animals

Animal	Dietary burden estimates, ppm							
	Canada-US		European Union		Australia		Japan	
	Maximum	Mean	Maximum	Mean	Maximum	Mean	Maximum	Mean
Beef cattle	6.89	5.55	7.16	1.63	18.2	17.3	2.72	2.40
Dairy cattle	9.73	6.87	9.45	4.16	20.6 [a b]	18.8 [c]	1.83	1.26
Broiler poultry	5.0	5.0	1.39	1.16	11.8	11.8	0.59	0.59
Laying hen	5.0	5.0	2.62	1.10	11.8 [d]	11.8 [e]	2.13	2.13

[a] Highest maximum cattle dietary burden suitable for MRL estimates for mammalian tissues.

[b] Highest maximum dairy cattle dietary burden suitable for MRL estimates for milk

[c] Highest mean cattle dietary burden suitable for STMR estimates for mammalian tissues and milk

[d] Highest maximum poultry dietary burden suitable for MRL estimates for poultry tissues and eggs

[e] Highest mean poultry dietary burden suitable for STMR estimates for poultry tissues and eggs

Farm animal feeding studies

Cattle

A cattle-feeding study with thiamethoxam was reviewed by the 2010 JMPR. Animals were dosed at 2, 6, or 20 ppm in the diet. Thiamethoxam residues were as follows:

Matrix	2 ppm in feed		6 ppm in feed		20 ppm in feed	
	Maximum	Mean	Maximum	Mean	Maximum	Mean
Muscle	< 0.01 mg/kg	< 0.01 mg/kg	0.01 mg/kg	< 0.01 mg/kg	0.06 mg/kg	0.035 mg/kg
Fat	Not analysed		Not analysed		< 0.01	< 0.01
Kidney	< 0.01	< 0.01	< 0.01	< 0.01	0.04	0.0266
Liver	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
Milk	0.01	0.0073	0.05	0.036	0.19	0.124

Interpolation between the 6-ppm and 20-ppm feeding levels gives the anticipated residues in mammalian commodities shown below.

Thiamethoxam feeding study	Feed level (ppm) for milk/egg residues	Residues (mg/kg) in milk	Feed level (ppm) for tissue residues	Residues (mg/kg)			
				Muscle	Liver	Kidney	Fat
MRL beef or dairy cattle							
Feeding study	20	0.124	20	0.06	< 0.01	0.04	< 0.01
Dietary burden and high residue	20.6	0.128	20.6	0.062	< 0.01	0.041	< 0.01
STMR beef or dairy cattle							
Feeding study	6 20	0.036 0.124	6 20	< 0.01 0.035	< 0.01 < 0.01	< 0.01 0.0266	< 0.01 < 0.01
Dietary burden and residue estimate	18.8	0.096	18.8	0.033	< 0.01	0.025	< 0.01

The Meeting estimated new maximum residue levels, replacing the previous recommendations for meat, milks and edible offal as follows: Meat (from mammals other than marine mammals) at 0.07 mg/kg, Milks at 0.15 mg/kg, Edible offal (mammalian) at 0.05 mg/kg and Mammalian fats (except milk fats) at 0.01(*) mg/kg.

Poultry

A poultry-feeding study for thiamethoxam is not available. In the laying hen metabolism study reviewed by the 2010 JMPR, test animals were dosed at 98 or 112 ppm thiamethoxam in the feed.

To evaluate residues relevant for compliance with the MRL and for dietary risk assessment, the Meeting used the highest maximum and mean dietary burden for poultry (both at 11.8 ppm) and the 98-ppm dose level to estimate residues in poultry commodities. Concentrations of thiamethoxam (residue definition for compliance with the MRL) were up to 0.195 mg/kg in muscle, 0.0435 mg/kg in fat+skin, 0.0184 mg/kg in liver and 0.033 mg/kg in eggs. The resulting estimated residues are 0.023 mg/kg in muscle, 0.0052 mg/kg in fat, 0.0022 mg/kg in liver and 0.004 mg/kg in eggs.

The Meeting confirmed its previous recommendations of 0.01(*) mg/kg for poultry edible offal and eggs, estimated a new maximum residue level of 0.01(*) mg/kg for poultry fat and estimated a new maximum residue level of 0.03 mg/kg for poultry meat to replace its previous recommendation.

In the metabolism study concentrations of thiamethoxam + CGA 265307 + MU3, expressed as thiamethoxam (residue definition for dietary assessment), were up to 0.534 mg/kg in muscle, 0.276 mg/kg in fat+skin, 2.96 mg/kg in liver and 0.232 mg/kg in eggs. Estimated HRs and STMRs are 0.064 mg/kg in muscle, 0.033 mg/kg in fat, 0.36 mg/kg in liver and 0.028 mg/kg in eggs.

RECOMMENDATIONS

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

Definition of the residue for compliance with the MRL and dietary risk assessment for plant commodities: *thiamethoxam.*

Definition of the residue for compliance with the MRL and dietary risk assessment for animal commodities (except poultry): *thiamethoxam and clothianidin* (considered separately).

Definition of the residue for dietary risk assessment for poultry: *sum of thiamethoxam, CGA 265307 and MU3, expressed as thiamethoxam and clothianidin* (clothianidin considered separately).

The residue is not fat-soluble.

Table 3 Recommendations for residues of thiamethoxam from the 2021 Extra JMPR

CCN	Crop/Commodity	Recommended Maximum residue level (mg/kg)		STMR or STMR-P, mg/kg	HR or HR-P, mg/kg
		New	Previous		
GC 0640	Barley	0.5		0.112	
CF 0640	Barley bran	1.5		0.269	
	Barley hay	8 (dw)		0.2 (as)	6.7 (as)
AS 0640	Barley straw and fodder, dry	8 (dw)	2	0.53 (as)	6.7 (as)
MO 0105	Edible offal (mammalian)	0.05	0.01(*)	0.025	0.041
PE 0112	Eggs	0.01(*)	0.01(*)	0.028	0.028
MF 0100	Mammalian fats (except milk fats)	0.01(*)		0.01	0.01
MM 0095	Meat (from mammals other than marine mammals	0.07	0.02	0.033	0.062
ML 0106	Milks	0.15	0.05	0.096	
	Oat hay	8 (dw)		0.2 (as)	6.7 (as)
AS 0647	Oat straw and fodder, dry	8 (dw)		0.53 (as)	6.7 (as)
GC 0647	Oats	0.5		0.112	
FP 0307	Persimmon, Japanese	0.6		0.165	0.26
PF 0111	Poultry fats	0.01(*)		0.033	0.033
PM 0110	Poultry meat	0.03	0.01(*)	0.064	0.064
PO 0111	Poultry, edible offal of	0.01(*)	0.01(*)	0.36	0.36
GC 0649	Rice	50		17	
CM 1206	Rice bran	30		9.35	
CM 1207	Rice hulls	300		95.2	
AS 0649	Rice straw and fodder, dry	3 (dw)		0.47 (as)	1 (as)
CM 0649	Rice, husked	5		1.7	
CM 1205	Rice, polished	3		0.704	
	Rye hay	8 (dw)		0.2 (as)	6.7 (as)
AS 0651	Sorghum straw and fodder (dry)	0.8 (dw)		0.14 (as)	0.49 (as)
GC 0651	Sorghum grain	0.6		0.079	

CCN	Crop/Commodity	Recommended Maximum residue level (mg/kg)		STMR or STMR-P, mg/kg	HR or HR-P, mg/kg
		New	Previous		
GS 0658	Sorgo or sorghum, sweet	0.6		0.033	0.24
AS 0447	Sweet corn fodder	0.25 (dw)		0.0165 (as)	0.099 (as)
GC 2090	Sweet corns, Subgroup of	0.01(*)		0.01	0.01
GC 0653	Triticale	0.15		0.032	
	Triticale hay	8 (dw)		0.2 (as)	6.7 (as)
AS 0653	Triticale straw and fodder, dry	8 (dw)		0.53 (as)	6.7 (as)
GC 0654	Wheat	0.15		0.032	
CF 0654	Wheat bran	0.4		0.0768	
CF 1210	Wheat germ	0.3		0.0464	
	Wheat hay	8 (dw)		0.2 (as)	6.7 (as)
AS 0654	Wheat straw and fodder, dry	8 (dw)	2	0.53 (as)	6.7 (as)
For dietary risk assessment and/or dietary burden calculations					
	Wheat and triticale flour			0.00336	
	Wheat aspirated grain fractions			0.467	
	Barley flour			0.0118	
	Triticale flour			0.00336	
	Sorghum (grain) flour			0.0548	
	Sorghum aspirated grain fractions			0.908	
	Sorghum (sweet) syrup	--	--	0.0267	

(as) – as received; (dw) – dry weight

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for thiamethoxam is 0–0.08 mg/kg bw. The International Estimated Daily Intakes (IEDIs) for thiamethoxam were estimated for the 17 GEMS/Food Consumption Cluster Diets using the STMR or STMR-P values estimated by the JMPR. The results are shown in Annex 3 of the 2021 Extra JMPR Report.

The IEDIs ranged from 1–7% of the maximum ADI. The Meeting concluded that long-term dietary exposure to residues of thiamethoxam from uses considered by the JMPR is unlikely to present a public health concern.

Acute dietary exposure

The ARfD for thiamethoxam is 1.0 mg/kg bw. The International Estimate of Short-Term Intakes (IESTIs) for thiamethoxam were calculated for the food commodities and their processed commodities for which HRs/HR-Ps or STMRs/STMR-Ps were estimated by the present Meeting and for which consumption data were available. The results are shown in Annex 4 of the 2021 Extra JMPR Report.

The IESTIs varied from 0–5% of the ARfD for children and 0–3% of the ARfD for the general population. The Meeting concluded that acute dietary exposure to residues of thiamethoxam from uses considered by the present Meeting is unlikely to present a public health concern.

REFERENCES

Reference Number	Author(s)	Year	Study Title
CGA293343_50046	Perez, R Perez, S Almarez, O Ibrahim, A	2009	Analytical Method for the Determination of Residues of Thiamethoxam (CGA293343) and Metabolite CGA322704, in Rice and Barley by LC/MS/MS Report No. ADPEN M1104 Syngenta file No. CGA293343_50046 GLP, Unpublished
CGA293343_11576	Class, T Richter, S	2012	Thiamethoxam – Laboratory Validation of the QuEChERS Multiple Residue Method for Thiamethoxam and CGA322704 in Crops. Report No. B 2661 G Syngenta file No. CGA293343_11576 GLP, Unpublished
TK00014208	Willard, T.	2013	Thiamethoxam FS (A9765N) and Thiamethoxam WG (A9584C) - Magnitude of the Residues on Sweet Corn Report No. TK00014208 Syngenta file No. A9765N_50065 GLP, Unpublished
TK0011969	Oakes, T.	2013	Thiamethoxam (A9765N), Thiamethoxam (A9584C) and Lambda-cyhalothrin (A12871Q) – Magnitude of the Residues in or on Barley, USA 2011 Report No. TK0011969 Syngenta file No. A9765N_50056 GLP, Unpublished
TK0021631	Oakes, T.	2013	Thiamethoxam - Magnitude of the Residues in Rice Following Combined Seed Treatment and Foliar Applications Report No. TK0021631 Syngenta file No. A9765N_50082 GLP, Unpublished
TK0176048	Salzman, F.	2016	Thiamethoxam WG (A9584C) and Lambda Cyhalothrin CS (A12871Q) - Magnitude of the Residues in or on Grain Sorghum and Sweet Sorghum, USA 2013 Report No. TK0176048 Syngenta file No. A9584C_50116 GLP, Unpublished
TK0020708	Willard, T.	2013	Thiamethoxam FS (A9765N), Thiamethoxam WG (A9584C), and Lambda-cyhalothrin CS (A12871Q) – Magnitude of the Residues in or on Wheat Report No. TK0020708 Syngenta file No. A9765N_50068 GLP, Unpublished

TRIFLOXYSTROBIN (213)

First draft prepared by Ms M Le, Pest Management Regulatory Agency, Canada

EXPLANATION

Trifloxystrobin is a strobilurin broad-spectrum contact fungicide that was first evaluated for toxicology and residues by the JMPR in 2004. The 2004 JMPR established an ADI of 0–0.04 mg/kg bw and decided that an ARfD is unnecessary. Trifloxystrobin underwent subsequent evaluations by the JMPR in 2012, 2015, and 2017.

The residue definition for compliance with the MRL for plant commodities is trifloxystrobin *per se* and for compliance with the MRL for animal commodities as well as for dietary risk assessment for both plant and animal commodities is the sum of trifloxystrobin and (*E,E*)-methoxyimino-{2-[1-(3-trifluoromethyl-phenyl) ethylidene-aminooxymethyl]-phenyl}acetic acid = CGA321113) (expressed as trifloxystrobin equivalents). *The residue is fat-soluble.*

Trifloxystrobin was scheduled at the Fifty-first Session of the CCPR for the evaluation of additional uses by the 2020 JMPR, which was postponed to the 2021 Extra JMPR. The Meeting received information on analytical methods, supervised residue trials, and processing studies for citrus fruits, raspberries, currants, lettuce, legume vegetables, dry peas, tree nuts, flax, and coffee.

RESIDUE ANALYSIS

Analytical methods

Previously Reviewed Analytical Methods

Method 200177

This method was reviewed by the 2004 and 2015 JMPR and determined to be suitable for the analysis of trifloxystrobin and CGA321113 in plant materials, with an LOQ of 0.01 mg/kg for each analyte. In the current evaluation, Method 200177 was used for the analysis of trifloxystrobin and CGA321113 residues in citrus fruit and tree nut matrices in the supervised residue trials provided.

Method validation and concurrent recoveries of trifloxystrobin and CGA321113 provided to the current Meeting, from citrus fruit and tree nut matrices, were determined at fortifications ranging from 0.01 mg/kg up to at least 5 mg/kg. Average recoveries in citrus fruits ranged from 83–103% for both trifloxystrobin and CGA321113 across orange (whole fruit), orange (peeled fruit), orange (peel), lemon (whole fruit), and grapefruit (whole fruit), with relative standard deviations of up to 5.6%. Average recoveries in tree nuts ranged from 86–110% with relative standard deviations of up to 10.5% for both trifloxystrobin and CGA321113 across almond hulls, almond nutmeat, and pecan nutmeat.

The method was adequately validated at an LOQ of 0.01 mg/kg for each analyte in almonds and pecans. The method was also adequately validated at an LOQ of 0.02 mg/kg for each analyte in oranges, lemons, and grapefruits. Although method validation data for each analyte in oranges was conducted at a fortification level of 0.01 mg/kg, given the limited number of samples validated at this level in oranges (i.e. n = 2 for each analyte), the Meeting has determined that the LOQ for all citrus fruits using Method 200177 is 0.02 mg/kg for each analyte (n = 14 for each analyte in all citrus fruit samples).

Method 01013 and Method 00765

Method 01013 was reviewed by the 2012 and 2015 JMPR and determined to be suitable for the analysis of trifloxystrobin and CGA321113 in citrus fruit, pea green seed, wheat grain, rape seed, and maize green material, with an LOQ of 0.01 mg/kg for each analyte in all matrices tested. In the current evaluation, Method 01013 was used for the extraction and analysis of trifloxystrobin and CGA321113 residues in head lettuce in the supervised residue trials provided. It is noted that additional representative chromatographs for Method 01013 were provided to the Meeting (Brumhard and Stuke, 2017, Amendment No. 0004 to Study Report MR-06/138) for trifloxystrobin or CGA321113 validated in pea (green seed), rape (seed), wheat (grain) and corn/maize (green material). Control samples of rape seed and corn green material showed background levels of both trifloxystrobin and CGA 321113, however as these were observed at levels of less than 10% of the LOQ (0.01 ppm for each analyte), no corrections of recoveries are required.

Method 00765 was reviewed by the 2015 JMPR and determined to be suitable for the analysis of trifloxystrobin and CGA321113 in cucumbers, green peppers, melons, and tomatoes, with an LOQ of 0.01 mg/kg for each analyte in all matrices tested. In the current evaluation, Method 00765 was used for the extraction and analysis of trifloxystrobin and CGA321113 residues in coffee matrices in the coffee processing study provided.

In the coffee field trials provided, Method 01013 was used for the extraction and Method 00765 was used for the analysis of trifloxystrobin and CGA321113 residues in coffee beans.

Concurrent recoveries of trifloxystrobin and CGA321113 from head lettuce that were extracted and analysed with Method 01013 were determined at fortifications ranging from 0.01 mg/kg up to at least 10 mg/kg. Mean recoveries from head lettuce ranged from 85–112% for both trifloxystrobin and CGA321113, with relative standard deviations of up to 10.2%.

Method validation and concurrent recoveries of trifloxystrobin and CGA321113 from coffee matrices that were extracted and analysed with Method 00765 were determined at fortifications of 0.01 mg/kg and 0.10 mg/kg. Mean recoveries from coffee matrices ranged from 92–101% for both trifloxystrobin and CGA321113, across coffee beans, instant coffee, and roasted coffee beans, with relative standard deviations of up to 10.1%.

Concurrent recoveries of trifloxystrobin and CGA321113 from coffee beans that were extracted with Method 01013 and analysed with Method 00765 were determined at fortifications of 0.01 mg/kg and 1 mg/kg. Mean recoveries from coffee beans ranged from 98–117% for both trifloxystrobin and CGA321113, with relative standard deviations of up to 7.8%. Although mean recoveries of both trifloxystrobin and CGA321113 from coffee beans (F13-018) fortified at 1 mg/kg exceed the acceptable 70–110% range, the precision data at this level were acceptable (i.e. 3.9% for trifloxystrobin and 2.2% for CGA 321113).

Methods 01013 and 00765 were adequately validated at an LOQ of 0.01 mg/kg for each analyte in coffee matrices. Method 01013 was adequately validated at an LOQ of 0.01 mg/kg for each analyte in head lettuce.

Method 01313

This method was reviewed by the 2015 JMPR and determined to be suitable for the analysis of trifloxystrobin and CGA321113 in corn green material, bean dry seed, wheat grain, rape seed, dried hops and orange fruit, with an LOQ of 0.01 mg/kg for each analyte in all matrices tested. In the current evaluation, Method 01313 was used for the analysis of trifloxystrobin and CGA321113 residues in bean and pea matrices in the supervised residue trials provided.

Concurrent recoveries of trifloxystrobin and CGA321113 from fresh kidney beans (green material, pod, and seed) and field pea (pod and dry seed) were determined at fortifications ranging from 0.01 mg/kg up to at least 1 mg/kg. Mean recoveries in pea matrices ranged from 94–116% for both trifloxystrobin and CGA321113 across field pea pods and dry field pea seeds, with relative standard deviations of up to 10.0%. Mean recoveries in bean matrices ranged from 87–108% for both trifloxystrobin and CGA321113 across beans (green material), beans (pods), and beans (seed), with relative standard deviations of up to 9.8%. The method was validated at an LOQ of 0.01 mg/kg for both analytes in fresh kidney beans (green material, pod, and seed) and field pea (pod and dry seed). Although there were a low number of samples at each fortification level in most of the matrices (with the exception of kidney bean pods), Method 01313 has previously been validated in a variety of commodity matrices (including high water and high protein content commodities).

Analytical Methods Reviewed by the Current Meeting

Method 01313/M001

This method (Stuke and Teubner, 2013, Study Report MR-12/073) is a modification of Method 01313. Briefly, residues of trifloxystrobin and CGA321113 are extracted in acetonitrile:water (4:1, v/v). The extract is filtered, adjusted to a PH of 6–7 with ammonium acetate (1 mol/L), internal standards were added, and then the solution was brought to volume with acetonitrile:water (4:1, v/v) and analysed by HPLC-MS/MS. Three MRM (multiple-reaction monitoring) transitions were monitored for trifloxystrobin (*m/z* 409/186, *m/z* 409/145, and *m/z* 409/206) and two MRM transitions were monitored for CGA 321113 (*m/z* 395/186 and *m/z* 395/145) in each matrix tested. The 1st MRM is for quantitation while the subsequent MRMs were confirmatory. Relative to Method 01313, this method adds stable isotope labelled internal standards for all analytes and implements a shaking extraction procedure combined with a reduction of the weighed sample amount to achieve a significantly higher sample through-put.

Method 01313/M001 was used for the analysis of raspberry, currant, lettuce, pea (fresh and dry), and bean (fresh) samples.

Method validation data were provided to the Meeting for broccoli head (high water content), rape seed (high oil content), dry kidney bean seed (high protein), grapes (high acid content), and wheat grain (high starch content) at fortifications of 0.01 mg/kg and 0.10 mg/kg for both trifloxystrobin and CGA321113. Mean method validation recoveries in these matrices ranged from 82–111%, with relative standard deviations of up to 15.9%.

Concurrent recovery data for Method 01313/M001 were also provided as part of the supervised residue trials for raspberries, currants, head lettuce, peas (pod, rest of plant, green seed, dry seed, straw), and beans (green material, pod) at fortifications ranging from 0.01 mg/kg up to 15 mg/kg for each analyte. Mean recoveries in these matrices ranged from 71–112%, with relative standard deviations (RSDs) of up to 21.3%. Although a high relative standard deviation was observed for CGA 321113 in pea (rest of plant) at a fortification level of 0.01 mg/kg from one of the field pea supervised residue trial studies, recovery data for this method from other high water content crops (i.e. broccoli, lettuce, and fresh legumes from other studies) at the same fortification level demonstrated acceptable precision/repeatability with relative standard deviations ranging from 4.9–13.5%. Although there were a few individual concurrent recoveries of each analyte that fell outside the acceptable range for the fortification level, overall the concurrent recovery data for raspberries, currants, head lettuce, peas (pod, rest of plant, green seed, dry seed, straw), and beans (green material, pod) confirm the suitability of the method in these matrices.

Method 01313/M001 was adequately validated at an LOQ of 0.01 mg/kg for each analyte in a variety of plant matrices covering high water, high oil, high protein, high starch, and high acid content commodities.

Method 01207

Method 01207 is a QuEChERS multiresidue method that was validated for the analysis of trifloxystrobin and CGA321113 in apple, orange, carrot, oilseed rape, and bean (Lakaschus *et al.*, 2013, Study Report S10-00279). Briefly, residues of trifloxystrobin and CGA321113 are extracted with cysteine-hydrochloride solution (1:1, w/v) and acetonitrile:water (4:1, v/v). A salt mixture of Mg_2SO_4/NaCl/Na_3 citrate 2 H_2O/Na_2H citrate 6 H_2O) (4/1/1/0.5, w/w/w/w) was added. For oilseed rape, extracts were deep frozen and the fat was separated from the solution. The extract was centrifuged, internal standards were added, and samples were analysed by LC-MS/MS. Two MRM (multiple-reaction monitoring) transitions were monitored for trifloxystrobin (*m/z* 409/186 and *m/z* 409/145) and two MRM transitions were monitored for CGA 321113 (*m/z* 395/186 and *m/z* 395/148) in each matrix tested. The 1st MRM is for quantitation while the second MRM is confirmatory.

Method 01207 was used for the analysis of raspberry and currant samples in the supervised residue crop field trials provided.

Method validation data were provided to the Meeting for apple (high water content), oilseed rape (high oil content), dry bean (high protein), orange (high acid content), and carrot (high starch content) at fortifications of 0.01 mg/kg and 0.10 mg/kg for both trifloxystrobin and CGA321113. Mean method validation recoveries in these matrices ranged from 79–105%, with relative standard deviations of up to 13%.

Concurrent recovery data for Method 01207 were also provided as part of the supervised residue trials for raspberries and currants at fortifications of 0.01 mg/kg and 0.10 mg/kg. Mean recoveries in these matrices ranged from 85–106%, with relative standard deviations (RSDs) of up to 8.1%. The concurrent recovery data for raspberries and currants confirm the suitability of the method in these matrices.

Method 01207 was adequately validated at an LOQ of 0.01 mg/kg for each analyte in a variety of plant matrices covering high water, high oil, high protein, high starch, and high acid content commodities.

GIR/MET/TRIFLOXY/01

The meeting received description and concurrent recovery data for analytical method GIR/MET/TRIFLOXY/01 for the analysis of trifloxystrobin and CGA321113 residues in raspberries and currants as part of the supervised crop field trial studies (Perny, 2017, Study Report R B5111 and Malet and Allard, 2012, Study Report RAFR03509). Briefly, residues of trifloxystrobin and CGA321113 are extracted in acetonitrile:water (80:20, v/v) with the addition of L-cysteine chlorohydrate. After the addition of expanded perlite, samples are filtered, brought up to volume with acetonitrile:water (80:20, v/v), diluted with methanol:water (40:60, v/v), and analysed by LC-MS/MS.

Concurrent recovery data for Method GIR/MET/TRIFLOXY/01 were providedas part of the supervised residue trials for raspberries and currants at fortifications of 0.005 mg/kg and 0.05 mg/kg for each analyte. Mean recoveries in these matrices ranged from 78–106%, with relative standard deviations (RSDs) of up to 17%. Although only 3 samples were analyzed in blackcurrants, at each fortification level for each analyte, mean recoveries and relative standard deviations were acceptable and an adequate number of samples were provided for raspberries (i.e. n = 5 for each analyte) at the same fortification levels.

Method GIR/MET/TRIFLOXY/01 was adequately validated at an LOQ of 0.005 mg/kg for each analyte in raspberries and currants.

TF-003-P16-01

The meeting received description and concurrent recovery data for analytical method TF-003-P16-01 for the analysis of trifloxystrobin and CGA321113 residues in flax matrices as part of the supervised crop field trial and processing studies (Ardiel, 2017, Study Report CEJAN014 and Ardiel, 2017, Study Report CEJAN016). Briefly, residues of trifloxystrobin and CGA321113 are extracted twice in acetonitrile:water (4:1, v/v). The combined extracts were amended with a stable, isotopically labelled internal standard mixture. An aliquot was diluted and analysed by high performance liquid chromatography/triple stage mass spectrometry (LC-MS/MS).

Concurrent recovery data for Method TF-003-P16-01 were provided as part of the supervised residue trials and processing studies for flax at fortifications ranging from 0.01 mg/kg up to 0.375 mg/kg for each analyte. Mean recoveries from flax seed, meal, and oil ranged from 77–104%, with relative standard deviations (RSDs) of up to 13%. Although only 3 samples were analyzed in each flax processed commodity, at each fortification level for each analyte, mean recoveries and relative standard deviations were acceptable.

Method TF-003-P16-01 was adequately validated at an LOQ of 0.01 mg/kg for each analyte in flax seed and processed commodities.

Method 00742/E001

The meeting received description and concurrent recovery data for analytical method 00742/E001 for the analysis of trifloxystrobin and CGA321113 residues in beans with pods as part of the processing study (Nüßlein and Eberhardt, 2003, RA-3037/02). Briefly, residues of trifloxystrobin and CGA321113 are extracted in acetonitrile:water, filtered, concentrated to the aqueous remainder and the purified by liquid/liquid partitioning, and then reconstituted into cyclohexane/ethyl acetate for analysis by reversed-phase HPLC with MS/MS-detection.

Method validation and concurrent recovery data for Method 00742/E001 were provided as part of the processing study for beans with pods at fortifications ranging from 0.02 mg/kg up to 2.0 mg/kg for each analyte. Mean recoveries from beans with pods ranged from 90–96%, with relative standard deviations (RSDs) of up to 10.3%.

Method 00742/E001 was adequately validated at an LOQ of 0.02 mg/kg for each analyte in beans with pods.

Recovery data for all of the anlaytical methods used to analyse samples from the supervised residue trials for trifloxystrobin reviewed by the current Meeting are summarized below.

Table 1 Summary of method validation (MV) and concurrent recovery (CR) data for trifloxystrobin and CGA321113 from plant matrices

Commodity	Analyte	Fortification level (mg/kg)	n	Recoveries (%)	Mean Recovery (%)	RSD (%)	Reference
Method 200177							
Orange, whole fruit	Trifloxystrobin	0.01	2	MV: 99, 92	96	-	RATFY005
		0.02	8	CR:103, 98, 104, 102, 100, 104, 106, 101	102	2.5	
		0.05	3	MV: 98, 100 CR: 102	100	2.0	
		0.20	2	MV: 101, 101	101	-	

Commodity	Analyte	Fortification level (mg/kg)	n	Recoveries (%)	Mean Recovery (%)	RSD (%)	Reference
		0.50	9	CR: 104, 102, 101, 101, 108, 103, 102, 104, 102	103	2.1	
	CGA 321113	0.01	2	MV: 87, 79	83	-	
		0.02	8	CR: 92, 99, 102, 104, 98, 97, 98, 98	98	3.6	
		0.05	3	MV: 96, 99 CR: 101	99	2.6	
		0.20	2	MV: 101, 103	102	-	
		0.50	9	CR: 102, 98, 98, 102, 103, 96, 97, 104, 100	100	2.9	
Lemon, whole fruit	Trifloxystrobin	0.02	3	CR: 96, 107, 104	102	5.6	
		0.50	3	CR: 100, 103, 107	103	3.4	
	CGA 321113	0.02	3	CR: 93, 101, 100	98	4.5	
		0.50	3	CR: 100, 99, 104	101	2.6	
Grapefruit, whole fruit	Trifloxystrobin	0.02	3	CR: 101, 104, 105	103	2.0	
		0.50	3	CR: 100, 102, 100	101	1.2	
	CGA 321113	0.02	3	CR: 100, 101, 101	101	0.6	
		0.50	3	CR: 100, 98, 97	98	1.6	
Orange, peeled fruit	Trifloxystrobin	0.01	1	CR: 101	-	-	
		0.05	1	CR: 98	-	-	
	CGA 321113	0.01	1	CR: 73	-	-	
		0.05	1	CR: 92	-	-	
Orange, peel	Trifloxystrobin	0.05	1	CR: 97	-	-	
		1.0	1	CR: 96	-	-	
	CGA 321113	0.05	1	CR: 98	-	-	
		1.0	1	CR: 98	-	-	
Almond hulls	Trifloxystrobin	0.01	10	CR: 106, 105, 92, 104, 101, 108, 101, 96, 99, 103	102	4.8	RAGMP168
		5.0	3	MV: 101, 105, 99	102	3.0	
	CGA 321113	0.01	10	CR: 106, 96, 96, 111, 105, 105, 105, 102, 97, 99	102	4.9	
		5.0	3	MV: 99, 102, 99	100	1.7	
Almond, nutmeat	Trifloxystrobin	0.01	8	MV: 92, 91, 87 CR: 100, 98, 97, 106, 103	97	6.6	
		0.05	3	MV: 100, 87, 82	90	10.4	
	CGA 321113	0.01	8	MV: 87, 85, 89 CR: 101, 99, 97, 101, 97	95	6.9	
		0.05	3	MV: 89, 84, 92	88	4.6	
Pecan, nutmeat	Trifloxystrobin	0.01	10	CR: 107, 100, 98, 99, 116, 104, 107, 125, 110, 95	106	8.6	RATFN132
		0.1	3	CR: 108, 112, 111	110	1.9	
	CGA 321113	0.01	10	CR: 78, 73, 95, 90, 93, 76, 87, 84, 100, 79	86	10.5	
		0.1	3	CR: 108, 108, 107	108	0.5	

Commodity	Analyte	Fortification level (mg/kg)	n	Recoveries (%)	Mean Recovery (%)	RSD (%)	Reference
Method 01013 (extraction and analysis)							
Lettuce, head	Trifloxystrobin	0.01	3	CR: 108, 111, 117	112	4.1	RA-2620/07
		0.10	3	CR: 101, 101, 99	100	1.2	
		1.0	3	CR: 93, 89, 86	89	3.9	
		10	3	CR: 80, 95, 80	85	10.2	
	CGA 321113	0.01	3	CR: 106, 94, 102	101	6.1	
		0.10	3	CR: 97, 90, 103	97	6.7	
		1.0	3	CR: 100, 98, 98	99	1.2	
		10	3	CR: 82, 91, 84	86	5.5	
Method 01013 (extraction) and Method 00765 (analysis)							
Coffee bean	Trifloxystrobin	0.01	5	CR: 113, 110, 93, 87, 86	98	13	F12-026
		1.0	5	CR: 101, 102, 101, 104, 110	104	3.7	
	CGA 321113	0.01	6	CR: 107, 109, 105, 101, 100, 96	103	4.7	
		1.0	6	CR: 93, 104, 105, 104, 105, 87	100	7.8	
Coffee bean	Trifloxystrobin	0.01	3	CR: 111, 120, 114	115	4.0	F13-018
		1.0	3	CR: 116, 111, 120	116	3.9	
	CGA 321113	0.01	3	CR: 108, 114, 105	109	4.2	
		1.0	3	CR: 116, 120, 115	117	2.2	
Method 00765 (extraction and analysis)							
Coffee bean	Trifloxystrobin	0.01	3	MV: 96, 81 CR: 98	92	10.1	RATFL003
		0.1	3	MV: 92, 95 CR: 105	97	7.0	
	CGA 321113	0.01	3	MV: 98, 95 CR: 82	92	9.3	
		0.10	3	MV: 103, 98 CR: 86	96	9.1	
Coffee instant	Trifloxystrobin	0.01	2	CR: 96, 95	96	-	
		0.10	2	CR: 84, 99	92	-	
	CGA 321113	0.01	2	CR: 99, 93	96	-	
		0.10	2	CR: 91, 111	101	-	
Coffee bean roasted	Trifloxystrobin	0.01	2	CR: 94, 100	97	-	
		0.10	2	CR: 98, 103	101	-	
	CGA 321113	0.01	2	CR: 98, 108	103	-	
		0.10	2	CR: 94, 103	99	-	
Method 01313							
Pea, field (pod)	Trifloxystrobin	0.01	4	CR: 98, 99, 105, 116	105	7.9	11-2000
		0.10	3	CR: 98, 102, 104	101	3.0	
		1.0	1	CR: 88	-	-	
	CGA 321113	0.01	4	CR: 94, 95, 100, 102	98	4.0	
		0.10	3	CR: 88, 96, 99	94	6.0	
		1.0	1	CR: 91	-	-	

Commodity	Analyte	Fortification level (mg/kg)	n	Recoveries (%)	Mean Recovery (%)	RSD (%)	Reference
Pea, field (dry seed)	Trifloxystrobin	0.01	4	CR: 114, 115, 117, 119	116	1.9	
		0.10	3	CR: 105, 106, 107	106	0.9	
		1.0	1	CR: 105	-	-	
	CGA 321113	0.01	4	CR: 97, 111, 112, 124	111	10.0	
		0.10	3	CR: 97, 100, 102	100	2.5	
		1.0	1	CR: 100	-	-	
Kidney bean (green material)	Trifloxystrobin	0.01	2	CR: 95, 96	96	-	10-2125
		1.0	2	CR: 97, 99	98	-	
	CGA 321113	0.01	1	CR: 99	-	-	
		1.0	1	CR: 90	-	-	
Kidney bean (pod)	Trifloxystrobin	0.01	3	CR: 83, 93, 97	91	7.9	
		0.10	1	CR: 71	-	-	
		1.0	1	CR: 106	-	-	
		10	1	CR: 94	-	-	
	CGA 321113	0.01	2	CR: 85, 89	87	-	
		0.10	1	CR: 94	-		
		1.0	1	CR: 89	-	-	
Kidney bean (pod)	Trifloxystrobin	0.01	1	CR: 90	-	-	10-2128
		0.10	1	CR: 94	-	-	
	CGA 321113	0.01	1	CR: 96	-	-	
		0.10	1	CR: 92	-	-	
Kidney bean (green seed)	Trifloxystrobin	0.01	1	CR: 88	-	-	
		0.10	1	CR: 98	-	-	
	CGA 321113	0.01	1	CR: 99	-	-	
		0.10	1	CR: 92	-	-	
Kidney bean (pod)	Trifloxystrobin	0.01	4	CR: 105, 108, 109, 111	108	2.3	11-2001
		0.10	4	CR: 94, 103, 106, 106	102	5.6	
		0.80	1	CR: 98	-	-	
		2.5	1	CR: 102	-	-	
		8.0	1	CR: 107	-	-	
	CGA 321113	0.01	4	CR: 95, 96, 109, 116	104	9.8	
		0.10	4	CR: 94, 99, 102, 107	101	5.4	
		0.80	1	CR: 96	-	-	
		2.5	1	CR: 94	-	-	
		8.0	1	CR: 97	-	-	
Method 01313/M001							
Broccoli (head)	Trifloxystrobin (*m/z* 409/186)	0.01	5	MV: 86, 98, 96, 99, 96	95	5.5	MR-12/073 [a]
		0.10	5	MV: 91, 98, 95, 87, 84	91	6.3	
	Trifloxystrobin (*m/z* 409/145)	0.01	5	MV: 89, 100, 94, 87, 100	94	6.4	
		0.10	5	MV: 100, 98, 97, 96, 91	96	3.5	
	Trifloxystrobin (*m/z* 409/206)	0.01	5	MV: 100, 110, 93, 110, 94	101	8.2	
		0.10	5	MV: 95, 97, 100, 97, 89	96	4.3	

Commodity	Analyte	Fortification level (mg/kg)	n	Recoveries (%)	Mean Recovery (%)	RSD (%)	Reference
	CGA 321113	0.01	5	MV: 90, 99, 93, 92,81	91	7.2	
	(*m/z* 395/186)	0.10	5	MV: 88, 91, 85, 86, 96	89	5.0	
	CGA 321113	0.01	5	MV: 92, 94, 84, 73, 69	82	13.5	
	(*m/z* 395/145)	0.10	5	MV: 87, 82, 83, 84, 88	85	3.1	
Rape (seed)	Trifloxystrobin	0.01	5	MV: 100, 94, 91, 100, 90	95	5.0	
	(*m/z* 409/186)	0.10	5	MV: 87, 93, 100, 93, 88	92	5.6	
	Trifloxystrobin	0.01	5	MV: 95, 85, 88, 91, 100	92	6.4	
	(*m/z* 409/145)	0.10	5	MV: 86, 94,97, 82, 83	88	7.6	
	Trifloxystrobin	0.01	5	MV: 87, 96, 85, 110, 100	96	10.6	
	(*m/z* 409/206)	0.10	5	MV: 89, 96, 91, 86, 90	90	4	
	CGA 321113	0.01	5	MV: 91, 97, 77, 78, 86	86	9.9	
	(*m/z* 395/186)	0.10	5	MV: 92, 79, 84, 91, 89	87	6.2	
	CGA 321113	0.01	5	MV: 98, 110, 100, 100, 100	102	4.7	
	(*m/z* 395/145)	0.10	5	MV: 91, 86, 86, 89, 91	89	2.8	
Kidney bean (dry seed)	Trifloxystrobin	0.01	5	MV: 91, 110, 100, 90, 110	100	9.7	
	(*m/z* 409/186)	0.10	5	MV: 100, 99, 94, 110, 96	98	2.5	
	Trifloxystrobin	0.01	5	MV: 110, 99, 98, 100, 99	101	4.9	
	(*m/z* 409/145)	0.10	5	MV: 100, 99, 100, 88, 98	97	5.3	
	Trifloxystrobin	0.01	5	MV: 100, 100, 100, 92, 93	97	4.3	
	(*m/z* 409/206)	0.10	5	MV: 100, 100, 110, 94, 90	99	7.7	
	CGA 321113	0.01	5	MV: 91, 86, 84, 100, 97	92	7.5	
	(*m/z* 395/186)	0.10	5	MV: 92, 93, 91, 91, 95	92	1.8	
	CGA 321113	0.01	5	MV: 100, 75, 110, 80, 100	93	15.9	
	(*m/z* 395/145)	0.10	5	MV: 94, 90, 94, 100, 84	92	6.4	
Grape (bunches of grapes)	Trifloxystrobin	0.01	5	MV: 130, 100, 100, 110, 110	110	11.1	
	(*m/z* 409/186)	0.10	5	MV: 100, 99, 94, 110, 96	100	6.2	
	Trifloxystrobin	0.01	5	MV: 97, 120, 100, 120, 120	111	10.6	
	(*m/z* 409/145)	0.10	5	MV: 110, 100, 98, 100, 110	104	5.7	
	Trifloxystrobin	0.01	5	MV: 120, 110, 91, 110, 110	108	9.7	
	(*m/z* 409/206)	0.10	5	MV: 110, 110, 110, 97, 100	105	6.1	
	CGA 321113	0.01	5	MV: 110, 110, 85, 120, 120	109	13.1	
	(*m/z* 395/186)	0.10	5	MV: 99, 98, 100, 100, 100	99	0.9	
	CGA 321113	0.01	5	MV: 81, 90, 76, 92, 87	85	7.8	
	(*m/z* 395/145)	0.10	5	MV: 89, 91, 96, 92, 90	92	2.9	
Wheat (grain)	Trifloxystrobin	0.01	5	MV: 100, 79, 87, 82, 85	87	9.3	
	(*m/z* 409/186)	0.10	5	MV: 96, 96, 95, 100, 99	97	2.2	
	Trifloxystrobin	0.01	5	MV: 100, 110, 100, 100, 110	104	5.3	
	(*m/z* 409/145)	0.10	5	MV: 92, 110, 98, 98,94	98	7.1	
	Trifloxystrobin	0.01	5	MV: 100, 120, 110, 87, 98	103	12.2	
	(*m/z* 409/206)	0.10	5	MV: 95, 100, 99, 98, 84	95	6.9	
	CGA 321113	0.01	5	MV: 91, 78, 110, 92, 97	94	12.3	
	(*m/z* 395/186)	0.10	5	MV: 86, 93, 87, 94, 88	90	4.1	

Commodity	Analyte	Fortification level (mg/kg)	n	Recoveries (%)	Mean Recovery (%)	RSD (%)	Reference
	CGA 321113 (*m/z* 395/145)	0.01	5	MV: 96, 120, 110, 100, 110	107	8.8	
		0.10	5	MV: 90, 91, 96, 98, 79	91	8.1	
Red currants	Trifloxystrobin	0.01	4	CR: 92, 93, 97, 104	97	5.6	15-2032 15-2033
		0.10	5	CR: 93, 96, 97, 98, 98	96	2.2	
		1.0	2	CR: 95, 101	98	-	
	CGA 321113	0.01	4	CR: 89, 90, 90, 103	93	7.2	
		0.10	5	CR: 92, 93, 93, 95, 96	94	1.8	
		1.0	2	CR: 97, 101	99	-	
Red currants	Trifloxystrobin	0.01	3	CR: 91, 91, 92	91	0.6	14-2025
		0.10	3	CR: 92, 93, 95	93	1.6	
		1.0	1	CR: 97	-	-	
		2.0	1	CR: 98	-	-	
	CGA 321113	0.01	3	CR: 85, 90, 93	89	4.5	
		0.10	3	CR: 89, 92, 94	92	2.7	
		1.0	1	CR: 95	-	-	
		2.0	1	CR: 94	-	-	
Raspberry	Trifloxystrobin	0.01	3	CR: 94, 94, 100	96	3.6	15-2034
		0.10	3	CR: 94, 95, 95	95	0.6	
		1.0	3	CR: 97, 97, 98	97	0.6	
	CGA 321113	0.01	3	CR: 91, 106, 110	102	9.8	
		0.10	3	CR: 90, 90, 103	94	8.0	
		1.0	3	CR: 96, 97, 102	98	3.3	
Raspberry	Trifloxystrobin	0.01	3	CR: 100, 101, 101	101	0.6	18-2051
		0.50	4	CR: 100, 100, 102, 102	101	1.1	
		5.0	6	CR: 94, 96, 97, 98, 99, 102	98	2.8	
	CGA 321113	0.01	3	CR: 93, 104, 108	102	7.6	
		0.50	4	CR: 96, 100, 101, 102	100	2.6	
		5.0	6	CR: 87, 91, 91, 98, 101, 103	95	6.7	
Lettuce, head	Trifloxystrobin	0.01	10	CR: 76, 83, 89, 93, 96, 98, 101, 102, 102, 108	95	10.3	14-2028
		0.10	6	CR: 93, 93, 95, 96, 99, 99	96	2.8	
		1.0	5	CR: 94, 98, 98, 99, 100	98	2.3	
		15	1	CR: 91	-	-	
	CGA 321113	0.01	10	CR: 83, 89, 89, 99, 99, 104, 106, 107, 107, 111	99	9.5	
		0.10	6	CR: 89, 92, 93, 93, 100, 102	95	5.3	
		1.0	5	CR: 93, 94, 95, 96, 97	95	1.7	
		15	1	CR: 87	-	-	
Lettuce, head	Trifloxystrobin	0.01	3	CR: 92, 94, 99	95	3.8	18-2048
		0.50	3	CR: 93, 97, 98	96	2.8	
		10	3	CR: 108, 118, 120	115	5.6	
	CGA 321113	0.01	3	CR: 93, 96, 107	99	7.5	
		0.50	3	CR: 96, 102, 103	100	3.8	

Commodity	Analyte	Fortification level (mg/kg)	n	Recoveries (%)	Mean Recovery (%)	RSD (%)	Reference
Pea (pod)	Trifloxystrobin	0.01	3	CR: 72, 74, 75	74	2.1	12-2031
		0.10	3	CR: 82, 84, 85	84	1.8	
	CGA 321113	0.01	3	CR: 71, 73, 78	74	4.9	
		0.10	3	CR: 78, 78, 79	78	0.7	
Pea (rest of plant)	Trifloxystrobin	0.01	9	CR: 73, 81, 83, 84, 86, 90, 98, 100, 101	88	10.8	
		0.10	3	CR: 73, 79, 81	78	5.4	
		0.50	1	CR: 76	-	-	
		5.0	1	CR: 96	-	-	
	CGA 321113	0.01	6	CR: 65, 71, 73, 99, 100, 108	86	21.3	
		0.10	3	CR: 75, 76, 77	76	1.3	
		0.50	1	CR: 70	-		
Pea (dry seed)	Trifloxystrobin	0.01	8	CR: 70, 70, 77, 80, 82, 82, 87, 104	82	13.3	
		0.10	3	CR: 78, 82, 85	82	4.3	
		0.50	1	CR: 80	-	-	
	CGA 321113	0.01	6	CR: 66, 66, 68, 92, 92, 98	80	18.9	
		0.10	3	CR: 69, 71, 84	75	10.9	
		0.50	1	CR: 68	-	-	
Pea (green seed)	Trifloxystrobin	0.01	6	CR: 74, 78, 92, 99, 108, 109	93	15.9	
		0.10	3	CR: 71, 73, 77	74	4.1	
		1.0	1	CR: 79	-	-	
	CGA 321113	0.01	3	CR: 98, 98, 107	101	5.1	
		0.10	3	CR: 76, 77, 83	79	4.8	
		1.0	1	CR: 82	-	-	
Pea (straw)	Trifloxystrobin	0.01	3	CR: 96, 105, 114	105	8.6	
		0.10	3	CR: 100, 100, 104	101	2.3	
		1.0	1	CR: 102	-	-	
		10	1	CR: 100	-	-	
	CGA 321113	0.01	3	CR: 102, 104, 109	105	3.4	
		0.10	3	CR: 88, 93, 100	94	6.4	
		1.0	1	CR: 109	-	-	
		10	1	CR: 111	-	-	
Pea (pod)	Trifloxystrobin	0.01	2	CR: 109, 115	112	-	12-2155
		0.10	1	CR: 104	-	-	
		0.50	1	CR: 82	-	-	
	CGA 321113	0.01	2	CR: 72, 83	78	-	
		0.10	1	CR: 94	-	-	
		0.50	1	CR: 73	-	-	
Pea (rest of plant)	Trifloxystrobin	0.01	2	CR: 103, 103	103	-	
		0.10	1	CR: 103	-	-	
		0.50	1	CR: 86	-	-	
	CGA 321113	0.01	2	CR: 82, 85	84	-	

Commodity	Analyte	Fortification level (mg/kg)	n	Recoveries (%)	Mean Recovery (%)	RSD (%)	Reference
		0.10	1	CR: 94	-	-	
		0.50	1	CR: 82	-	-	
Pea (dry seed)	Trifloxystrobin	0.01	1	CR: 76	-	-	
		0.10	1	CR: 87	-	-	
		0.50	1	CR: 79	-	-	
	CGA 321113	0.01	1	CR: 79	-	-	
		0.10	1	CR: 92	-	-	
		0.50	1	CR: 81	-	-	
Pea (green seed)	Trifloxystrobin	0.01	2	CR: 91, 96	94	-	
		0.10	1	CR: 94	-	-	
		0.50	1	CR: 84	-	-	
	CGA 321113	0.01	2	CR: 75, 75	75	-	
		0.10	1	CR: 93	-	-	
		0.50	1	CR: 82	-	-	
Pea (straw)	Trifloxystrobin	0.01	1	CR: 101	-	-	
		0.10	1	CR: 91	-	-	
		0.50	1	CR: 92	-	-	
		10	1	CR: 86	-	-	
	CGA 321113	0.01	1	CR: 79	-	-	
		0.10	1	CR: 89	-	-	
		0.50	1	CR: 89	-	-	
Pea (pod)	Trifloxystrobin	0.01	3	CR: 93, 100, 105	99	6.1	
		0.10	3	CR: 106, 106, 111	108	2.7	
		1.0	1	CR: 107	-	-	
	CGA 321113	0.01	3	CR: 85, 88, 100	91	8.7	
		0.10	3	CR: 98, 104, 109	104	5.3	
		1.0	1	CR: 102	-	-	
Pea (green material)	Trifloxystrobin	0.01	5	CR: 95, 100, 100, 100, 106	100	3.9	
		0.10	5	CR: 98, 99, 99, 101, 104	100	2.4	
		5.0	1	CR: 84	-	-	
		15	1	CR: 102	-	-	
	CGA 321113	0.01	5	CR: 88, 89, 97, 108, 114	99	11.6	15-2030
		0.10	5	CR: 95, 99, 101, 103, 106	101	4.1	
		5.0	1	CR: 86	-		
Pea (dry seed)	Trifloxystrobin	0.01	3	CR: 96, 102, 103	100	3.8	
		0.10	3	CR: 101, 103, 103	102	1.1	
		5.0	1	CR: 93	-	-	
	CGA 321113	0.01	3	CR: 104, 104, 112	107	4.3	
		0.10	3	CR: 101, 103, 107	104	3.5	
		5.0	1	CR: 96	-	-	
Pea (green seed)	Trifloxystrobin	0.01	3	CR: 104, 110, 110	108	3.2	
		0.10	3	CR: 103, 104, 105	104	1.0	

Commodity	Analyte	Fortification level (mg/kg)	n	Recoveries (%)	Mean Recovery (%)	RSD (%)	Reference
		5.0	1	CR: 84	-	-	
	CGA 321113	0.01	3	CR: 87, 102, 103	97	9.2	
		0.10	3	CR: 98, 101, 107	102	4.5	
		5.0	1	CR: 88	-	-	
Pea (pod)	Trifloxystrobin	0.01	1	CR: 89	-	-	
		0.1	1	CR: 101	-	-	
		1.0	1	CR: 77	-	-	
	CGA 321113	0.01	1	CR: 79	-	-	
		0.10	1	CR: 94	-	-	
		1.0	1	CR: 76	-	-	
Pea (rest of plant)	Trifloxystrobin	0.01	2	CR: 105, 109	107	-	
		0.10	1	CR: 102	-	-	
		1.0	1	CR: 81	-	-	
		5.0	1	CR: 90	-	-	
		15	1	CR: 77	-	-	
	CGA 321113	0.01	2	CR: 86, 94	90	-	
		0.10	1	CR: 106	-	-	
		1.0	1	CR: 79	-	-	
Pea (dry seed)	Trifloxystrobin	0.01	1	CR: 111	-	-	
		0.10	1	CR: 97	-	-	
		1.0	1	CR: 93	-	-	12-2032
	CGA 321113	0.01	1	CR: 91	-	-	
		0.10	1	CR: 105	-	-	
		1.0	1	CR: 90	-	-	
Pea (green seed)	Trifloxystrobin	0.01	2	CR: 101, 106	104	-	
		0.10	1	CR: 106	-	-	
		1.0	1	CR: 99	-	-	
	CGA 321113	0.01	2	CR: 92, 93	93	-	
		0.10	1	CR: 101	-	-	
		1.0	1	CR: 96	-	-	
Pea (straw)	Trifloxystrobin	0.01	1	CR: 110	-	-	
		0.10	1	CR: 102	-	-	
		0.50	1	CR: 115	-	-	
		10	1	CR: 102	-	-	
	CGA 321113	0.01	2	CR: 96, 100	98	-	
		0.10	1	CR: 103	-	-	
		0.50	1	CR: 114	-	-	
		10	1	CR: 100	-	-	
Bean (green material)	Trifloxystrobin	0.01	3	CR: 69, 83, 86	79	11.4	12-2030
		0.10	3	CR: 73, 75, 81	76	5.5	
		0.20	1	CR: 80	-	-	
		1.0	1	CR: 78	-	-	

Commodity	Analyte	Fortification level (mg/kg)	n	Recoveries (%)	Mean Recovery (%)	RSD (%)	Reference
		10	1	CR: 88	-	-	
	CGA 321113	0.01	3	CR: 73, 81, 81	78	5.9	
		0.10	3	CR: 72, 75, 80	76	5.3	
		0.20	1	CR: 71	-	-	
Bean (pod)	Trifloxystrobin	0.01	3	CR: 77, 83, 89	83	7.2	
		0.10	3	CR: 82, 89, 91	87	5.4	
		0.50	1	CR: 67	-	-	
	CGA 321113	0.01	3	CR: 67, 72, 75	71	5.7	
		0.10	3	CR: 71, 76, 77	75	4.3	
		0.50	1	71	-	-	
Method 01207							
Apple	Trifloxystrobin (*m/z* 409→186)	0.01	5	MV: 88, 85, 80, 86, 86	85	3.5	
		0.10	5	MV: 77, 87, 84, 81, 78	81	5.1	
	Trifloxystrobin (*m/z* 409→145)	0.01	5	MV: 81, 90, 77, 84, 83	83	5.7	
		0.10	5	MV: 77, 84, 84, 78, 79	80	4.2	
	CGA 321113 (*m/z* 395→186)	0.01	5	MV: 97, 96, 100, 105, 96	99	3.9	
		0.10	5	MV: 98, 104, 105, 101, 96	101	3.8	
	CGA 321113 (*m/z* 395→148)	0.01	5	MV: 106, 99, 82, 96, 93	95	9.3	
		0.10	5	MV: 99, 103, 102, 95, 85	97	7.5	
Orange	Trifloxystrobin (*m/z* 409→186)	0.01	5	MV: 83, 91, 74, 86, 79	83	7.9	
		0.10	5	MV: 85, 83, 86, 76, 81	82	4.8	
	Trifloxystrobin (*m/z* 409→145)	0.01	5	MV: 87, 90, 88, 98, 92	91	4.8	
		0.10	5	MV: 84, 82, 89, 76, 86	83	5.8	
	CGA 321113 (*m/z* 395→186)	0.01	5	MV: 100, 104, 101, 101, 101	101	1.5	
		0.10	5	MV: 103, 99, 103, 86, 107	100	8.1	
	CGA 321113 (*m/z* 395→148)	0.01	5	MV: 93, 114, 111, 105, 103	105	7.7	
		0.10	5	MV: 105, 97, 95, 88, 96	96	6.3	S10-00279 [b]
Carrot	Trifloxystrobin (*m/z* 409→186)	0.01	5	MV: 84, 68, 90, 90, 99	86	13	
		0.10	5	MV: 82, 85, 78, 83, 82	82	3.1	
	Trifloxystrobin (*m/z* 409→145)	0.01	5	MV: 86, 72, 85, 97, 84	85	10	
		0.10	5	MV: 84, 80, 79, 82, 81	81	2.4	
	CGA 321113 (*m/z* 395→186)	0.01	5	MV: 105, 86, 105, 115, 108	104	10	
		0.10	5	MV: 103, 102, 101, 110, 105	104	3.4	
	CGA 321113 (*m/z* 395→148)	0.01	5	MV: 111, 80, 103, 105, 97	99	12	
		0.10	5	MV: 90, 100, 91, 85, 89	91	6.1	
Dry bean	Trifloxystrobin (*m/z* 409→186)	0.01	5	MV: 89, 88, 83, 84, 83	85	3.4	
		0.10	5	MV: 81, 78, 80, 78, 79	79	1.6	
	Trifloxystrobin (*m/z* 409→145)	0.01	5	MV: 93, 90, 91, 88, 87	90	2.7	
		0.10	5	MV: 87, 77, 81, 80, 76	80	5.4	
	CGA 321113 (*m/z* 395→186)	0.01	5	MV: 88, 87, 87, 90, 88	88	1.4	
		0.10	5	MV: 86, 80, 83, 85, 83	83	2.8	
		0.01	5	MV: 93, 77, 87, 80, 80	83	7.8	

Commodity	Analyte	Fortification level (mg/kg)	n	Recoveries (%)	Mean Recovery (%)	RSD (%)	Reference
	CGA 321113 (*m/z* 395→148)	0.10	5	MV: 83, 78, 81, 85, 79	81	3.5	
Oilseed rape	Trifloxystrobin (*m/z* 409→186)	0.01	5	MV: 86, 83, 87, 89, 83	86	3.0	
		0.10	5	MV: 83, 80, 82, 83, 84	82	1.8	
	Trifloxystrobin (*m/z* 409→145)	0.01	5	MV: 92, 85, 80, 85, 81	85	5.6	
		0.10	5	MV: 79, 81, 80, 82, 81	81	1.4	
	CGA 321113 (*m/z* 395→186)	0.01	5	MV: 84, 85, 88, 87, 88	86	2.1	
		0.10	5	MV: 90, 87, 89, 87, 90	89	1.7	
	CGA 321113 (*m/z* 395→148)	0.01	5	MV: 83, 80, 88, 97, 81	86	8.1	
		0.10	5	MV: 83, 89, 86, 88, 89	87	2.9	
Red currant	Trifloxystrobin (*m/z* 409→186)	0.01	3	CR: 99, 104, 100	101	2.6	BCS-G402-11
		0.10	3	CR: 88, 92, 93	91	2.9	
	Trifloxystrobin (*m/z* 409→145)	0.01	3	CR: 98, 102, 98	99	2.3	
		0.10	3	CR: 90, 90, 92	91	1.3	
	CGA 321113 (*m/z* 395→186)	0.01	3	CR: 95, 90, 88	91	4.0	
		0.10	3	CR: 88, 86, 88	87	1.3	
	CGA 321113 (*m/z* 395→148)	0.01	3	CR: 82, 91, 93	89	6.6	
		0.10	3	CR: 89, 88, 93	90	2.9	
Red currant	Trifloxystrobin	0.01	2	CR: 99, 99	99	-	PTZ-NLI-11796
		0.10	1	CR: 95	-	-	
		1.0	1	CR: 76	-	-	
	CGA 321113	0.01	1	CR: 88	-	-	
		0.10	1	CR: 97	-	-	
Raspberry	Trifloxystrobin (*m/z* 409→186)	0.01	3	CR: 103, 107, 107	106	2.2	BCS-G401-11
		0.10	3	CR: 88, 88, 91	89	1.9	
	Trifloxystrobin (*m/z* 409→145)	0.01	3	CR: 89, 104, 101	98	8.1	
		0.10	3	CR: 85, 84, 89	86	3.1	
	CGA 321113 (*m/z* 395→186)	0.01	3	CR: 91, 99, 94	95	4.3	
		0.10	3	CR: 85, 86, 88	86	1.8	
	CGA 321113 (*m/z* 395→148)	0.01	3	CR: 94, 93, 103	97	5.7	
		0.10	3	CR: 82, 84, 88	85	3.6	
Raspberry	Trifloxystrobin	0.01	2	CR: 98, 102	100	-	PTZ-NLI-11797
		0.10	1	CR: 97	-	-	
		1.5	1	CR: 80	-	-	
	CGA 321113	0.01	1	CR: 90	-	-	
		0.10	1	CR: 91	-	-	
Method GIR/MET/TRIFLOXY/01							
Blackcurrant	Trifloxystrobin	0.005	3	CR: 111, 107, 102	106	4	R B5111
		0.05	3	CR: 101, 106, 102	103	2	
	CGA 321113	0.005	3	CR: 105, 86, 106	99	11	

Commodity	Analyte	Fortification level (mg/kg)	n	Recoveries (%)	Mean Recovery (%)	RSD (%)	Reference
		0.05	3	CR: 91, 87, 98	92	6	
Raspberry	Trifloxystrobin	0.005	5	CR: 98, 100, 86, 77, 88	89	11	RAFR03509
		0.05	5	CR: 86, 105, 75, 73, 71	82	17	
	CGA 321113	0.005	5	CR: 88, 88, 79, 80, 78	82	6	
		0.05	5	CR: 82, 92, 74, 72, 68	78	12	
Method TF-003-P16-01							
Flax seed	Trifloxystrobin	0.01	9	CR: 78, 78, 82, 91, 81, 93, 88, 82, 92	85	7	CEJAN014
		0.35	3	CR: 96, 94, 93	94	2	
	CGA 321113	0.01	9	CR: 83, 90, 96, 94, 94, 95, 100, 97, 94	94	5	
		0.35	3	CR: 89, 94, 97	93	4	
Flax seed	Trifloxystrobin	0.01	3	CR: 92, 89, 100	94	6	CEJAN016
		0.25	3	CR: 100, 103, 99	101	2	
	CGA 321113	0.01	3	CR: 66, 84, 82	77	13	
		0.25	3	CR: 94, 96, 94	95	1	
Flax meal	Trifloxystrobin	0.01	3	CR: 86, 94, 78	86	9	
		0.25	3	CR: 99, 97, 104	100	4	
	CGA 321113	0.01	3	CR: 86, 83, 87	85	2	
		0.25	3	CR: 93, 94, 87	91	4	
Flax, oil cold pressed	Trifloxystrobin	0.01	3	CR: 97, 87, 95	93	6	
		0.25	3	CR: 94, 93, 95	94	1	
		0.375	3	CR: 87, 88, 96	90	5	
	CGA 321113	0.01	3	CR: 87, 104, 105	99	10	
		0.25	3	CR: 91, 95, 94	93	2	
		0.375	3	CR: 104, 104, 100	103	2	
Flax, oil solvent extracted	Trifloxystrobin	0.01	3	CR: 97, 98, 97	97	1	
		0.25	3	CR: 108, 96, 107	104	6	
	CGA 321113	0.01	3	CR: 89, 88, 81	86	5	
		0.25	3	CR: 104, 103, 97	101	4	
Method 00742/E001							
Bean with pod	Trifloxystrobin	0.02	4	92, 92, 94, 104	96	6.0	RA-3037/ 02 [c]
		0.2	13	91, 91, 93, 90, 92, 106, 93, 94, 96, 88, 94, 90, 84	92	5.5	
		2.0	3	79, 95, 95	90	10.3	
	CGA 321113	0.02	4	92, 94, 93, 96	94	1.8	
		0.2	13	88, 90, 91, 90, 92, 92, 95, 91, 91, 86, 91, 87, 82	90	3.7	
		2.0	3	81, 96, 96	91	9.5	

n = number of -replicates; RSD = relative standard deviation

[a] In this study three MRM (multiple-reaction monitoring) transitions were monitored for trifloxystrobin and two MRM transitions were monitored for CGA 321113 in each matrix tested. The 1st MRM is for quantitation while the subsequent MRMs were confirmatory.

[b] In this study two MRM transitions were monitored for both trifloxystrobin and CGA 321113 in each matrix tested. The 1st MRM is for quantitation while the second MRM was confirmatory.

[c] The study report indicated that these recovery data were generated both prior to and during sample analysis however the recoveries were summarized together in the report with no distinction between the different types of recoveries.

Stability of residues in stored analytical samples

The stability of residues in samples on frozen storage was evaluated by the 2004 JMPR for a range of commodities. Residues of trifloxystrobin and CGA 321113 were stable under freezer storage conditions for at least 24 months in the case of grapefruit, cucumber, potato and wheat commodities (grain, straw and whole plant) or 18 months for apple (fruit, wet pomace), peanut (nutmeat, oil) and grape juice. The stability data covered commodities that are representative of high water content (apple, cucumber, grape juice), high acid content (grapefruit), high oil content (peanut) and high starch content (potato, wheat) and can be extrapolated to the commodities considered at the current Meeting. Although storage stability data are not available for any high protein content commodity, the 2004 JMPR concluded that no significant decrease of residues of trifloxystrobin or CGA 321113 was observed after the test period of 18 or 24 months. In addition, stability of trifloxystrobin and CGA 321113 was observed in wheat straw (i.e. a commodity that does not fall into any of the 5 OECD commodity categories) for at least 24 months. As such, the existing stability data for trifloxystrobin and CGA 321113 can be extrapolated to high protein content commodities.

Maximum storage to analysis intervals for samples in the supervised field trials provided were: 16.8 months for citrus, 9.0 months for raspberries, 13.1 months for currants, 17 months for lettuce, 19.8 months for fresh and dry peas, 16.7 months for fresh beans, 10.5 months for almonds, 6.4 months for pecans, 5.1 months for flax and 5.9 months for coffee. The periods of demonstrated stability cover the frozen storage intervals used in the residue studies on crops.

USE PATTERN

The registered uses of trifloxystrobin relevant to the supervised residue studies made available to the current Meeting are summarized in Table 2.

Table 2 Registered uses of trifloxystrobin (SC formulations)

Crop	Country	F/G	Application							PHI (days)
			Method	GS/Timing	Max. No.	Rate/ application (g ai/ha)	Interval (days)	Water (L/ha)	Max. Rate/ season (g ai/ha)	
001 Citrus Fruit										
Citrus Fruit, Crop Group 10 [a]	USA	F	Foliar	Throughout growing season.	4	67-135	7-21	≥ 468 (grnd) ≥ 94 (air)	540	7
Citrus Fruit, Crop Group 10-10 [b]	USA	F	Foliar	Critical timing for disease control.	4	140	7-21	NS (grnd); ≥ 94 (air)	499 [560] [c]	7
004A Cane Berries										
Raspberries, Blackberries	Austria	F G	Foliar	BBCH 15-89	2	200	7	1000 200-1200	400	3
Raspberries and other *Rubus* species	France	F	Foliar	BBCH 13-69	1	200	-	NS	200	3
Blackberry and	NL	F	Foliar	BBCH 40-69	2	150	21	NS	300	3

Crop	Country	F/G	Application							PHI (days)
			Method	GS/Timing	Max. No.	Rate/ application (g ai/ha)	Interval (days)	Water (L/ha)	Max. Rate/ season (g ai/ha)	
raspberry family (*Rubus* spp.)		G		-		150-200	7		400	
Raspberry, Blackberry	Portugal	G	Foliar	-	2	150-200	7	300-1000	400	3
Raspberries, Blackberries	Spain	F	Foliar	BBCH 13-89	1	150-200	-	300-1000	200	3
004B Bush Berries										
Currants, Gooseberries, Blueberries	Austria	F	Foliar	BBCH 15-89	2	200	7	1000	400	7
		G						200-1200		
Blackcurrant	France	F	Foliar	BBCH 13-69	1	200	-	NS	200	7
Berries excluding Kiwiberry [d]	NL	F	Foliar	-	2	150	14	NS	300	7
		G		-		150-200	7		400	
Blueberry, Currant	Portugal	G	Foliar	-	2	150-200	7	300-1000	400	7
Blueberry, Currant Highbush blueberry	Spain	F	Foliar	BBCH 13-89	2	150-200	7	300-1000	200	7
013A Leafy Greens										
Lettuce	Austria	F	Foliar	BBCH 13-49	1	200	-	300-600	200	7
Endive				BBCH 13-19				200-800		
Lettuce, lamb´s lettuce	Austria	G	Foliar	BBCH 12-49	2	200	7	500-1000	400	7
Chard, lettuce, endive, chicory	Brazil	F	Foliar	From transplanting.	3	60-75	7	300-1000	225	1
Lettuce, lamb's lettuce	France	F	Foliar	BBCH 40-49	1	200	-	NS	200	7
Lettuce		G		BBCH 13-49						
Lettuce (*Lactuca* spp.), Lamb's lettuce	NL	F	Foliar	-	1	200	-	NS	200	7
		G		-	2		7		400	
Endive	NL	F	Foliar	-	1	200	-	NS	200	7
Lettuce	Portugal	F	Foliar	-	1	150-200	-	300-1000	200	7
Lettuce	Spain	F	Foliar	BBCH 40-49	1	150-200	-	300-1000	200	7
				-	2	150-200	7		400	
Leafy green vegetables [e]	USA	F	Foliar	Begin preventatively and continue as needed.	NS	135	5-10	NS	270	0
			Banded							20
Leafy greens except watercress [f]	USA	F	Foliar	Critical timing for disease control.	NS	140	14	NS	280 [420] [c]	0
Lettuce (head and leaf)			Banded							20
014 Legume Vegetables and 015 Pulses										

Crop	Country	F/G	Application							PHI (days)
			Method	GS/Timing	Max. No.	Rate/ application (g ai/ha)	Interval (days)	Water (L/ha)	Max. Rate/ season (g ai/ha)	
Kidney bean (fresh or dried, with or without pods)	Austria	F	Foliar	BBCH 59-69	2	200	7-14	400-600	400	7
French climbing bean with pod;	Austria	F	Foliar	BBCH 55-79	2	200	14	200-800	400	14
Beans without pod (fresh or dried) [g]; Field bean without pod (fresh or dried) [h]	Austria	F	Foliar	BBCH 55-79	2	200	14	200-800	400	14
Peas with/ without pods (fresh or dried)	Austria	F	Foliar	BBCH 55-79	2	200	14	200-800	400	14
Fresh beans without pods: Field bean and Flageolet bean	France	F	Foliar	BBCH 55-89	1	200	-	NS	200	7
Bean with pods, fresh	France	F	Foliar	BBCH 55-89	1	200	-	NS	200	7
				BBCH 61-75	1	200	-	NS	200	14
Pea with pod, fresh; Pea, for canning	France	F	Foliar	BBCH 55-89	1	200	-	NS	200	7
Field bean, dry; Bean, dry; Lentils; Chickpea; Pea, dry	France	F	Foliar	BBCH 55-89	1	200	-	NS	200	21
Beans and Peas with/without pods (fresh or dried)	NL	F	Foliar	-	2	200	14	NS	400	14
022 Tree Nuts										
Almonds	USA	F	Foliar	Begin applications at pink bud stage (~5% bloom) and apply again at full bloom and at petal fall.	4	67-135	7-21	≥ 468 (grnd) ≥ 94 (air)	540	14
Almonds	USA	F	Foliar	Critical timing for disease control.	4	92-139	7-14	NS (grnd); ≥ 94 (air)	498 [560] [c]	14
Pecans	USA	F	Foliar	Begin at bud break and continue through pollination.	6	67-135	14-21	≥ 468 (grnd) ≥ 94 (air)	797	30
Pecans	USA	F	Foliar	Critical timing for disease control.	6	74-139	14-21	NS (grnd); ≥ 94 (air)	498 [840] [c]	14
Pistachios	USA	F	Foliar	Begin preventatively and continue as needed.	4	67-135	14-21	≥ 468 (gr) ≥ 94 (air)	540	28

Crop	Country	F/G	Application							PHI (days)
			Method	GS/Timing	Max. No.	Rate/ application (g ai/ha)	Interval (days)	Water (L/ha)	Max. Rate/ season (g ai/ha)	
Pistachios	USA	F	Foliar	Critical timing for disease control.	4	92-139	14-21	NS (gr); ≥ 94 (air)	423	14
Tree nuts, Crop Group 14 [i]	USA	F	Foliar	Begin preventatively and continue as needed.	4	67-135	7-21	≥ 468 (gr) ≥ 94 (air)	540	60
Tree nuts, Crop Group 14-12 [j]	USA	F	Foliar	Critical timing for disease control.	4	139	7-21	NS (gr); ≥ 94 (air)	280 [560] [c]	14
023 Oilseeds										
Flax	Canada	F	Foliar	From early flowering (R1) to complete pod fill (R5).	1	132	-	≥100 (gr) ≥ 50 (air)	132	36
024 Seed for Beverages and Sweets										
Coffee	Brazil	F	Foliar	Main flowering; swab phase (maturing of the floral buds).	3	75-100 [k]	21	400-500 (gr) 30-40(air)	600	30

NL = The Netherlands; grnd = ground application; NS = not specified; F = field or outdoors with no cover or form of protection over the crop or "unprotected" on the label from the Netherlands.; G = greenhouse (indoor) for the labels from Austria, Portugal, and Spain or "protected" on the label from the Netherlands.

[a] Includes: calamondin; citrus citron; citrus hybrids; grapefruit; kumquat; lemon; lime; mandarin; orange, sour; orange sweet; pummelo; and Satsuma mandarin.

[b] Includes: Australian Desert Lime; Australian Finger Lime; Australian Round Lime; Brown River Finger Lime; Calamondin; Citron; Citrus Hybrids; Grapefruit; Japanese Summer Grapefruit; Kumquat; Lemon; Lime; Mediterranean Mandarin; Mount White Lime; New Guinea Wild Lime; Orange, Sour; Orange, Sweet; Pummelo; Russell River Lime; Satsuma Mandarin; Sweet Lime; Tachibana Orange; Tahiti Lime; Tangelo; Tangerine (Mandarin); Tangor; Trifoliate Orange; Uniq Fruit; cultivars, varieties and/or hybrids of these.

[c] Rate in square brackets represents the maximum seasonal application rate for all methods of applications for trifloxystrobin (e.g. foliar and soil uses).

[d] As per clarification, "Berries" would include: currants, gooseberries, blueberries (including bilberries and foxberries), cranberries, mulberries, rose hips, and elderberries).

[e] Amaranth (leafy amaranth, Chinese spinach, tampala), Arugula (Roquette), Chervil, Chrysanthemum (edible leaved and garland), Corn salad, Cress (garden), Cress (upland, yellow rocket, winter cress), Dandelion, Dock (sorrel), Endive (escarole), Lettuce (head and leaf), Orach, Parsley, Purslane (garden and winter), Radicchio (red chicory), Spinach [including New Zealand and vine (Malabar spinach, Indian spinach)].

[f] Amaranth (leafy amaranth, Chinese spinach, tampala); Arugula (Roquette); Chervil; Chrysanthemum (edible leaved and garland); Corn salad; Cress (garden); Cress (upland, yellow rocket, winter cress); Dandelion; Dock (sorrel); Endive (escarole); Lettuce (head and leaf); Orach; Parsley; Purslane (garden and winter); Radicchio (red chicory); Spinach [including New Zealand and vine (Malabar spinach, Indian spinach)].

[g] French climbing bean, lima bean, asparagus bean, butter bean, and giant bean without pod. Can be fresh or dried as per clarification.

[h] As per clarification, this is a general term that may include different species like *Phaseolus* and *Vicia*.

[I] Beechnuts; Brazil Nuts; Butternuts; Cashew; Chestnuts; Chinquapins; Filberts; Hickory Nuts; Macadamia Nuts; Walnuts.

[j] African Nut-Tree; Beechnut; Brazil Nut; Brazilian Pine; Bunya; Bur Oak; Butternut; Cajou Nut; Candlenut; Cashew; Chestnut; Chinquapin; Coconut; Coquito Nut; Dika Nut; Ginkgo; Guiana Chestnut; Hazelnut; Heartnut; Hickory Nut; Japanese Horse-Chestnut; Macadamia Nut; Mongongo Nut; Monkey-Pot; Monkey Puzzle Nut; Okari Nut; Pachira Nut; Peach Palm Nut; Pequi; Pili Nut; Pine Nut; Sapucaia Nut; Tropical Almond; Walnut, Black; Walnut, English; Yellowhorn; Cultivars, varieties, and/or hybrids of these.

[k] Add methylated soybean oil at 0.25% v/v.

RESULTS OF SUPERVISED RESIDUE TRIALS ON CROPS

The Meeting received information on supervised field trials for trifloxystrobin on the following crops or crop groups:

Crop		Table No.
Citrus fruits		
	Oranges, Lemons, and Grapefruits	3
Caneberries		
	Raspberries	4
Bushberries		
	Currants	5
Leafy greens		
	Leaf lettuce	6
Legume Vegetables		
	Beans with pods	7
	Peas with pods	8
	Beans without pods	9
	Peas without pods	10
Pulses		
	Dry peas	11
Tree nuts		
	Almonds and Pecans	12
Oilseeds		
	Flax	13
Coffee		14
Legume animal feeds		
	Bean forage	15
	Pea vines	16
	Pea hay	17
Almond hulls		18

Residue values from the trials conducted according to the critical GAP (or a suitable alternative GAP) have been used for the estimation of maximum residue levels, STMR, and HR (where applicable). Those results included in the evaluation as adequately supporting the GAP are underlined. Non quantifiable residues are shown as below the reported LOQ (e.g. < 0.02 mg/kg). Where multiple analyses were conducted on a single sample, the average value is reported. Where multiple samples were taken from a single plot, the individual and average values are reported. For all trials except citrus fruits (which had two treated samples), only a single composite treated sample was analysed.

The residues presented in the tables are given as trifloxystrobin and the metabolite CGA321113, expressed as themselves. The sum of trifloxystrobin and CGA321113 was calculated and expressed as trifloxystrobin on the basis of the relative molecular masses. A conversion factor of 1.036 is required to

express CGA321113 as trifloxystrobin. As CGA321113 does not generally constitute a significant proportion of the residue in crops, when the levels of trifloxystrobin or CGA321113 were below the LOQ, their sum was calculated as in the examples provided by the 2004 JMPR and copied below.

Trifloxystrobin (mg/kg)	CGA321113 (mg/kg)	Total (expressed as trifloxystrobin) (mg/kg)
< 0.02	< 0.02	< 0.02
< 0.02	0.03	0.05
0.10	< 0.02	0.10
0.92	0.16	1.1

Citrus fruit

Table 3 Residues of trifloxystrobin and CGA321113 (mg/kg) in citrus (Mackie, 2006, RATFY005) following four foliar applications of trifloxystrobin in a WG-formulation

Location, year, variety CITRUS	N (int)	Rate (g ai/ha)	Spray volume (L/ha)	GS	DALA	Sample	Trifloxystrobin (mg/kg) [average]	CGA321113 (mg/kg) [average]	Total, mg/kg (mg/kg) [average]
US GAP	4 (7-21)	140	NS (grnd), ≥ 94 (air)		7				Max. rate is 560 g ai/ha/season
Orange									
Oveido, FL, USA, 2004, Navel [a]	4 (19-21)	140 138 140 139	2109 2083 2121 2109	79 81 83 85	7	Fruit	0.148, 0.092 [0.12]	<0.02, <0.02 [<0.02]	0.148, 0.092 [0.12]
Oveido, FL, USA, 2004, Hamlin [a]	4 (19-21)	140 138 138 140	561 554 564 561	79 81 83 85	0	Fruit	0.185,0.170 [0.18]	<0.02, <0.02 [<0.02]	0.185,0.170 [0.18]
					3		0.195, 0.184 [0.19]	<0.02, <0.02 [<0.02]	0.195, 0.184 [0.19]
					7		0.107, 0.158 [0.13]	<0.02, <0.02 [<0.02]	0.107, 0.158 [0.13]
					10		0.076, 0.086 [0.081]	<0.02, <0.02 [<0.02]	0.076, 0.086 [0.081]
					14		0.066, 0.060 [0.063]	<0.02, <0.02 [<0.02]	0.066, 0.060 [0.063]
					7	Whole fruit	0.110, 0.119, 0.119 [0.116]	<0.01, <0.01, <0.01 [<0.01]	0.110, 0.119, 0.119 [0.116]
						Flesh	0.0268, 0.0198, 0.0328 [0.0265]	<0.01, <0.01, <0.01 [<0.01]	0.0268, 0.0198, 0.0328 [0.0265]
						Peel	0.398, 0.435, 0.455 [0.429]	<0.01, <0.01, <0.01 [<0.01]	0.398, 0.435, 0.455 [0.429]

Location, year, variety CITRUS	N (int)	Rate (g ai/ha)	Spray volume (L/ha)	GS	DALA	Sample	Trifloxystrobin (mg/kg) [average]	CGA321113 (mg/kg) [average]	Total, mg/kg (mg/kg) [average]
US GAP	4 (7-21)	140	NS (grnd), ≥ 94 (air)		7				Max. rate is 560 g ai/ha/season
	4 (19-21)	138 140 139 141	2084 2103 2096 2115	79 81 83 85	7	Fruit	0.206, 0.163 [0.18]	<0.02, <0.02 [<0.02]	0.206, 0.163 [0.18]
Oveido, FL, USA, 2005, Valencia [a]	4 (21)	141 140 139 140	563 560 558 559	79 79 83 83	7	Fruit	0.117, 0.124 [0.12]	<0.02, <0.02 [<0.02]	0.117, 0.124 [0.12]
	4 (21)	138 139 139 139	2077 2095 2086 2086	79 79 83 83	7	Fruit	0.142, 0.126 [0.13]	<0.02, <0.02 [<0.02]	0.142, 0.126 [0.13]
Winter Garden, FL, USA, 2004, Hamlin	4 (21)	140 140 141 140	537 486 526 514	79 79 83 83	7	Fruit	0.137, 0.198 [0.17]	<0.02, <0.02 [<0.02]	0.137, 0.198 [0.17]
	4 (20)	141 140 142 141	2657 2668 2626 2639	83 83 83 83	7	Fruit	0.084, 0.079 [0.082]	<0.02, <0.02 [<0.02]	0.084, 0.079 [0.082]
Clermont, FL, USA, 2004, Midsweet	4 (19)	141 141 140 141	462 502 489 517	83 83 83 83	7	Fruit	0.088, 0.124 [0.11]	<0.02, <0.02 [<0.02]	0.088, 0.124 [0.11]
	4 (19)	141 141 140 140	2542 2494 2501 2526	83 83 83 83	7	Fruit	0.058, 0.061 [0.060]	<0.02, <0.02 [<0.02]	0.058, 0.061 [0.060]
Mt. Dora, FL, USA, 2004, Hamlin	4 (20-21)	138 141 141 140	374 378 442 457	74 74 79 85	7	Fruit	0.085, 0.116 [0.10]	<0.02, <0.02 [<0.02]	0.085, 0.116 [0.10]
	4 (20-21)	140 146 141 141	2524 3599 2486 2529	74 74 79 85	7	Fruit	0.098, 0.095 [0.097]	<0.02, <0.02 [<0.02]	0.098, 0.095 [0.097]
Lake Placid, FL, USA, 2004, Hamlin	4 (20-21)	139 141 140 141	330 337 381 405	74 74 79 85	7	Fruit	0.090, 0.113 [0.10]	<0.02, <0.02 [<0.02]	0.090, 0.113 [0.10]

Location, year, variety CITRUS	N (int)	Rate (g ai/ha)	Spray volume (L/ha)	GS	DALA	Sample	Trifloxystrobin (mg/kg) [average]	CGA321113 (mg/kg) [average]	Total, mg/kg (mg/kg) [average]
US GAP	4 (7-21)	140	NS (grnd), ≥ 94 (air)		7				Max. rate is 560 g ai/ha/season
	4 (20-21)	138 141 139 141	2198 2143 2215 2211	74 74 79 85	7	Fruit	0.104, 0.100 [0.10]	<0.02, <0.02 [<0.02]	0.104, 0.100 [0.10]
Vero Beach, FL, USA, 2004, Hamlin	4 (20-21)	141 140 143 142	334 333 382 407	74 74 79 85	7	Fruit	0.056, 0.070 [0.063]	<0.02, <0.02 [<0.02]	0.056, 0.070 [0.063]
	4 (20-21)	142 141 144 142	2260 2139 2241 2224	74 74 79 85	7	Fruit	0.137, 0.103 [0.12]	<0.02, <0.02 [<0.02]	0.137, 0.103 [0.12]
Hobe Sound, FL, USA, 2004, Valencia	4 (21)	140 143 145 142	477 477 476 448	79 79 81 83	7	Fruit	0.232, 0.320 [0.28]	<0.02, <0.02 [<0.02]	0.232, 0.320 [0.28]
	4 (21)	140 138 138 136	2261 2377 2253 2200	79 79 81 83	7	Fruit	0.079, 0.113 [0.096]	<0.02, <0.02 [<0.02]	0.079, 0.113 [0.096]
Raymondville, TX, USA, 2004, N-33 Navel	4 (21)	141 142 141 143	559 562 567 571	79 79 79 83	7	Fruit	0.050, 0.079 [0.065]	<0.02, <0.02 [<0.02]	0.050, 0.079 [0.065]
	4 (21)	141 141 140 141	2788 2788 2838 2829	79 79 79 83	7	Fruit	0.057, 0.054 [0.056]	<0.02, <0.02 [<0.02]	0.057, 0.054 [0.056]
Fresno, CA, USA, 2004, Washington Navel	4 (20-21)	142 140 140 140	403 435 442 439	79 79 81 83	0	Fruit	0.155, 0.176 [0.17]	<0.02, <0.02 [<0.02]	0.155, 0.176 [0.17]
					3		0.121, 0.086 [0.10]	<0.02, <0.02 [<0.02]	0.121, 0.086 [0.10]
					7		0.167, 0.127 [0.15]	<0.02, <0.02 [<0.02]	0.167, 0.127 [0.15]
					9		0.074, 0.086 [0.080]	<0.02, <0.02 [<0.02]	0.074, 0.086 [0.080]
					13		0.085, 0.063 [0.074]	<0.02, <0.02 [<0.02]	0.085, 0.063 [0.074]

Location, year, variety CITRUS	N (int)	Rate (g ai/ha)	Spray volume (L/ha)	GS	DALA	Sample	Trifloxystrobin (mg/kg) [average]	CGA321113 (mg/kg) [average]	Total, mg/kg (mg/kg) [average]
US GAP	4 (7-21)	140	NS (grnd), ≥ 94 (air)		7				Max. rate is 560 g ai/ha/season
	4 (20-21)	141 141 142 141	2555 2592 2636 2608	79 79 81 83	7	Fruit	0.104, 0.064 [0.084]	<0.02, <0.02 [<0.02]	0.104, 0.064 [0.084]
Porterville, CA, USA, 2004, Washington Nave	4 (20-21)	140 141 142 143	562 565 571 575	81 81 83 85	7	Fruit	0.044, 0.022 [0.033]	<0.02, <0.02 [<0.02]	0.044, 0.022 [0.033]
	4 (20-21)	142 141 140 141	2472 2484 2476 2477	81 81 83 85	7	Fruit	0.177, 0.201 [0.19]	<0.02, <0.02 [<0.02]	0.177, 0.201 [0.19]
Woodlake, CA, USA, 2005, Late Lane	4 (19-20)	138 137 141 140	514 504 503 504	83 83 85 85	7	Fruit	0.084, 0.073 [0.079]	<0.02, <0.02 [<0.02]	0.084, 0.073 [0.079]
	4 (19-20)	139 136 140 141	2170 2136 2173 2195	83 83 85 85	7	Fruit	0.038, 0.051 [0.045]	<0.02, <0.02 [<0.02]	0.038, 0.051 [0.045]
Lemon									
Clermont, FL, USA, 2004, Meyer	4 (20-22)	139 140 140 139	560 563 564 563	72 74 79 83	7	Fruit	0.046, 0.063 [0.055]	<0.02, <0.02 [<0.02]	0.046, 0.063 [0.055]
	4 (20-22)	141 140 139 139	1975 1969 1958 1951	72 74 79 83	7	Fruit	0.051, 0.062 [0.057]	<0.02, <0.02 [<0.02]	0.051, 0.062 [0.057]
Porterville, CA, USA, 2005, Lisbon[b]	4 (20)	140 142 139 140	595 606 571 573	83 83 85 85	0	Fruit	0.492, 0.302 [0.340]	<0.02, <0.02 [<0.02]	0.492, 0.302 [0.40]
					3		0.275, 0.270 [0.27]	<0.02, <0.02 [<0.02]	0.275, 0.270 [0.27]
					7		0.240, 0.185 [0.21]	<0.02, <0.02 [<0.02]	0.240, 0.185 [0.21]
					10		0.305, 0.167 [0.24]	<0.02, <0.02 [<0.02]	0.305, 0.167 [0.24]
					14		0.197, 0.262 [0.23]	<0.02, <0.02 [<0.02]	0.197, 0.262 [0.23]

Location, year, variety CITRUS	N (int)	Rate (g ai/ha)	Spray volume (L/ha)	GS	DALA	Sample	Trifloxystrobin (mg/kg) [average]	CGA321113 (mg/kg) [average]	Total, mg/kg (mg/kg) [average]
US GAP	4 (7-21)	140	NS (grnd), ≥ 94 (air)		7				Max. rate is 560 g ai/ha/season
	4 (20)	141 142 140 140	2524 2531 2497 2494	83 83 85 85	7	Fruit	0.272, 0.233 [0.25]	<0.02, <0.02 [<0.02]	0.272, 0.233 [0.25]
Porterville, CA, USA, 2005, Lisbon [b]	4 (20)	139 141 139 140	293 600 568 573	83 83 85 85		Fruit	0.172, 0.142 [0.16]	<0.02, <0.02 [<0.02]	0.172, 0.142 [0.16]
	4 (20)	138 142 140 140	2475 2541 2490 2488	83 83 85 85	7	Fruit	0.181, 0.164 [0.17]	<0.02, <0.02 [<0.02]	0.181, 0.164 [0.17]
Porterville, CA, USA, 2004, Pryor [b]	4 (19-21)	140 138 138 141	458 459 400 389	74 79 83 85	6	Fruit	0.312, 0.224 [0.27]	<0.02, <0.02 [<0.02]	0.312, 0.224 [0.27]
	4 (19-21)	140 140 141 141	2132 2280 2150 2172	74 79 83 85	6	Fruit	0.260, 0.348 [0.30]	<0.02, <0.02 [<0.02]	0.260, 0.348 [0.30]
Nipomo, CA, USA, 2004, Eureka	4 (20-22)	140 141 141 141	513 514 513 499	73 79 79 79	7	Fruit	0.170, 0.170 [0.17]	<0.02, <0.02 [<0.02]	0.170, 0.170 [0.17]
	4 (20-22)	141 141 142 140	2429 2407 2418 2365	73 79 79 79	7	Fruit	0.083, 0.066 [0.075]	<0.02, <0.02 [<0.02]	0.083, 0.066 [0.075]
Grapefruit									
Oviedo, FL, USA, 2004, Flame red	4 (19-21)	142 142 143 139	568 568 573 566	79 81 83 85	0	Fruit	0.124, 0.170 [0.15]	<0.02, <0.02 [<0.02]	0.124, 0.170 [0.15]
					3		0.120, 0.067 [0.094]	<0.02, <0.02 [<0.02]	0.120, 0.067 [0.094]
					7		0.156, 0.095 [0.13]	<0.02, 0.033 [0.027]	0.156, 0.128 [0.14]
					10		0.066, 0.050 [0.058]	<0.02, <0.02 [<0.02]	0.066, 0.050 [0.058]
					14		0.058, 0.039 [0.049]	<0.02, <0.02 [<0.02]	0.058, 0.039 [0.049]

Location, year, variety CITRUS	N (int)	Rate (g ai/ha)	Spray volume (L/ha)	GS	DALA	Sample	Trifloxystrobin (mg/kg) [average]	CGA321113 (mg/kg) [average]	Total, mg/kg (mg/kg) [average]
US GAP	4 (7-21)	140	NS (grnd), ≥ 94 (air)		7				Max. rate is 560 g ai/ha/season
	4 (19-21)	140 140 139 139	2105 2111 2110 2117	79 81 83 85	7	Fruit	0.164, 0.170 [0.17]	<0.02, <0.02 [<0.02]	0.164, 0.170 [0.17]
Howie in the Hills, FL, USA, 2004, Flame	4 (20)	140 141 140 140	536 487 524 513	83 83 83 83	7	Fruit	0.081, 0.037 [0.059]	<0.02, <0.02 [<0.02]	0.081, 0.037 [0.059]
	4 (20)	140 140 142 140	1920 1932 1912 1920	83 83 83 83	7	Fruit	0.046, 0.048 [0.047]	<0.02, <0.02 [<0.02]	0.046, 0.048 [0.047]
Hobe Sound, FL, USA, 2004, White Marsh	4 (21)	140 142 137 139	560 568 539 536	83 83 83 83	7	Fruit	0.078, 0.077 [0.078]	<0.02, <0.02 [<0.02]	0.078, 0.077 [0.078]
	4 (21)	141 139 139 140	2724 2858 2748 2744	83 83 83 83	7	Fruit	0.092, 0.107 [0.10]	<0.02, <0.02 [<0.02]	0.092, 0.107 [0.10]
Raymondville, TX, USA, 004, Rio Red	4 (21)	141 141 140 143	560 558 564 572	79 79 79 83	7	Fruit	0.036, 0.021 [0.029]	<0.02, <0.02 [<0.02]	0.036, 0.021 [0.029]
	4 (21)	142 141 140 142	2803 2783 2837 2849	79 79 79 83	7	Fruit	0.028, 0.030 [0.029]	<0.02, <0.02 [<0.02]	0.028, 0.030 [0.029]
Nipomo, CA, USA, 2004, Star Ruby	4 (20-21)	138 138 141 135	491 494 508 497	74 79 79 83	7	Fruit	0.035, 0.048 [0.042]	<0.02, <0.02 [<0.02]	0.035, 0.048 [0.042]
	4 (20-21)	142 138 133 140	2447 2387 2318 2432	74 79 79 83	7	Fruit	0.051, 0.033 [0.042]	<0.02, <0.02 [<0.02]	0.051, 0.033 [0.042]
Porterville, CA, USA, 2004, Mellogold	4 (21)	139 140 139 140	568 564 564 574	79 79 81 83	7	Fruit	0.011, 0.015 [0.013]	<0.02, <0.02 [<0.02]	0.011, 0.015 [0.013]

Location, year, variety CITRUS	N (int)	Rate (g ai/ha)	Spray volume (L/ha)	GS	DALA	Sample	Trifloxystrobin (mg/kg) [average]	CGA321113 (mg/kg) [average]	Total, mg/kg (mg/kg) [average]
US GAP	4 (7-21)	140	NS (grnd), ≥ 94 (air)		7				Max. rate is 560 g ai/ha/season
	4 (21)	140 140 140 140	2524 2491 2490 2510	79 79 81 83	7	Fruit	0.068, 0.103 [0.086]	<0.02, <0.02 [<0.02]	0.068, 0.103 [0.086]

[a] The Naval and Hamlin variety orange trials were conducted at the same location and with final application occurring on the same day. These trials are not considered independent for the purposes of estimating maximum residue limits. The Valencia variety orange trial is considered independent from the other two trials conducted at this location on the basis of the different varieties and last applications being made 125 days apart.

[b] The Lisbon variety lemon trials were conducted at the same location and with final application occurring on the same day. These trials are not considered independent for the purposes of estimating maximum residue limits. The Pryor lemon variety trial is considered independent from the other two trials conducted at this location on the basis of the different varieties and last applications being made 106 days apart.

Raspberries

Table 4 Residues of trifloxystrobin and CGA321113 (mg/kg) in raspberries following two foliar applications of trifloxystrobin in a SC-formulation

Location, year, variety RASPBERRY	N (int)	Rate (g ai/ha)	Spray volume (L/ha)	GS	DALA	Sample	Trifloxystrobin (mg/kg)	CGA321113 (mg/kg)	Total (mg/kg)	Study No. (Protected/ Unprotected)
The NL GAP	2 (7)	200	200-1200		3					Greenhouse/ Protected
Austria, Portugal GAP	2 (7)	200	200-1200		3					Field/ Unprotected
Damazan, France, 2015, Kwenza	2 (7)	205 197	1232 1184	81 83-85	0 3 7	Fruit	0.40 0.25 0.12	0.016 0.042 0.025	0.42 0.29 0.15	RAFR00215 (Protected [a])
Cendrieux, France, 2015, Kweli	2 (6)	204 200	1224 1197	79 81	0 3 7	Fruit	0.46 0.086 0.048	0.047 0.017 0.033	0.51 0.10 0.081	
Capdrot, France, 2015, Kwenza	2 (7)	184 199	1101 1196	85-87 87	0 1 3 7	Fruit	1.3 0.50 0.48 0.23	0.026 0.019 0.045 0.023	1.3 0.52 0.53 0.25	
Bannes, France, 2015, Imara	2 (7)	206 208	1031 1041	87 87	0 1 3 7	Fruit	0.32 0.25 0.17 0.16	0.006 <0.005 0.008 0.009	0.33 0.25 0.18 0.17	
Juillac, France, 2010, Meeker	2 (7)	199 213	792 848	81 87	0 1 3	Fruit	1.8 1.1 0.98	0.13 0.114 0.16	1.9 1.2 1.1	RAFR00810 (Protected [b])

Location, year, variety RASPBERRY	N (int)	Rate (g ai/ha)	Spray volume (L/ha)	GS	DALA	Sample	Trifloxystrobin (mg/kg)	CGA321113 (mg/kg)	Total (mg/kg)	Study No. (Protected/ Unprotected)
The NL GAP	2 (7)	200	200-1200		3					Greenhouse/ Protected
Austria, Portugal GAP	2 (7)	200	200-1200		3					Field/ Unprotected
Saugon, France, 2010, Heritage	2 (8)	220	875	81	0	Fruit	0.44	0.043	0.48	
		220	875	87	1		0.54	0.085	0.63	
					3		0.33	0.050	0.38	
Saugon, France, 2009, Heritage	2 (7)	211	840	NS	0	Fruit	0.97	0.045	1.0	RAFR03509 (Protected [b])
		214	850		1		0.87	0.047	0.92	
					3		0.51	0.088	0.60	
Juillac, France, 2009, Meeker	2 (7)	202	804	NS	0	Fruit	1.0	0.14	1.1	
		209	832		1		0.57	0.12	0.69	
					3		0.52	0.069	0.59	
Perwez, Belgium, 2011, Malling Exploit	2 (7)	194	873	81	-0	Fruit	0.13	0.03	0.16	BCS-G401-11 (Protected [c])
		207	933	87	0		0.77	0.03	0.80	
					1		0.58	0.04	0.62	
					3		0.57	0.05	0.62	
					7		0.30	0.04	0.34	
					14		0.15	0.03	0.18	
					21		0.05	0.03	0.08	
Haaften, the Netherlands, 2011, Brillant	2 (7)	200	1000	75-87	-0	Fruit	0.90	0.05	0.95	PTZ-NLI-11797 (Protected [c])
		200	1000	85-87	0		1.3	0.06	1.4	
					1		1.3	0.05	1.4	
					3		1.4	0.04	1.4	
					7		0.76	0.05	0.81	
					14		0.17	0.02	0.19	
					21		0.13	0.01	0.14	
Mauguio, France, 015, Bois Blanc	2 (7)	216	865	89	-0	Fruit	0.28	0.079	0.36	15-2034 (Unprotected)
		200	800	89	0		1.0	0.077	1.1	
					1		0.65	0.084	0.73	
					3		0.70	0.10	0.80	
					7		0.21	0.078	0.29	
					14		0.035	0.014	0.049	
Ambres, France, 2015, Autumn Bliss	2 (7)	200	1000	85	-0	Fruit	0.32	0.055	0.38	
		200	1000	85	0		0.89	0.034	0.92	
					1		0.62	0.057	0.68	
					3		0.64	0.068	0.71	
					7		0.20	0.042	0.24	
					14		0.044	0.012	0.056	

Location, year, variety RASPBERRY	N (int)	Rate (g ai/ha)	Spray volume (L/ha)	GS	DALA	Sample	Trifloxystrobin (mg/kg)	CGA321113 (mg/kg)	Total (mg/kg)	Study No. (Protected/ Unprotected)
The NL GAP	2 (7)	200	200-1200		3					Greenhouse/ Protected
Austria, Portugal GAP	2 (7)	200	200-1200		3					Field/ Unprotected
Tabuyo del Monte, Spain, 2015, Heritage	2 (10)	200 200	1200 1200	85 87	-0	Fruit	0.28	0.019	0.30	
					0		0.72	0.019	0.74	
					1		0.48	<0.01	0.48	
					3		0.35	0.018	0.37	
					7		0.16	0.017	0.18	
					14		0.046	0.010	0.056	
Guijo de Santa Barbara, Spain, 015, Himbo Top	2 (7)	200 200	1200 1200	85 85	-0	Fruit	0.042	<0.01	0.042	
					0		0.34	<0.01	0.34	
					1		0.22	0.015	0.24	
					3		0.20	0.027	0.23	
					7		0.076	0.015	0.091	
					14		0.030	<0.01	0.030	
Cak, Hungary, 2018, Fertodi zamatos	2 (7)	195 193	778 770	85 85	-0	Fruit	0.21	0.029	0.24	18-2051 (Unprotected)
					0		0.48	0.024	0.50	
					1		0.22	0.019	0.24	
					3		0.36	0.026	0.39	
					7		0.31	0.026	0.34	
					15		0.064	<0.01	0.064	
Dziurków, Poland, 2018, Polana	2 (7)	209 209	731 732	85 87	-0	Fruit	0.30	0.024	0.32	
					0		1.1	0.026	1.1	
					1		1.0	0.050	1.0	
					3		1.0	0.058	1.1	
					7		0.52	0.047	0.57	
					14		0.35	0.042	0.39	
Untergruppenbach, Germany, 2018, Green Ample	2 (7)	201 194	803 777	81 87	-0	Fruit	0.17	0.038	0.21	
					0		0.19	0.016	0.21	
					1		0.15	0.027	0.18	
					3		0.098	0.020	0.12	
					7		0.054	0.020	0.074	
					14		0.028	<0.01	0.028	
La Chapelle de Guinchay, France, 2018, Zeva	2 (7)	191 192	763 767	85 87	-0	Fruit	0.99	0.033	1.0	
					0		2.8	0.029	2.8	
					1		1.4	0.024	1.4	
					3		1.5	0.039	1.5	
					7		0.66	0.050	0.71	
					13		0.26	0.036	0.30	

NS = not specified; "-0" = harvested before the last application.

[a] Trials were conducted in an enclosed glass greenhouse and grown in coconut or mineral substrate.

[b] Berries were grown outdoor under a plastic tunnel or cold shelter.

[c] Trials were conducted under plastic umbrella.

Currants

Table 5 Residues of trifloxystrobin and CGA321113 (mg/kg) in currants following two foliar applications of trifloxystrobin in a SC-formulation

Location, year, variety CURRANT	N (int)	Rate (g ai/ha)	Spray volume (L/ha)	GS	DALA	Sample	Trifloxystrobin (mg/kg)	CGA321113 (mg/kg)	Total (mg/kg)	Study No. (Protected/ Unprotected)
The NL GAP	2 (7)	200	200-1200		7					Greenhouse/ Protected
Austria, Spain GAP	2 (7)	200	200-1200		7					Field/ Unprotected
Pergine Val Sugana, Italy, 2014, Rovada (red currant)	2 (7)	198 202	693 707	85 87	-0[A] 0 1 3 8 15	Fruit	0.33 0.57 0.31 0.20 0.31 0.25	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01	0.33 0.57 0.31 0.20 0.31 0.25	14-2025 (Protected [a])
Verona, Italy, 2014, Titania (black currant)	2 (6)	201 203	705 710	85 85	-0 0 1 3 7 15	Fruit	0.36 1.5 1.2 0.49 0.35 0.29	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01	0.36 1.5 1.2 0.49 0.35 0.29	
Mondescourt, France, 2015, Rovada (red currant)	2 (7)	200 200	500 500	81 85	-0 0 1 3 7 14	Fruit	0.28 0.58 0.57 0.19 0.34 0.51	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01	0.28 0.58 0.57 0.19 0.34 0.51	15-2032 (Protected [b])
Ugny le Gay, France, 2015, Rovada (red currant)	2 (7)	200 200	500 500	81 85	-0 0 1 3 7 14	Fruit	0.26 0.55 0.39 0.35 0.27 0.23	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01	0.26 0.55 0.39 0.35 0.27 0.23	
Carreño, Spain, 2015, Negra (black currant)	2 (6)	200 200	700 700	85 87	-0 0 1 3 7 14	Fruit	0.45 0.86 0.68 0.34 0.36 0.022	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01	0.45 0.86 0.68 0.34 0.36 0.022	15-2032 (Protected [c])

Location, year, variety CURRANT	N (int)	Rate (g ai/ha)	Spray volume (L/ha)	GS	DALA	Sample	Trifloxystrobin (mg/kg)	CGA321113 (mg/kg)	Total (mg/kg)	Study No. (Protected/ Unprotected)
The NL GAP	2 (7)	200	200-1200		7					Greenhouse/ Protected
Austria, Spain GAP	2 (7)	200	200-1200		7					Field/ Unprotected
Guijo de Santa Barbara, Spain, 2015, Rovada (red currant)	2 (8)	200 200	1000 1000	85 87	-0 0 1 3 7 14	Fruit	0.035 0.27 0.20 0.11 0.15 0.091	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01	0.035 0.27 0.20 0.11 0.15 0.091	
Perwez, Belgium, 2011, Rosetta (red currant)	2 (7)	206 212	927 953	81 81-85	-0 0 1 3 7 14 21	Fruit	0.71 1.5 1.3 1.1 0.77 0.58 0.51	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01	0.71 1.5 1.3 1.1 0.77 0.58 0.51	BCS-G402-11 (Protected [d])
Westwoud, the Netherlands, 2011, Rovada (red currant)	2 (7)	200 200	1000 1000	85 87	0* 0 1 3 7 14 21	Fruit	0.46 0.56 0.45 0.36 0.26 0.27 0.31	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01	0.46 0.56 0.45 0.36 0.26 0.27 0.31	PTZ-NLI-11796 (Protected [d])
Neuillé-Pont-Pierre, France, 2015, Blackdown (black currant)	2 (7)	184 191	461 478	85 87	0 1 3 7	Fruit	0.40 0.40 0.28 0.13	<0.005 <0.005 <0.005 <0.005	0.40 0.40 0.28 0.13	R B5111 (Unprotected)
Thorée-les-pins, France, 2015, Andorine (black currant)	2 (7)	205 195	513 487	85 87	0 1 3 6	Fruit	0.52 0.40 0.35 0.22	<0.005 <0.005 <0.005 <0.005	0.52 0.40 0.35 0.22	
Merceuil, France, 2015, Noir de Bourgogne (black currant)	2 (7)	200 204	500 510	87 87	0 1 3 7	Fruit	2.0 2.0 1.5 1.2	<0.005 0.007 0.009 0.009	2.0 2.0 1.5 1.2	
Beaune, France, 2015, Bourgogne (black currant)	2 (7)	202 199	506 497	87 87	0 1 3 7	Fruit	1.7 1.1 0.92 0.64	0.015 0.026 0.018 0.011	1.7 1.1 0.94 0.65	

Location, year, variety CURRANT	N (int)	Rate (g ai/ha)	Spray volume (L/ha)	GS	DALA	Sample	Trifloxystrobin (mg/kg)	CGA321113 (mg/kg)	Total (mg/kg)	Study No. (Protected/ Unprotected)
The NL GAP	2 (7)	200	200-1200		7					Greenhouse/ Protected
Austria, Spain GAP	2 (7)	200	200-1200		7					Field/ Unprotected
Marsillargues, France, 2015, Junifer (red currant)	2 (7)	200 200	800 800	81 85	-0 0 1 3 7 14	Fruit	2.2 2.9 3.7 2.3 2.1 1.0	<0.01 <0.01 <0.01 <0.01 <0.01 0.012	2.2 2.9 3.7 2.3 2.1 1.0	15-2033 (Unprotected)
Lancie, France, 2015, Noir de Bourgogne (black currant)	2 (7)	200 200	500 500	85 87	-0 0 1 3 7 14	Fruit	0.38 1.2 0.98 0.87 0.46 0.46	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01	0.38 1.2 0.98 0.87 0.46 0.46	
Carreño, Spain, 2015, Negra (black currant)	2 (6)	200 200	700 700	85 87	-0 0 1 3 7 14	Fruit	0.23 0.56 0.48 0.30 0.29 0.24	<0.01 <0.01 <0.01 <0.01 <0.01 0.010	0.23 0.56 0.48 0.30 0.29 0.25	
Guijo de Santa Barbara, Spain, 22015, Rovada (red currant)	2 (6)	200 200	700 700	85 87	-0 0 1 3 7 16	Fruit	0.13 0.30 0.23 0.15 0.14 0.060	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01	0.13 0.30 0.23 0.15 0.14 0.060	

"-0" = harvested before the last application.

[a] Walk-in greenhouse with plastic cover.

[b] Trials were conducted in an open plastic greenhouse (open along the sides and the end).

[c] Trials were conducted in an open plastic greenhouse (closed the sides and open at the end).

[d] This trial was conducted under plastic umbrella.

Lettuce

Table 6 Residues of trifloxystrobin and CGA321113 (mg/kg) in leaf lettuce following two foliar applications of trifloxystrobin in a SC-formulation

Location, year, variety LEAF LETTUCE	N (int	Rate (g ai/ha)	Spray volume (L/ha)	GS	DALA	Sample	Trifloxystrobin (mg/kg)	CGA321113 (mg/kg)	Total (mg/kg)	Study No. (Protected/ Unprotected)
Austria	2 (7)	200	300- 1000		7					Greenhouse/ Protected
Leichlingen, Germany, 2014, Lugano Lollo bionda (open leaf)	2 (7)	200 200	300 300	45 47	-0 0 3 7 14	leaf	2.8 6.0 4.4 3.1 1.7	0.091 0.11 0.17 0.14 0.082	2.9 6.1 4.6 3.2 1.8	14-2028 (Protected [a])
Ridderkerk, the Netherlands, 2014, Satine Lollo Rosso (open leaf)	2 (7)	200 200	1000 1000	46 47	-0 0 3 7 14	leaf	2.4 13 5.3 4.5 1.2	0.19 0.64 0.57 0.19 0.062	2.6 14 5.9 4.7 1.3	
Saint-Amand, Belgium, 2014, Sansula Oakleaf (open leaf)	2 (7)	200 200	900 900	41 45	-0 0 3 7 14	leaf	0.84 5.3 2.5 1.2 0.65	0.071 0.12 0.18 0.10 0.051	0.91 5.4 2.7 1.3 0.70	
Castelsarrasin, France, 2014, Parinice Oakleaf (open leaf)	2 (7)	200 200	800 800	19 42	-0 0 3 7 14	leaf	0.65 5.3 3.5 0.85 0.053	0.11 0.56 0.14 0.052 <0.01	0.76 5.9 3.6 0.90 0.053	
Ridderkerk, the Netherlands, 2014, Korentina (open leaf)	2 (7)	200 200	600 600	45 46	-0 0 3 7 14	leaf	5.8 12 13 9.9 8.6	0.10 0.16 0.14 0.10 0.12	5.9 12 13 10 8.7	
Longué, France, 2014, Kimpala Oakleaf (open leaf)	2 (7)	200 200	600 600	47 48	-0 0 3 7 14	leaf	2.2 6.3 5.3 3.8 1.1	0.059 0.10 0.057 0.034 0.022	2.3 6.4 5.4 3.8 1.1	
Wervershoof, the Netherlands, 2007, Lolo Rosso (leaf lettuce)	2 (7)	200 200	1000 1000	47 49	-0 0 3 7 14 21	leaf	1.8 6.7 4.0 2.6 1.3 0.58	0.19 0.20 0.31 0.24 0.072 0.041	2.0 6.9 4.3 2.8 1.4 0.62	RA-2620/07 (Protected [a])

Location, year, variety LEAF LETTUCE	N (int)	Rate (g ai/ha)	Spray volume (L/ha)	GS	DALA	Sample	Trifloxystrobin (mg/kg)	CGA321113 (mg/kg)	Total (mg/kg)	Study No. (Protected/ Unprotected)
Austria	2 (7)	200	300-1000		7					Greenhouse/ Protected
Leichlingen, Germany, 2007, Alexandria (butterhead lettuce)	2 (7)	200 200	600 600	43 45	-0 0 3 7 14 21	leaf	2.9 11 6.9 3.5 1.8 0.78	0.041 0.072 0.041 0.031 0.031 0.021	2.9 11 6.9 3.5 1.8 0.80	
Ouzilly, France, 2007, Santoro (butterhead lettuce)	2 (7)	200 200	600 600	41 45	-0 0 3 7 14 21	leaf	0.97 5.2 3.0 1.9 0.68 0.09	0.031 0.093 0.041 0.041 0.021 <0.01	1.0 5.3 3.0 1.9 0.70 0.090	
Meckenbeuren, Germany, 2007, Alexandria (butterhead lettuce)	2 (7)	200 200	300 300	43 45	-0 0 3 7 14 21	leaf	1.5 5.0 2.2 1.3 0.90 0.37	0.031 0.052 0.041 0.021 0.010 <0.01	1.5 5.1 2.2 1.3 0.91 0.37	
Leichlingen, Germany, 2018, Macai RZ (open leaf)	2 (7)	193 208	579 623	44 47	-0 0 3 7 14	leaf	3.3 10 8.6 6.2 1.2	0.16 0.17 0.18 0.47 0.090	3.5 10 8.8 6.7 1.3	18-2048 (Protected [a])
Alkmaar, the Netherlands, 2018, Lollo Rossa Satine (open leaf)	2 (7)	200 192	500 480	47 48	-0 0 3 7 14	leaf	3.5 9.0 9.4 8.6 7.0	0.22 0.27 0.19 0.11 0.20	3.7 9.3 9.6 8.7 7.2	
Toulouse, France, 2018, Sumitie RZ Batavia Blonde (open leaf)	2 (7)	2016 197	825 788	45 46	-0 0 3 7 14	leaf	1.9 8.0 5.0 3.2 0.87	0.052 0.072 0.071 0.056 0.037	2.0 8.1 5.1 3.3 0.91	
Villers-Perwin, Belgium, 2014, Sansula (oak leaf) (open leaf)	2 (7)	200 200	900 900	45 48	-0 0 3 7 14	leaf	0.56 2.0 1.7 0.77 0.10	0.060 0.16 0.10 0.068 0.019	0.62 2.2 1.8 0.84 0.12	14-2029 (Unprotected)

Location, year, variety LEAF LETTUCE	N (int)	Rate (g ai/ha)	Spray volume (L/ha)	GS	DALA	Sample	Trifloxystrobin (mg/kg)	CGA321113 (mg/kg)	Total (mg/kg)	Study No. (Protected/ Unprotected)
Austria	2 (7)	200	300-1000		7					Greenhouse/ Protected
Dannstadt-Schauernheim, Germany, 2014, Cavernet (Lollo Rosso) (open leaf)	2 (7)	200 200	600 600	45 47	-0 0 3 7 14	leaf	0.54 2.4 1.6 1.0 0.18	0.082 0.10 0.10 0.12 0.046	0.62 2.5 1.7 1.1 0.23	
Zwaagdijk , the Netherlands, 2014, Loka (Lollo Rossa) (open leaf)	2 (7)	200 200	500 500	45 46	-0 0 3 7 14	leaf	2.6 8.6 3.5 1.5 0.18	0.18 0.18 0.34 0.24 0.065	2.8 8.8 3.8 1.7 0.25	
Lignières de Touraine, France, 2014, Kiribati (oak leaf) (open leaf)	2 (7)	200 200	600 600	47 48	-0 0 3 7 14	leaf	1.2 5.8 2.6 0.16 <0.01	0.035 0.071 0.030 0.010 <0.01	1.2 5.9 2.6 0.17 <0.01	
Leichlingen, Germany, 2014, Aleppo (Lollo bionda) (open leaf)	2 (7)	200 200	300 300	44 47	-0 0 3 7 14	leaf	1.7 5.3 3.1 0.77 0.12	0.082 0.10 0.10 0.075 0.016	1.8 5.4 3.2 0.85 0.14	
Neuruppin, Germany, 2014, Lollo Rosso (open leaf)	2 (9)	200 200	500 500	41 41	-0 0 3 8 15	leaf	1.2 0.56 0.21 0.036 0.028	<0.01 <0.01 <0.01 <0.01 <0.01	1.2 0.56 0.21 0.036 0.028	14-2184 (Unprotected)
Hooghalen, the Netherlands, 2014, Smile (open leaf)	2 (7)	200 200	400 400	48 49	-0 0 3 7 14	leaf	0.54 3.0 2.9 1.2 0.51	0.065 0.10 0.13 0.078 0.045	0.61 3.1 3.0 1.3 0.56	
Ferenctanya, Hungary, 2014, Linaro RZ (open leaf)	2 (7)	200 200	500 500	43 47	-0 0 3 7 13	leaf	0.023 4.6 2.0 0.24 0.037	0.063 0.17 0.13 0.083 0.021	0.086 4.8 2.1 0.32 0.058	
Banbury, United Kingdom, 2014, Anaconda (open leaf)	2 (8)	200 200	350 350	45 46	-0 0 3 7 16	leaf	0.38 2.3 0.46 0.28 0.010	0.031 0.032 0.035 0.027 <0.01	0.41 2.3 0.50 0.31 0.010	

Location, year, variety LEAF LETTUCE	N (int)	Rate (g ai/ha)	Spray volume (L/ha)	GS	DALA	Sample	Trifloxystrobin (mg/kg)	CGA321113 (mg/kg)	Total (mg/kg)	Study No. (Protected/ Unprotected)
Austria	2 (7)	200	300-1000		7					Greenhouse/ Protected
Alginet, Spain, 2014, Paladio (oak leaf) (open leaf)	2 (7)	200 200	750 750	48 49	-0 0 3 7 14	leaf	0.82 2.4 1.5 0.90 0.22	0.028 0.053 0.027 0.016 <0.01	0.85 2.5 1.5 0.92 0.22	14-2030 (Unprotected)
C.da Pigno, Catania, Italy, 2014, Naupius – Canasta (Batavia) (open leaf)	2 (7)	200 200	600 600	46 48	-0 0 3 7 14	leaf	1.3 3.9 2.6 1.2 0.43	0.052 0.17 0.043 0.028 0.021	1.4 4.1 2.6 1.2 0.45	
St Etienne du gres, France, 2014, Kiribati (oak leaf) (open leaf)	2 (7)	200 200	600 600	48 49	-0 0 3 7 14	leaf	0.82 3.4 2.3 1.1 0.43	0.051 0.086 0.071 0.053 0.036	0.87 3.5 2.4 1.2 0.47	
Aronas - Katerini – Pieria, Greece, 2014, Manchester (loose/open leaf)	2 (7)	200 200	600 600	45 46	-0 0 3 7 14	leaf	1.1 4.7 1.8 2.1 1.1	0.13 0.30 0.16 0.095 0.10	1.2 5.0 2.0 2.2 1.2	
Gela (CL), C.da Mignechi, Italy, 2014, Nauplus – Canasta (Batavia) (open leaf)	2 (7)	200 200	600 600	45 46	-0 0 3 7 14	leaf	0.95 4.9 4.6 1.3 0.35	0.075 0.094 0.071 0.085 0.033	1.0 5.0 4.7 1.4 0.38	
Mediglia, Italy, 2014, Gentilina (open leaf variety)	2 (7)	192 197	479 491	44 47	-0 0 3 7 14	leaf	0.39 3.7 1.0 0.090 <0.01	0.024 0.095 0.054 0.014 <0.01	0.41 3.8 1.1 0.10 <0.01	14-2185 (Unprotected)
Pozoblanco (Cordoba), Spain, 2014, Aitana (open leaf variety)	2 (7)	213 205	533 512	43 46	-0 0 3 7 14	leaf	0.11 3.3 2.4 1.1 0.051	0.016 0.034 0.027 0.013 <0.01	0.13 3.3 2.4 1.1 0.051	
Zafarraya, Spain, 2014, Isasa (Romana) (open leaf variety)	2 (7)	205 202	512 505	37 43	-0 0 3 7 14	leaf	1.7 5.3 3.2 2.1 0.65	0.057 0.080 0.039 0.041 0.018	1.8 5.4 3.2 2.1 0.67	

Location, year, variety LEAF LETTUCE	N (int)	Rate (g ai/ha)	Spray volume (L/ha)	GS	DALA	Sample	Trifloxystrobin (mg/kg)	CGA321113 (mg/kg)	Total (mg/kg)	Study No. (Protected/ Unprotected)
Austria	2 (7)	200	300-1000		7					Greenhouse/ Protected
Nea Magnisia, Greece, 2014, Manchester F1 (open leaf variety)	2 (7)	200 200	800 801	44 45	-0 0 3 7 14	leaf	0.044 3.9 1.9 0.61 0.054	0.019 0.060 0.081 0.028 <0.01	0.063 4.0 2.0 0.64 0.054	

"-0" = harvested before the last application.

[a] Trials were conducted in a greenhouse.

Legume Vegetables

Table 7 Residues of trifloxystrobin and CGA321113 (mg/kg) in beans with pods following two foliar applications of trifloxystrobin in a SC-formulation

Location, year, variety BEANS W/ PODS	N (int)	Rate (g ai/ha)	Spray volume (L/ha)	GS	DALA	Sample	Trifloxystrobin (mg/kg)	CGA321113 (mg/kg)	Total (mg/kg)	Study No.
Austria	2 (7)	200	400-600		7					Kidney beans
The NL	2 (14)	200	400-600		14					Beans with pods
Heimerzheim, Germany, 2010, Kidney bean, Orinoko (yellow French bean)	2 (7)	200 200	300 300	61 63	15 21 28	Beans with pod	0.02 <0.01 0.01	<0.01 <0.01 <0.01	0.02 <0.01 0.01	10-2125
Villers-Perwin, Belgium, 2010, Kidney bean, Beaufort	2 (7)	200 200	400 400	60 67	7 14 21 28	Beans with pod	0.03 0.02 0.01 <0.01	0.01 <0.01 <0.01 <0.01	0.04 0.02 0.01 <0.01	
Alginet, Spain, 2010, Kidney bean, Cleo (Dwarf bean; Judia enana)	2 (7)	200 200	500 500	71 75	-0 0 7 14 22 28	Beans with pod	0.11 0.33 0.11 0.14 0.34 0.11	<0.01 <0.01 <0.01 <0.01 0.01 0.01	0.11 0.33 0.11 0.14 0.35 0.12	
Ladispoli, Italy, 2010, Kidney bean, Pongo	2 (7)	200 200	500 500	63 71	-0 0 7 14 21 28	Beans with pod	<0.01 0.33 0.02 <0.01 <0.01 <0.01	<0.01 <0.01 0.01 <0.01 <0.01 <0.01	<0.01 0.33 0.030 <0.01 <0.01 <0.01	

Location, year, variety BEANS W/ PODS	N (int)	Rate (g ai/ha)	Spray volume (L/ha)	GS	DALA	Sample	Trifloxystrobin (mg/kg)	CGA321113 (mg/kg)	Total (mg/kg)	Study No.
Austria	2 (7)	200	400-600		7					Kidney beans
The NL	2 (14)	200	400-600		14					Beans with pods
Toulouse, France, 2010, Kidney bean, Argus (French bean)	2 (7)	200 200	500 500	65 72	-0 0 7 14 21 28	Beans with pod	<0.01 0.12 0.03 0.02 0.02 0.01	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01	<0.01 0.12 0.03 0.02 0.02 0.01	
Ribafria, Portugal, 2010, Kidney bean, Bolinhas (French bean)	2 (7)	212 212	530 530	75 75	-0 0 7 14 21 28	Beans with pod	0.06 0.22 0.07 0.09 0.03 0.03	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01	0.06 0.22 0.07 0.09 0.03 0.03	
Criquebeuf sur Seine, France, 2010, Kidney bean, Flagrano (flageolet)	2 (7)	200 200	300 300	73 77	-0 0 7	Beans with pod	0.12 0.50 0.21	0.021 0.021 0.021	0.14 0.52 0.23	10-2128
Damery, France, 2010, Kidney bean, Flagrano (flageolet)	2 (7)	200 200	300 300	75 76	-0 0 7	Beans with pod	0.01 0.24 0.16	0.01 0.01 0.031	0.02 0.25 0.19	
Toulouse - Croix daurade, France, 2011, Kidney bean, Argus french bean	2 (9)	200 200	500 500	66 73	-0 0 7 14 21 28	Beans with pod	0.025 0.43 0.045 0.023 0.019 0.013	0.019 0.040 0.014 <0.01 <0.01 <0.01	0.044 0.47 0.059 0.023 0.019 0.013	11-2001
Alginet, Spain, 2011, Kidney bean, Cleo dwarf bean	2 (7)	200 200	500 500	69 69	-0 0 7 14 21 30	Beans with pod	0.062 0.87 0.21 0.046 0.036 0.021	0.043 0.057 0.047 0.018 0.016 0.011	0.11 0.93 0.26 0.064 0.052 0.032	
Andria, Italy, 2011, Kidney bean, Blue lake	2 (7)	200 200	500 500	65 71	-0 0 7 14 21 28	Beans with pod	0.011 4.4 0.063 <0.01 <0.01 <0.01	<0.01 0.020 <0.01 <0.01 <0.01 <0.01	0.011 4.4 0.063 <0.01 <0.01 <0.01	

Location, year, variety BEANS W/ PODS	N (int)	Rate (g ai/ha)	Spray volume (L/ha)	GS	DALA	Sample	Trifloxystrobin (mg/kg)	CGA321113 (mg/kg)	Total (mg/kg)	Study No.
Austria	2 (7)	200	400-600		7					Kidney beans
The NL	2 (14)	200	400-600		14					Beans with pods
Atowia da Enreia, Portugal, 2011, Kidney bean, Bolinhas	2 (7)	200 200	500 500	75 75	-0 0 7 14 21 28	Beans with pod	0.079 0.40 0.12 0.050 0.030 0.030	<0.01 <0.01 <0.01 <0.01 <0.01 0.011	0.079 0.40 0.12 0.050 0.030 0.041	
Werl-Mawicke, Germany, 2012, French bean, Primel bean	2 (7)	200 200	300 300	63 69	7 10 14 21	Beans with pod	0.038 0.028 0.012 <0.01	0.013 <0.01 <0.01 <0.01	0.051 0.028 0.012 <0.01	12-2030
Fondettes, France, 2012, French bean, Contender	2 (7)	200 200	500 500	69 75	-0 0 7 10 14 21	Beans with pod	0.035 0.28 0.074 0.079 0.076 0.038	<0.01 <0.01 0.017 0.020 <0.01 <0.01	0.035 0.28 0.091 0.099 0.076 0.038	

"-0" = harvested before the last application.

Table 8 Residues of trifloxystrobin and CGA321113 (mg/kg) in peas with pods following two foliar applications of trifloxystrobin in a SC-formulation

Location, year, variety PEAS W/ PODS	N (int)	Rate (g ai/ha)	Spray volume (L/ha)	GS	DALA	Sample	Trifloxystrobin (mg/kg)	CGA321113 (mg/kg)	Total (mg/kg)	Study No.
France GAP	1	200			7					Peas with pod
Austria and the NL GAP	2 (14)	200	200-800		14					Peas with pods
Burscheid, Germany, 2012, Respect	2 (7)	200 200	300 300	65 71	-0 0 7	Peas w/ pod, fresh	0.081 0.26 0.063	<0.01 <0.01 <0.01	0.081 0.26 0.063	12-2031
Chaussy, France, 2012, Genial	2 (7)	200 200	300 300	73 75	-0 0 7	Peas w/ pod, fresh	0.046 0.30 0.075	<0.01 <0.01 <0.01	0.046 0.30 0.075	
Beucha-Wolfshain, Germany, 2012, Rocket	2 (7)	200 200	300 300	65 75	-0 0 7	Peas w/ pod, fresh	0.19 0.38 0.066	<0.01 <0.01 <0.01	0.19 0.38 0.066	
Villers-Perwin, Belgium, 2012, Ravenna	2 (7)	200 200	400 400	65 67-73	-0 0 7	Peas w/ pod, fresh	0.037 0.14 0.028	<0.01 <0.01 <0.01	0.037 0.14 0.028	

Location, year, variety PEAS W/ PODS	N (int)	Rate (g ai/ha)	Spray volume (L/ha)	GS	DALA	Sample	Trifloxystrobin (mg/kg)	CGA321113 (mg/kg)	Total (mg/kg)	Study No.
France GAP	1	200			7					Peas with pod
Austria and the NL GAP	2 (14)	200	200-800		14					Peas with pods
Cambridge, United Kingdom, 2012, Tommy	2 (9)	200 200	300 300	74 76	-0 0 6	Peas w/ pod, fresh	0.021 0.27 0.24	<0.01 <0.01 <0.01	0.021 0.27 0.24	
Langförden, Germany, 2012, Alvesta	2 (7)	200 200	300 300	69 76	-0 0 7	Peas w/ pod, fresh	0.054 0.15 0.029	<0.01 <0.01 <0.01	0.054 0.15 0.029	
Lapalud, France, 2012, Isard	2 (7)	200 200	400 400	70 72	-0 0 6	Peas w/ pod, fresh	0.063 0.42 0.28	<0.01 <0.01 <0.01	0.063 0.42 0.28	12-2032
Dos Hermanas, Spain, 2012, Cartouche	2 (7)	200 188	300 282	75 77	0 7	Peas w/ pod, fresh	0.27 0.25	<0.01 <0.01	0.27 0.25	
Ladispoli (RM), Italy, 2012, Attika	2 (7)	200 200	300 300	67 70	-0 0 7	Peas w/ pod, fresh	0.035 0.22 0.058	<0.01 <0.01 <0.01	0.035 0.22 0.058	
Nea Messimvria, Greece, 2012, LiVioletta	2 (7)	200 200	400 400	71 75	-0 0 7	Peas w/ pod, fresh	0.073 0.57 0.26	<0.01 <0.01 <0.01	0.073 0.57 0.26	
Alginet, Spain, 2012, Lincoln	2 (9)	200 200	500 500	74 75	-0 0 7	Peas w/ pod, fresh	0.20 0.58 0.41	<0.01 0.011 0.020	0.20 0.59 0.43	
Salobrena Granada, Spain, 2012, Utrillo	2 (7)	200 200	400 400	71 74	-0 0 6	Peas w/ pod, fresh	0.86 1.1 0.65	<0.01 <0.01 <0.01	0.86 1.1 0.65	12-2155
Malaga, Spain, 2012-2013, Utrillo	2 (7)	200 200	400 400	72 75	-0 0 7	Peas w/ pod, fresh	0.24 0.70 0.36	<0.01 <0.01 <0.01	0.24 0.70 0.36	
Papiana Marsciano, 2012, Gran Rugoso Tondo	2 (7)	200 200	500 500	74 78	-0 0 7	Peas w/ pod, fresh	0.30 0.73 0.36	0.019 0.018 0.018	0.32 0.75 0.38	
Abenraa, Denmark, 2015, Maxigold	2 (7)	208 208	312 312	65 71	0 7 13	Peas w/ pod, fresh	0.62 0.23 0.23	<0.01 <0.01 <0.01	0.62 0.23 0.23	15-2030
Kehl-Bodersweier, Germany, 2015, Astronaut	2 (7)	199 198	398 397	72 75	0 6	Peas w/ pod, fresh	0.63 0.49	<0.01 0.024	0.63 0.51	
Salobreña, Spain, 2015-2016, Dorian	2 (6)	196 208	391 415	73 75	0 7 13	Peas w/ pod, fresh	0.79 0.83 0.44	<0.01 0.013 0.011	0.79 0.84 0.45	

Location, year, variety PEAS W/ PODS	N (int)	Rate (g ai/ha)	Spray volume (L/ha)	GS	DALA	Sample	Trifloxystrobin (mg/kg)	CGA321113 (mg/kg)	Total (mg/kg)	Study No.
France GAP	1	200			7					Peas with pod
Austria and the NL GAP	2 (14)	200	200-800		14					Peas with pods
Mediglia, Italy, 2016, Nano Progress	2 (7)	204 196	409 391	71 73	0 7 14	Peas w/ pod, fresh	0.39 0.12 0.037	0.020 <0.01 <0.01	0.41 0.12 0.037	

"-0" = harvested before the last application.

Table 9 Residues of trifloxystrobin and CGA321113 (mg/kg) in bean without pods following two foliar applications of trifloxystrobin in a SC-formulation

Location, year, variety BEANS W/OUT PODS	N (int)	Rate (g ai/ha)	Spray volume (L/ha)	GS	DALA	Sample	Trifloxystrobin (mg/kg)	CGA321113 (mg/kg)	Total (mg/kg)	Study No.
Austria GAP	2 (7)	200	400-600		7					Kidney beans
Austria and the NL GAP	2 (14)	200	200-800		14					Beans without pods
Criquebeuf sur Seine, France, 2010, Kidney bean, Flagrano (flageolet)	2 (7)	200 200	300 300	73 77	14 21 28	Bean, seed, green	0.02 0.01 <0.01	<0.01 0.021 0.021	0.02 0.031 0.031	10-2128
Damery, France, 2010, Kidney bean, Flagrano (flageolet)	2 (7)	200 200	300 300	75 76	14 21 28	Bean, seed, green	0.04 0.03 0.02	0.021 0.031 0.031	0.061 0.061 0.051	

Table 10 Residues of trifloxystrobin and CGA321113 (mg/kg) in peas without pods following two foliar applications of trifloxystrobin in a SC-formulation

Location, year, variety PEAS W/OUT PODS	N (int)	Rate (g ai/ha)	Spray volume (L/ha)	GS	DALA	Sample	Trifloxystrobin (mg/kg)	CGA321113 (mg/kg)	Total (mg/kg)	Study No.
Austria and the NL GAP	2 (14)	200	200-800		14					
Burscheid, Germany, 2012, Respect	2 (7)	200 200	300 300	65 71	7 10 14 21	Pea, seed, green	<0.01 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01	12-2031

Location, year, variety PEAS W/OUT PODS	N (int)	Rate (g ai/ha)	Spray volume (L/ha)	GS	DALA	Sample	Trifloxystrobin (mg/kg)	CGA321113 (mg/kg)	Total (mg/kg)	Study No.
Austria and the NL GAP	2 (14)	200	200-800		14					
Chaussy, France, 2012, Genial	2 (7)	200 200	300 300	73 75	7 10 14 21	Pea, seed, green	0.010 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01	0.010 <0.01 <0.01 <0.01	
Beucha-Wolfshain, Germany, 2012, Rocket	2 (7)	200 200	300 300	65 75	7 10 14	Pea, seed, green	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	
Villers-Perwin, Belgium, 2012, Ravenna	2 (7)	200 200	400 400	65 67-73	7 10 14	Pea, seed, green	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	
Cambridge, United Kingdom, 2012, Tommy	2 (9)	200 200	300 300	74 76	6 10 13	Pea, seed, green	0.010 <0.01 <0.01	<0.01 <0.01 <0.01	0.010 <0.01 <0.01	
Langförden, Germany, 2012, Alvesta	2 (7)	200 200	300 300	69 76	7 10 13	Pea, seed, green	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	
Lapalud, France, 2012, Isard	2 (7)	200 200	400 400	70 72	6 10 14 21	Pea, seed, green	0.024 0.014 <0.01 0.013	<0.01 <0.01 <0.01 <0.01	0.024 0.014 <0.01 0.013	12-2032
Dos Hermanas, Spain, 2012, Cartouche	2 (7)	200 188	300 282	75 77	7 10 14	Pea, seed, green	0.014 <0.01 <0.01	<0.01 <0.01 0.017	0.014 <0.01 0.027	
Ladispoli (RM), Italy, 2012, Attika	2 (7)	200 200	300 300	67 70	7 10 14	Pea, seed, green	0.010 <0.01 <0.01	<0.01 <0.01 <0.01	0.010 <0.01 <0.01	
Nea Messimvria, Greece, 2012, LiVioletta	2 (7)	200 200	400 400	71 75	7 10 14	Pea, seed, green	0.032 0.014 0.010	<0.01 <0.01 <0.01	0.032 0.014 0.010	
Alginet, Spain, 2012, Lincoln	2 (9)	200 200	500 500	74 75	7 9 13	Pea, seed, green	0.039 0.028 0.028	<0.01 <0.01 0.013	0.039 0.028 0.041	
Salobrena Granada, Spain, 2012, Utrillo	2 (7)	200 200	400 400	71 74	6 9 14	Pea, seed, green	0.037 0.021 0.014	<0.01 <0.01 <0.01	0.037 0.021 0.014	12-2155
Malaga, Spain, 2012-2013, Utrillo	2 (7)	200 200	400 400	72 75	7 9 14	Pea, seed, green	0.020 0.016 0.018	<0.01 <0.01 <0.01	0.020 0.016 0.018	

Location, year, variety PEAS W/OUT PODS	N (int)	Rate (g ai/ha)	Spray volume (L/ha)	GS	DALA	Sample	Trifloxystrobin (mg/kg)	CGA321113 (mg/kg)	Total (mg/kg)	Study No.
Austria and the NL GAP	2 (14)	200	200-800		14					
Papiana Marsciano, 2012, Gran Rugoso Tondo	2 (7)	200 200	500 500	74 78	7 10 14	Pea, seed, green	0.032 0.020 0.012	<0.01 <0.01 <0.01	0.032 0.020 0.012	
Abenraa, Denmark, 2015, Maxigold	2 (7)	208 208	312 312	65 71	13 19	Pea, seed, green	0.012 <0.01	<0.01 <0.01	0.012 <0.01	15-2030
Kehl-Bodersweier Germany, 2015, Astronaut	2 (7)	199 198	398 397	72 75	6	Pea, seed, green	0.010	<0.01	0.010	
Salobreña, Spain, 2015-2016, Dorian	2 (6)	196 208	391 415	73 75	13 21	Pea, seed, green	0.037 0.011	<0.01 <0.01	0.037 0.011	
Mediglia, Italy, 2016, Nano Progress	2 (7)	204 196	409 391	71 73	14 21	Pea, seed, green	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	

Pulses

Table 11 Residues of trifloxystrobin and CGA321113 (mg/kg) in dry peas following two foliar applications of trifloxystrobin in a SC-formulation

Location, year, variety DRY PEAS	N (int)	Rate (g ai/ha)	Spray volume (L/ha)	GS	DALA	Sample	Trifloxystrobin (mg/kg)	CGA321113 (mg/kg)	Total (mg/kg)	Study No.
Austria and NL GAP	2 (14)	200	200-800		14					Peas with/without pods
Ambleville, France, 2011, Athos	2 (7)	200 200	300 300	80 80	14 21 28	Pea, seed, dry	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	11-2000
Burscheid, Germany, 2011, Mascara	2 (7)	200 200	300 300	79 80	14 21 28	Pea, seed, dry	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	
Burscheid, Germany, 2012, Respect	2 (7)	200 200	300 300	65 71	39	Pea, seed, dry	<0.01	<0.01	<0.01	12-2031
Chaussy, France, 2012, Genial	2 (7)	200 200	300 300	73 75	35	Pea, seed, dry	<0.01	<0.01	<0.01	
Beucha-Wolfshain, Germany, 2012, Rocket	2 (7)	200 200	300 300	65 75	21 43	Pea, seed, dry	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	

Location, year, variety DRY PEAS	N (int)	Rate (g ai/ha)	Spray volume (L/ha)	GS	DALA	Sample	Trifloxystrobin (mg/kg)	CGA321113 (mg/kg)	Total (mg/kg)	Study No.
Austria and NL GAP	2 (14)	200	200-800		14					Peas with/without pods
Villers-Perwin, Belgium, 2012, Ravenna	2 (7)	200 200	400 400	65 67-73	21 37	Pea, seed, dry	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	
Cambridge, United Kingdom, 2012, Tommy	2 (9)	200 200	300 300	74 76	20	Pea, seed, dry	<0.01	<0.01	<0.01	
Langförden, Germany, 2012, Alvesta	2 (7)	200 200	300 300	69 76	22 32	Pea, seed, dry	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	
Lapalud, France, 2012, Isard	2 (7)	200 200	400 400	70 72	40	Pea, seed, dry	<0.01	<0.01	<0.01	12-2032
Dos Hermanas, Spain, 2012, Cartouche	2 (7)	200 188	300 282	75 77	21	Pea, seed, dry	<0.01	0.013	0.023	
Ladispoli (RM), Italy, 2012, Attika	2 (7)	200 200	300 300	67 70	21	Pea, seed, dry	0.015	<0.01	0.015	
Nea Messimvria, Greece, 2012, LiVioletta	2 (7)	200 200	400 400	71 75	21 33	Pea, seed, dry	<0.01 <0.01	0.012 0.012	0.022 0.022	
Alginet, Spain, 2012, Lincoln	2 (9)	200 200	500 500	74 75	21	Pea, seed, dry	0.081	0.057	0.14	
Salobrena Granada, Spain, 2012, Utrillo	2 (7)	200 200	400 400	71 74	22	Pea, seed, dry	0.048	<0.01	0.048	12-2155
Malaga, Spain, 2012-2013, Utrillo	2 (7)	200 200	400 400	72 75	20 34	Pea, seed, dry	<0.01 0.015	<0.01 0.010	<0.01 0.025	
Papiana Marsciano, 2012, Gran Rugoso Tondo	2 (7)	200 200	500 500	74 78	21 28	Pea, seed, dry	0.093 0.014	0.013 <0.01	0.10 0.014	
Abenraa, Denmark, 2015, Maxigold	2 (7)	208 208	312 312	65 71	36	Pea, seed, dry	<0.01	0.011	0.021	15-2030
Kehl-Bodersweier, Germany, 2015, Astronaut	2 (7)	199 198	398 397	72 75	14 21	Pea, seed, dry	<0.01 <0.01	0.012 0.012	0.022 0.022	
Salobreña, Spain, 2015-2016, Dorian	2 (6)	196 208	391 415	73 75	29	Pea, seed, dry	0.029	0.028	0.057	
Mediglia, Italy, 2016, Nano Progress	2 (7)	204 196	409 391	71 73	48	Pea, seed, dry	<0.01	<0.01	<0.01	

Tree Nuts

Table 12 Residues of trifloxystrobin and CGA321113 (mg/kg) in tree nuts following three foliar applications of trifloxystrobin in a SC-formulation

Location, year, variety TREE NUT	N (int)	Rate (g ai/ha)	Spray volume (L/ha)	GS	DALA	Sample	Trifloxystrobin (mg/kg)	CGA321113 (mg/kg)	Total (mg/kg)	Study No.
US GAP	4 (7-21)	140	NS (grnd), ≥ 94 (air)		14					Max. rate is 560 g ai/ha/season
Almonds										
Orland, CA, USA 2010, Non-Pareil	3 (7, 7)	159 194 194	2000 2002 1998	79 79 89	0 7 14 21 28	Nutmeat	<0.01 <0.01 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01 <0.01	RAGMP168
	3 (7, 7)	161 198 198	468 468 467	79 79 89	0 7 14 21 28	Nutmeat	<0.01 <0.01 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01 <0.01	
Madera, CA, USA, 2010, Non-Pareil	3 (7, 7)	161 203 207	2076 2101 2138	78 79 79	14	Nutmeat	<0.01	<0.01	<0.01	
	3 (7, 7)	161 203 207	426 420 428	78 79 79	14	Nutmeat	<0.01	<0.01	<0.01	
Lost Hills, CA, USA, 2010, Monterey	3 (7, 7)	161 200 201	2282 2291 2279	85 85 85	14	Nutmeat	<0.01	<0.01	<0.01	
	3 (7, 7)	160 161 201	362 359 356	85 85 85	14	Nutmeat	<0.01	<0.01	<0.01	
Hickman, CA, USA, 2010, Nonpareil	3 (7, 7)	159 199 201	3072 2936 2989	81 85 85	14	Nutmeat	<0.01	0.016	0.03	
	3 (7, 7)	158 200 200	520 522 521	81 85 85	14	Nutmeat	<0.01	0.024	0.03	
Kerman, CA, USA, 2010, Padre	3 (8, 9)	158 200 203	2760 2797 2832	79 79 89	14	Nutmeat	<0.01	<0.01	<0.01	
	3 (8, 9)	161 202 194	496 499 478	79 79 89	14	Nutmeat	0.011	<0.01	0.011	
PECANS										

Location, year, variety TREE NUT	N (int)	Rate (g ai/ha)	Spray volume (L/ha)	GS	DALA	Sample	Trifloxystrobin (mg/kg)	CGA321113 (mg/kg)	Total (mg/kg)	Study No.
US GAP	4 (7-21)	140	NS (grnd), ≥ 94 (air)		14					Max. rate is 560 g ai/ha/season
Almonds										
Tifton, GA, USA, 2015, Sumner	3 (7, 7)	157 201 201	888 909 902	88 89 89	0 8 14 21 28	Nutmeat	0.017 <0.01 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01 <0.01	0.017 <0.01 <0.01 <0.01 <0.01	RATFN132
	3 (7, 7)	165 199 199	2162 2087 2084	88 89 89	0 8 14 21 28	Nutmeat	0.013 <0.01 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01 <0.01	0.013 <0.01 <0.01 <0.01 <0.01	
Chula, GA, USA, 2015, Pawnee	3 (5, 7)	157 211 192	817 812 868	88 88 89	14	Nutmeat	0.012	<0.01	0.012	
	3 (5, 7)	159 199 208	1878 1899 2174	88 88 --	14	Nutmeat	<0.01	<0.01	<0.01	
Bertrand, MO, USA, 2015, Pawnee	3 (7, 7)	163 202 202	763 753 757	78 79 81	14	Nutmeat	<0.01	<0.01	<0.01	
	3 (7, 7)	161 201 201	1884 1883 1881	78 79 81	14	Nutmeat	<0.01	<0.01	<0.01	
Pearsall, TX, USA, 2015, Cheyenne	3 (7, 7)	156 202 202	869 715 776	85 85 85	0 7 14 19 27	Nutmeat	0.016 <0.01 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01 <0.01	0.016 <0.01 <0.01 <0.01 <0.01	
	3 (7, 7)	156 202 202	1936 2444 2357	80 85 85	0 7 14 19 27	Nutmeat	<0.01 <0.01 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01 <0.01	
Levelland, TX, 2015, Western Schley	3 (8, 5)	158 197 198	828 829 832	85 85 85	14	Nutmeat	<0.01	<0.01	<0.01	
	3 (8, 5)	155 198 200	1904 1930 1948	85 85 85	14	Nutmeat	<0.01	<0.01	<0.01	

Flax

Table 13 Residues of trifloxystrobin and CGA321113 (mg/kg) in flax following one foliar application of trifloxystrobin in a SC-formulation

Location, year, variety FLAX	N (int)	Rate (g ai/ha)	Spray volume (L/ha)	GS	DALA	Sample	Trifloxystrobin (mg/kg)	CGA321113 (mg/kg)	Total (mg/kg)	Study No.
Canada GAP	1	132	50 (air), 100 (ground)		36					
York, NE, USA, 2016, Gold ND	1	132	140	65	35	Seed	<0.01	<0.01	<0.01	CEJAN014
Northwood, Grand Forks, ND, USA, 2016, CDC Neela	1	129	120	67	42	Seed	<0.01	<0.01	<0.01	
Saskatoon, SK, Canada, 2016, CDC Glass	1	134	120	69	29 35 42 49	Seed	0.10 0.040 0.040 0.011	0.0140 <0.01 <0.01 <0.01	0.11 0.040 0.040 0.011	
Taber, AB, Canada, 2016, CDC Glass	1	134	120	65-67	35	Seed	<0.01	<0.01	<0.01	
Carrington, Foster, ND, USA, 2016, CDC Neela	1	130	120	68	38	Seed	<0.01	<0.01	<0.01	
Outlook, SK, Canada, 2016, Bethune	1	133	110	69	34	Seed	0.28	0.011	0.29	
Hanley, SK, Canada, 2016,CDC Sanctuary	1	135	120	66-69	37	Seed	0.042	<0.01	0.042	
Rosthern, SK, Canada, 2016, Bethune	1	130	110	69	30 37 44 51	Seed	0.012 0.013 <0.01 0.01	<0.01 <0.01 <0.01 <0.01	0.013 0.013 <0.01 0.01	
Hepburn, SK, Canada, 2016, CDC Bethune	1	135	120	64-67	38	Seed	0.023	<0.01	0.023	
Alvena, SK, Canada, 2016, Bethune	1	134	110	69	37	Seed	0.015	<0.01	0.015	
Wakaw, SK, Canada, 2016, Bethune	1	131	110	69	35	Seed	0.054	0.01	0.055	

Coffee

Table 14 Residues of trifloxystrobin and CGA321113 (mg/kg) in coffee following three foliar applications (with adjuvant) of trifloxystrobin in a SC-formulation

Location, year, variety COFFEE	N (int)	Rate (g ai/ha)	Spray volume (L/ha)	GS	DALA	Sample	Trifloxystrobin (mg/kg)	CGA321113 (mg/kg)	Total (mg/kg)	Study No.
Brazil GAP	3 (21)	75 – 100	30-40 (air), 400-500 (ground)		30					0.25% v/v methylated soybean oil
Paulínia – SP, Brazil, 2012, Mundo novo	3 (20, 20)	96.9 100 102	400 400 400	77 81 83	0 15 30 45	Bean, dry (de-pulped)	<0.01 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01	F12-026
Araguari – MG, Brazil, 2012, Mundo novo	3 (20, 20)	99.0 102 101	400 400 400	75 79 81	0 15 30 45	Bean, dry (de-pulped)	0.04 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01	0.04 <0.01 <0.01 <0.01	
Ribeirão Preto – SP, Brazil, 2012, Catuaí	3 (20, 20)	103 103 96.4	400 400 400	77 77 85	0 15 30 45	Bean, dry (de-pulped)	0.01 <0.01 <0.01 <0.01	<0.01 <0.01 0.02 0.01	0.01 <0.01 0.03 0.02	
Cristais Paulista – SP, Brazil, 2012, Mundo novo	3 (20, 20)	102 102 105	400 400 400	81 81 85	30	Bean, dry (de-pulped)	<0.01	<0.01	<0.01	
Campinas – SP, Brazil, 2012, Catuaí Vermelho	3 (20, 20)	100 98.4 105	400 400 400	79 82 85	30	Bean, dry (de-pulped)	<0.01	0.01	0.02	
Ribeirão Preto – SP, Brazil, 2013, Catuaí Vermelho	3 (20, 20)	101 101 101	400 400 400	75 79 81	30 45 60	Bean, dry (de-pulped)	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	F13-018
Santo Antônio da Alegria – SP, Brazil, 2013, Mundo novo	3 (20, 20)	100 102 104	400 400 400	75 77 79	30 45 60	Bean, dry (de-pulped)	<0.01 0.01 <0.01	<0.01 <0.01 <0.01	<0.01 0.01 <0.01	

Animal Feeds

Legume Animal Feeds

Table 15 Residues of trifloxystrobin and CGA321113 (mg/kg) in bean forage following two foliar applications of trifloxystrobin in a SC-formulation

Location, year, variety BEAN FORAGE	N (int	Rate (g ai/ha)	Spray volume (L/ha)	GS	DALA	Sample	Trifloxystrobin (mg/kg)	CGA321113 (mg/kg)	Total (mg/kg)	Study No.
Austria GAP	2 (7)	200	400-600		7					Kidney beans
Heimerzheim, Germany, 2010, Kidney bean, Orinoko (yellow French bean)	2 (7)	200 200	300 300	61 63	-0 0 8	Bean, green material	0.92 6.3 0.70	0.08 0.16 0.072	1.0 6.5 0.77	10-2125
Villers-Perwin, Belgium, 2010, Kidney bean, Beaufort	2 (7)	200 200	400 400	60 67	-0 0	Bean, green material	1.5 6.1	0.072 0.11	1.6 6.2	
Werl-Mawicke, Germany, 2012, French bean, Primel bean	2 (7)	200 200	300 300	63 69	-0 0	Bean, green material	3.3 9.5	0.089 0.11	3.4 9.6	12-2030

"-0" = harvested before the last application.

Table 16 Residues of trifloxystrobin and CGA321113 (mg/kg) in pea vines following two foliar applications of trifloxystrobin in a SC-formulation

Location, year, variety PEA VINES	N (int	Rate (g ai/ha)	Spray volume (L/ha)	GS	DALA	Sample	Trifloxystrobin (mg/kg)	CGA321113 (mg/kg)	Total (mg/kg)	Study No.
Austria and ND GAP	2 (14)	200	200-800		14					Peas with/ without pods
Burscheid, Germany, 2012, Respect	2 (7)	200 200	300 300	65 71	-0 0 7 14 21	Pea, rest of plant	1.3 3.9 0.44 0.26 0.23	0.017 0.021 0.030 0.023 0.033	1.3 3.9 0.47 0.28 0.26	12-2031
Chaussy, France, 2012, Genial	2 (7)	200 200	300 300	73 75	-0 0 7 14 21	Pea, rest of plant	0.39 3.5 1.4 0.99 0.36	0.019 0.022 0.045 0.040 0.034	0.41 3.5 1.4 1.0 0.39	
Beucha-Wolfshain, Germany, 2012, Rocket	2 (7)	200 200	300 300	65 75	-0 0 7 14	Pea, rest of plant	1.4 3.1 1.6 1.0	0.011 0.011 0.023 0.022	1.4 3.1 1.6 1.0	

Location, year, variety PEA VINES	N (int)	Rate (g ai/ha)	Spray volume (L/ha)	GS	DALA	Sample	Trifloxystrobin (mg/kg)	CGA321113 (mg/kg)	Total (mg/kg)	Study No.
Austria and ND GAP	2 (14)	200	200-800		14					Peas with/ without pods
Villers-Perwin, Belgium, 2012, Ravenna	2 (7)	200 200	400 400	65 67-73	-0 0 7 14	Pea, rest of plant	1.3 3.9 1.0 0.25	0.011 0.013 0.038 0.021	1.3 3.9 1.0 0.27	
Cambridge, United Kingdom, 2012, Tommy	2 (9)	200 200	300 300	74 76	-0 0 6 13	Pea, rest of plant	0.32 7.7 7.1 5.8	0.058 0.070 0.18 0.31	0.38 7.8 7.3 6.1	
Langförden, Germany, 2012, Alvesta	2 (7)	200 200	300 300	69 76	-0 0 7 13	Pea, rest of plant	1.5 4.8 1.9 1.5	0.034 0.050 0.080 0.17	1.5 4.8 2.0 1.7	
Lapalud, France, 2012, Isard	2 (7)	200 200	400 400	70 72	-0 0 6 14 21	Pea, rest of plant	0.56 4.2 3.7 0.74 0.91	0.039 0.10 0.039 0.041 0.038	0.60 4.3 3.7 0.78 0.95	12-2032
Dos Hermanas, Spain, 2012, Cartouche	2 (7)	200 188	300 282	75 77	-0 0 7 14	Pea, rest of plant	4.3 8.6 5.5 3.7	0.053 0.052 0.13 0.22	4.4 8.7 5.6 3.9	
Ladispoli (RM), Italy, 2012, Attika	2 (7)	200 200	300 300	67 70	-0 0 7 14	Pea, rest of plant	0.70 4.0 2.7 5.4	0.022 0.028 0.049 0.12	0.72 4.0 2.7 5.5	
Nea Messimvria, Greece, 2012, LiVioletta	2 (7)	200 200	400 400	71 75	-0 0 7 14	Pea, rest of plant	0.58 3.8 2.8 0.90	0.041 0.062 0.099 0.13	0.62 3.9 2.9 1.0	
Alginet, Spain, 2012, Lincoln	2 (9)	200 200	500 500	74 75	-0 0 7 13	Pea, rest of plant	2.1 4.3 3.5 5.3	0.049 0.059 0.099 0.13	2.1 4.4 3.6 5.4	
Salobrena Granada, Spain, 2012, Utrillo	2 (7)	200 200	400 400	71 74	-0 0 6 14	Pea, rest of plant	5.6 8.8 6.7 5.6	0.028 0.032 0.047 0.048	5.6 8.8 6.7 5.6	12-2155
Malaga, Spain, 2012-2013, Utrillo	2 (7)	200 200	400 400	72 75	-0 0 7 14	Pea, rest of plant	1.4 3.6 1.8 2.2	0.019 0.023 0.032 0.086	1.4 3.6 1.8 2.3	

Location, year, variety PEA VINES	N (int)	Rate (g ai/ha)	Spray volume (L/ha)	GS	DALA	Sample	Trifloxystrobin (mg/kg)	CGA321113 (mg/kg)	Total (mg/kg)	Study No.
Austria and ND GAP	2 (14)	200	200-800		14					Peas with/ without pods
Papiana Marsciano, 2012, Gran Rugoso Tondo	2 (7)	200 200	500 500	74 78	-0 0 7 14	Pea, rest of plant	4.5 6.0 4.5 [a] 4.5	0.12 0.18 0.17 0.20	4.6 6.2 4.7 4.7	
Abenraa, Denmark, 2015, Maxigold	2 (7)	208 208	312 312	65 71	-0 0 7 13 19	Pea, rest of plant	2.8 6.3 4.6 2.8 1.9	0.013 0.020 0.084 0.11 0.11	2.8 6.3 4.7 2.9 2.0	15-2030
Kehl-Bodersweier, Germany, 2015, Astronaut	2 (7)	199 198	398 397	72 75	-0 0 6	Pea, rest of plant	0.49 3.9 3.2	0.036 0.055 0.12	0.53 4.0 3.3	
Salobreña, Spain, 2015-2016, Dorian	2 (6)	196 208	391 415	73 75	-0 0 7 13 21	Pea, rest of plant	4.3 8.8 11 11 3.9	0.039 0.047 0.094 0.082 0.063	4.3 8.8 11 11 4.0	
Mediglia, Italy, 2016, Nano Progress	2 (7)	204 196	409 391	71 73	-0 0 7 14 21	Pea, rest of plant	4.1 0.61 1.5 1.1 0.42	0.19 0.023 0.028 0.024 0.028	4.3 0.63 1.5 1.1 0.45	

"-0" = harvested before the last application.

[a] Residues of trifloxystrobin of 0.014 mg/kg detected in the control sample.

Table 17 Residues of trifloxystrobin and CGA321113 (mg/kg) in pea hay following two foliar applications of trifloxystrobin in a SC-formulation

Location, year, variety PEA HAY	N (int)	Rate (g ai/ha)	Spray volume (L/ha)	GS	DALA	Sample	Trifloxystrobin (mg/kg)	CGA321113 (mg/kg)	Total (mg/kg)	Study No.
Austria and ND GAP	2 (14)	200	200-800		14					
Alternative France GAP	1	200			21					
Ambleville, France, 2011, Athos	2 (7)	200 200	300 300	80 80	-0 0 0 [a] 7	Pea, pod dried	0.17 0.52 0.22 0.22	<0.01 <0.01 <0.01 <0.01	0.17 0.52 0.22 0.22	11-2000

Location, year, variety PEA HAY	N (int)	Rate (g ai/ha)	Spray volume (L/ha)	GS	DALA	Sample	Trifloxystrobin (mg/kg)	CGA321113 (mg/kg)	Total (mg/kg)	Study No.
Austria and ND GAP	2 (14)	200	200-800		14					
Alternative France GAP	1	200			21					
Burscheid, Germany, 2011, Mascara	2 (7)	200 200	300 300	79 80	-0 0 7	Pea, pod dried	0.063 0.91 0.15	<0.01 <0.01 <0.01	0.063 0.91 0.15	
Burscheid, Germany, 2012, Respect	2 (7)	200 200	300 300	65 71	39	Pea, straw	0.40	0.049	0.45	12-2031
Chaussy, France, 2012, Genial	2 (7)	200 200	300 300	73 75	35	Pea, straw	0.97	0.16	1.1	
Beucha-Wolfshain, Germany, 2012, Rocket	2 (7)	200 200	300 300	65 75	21 43	Pea, straw	0.88 1.2	0.052 0.16	0.93 1.4	
Villers-Perwin, Belgium, 2012, Ravenna	2 (7)	200 200	400 400	65 67-73	21 37	Pea, straw	0.31 1.3	0.051 0.47	0.36 1.8	
Cambridge, United Kingdom, 2012, Tommy	2 (9)	200 200	300 300	74 76	20	Pea, straw	6.5	1.4	7.9	
Langförden, Germany, 2012, Alvesta	2 (7)	200 200	300 300	69 76	22 32	Pea, straw	2.2 5.1	0.30 1.7	2.5 6.8	
Lapalud, France, 2012, Isard	2 (7)	200 200	400 400	70 72	40	Pea, straw	1.9	0.18	2.1	12-2032
Dos Hermanas, Spain, 2012, Cartouche	2 (7)	200 188	300 282	75 77	21	Pea, straw	3.8	0.17	4.0	
Ladispoli (RM), Italy, 2012, Attika	2 (7)	200 200	300 300	67 70	21	Pea, straw	8.7	0.26	9.0	
Nea Messimvria, Greece, 2012, LiVioletta	2 (7)	200 200	400 400	71 75	21 33	Pea, straw	1.1 1.4	0.10 0.43	1.2 1.8	
Alginet, Spain, 2012, Lincoln	2 (9)	200 200	500 500	74 75	21	Pea, straw	13	0.79	14	
Salobrena Granada, Spain, 2012, Utrillo	2 (7)	200 200	400 400	71 74	22	Pea, straw	9.9	0.060	10	12-2155
Malaga, Spain, 2012-2013, Utrillo	2 (7)	200 200	400 400	72 75	20 34	Pea, straw	3.7 6.0	0.052 0.11	3.8 6.1	
Papiana Marsciano, 2012, Gran Rugoso Tondo	2 (7)	200 200	500 500	74 78	21 28	Pea, straw	8.1 6.3	0.37 0.40	8.5 6.7	

"-0" = harvested before the last application.

[a] Additional 0 DALA sample was taken in the afternoon after the morning rainfall.

Almond Hulls

Table 18 Residues of trifloxystrobin and CGA321113 (mg/kg) in almond hulls following three foliar applications of trifloxystrobin in a SC-formulation

Location, year, variety ALMOND HULLS	N (int)	Rate (g ai/ha)	Spray volume (L/ha)	GS	DALA	Sample	Trifloxystrobin (mg/kg)	CGA321113 (mg/kg)	Total (mg/kg)	Study No.
US GAP	4 (7-21)	140	NS (grnd), 94 (air)		14					Max. rate of 560 g ai/ha/season
Orland, CA, USA 2010, Non-Pareil	3 (7, 7)	159 194 194	2000 2002 1998	79 79 89	0 7 14 21 28	Hull	1.9 0.056 0.55 1.5 0.70	<0.01 <0.01 <0.01 <0.01 <0.01	1.9 0.056 0.55 1.5 0.70	RAGMP168
	3 (7, 7)	161 198 198	468 468 467	79 79 89	0 7 14 21 28	Hull	0.82 0.037 0.87 1.4 0.51	<0.01 <0.01 0.019 <0.01 0.010	0.82 0.037 0.89 1.4 0.51	
Madera, CA, USA, 2010, Non-Pareil	3 (7, 7)	161 203 207	2076 2101 2138	78 79 79	14	Hull	1.5	0.035	1.6	
	3 (7, 7)	161 203 207	426 420 428	78 79 79	14	Hull	2.4	0.036	2.4	
Lost Hills, CA, USA, 2010, Monterey	3 (7, 7)	161 200 201	2282 2291 2279	85 85 85	14	Hull	<0.01	0.29	0.30	
	3 (7, 7)	160 161 201	362 359 356	85 85 85	14	Hull	<0.01	0.098	0.11	
Hickman, CA, USA, 2010, Nonpareil	3 (7, 7)	159 199 201	3072 2936 2989	81 85 85	14	Hull	0.012	3.7	3.7	
	3 (7, 7)	158 200 200	520 522 521	81 85 85	14	Hull	<0.01	2.3	2.3	
Kerman, CA, USA, 2010, Padre	3 (8, 9)	158 200 203	2760 2797 2832	79 79 89	14	Hull	3.0	0.062	3.1	
	3 (8, 9)	161 202 194	496 499 478	79 79 89	14	Hull	4.6	0.064	4.7	

FATE OF RESIDUES IN STORAGE AND PROCESSING

Residues after processing

Beans with Pods

Two processing trials on beans with pods were conducted in Europe during 2002 (Nüßlein and Eberhardt, 2003, RA-3037/02; Nüßlein and Fischer, 2003, RA-2037/02). Trifloxystrobin, formulated as a WG (containing 50% trifloxystrobin) was applied to climbing French beans in the greenhouse as three foliar applications at 125 g ai/ha with an interval of 7 days between applications. Fresh beans with pods were harvested 1 day after the last application (DALA) and were washed in standing water under slow movement. After washing, the beans were cut in small pieces and cooked for 15–20 minutes in salt water. Samples of washing water and cooking water were also taken.

All samples were stored frozen for a maximum of 173 days and then analysed for residues of trifloxystrobin and CGA 321113 using the validated method 00742/E001.

Table 19 Residues of trifloxystrobin and CGA321113 (mg/kg) in beans with pods processed commodities (Nüßlein and Eberhardt, 2003, RA-3037/02; Nüßlein and Fischer, 2003, RA-2037/02)

Location, year, variety BEANS W/ PODS	N (int)	Rate (g ai/ha)	DALA	Sample	Trifloxystrobin [a], mg/kg [mean]	CGA321113 [a, b], mg/kg [mean]	Total, mg/kg [mean]	PFtri	PFtot
Langenfeld Reusrath, Germany, 2002, Markant	3 (7, 7)	125 125 125	1	Bean w/ pod	0098	<0.02	0098	-	-
				Cooked bean	0.032, 0.041 [0.037]	<0.02, <0.02 [<0.02]	0.032, 0.041 [0.037]	0.38	0.38
				Washed bean	0.046, 0.060 [0.053]	<0.02, <0.02 [<0.02]	0.046, 0.060 [0.053]	0.54	0.54
				Washing water	0.035, 0.073 [0.054]	<0.02, <0.02 [<0.02]	0.035, 0.073 [0.054]	-	-
				Cooking water	<0.02, <0.02 [<0.02]	<0.02, <0.02 [<0.02]	<0.02, <0.02 [<0.02]	-	-
Palidoro-Fiumicino, Italy, 2002, Emerite	3 (7, 7)	125 125 125	1	Bean w/ pod	0.184	<0.02	0.184	-	-
				Cooked bean	0.148, 0.134 [0.141]	<0.02, <0.02 [<0.02]	0.148, 0.134 [0.141]	0.77	0.77
				Washed bean	0.103, 0.136 [0.120]	<0.02, <0.02 [<0.02]	0.103, 0.136 [0.120]	0.65	0.65
				Washing water	0.047, 0.036 [0.042]	<0.02, <0.02 [<0.02]	0.047, 0.036 [0.042]	-	-
				Cooking water	<0.02, <0.02 [<0.02]	<0.02, <0.02 [<0.02]	<0.02, <0.02 [<0.02]	-	-

[a] All values are the means of duplicate analyses except for the RAC "bean with pod".

[b] CGA321113 determined as CGA321113 and calculated as trifloxystrobin

PFtri = processing factor for trifloxystrobin

PFtot = processing factor for the sum of trifloxystrobin and CGA321113

Flax

Two processing trials on flax were conducted in Canada during the 2016 growing season (Ardiel, 2017, CEJAN016). Trifloxystrobin, formulated as an SC formulation (containing 150 g trifloxystrobin/L and 175 g prothioconazole/L) was applied as a single foliar broadcast application at 672–674 g trifloxystrobin/ha. Samples of flax seed were collected 85–92 days after the last application (DALA). Seeds were processed into meal, cold pressed flax oil, and solvent extracted crude oil using methods which are representative of commercial practice.

Flax seed samples were weighed (26.4–32.1 kg) and dried in an oven at 54–71 °C to a moisture content of 7–10%. Following drying, samples were cleaned by aspiration and screening.

The moisture content of the flax seed was adjusted to 14% with water, flaked in flaking rollers, and then mechanically pressed to remove a portion of the crude oil. The resulting fractions were pressed crude oil and presscake/meal (with residual crude oil).

The pressed crude oil was filtered and then heated and mixed under vacuum to 85–99 °C and held for 20–30 minutes to remove residual water from the crude oil (drying).

The presscake with residual oil was ground using a mill. The ground presscake was placed in stainless steel batch extractors and submerged in hexane at 49–60 °C. After 30 minutes, the miscella (crude oil and hexane) was drained and hexane added to repeat the cycle two more times at the same temperature range for 15 minutes each. After the final draining, the extracted material (meal) was desolventized with warm air resulting in flaxseed meal.

The miscella was passed through a laboratory vacuum evaporator to separate the crude oil and hexane. The crude oil was then heated to 73–91 °C to remove the hexane and the resulting fraction was the solvent extracted crude oil.

Samples of seed, meal and oil were stored frozen for a maximum of 53 days and then analysed for residues of trifloxystrobin and CGA 321113 using the validated method TF-003-P16-01.

Table 20 Residues of trifloxystrobin and CGA321113 (mg/kg) in flax seed processed commodities (means of three individual analyses) (Ardiel, 2017, CEJAN016)

Location, year, variety FLAX SEED	N	Rate (g ai/ha)	DALA	Sample	Trifloxystrobin, mg/kg	CGA321113, mg/kg [a]	Total, mg/kg	PFtri	PFtot
Hanley, SK, Canada, 2016, CDC Sanctuary	1	674	92	Seed	0.0663	0.0198 [b]	0.0861	-	-
				Meal	<0.01	0.0906	0.1006	<0.15	1.2
				Oil, pressed	0.0544	0.0221	0.0765	0.82	0.89
				Oil, solvent extracted	0.0399	0.0171	0.057	0.60	0.66
Rosthern, SK, Canada, 2016, Bethune	1	672	89	Seed	0.1600	0.0193	0.1793	-	-
				Meal	0.0227	0.0368	0.0595	0.14	0.33
				Oil, pressed	0.1331	<0.01	0.1331	0.83	0.74
				Oil, solvent extracted	0.1573	<0.01	0.1573	0.98	0.88

[A] CGA321113 determined as CGA321113 and calculated as trifloxystrobin

[B] One of the control samples had observed residues of 0.0104 mg/kg. No explanation was provided for the observed residues.

PFtri = processing factor for trifloxystrobin

PFtot = processing factor for the sum of trifloxystrobin and CGA321113

Coffee

One processing trial on coffee was conducted in Brazil during the 2009 growing season (Hoag, 2010, RATFL003). Trifloxystrobin, formulated as an SC formulation (containing 375 g trifloxystrobin/L and 160 g cyproconazole) was applied as two foliar airblast applications at 745–749 g trifloxystrobin/ha, with an application interval of 38 days, totalling 1494 g trifloxystrobin/ha. Samples of ripe coffee cherries were hand harvested 28 days after the last application (DALA). The cherries were air dried for 21 days and the outer hulls were removed. The hulled coffee green bean samples were processed into instant coffee and roasted coffee using methods which are representative of commercial practice.

Green bean samples (22.60–24.98 kg) were roasted in an oven for 6 minutes at 215–245 °C and allowed to cool prior to milling. A sample of the milled ground coffee was screened for 5 minutes using US #12, 16, 20, and 30 mesh screens. The resulting fraction was the ground roasted coffee.

The ground roasted coffee was brewed using a series of 4 pots. The ground roasted coffee was transferred counter current to the liquid. The brewed ground roasted coffee was pressed to separate the spent grounds from the coffee extract. The coffee extract was then cycled back into the brewing process. The coffee extract was centrifuged for 5 minutes at ~1500 rpm to remove fines and the extract was then frozen in 1" deep freezing trays. After freezing, the trays were placed in a freeze dryer until a vacuum of 20 milli Torr or less and a condenser temperature of -62 °C or less was achieved. The freeze-dried coffee was milled and the resulting fraction was the instant coffee.

Samples of green bean, instant coffee, and ground roasted coffee were stored frozen for a maximum of 101 days and then analysed for residues of trifloxystrobin and CGA 321113 using the validated method 00765.

Table 21 Residues of trifloxystrobin and CGA321113 (mg/kg) in coffee bean processed commodities (means of three individual analyses) (Ardiel, 2017, CEJAN016)

Location, year, variety COFFEE BEAN	N	Rate (g ai/ha)	DALA	Sample	Trifloxystrobin, mg/kg	CGA321113, mg/kg [a]	Total, mg/kg	PFtri	PFtot
Rio Claro, São Paulo, Brazil, 2009, Obata IAC-1669-20	2	749 745	28	Coffee green beans	<0.01	0.0565	0.0656	-	-
				Coffee Instant	<0.01	<0.01	<0.01	-[b]	0.15
				Coffee Roasted	<0.01	<0.01	<0.01	-[b]	0.15

[A] CGA321113 determined as CGA321113 and calculated as trifloxystrobin

[B] No processing factors for instant or roasted coffee could be derived for residues of trifloxystrobin since no quantifiable residues were observed in the RAC (green beans), despite the use of exaggerated application rates (approximately 2.5x the maximum label rate).

PFtri = processing factor for trifloxystrobin

PFtot = processing factor for the sum of trifloxystrobin and CGA321113

APPRAISAL

Trifloxystrobin is a strobilurin broad-spectrum contact fungicide that was first evaluated for toxicology and residues by the JMPR in 2004. The Meeting derived an ADI of 0–0.04 mg/kg bw and concluded that an ARfD is unnecessary. The residue definition for compliance with the MRL for plant commodities is trifloxystrobin *per se* and for compliance with the MRL for animal commodities as well as for dietary risk assessment for both plant and animal commodities is the sum of trifloxystrobin and (*E,E*)-methoxyimino-{2-[1-(3-trifluoromethyl-phenyl) ethylidene-aminooxymethyl]-phenyl}acetic acid = CGA321113) (expressed as trifloxystrobin equivalents). The residue is fat-soluble.

Trifloxystrobin was scheduled at the Fifty-first Session of the CCPR for the evaluation of additional uses by the 2020 JMPR, which was postponed to the 2021 Extra JMPR, for citrus fruits, cane berries, bush berries, leaf lettuce, legume vegetables, dry beans and peas, tree nuts, linseed and coffee.

Methods of analysis

Methods 01013, 01313, 00765 and 200177, which were previously evaluated by the 2004, 2012 and 2015 JMPR, were used for the analysis of trifloxystrobin and CGA321113 in citrus fruits, tree nuts, head lettuce, coffee beans, fresh beans and dry peas. The current Meeting received study reports and method validation data for method 01313/M001 and QuEChERS multiresidue method 01207 which were used for the analysis of trifloxystrobin and CGA321113 in raspberries, currants, lettuce, peas and beans. The current Meeting also received concurrent recovery data for analytical methods GIR/MET/TRIFLOXY/01, TF-003-P16-01 and 00742/E001 for the analysis of trifloxystrobin and CGA321113 in raspberries, currants, flax and beans with pods. In general, the new methods considered by the current Meeting involved extraction with acetonitrile/water and final determination was achieved using LC-MS/MS. Among all available methods, the validated LOQ for trifloxystrobin and CGA321113 ranged from 0.005–0.02 mg/kg for each analyte. Mean recoveries were, with a few exceptions, within the acceptable range of 70–120% with a RSD of < 20%.

The Meeting concluded that for the commodities considered by the Meeting, the methods used in the new residue trials were sufficiently validated and suitable to measure trifloxystrobin and CGA321113 in plant commodities.

Stability of pesticide residues in stored analytical samples

The stability of residues in samples on frozen storage was evaluated by the 2004 JMPR for a range of commodities. Samples in the trials considered by the current Meeting were stored frozen for periods less than the period of stability demonstrated in studies supplied to the 2004 JMPR and were satisfactory.

Results of supervised residue trials on crops

The Meeting received information on supervised field trials on citrus (orange, lemon, grapefruit), raspberry, currant, leaf lettuce, beans (fresh), peas (fresh and dried), tree nuts (almond and pecan), linseed and coffee.

Residues for maximum residue level estimation are expressed in mg trifloxystrobin/kg. Residues for dietary risk assessment include parent trifloxystrobin and metabolite CGA321113. The sum of trifloxystrobin and CGA321113 was calculated and expressed as trifloxystrobin on the basis of the relative molecular masses. A conversion factor of 1.036 is required to express CGA321113 as trifloxystrobin. As CGA321113 does not generally constitute a significant proportion of the residue in crops, when the levels of trifloxystrobin or CGA321113 were below the LOQ, their sum was calculated as in the examples provided by the 2004 JMPR and copied below.

Trifloxystrobin (mg/kg)	CGA321113 (mg/kg)	Total (expressed as trifloxystrobin) (mg/kg)

< 0.02	< 0.02	< 0.02
< 0.02	0.03	0.05
0.10	< 0.02	0.10
0.92	0.16	1.1

Citrus Fruits

Citrus fruits were previously evaluated by the 2004 JMPR where a maximum residue level of 0.5 mg/kg and an STMR of 0.095 mg/kg were estimated based on the GAP from the USA, comprising 4 foliar applications at 140 g ai/ha with a PHI of 30 days. This recommendation was based on residue trials from the USA on oranges, lemons and grapefruits (4 × 170 g ai/ha, 26–32-day PHIs).

The current Meeting received a new critical GAP for the USA involving a shorter PHI of 7 days and a minimum re-treatment interval of 7 days. No trials provided to the Meeting matched this GAP with respect to the RTI. The RTI used in the citrus supervised residue trials provided to the Meeting (i.e. 21-days) is significantly longer than the 7-day RTI from the critical GAP. Residue decline data provided and available to the Meeting for citrus fruits indicated that the trials underestimate residues expected in citrus fruits by more than 25% when treated according to the critical GAP.

As the Meeting received a new critical GAP that was not supported by the available field trials and no suitable GAP from another country was provided, following current assessment practices the Meeting withdrew its previous recommendation for citrus fruits.

Cane berries

Raspberries

The critical GAP for protected cane berries in the Netherlands and field grown raspberries and blackberries in Austria and Portugal is 2 foliar applications (SC formulation) × 200 g ai/ha, 7-day RTI and 3-day PHI.

The Meeting received supervised residue trials conducted in Europe on raspberries (protected and unprotected) matching the critical GAP.

For estimation of maximum residue levels, residue levels of trifloxystrobin (parent only) in ranked order were:

Raspberry (protected) (n = 10): 0.086, 0.17, 0.25, 0.33, 0.48, 0.51, 0.52, 0.57, 0.98 and 1.4 mg/kg.

Raspberry (unprotected) (n = 8): 0.098, 0.20, 0.35, 0.36, 0.64, 0.70, 1.0 and 1.5 mg/kg.

For estimation of STMRs, total residue levels of trifloxystrobin and CGA321113 in ranked order were:

Raspberry (protected) (n = 10): 0.10, 0.18, 0.29, 0.38, 0.53, 0.59, 0.60, 0.62, 1.1 and 1.4 mg/kg.

Raspberry (unprotected) (n = 8): 0.12, 0.23, 0.37, 0.39, 0.71, 0.80, 1.1 and 1.5 mg/kg.

The Mann-Whitney U-test determined that the protected and unprotected raspberry datasets were not statistically different. Therefore, the Meeting decided to combine the datasets of protected and unprotected raspberries.

Trifloxystrobin (parent only) residues in raspberries (protected and unprotected) in ranked order were (n = 18): 0.086, 0.098, 0.17, 0.20, 0.25, 0.33, 0.35, 0.36, 0.48, 0.51, 0.52, 0.57, 0.64, 0.70, 0.98, 1.0, 1.4 and 1.5 mg/kg.

Total residue levels of trifloxystrobin and CGA321113 (protected and unprotected) in raspberries in ranked order were (n = 18): 0.10, 0.12, 0.18, 0.23, 0.29, 0.37, 0.38, 0.39, 0.53, 0.59, 0.60, 0.62, 0.71, 0.80, 1.1 (2), 1.4 and 1.5 mg/kg.

Noting that raspberry is a representative commodity for the cane berries subgroup and that the Netherlands GAP includes all commodities in this subgroup, the Meeting estimated a maximum residue level of 3 mg/kg and an STMR value of 0.56 mg/kg for the subgroup of cane berries.

Bush berries

Currants

The critical GAP for protected berries (including currants, gooseberries, blueberries, bilberries, foxberries and rose hips) from the Netherlands and field-grown currants, gooseberries and blueberries from Austria and Spain is 2 foliar applications (SC formulation) × 200 g ai/ha, 7-day RTI and 7-day PHI.

The Meeting received supervised residue trials conducted in Europe on currants (protected and unprotected) matching the critical GAP.

For estimation of maximum residue levels, residue levels of trifloxystrobin (parent only) in ranked order were:

Currants (protected) (n = 8): 0.15, 0.27, 0.31 (2), 0.35, 0.36, 0.51 and 0.77 mg/kg.

Currants (unprotected) (n = 8): 0.13, 0.14, 0.22, 0.29, 0.46, 0.64, 1.2 and 2.1 mg/kg.

For estimation of STMRs, total residue levels of trifloxystrobin and CGA321113 in ranked order were:

Currants (protected) (n = 8): 0.15, 0.27, 0.31 (2), 0.35, 0.36, 0.51 and 0.77 mg/kg.

Currants (unprotected) (n = 8): 0.13, 0.14, 0.22, 0.29, 0.46, 0.65, 1.2 and 2.1 mg/kg.

The Mann-Whitney U-test determined that the protected and unprotected currants datasets were not statistically different. Therefore, the Meeting decided to combine the datasets of protected and unprotected currants.

Trifloxystrobin (parent only) residues in currants (protected and unprotected) in ranked order were (n = 16): 0.13, 0.14, 0.15, 0.22, 0.27, 0.29, 0.31 (2), 0.35, 0.36, 0.46, 0.51, 0.64, 0.77, 1.2 and 2.1 mg/kg.

Total residue levels of trifloxystrobin and CGA321113 in currants in ranked order were (n = 16): 0.13, 0.14, 0.15, 0.22, 0.27, 0.29, 0.31 (2), 0.35, 0.36, 0.46, 0.51, 0.65, 0.77, 1.2 and 2.1 mg/kg.

Noting that currants is a representative commodity for the bush berries subgroup and that the Netherlands GAP includes currants, gooseberries, blueberries, bilberries, foxberries (red bilberries) and rose hips, the Meeting estimated a maximum residue level of 3 mg/kg and an STMR value of 0.33 mg/kg for currants, blueberries, gooseberries, billberries, red bilberries and rose hips.

Lettuce, leaf

The critical GAP for lettuce is from Austria on protected lettuce; 2 foliar applications (SC formulation) at 200 g ai/ha, 7-day RTI, with a 7-day PHI. The Meeting received supervised residue trials conducted in Europe on protected leaf lettuce matching the critical GAP.

For estimation of maximum residue levels, residue levels of trifloxystrobin (parent only) in ranked order were:

Lettuce, leaf (protected) (n = 13): 0.85, 1.2, 1.3, 1.9, 2.6, 3.1, 3.2, 3.5, 3.8, 4.5, 6.2, 8.6 and 9.9 mg/kg.

For estimation of STMRs, total residue levels of trifloxystrobin and CGA321113 in ranked order were:

Lettuce, leaf (protected) (n = 13): 0.90, 1.3 (2), 1.9, 2.8, 3.2, 3.3, 3.5, 3.8, 4.7, 6.7, 8.7 and 10 mg/kg.

The Meeting estimated a maximum residue level of 15 mg/kg and STMR of 3.3 mg/kg for leaf lettuce. The Meeting noted that the GAP in Austria also covers protected lambs lettuce (corn salad). As leaf lettuce is a representative leafy greens commodity, the Meeting agreed to extrapolate the maximum residue level of 15 mg/kg and an STMR of 3.3 mg/kg to corn salad based on the dataset of protected leaf lettuce.

Legume vegetables

Subgroup of beans with pods:

The critical GAP for kidney beans (common bean (poroto)) is from Austria; 2 foliar applications (SC formulation) at 200 g ai/ha, with a 7-day RTI and 7-day PHI.

The Meeting received supervised residue trials conducted in Europe on beans with pods matching the critical GAP.

Trifloxystrobin (parent only) residues in beans with pods in ranked order were (n = 13): 0.02, 0.03 (2), 0.038, 0.045, 0.063, 0.079, 0.09, 0.12, 0.16, 0.21 (2) and 0.34 mg/kg.

Total residue levels of trifloxystrobin and CGA321113 in beans with pods in ranked order were (n = 13): 0.03 (2), 0.04, 0.051, 0.059, 0.063, 0.09, 0.099, 0.12, 0.19, 0.23, 0.26 and 0.35 mg/kg.

The Meeting considered the data would support a maximum residue level of 0.5 mg/kg and an STMR of 0.09 mg/kg for common bean (poroto).

The Meeting noted a GAP for the subgroup of beans with pods from the Netherlands; 2 foliar applications (SC formulation) × 200 g ai/ha, 14-day RTI and 14-day PHI.

The Meeting also received supervised residue trials conducted in Europe on beans with pods approximating the Netherlands GAP but with RTIs of 7-days. Although the residue trials have a shorter RTI compared to the Netherlands GAP, based on residue decline data for beans with pods the Meeting estimated that residues from the field trials would be within 25% of the Netherlands GAP.

Trifloxystrobin (parent only) residues in beans with pods in ranked order were (n = 10): < 0.01 (2), 0.012, 0.02 (2), 0.023, 0.046, 0.05, 0.09 and 0.34 mg/kg.

Total residue levels of trifloxystrobin and CGA321113 in beans with pods in ranked order were (n = 10): < 0.01 (2), 0.012, 0.02 (2), 0.023, 0.05, 0.064, 0.09 and 0.35 mg/kg.

The Meeting considered the data would support a maximum residue level of 0.5 mg/kg and an STMR of 0.022 mg/kg based on the Netherlands GAP.

Noting that the above estimates are the same the Meeting decided to recommend a maximum residue level of 0.5 mg/kg and an STMR of 0.09 mg/kg for the subgroup of beans with pods.

Subgroup of peas with pods

Based on the available residue data, the Meeting concluded that the critical GAP for peas with pods is from France; 1 foliar application using an SC formulation, at 200 g ai/ha, with a PHI of 7 days.

The Meeting received supervised residue trials conducted in Europe on peas with pods, where 2 foliar spray applications (SC formulation) were made at a nominal rate of 200 g ai/ha per application and a 7-day RTI. In 13 trials, peas with pods were harvested at a "-0" day PHI (i.e. just before

the last application; 7 days after the first application) which matches the critical GAP from France. As the level of CGA321113 was below the LOQ in all samples, the data populations for enforcement and risk assessment purposes are identical.

Trifloxystrobin (and total trifloxystrobin and CGA321113) residues in peas with pods in ranked order were (n = 13): 0.021, 0.035, 0.037, 0.046, 0.054, 0.063, 0.073, 0.081, 0.19, 0.20, 0.24, 0.30 and 0.86 mg/kg.

The Meeting estimated a maximum residue level of 1.5 mg/kg and an STMR value of 0.073 mg/kg for the subgroup of peas with pods.

Subgroup of succulent beans without pods

The critical GAP for succulent kidney beans without pods is from Austria; 2 foliar applications (SC formulation) at 200 g ai/ha, with a 7-day RTI and a 7-day PHI.

The Meeting received two supervised residue trials conducted in Europe on beans without pods that differed from the critical GAP with regards to the pre-harvest interval. As there were no trials conducted in accordance with the critical GAP and there was an insufficient number of trials to support any other GAP provided for succulent beans without pods, the Meeting did not estimate a maximum residue level or STMR for the subgroup of succulent beans without pods.

Subgroup of succulent peas without pods

The critical GAPs for succulent peas without pods are from Austria and the Netherlands; 2 foliar applications (SC formulation) at 200 g ai/ha, with a 14-day RTI and a 14-day PHI.

The Meeting received supervised residue trials conducted in Europe on peas without pods that differed from the critical GAP with regards to the re-treatment interval (i.e. 7-day RTIs in the trials versus the 14-day RTI from the critical GAP). The residue decline data for succulent peas without pods were insufficient to determine whether the residues in the trials approximated residues according to the critical GAPs.

As there were no trials conducted in accordance with the critical GAP or any other GAP provided for succulent peas without pods, the Meeting did not estimate a maximum residue level or STMR for the subgroup of succulent peas without pods.

Pulses

Subgroup of dry beans and Subgroup of dry peas

The critical GAP for the subgroup of dry beans and subgroup of dry peas is from Austria and the Netherlands; 2 foliar applications (SC formulation) at 200 g ai/ha, with a 14-day RTI and a 14-day PHI.

The Meeting received supervised residue trials conducted in Europe on dry peas, where 2 foliar applications (SC formulation) were made at a nominal rate of 200 g ai/ha per application and 7-day RTI. Dry pea seeds were harvested at PHIs of 14, 20–22 and 28–48 days. Only three trials were provided in which peas without pods were harvested at a 14-day PHI which are insufficient to support a recommendation based on the Austria and the Netherlands GAPs.

As none of the European dry pea trials reflected the critical GAP or any other GAP provided for dry beans or peas, the Meeting could not estimate a maximum residue level or STMR for the subgroup of dry beans or the subgroup of dry peas.

Tree nuts

The critical GAP for tree nuts is from the USA; 4 foliar applications (SC formulation) at140 g ai/ha, with a 7-day RTI and a 14-day PHI. The Meeting received supervised residue trials conducted in the USA on almonds and pecans where 3 foliar spray applications (SC formulation) were made at a nominal rate of 160 g ai/ha (first application) and 200 g ai/ha (second and third applications), 7-day RTI and a 14-day PHI.

As none of the USA tree nut trials reflected the critical GAP, with regard to application rate and number of applications and the proportionality approach could not be considered, the Meeting was unable to estimate a maximum residue level or STMR for the tree nuts group.

Linseed

The critical GAP for linseed (flax) is from Canada; 1 foliar application (SC formulation) at 132 g ai/ha and a 36-day PHI. The Meeting received supervised residue trials conducted in Canada on flax matching the critical GAP.

Trifloxystrobin (parent only) residues in flax seed in ranked order were (n = 11): < 0.01 (4), 0.013, 0.015, 0.023, 0.04, 0.042, 0.054 and 0.28 mg/kg.

Total residue levels of trifloxystrobin and CGA321113 in flax seed in ranked order were (n = 11): < 0.01 (4), 0.013, 0.015, 0.023, 0.04, 0.042, 0.055 and 0.29 mg/kg.

The Meeting estimated a maximum residue level of 0.4 mg/kg and an STMR of 0.015 mg/kg for linseed.

Coffee beans

The critical GAP for coffee is from Brazil; 3 foliar applications (SC formulation) at 100 g ai/ha, with a 21-day RTI and a 30-day PHI with 0.25% v/v methylated soybean oil. The Meeting received supervised residue trials conducted in Brazil on coffee matching the critical GAP.

Trifloxystrobin (parent only) residues in coffee beans in ranked order were (n = 7): < 0.01 (6) and 0.01 mg/kg.

Total residue levels of trifloxystrobin and CGA321113 in dry coffee beans in ranked order were (n = 7): < 0.01 (4), 0.01, 0.02 and 0.03 mg/kg.

The Meeting estimated a maximum residue level of 0.015 mg/kg and an STMR value of 0.01 mg/kg for coffee beans.

Residues in animal feeds

Bean forage

The critical GAP for beans is from Austria: 2 foliar applications (SC formulation) at 200 g ai/ha, with a 7 day RTI and 7-day PHI. The Meeting received three supervised residue trials conducted in Europe on bean green material (bean forage).

The Meeting concluded that there are an insufficient number of trials conducted at the critical GAP or any other GAP provided for beans to estimate a median or highest residue for bean forage.

Pea vines

The critical GAPs for peas are from Austria and the Netherlands: 2 foliar applications (SC formulation) at 200 g ai/ha, with a 14-day RTI and a 14-day PHI. The Meeting received supervised residue trials conducted

Europe on pea rest of plant and green material (pea vines) where 2 foliar spray applications (SC formulation) were made at a nominal rate of 200 g ai/ha, with a 7-day RTI and a 14-day PHI.

Although the residue trials have a shorter RTI compared to the critical GAPs, based on the residue decline data for pea vines the Meeting estimated that residues from the field trials would be within 25% of the critical GAP.

Trifloxystrobin (parent only) residues in pea vines in ranked order were (n = 17): 0.25, 0.26, 0.9, 0.91, 0.99, 1.0, 1.1, 1.5, 2.2, 2.8, 3.7, 4.5, 5.3, 5.4, 5.6, 5.8 and 11 mg/kg.

Total residue levels of trifloxystrobin and CGA321113 in pea vines in ranked order were (n = 17): 0.27, 0.28, 0.95, 1.0 (3), 1.1, 1.7, 2.3, 2.9, 3.9, 4.7, 5.4, 5.5, 5.6, 6.1 and 11 mg/kg.

The Meeting estimated a highest residue of 11 mg/kg and a median value of 2.3 mg/kg for pea vines.

Pea hay

The Meeting received GAP information for pea hay from Austria, France and the Netherlands. None of the available trials matched these GAPs. The Meeting could not estimate a maximum residue level, highest residue, or median value for pea hay.

Almond hulls

None of the almond supervised residue trials were conducted according to the US GAP; 4 × 140 g ai/ha, an RTI of 7–21 days and a 14-day PHI. The Meeting was unable to estimate a maximum residue level or median value for almond hulls.

Fate of residues in processing

Processing data on beans with pods, linseed and coffee were provided. All data relevant for an estimation of maximum residue levels in processed commodities or for dietary exposure calculations are summarized in the following table.

Table 1 Processing factors and residue estimates for trifloxystrobin and CGA321113

Raw commodity	Residue in RAC, mg/kg		Processed commodity	Processing Factors		Residue in processed commodity, mg/kg	
	Max	STMR		Trifloxystrobin [mean or best estimate]	Total [a] [mean or best estimate]	Max-P	STMR-P
Linseed	0.4	0.015	Meal	< 0.15, 0.14 [< 0.15]	1.2, 0.33 [0.77]	--	0.012
			Oil, crude (pressed)	0.82, 0.83 [0.83]	0.89, 0.74 [0.82]	--	--
			Oil, refined (solvent extracted)	0.60, 0.98 [0.79]	0.66, 0.88 [0.77]	--	0.012
Coffee, green beans	0.01*	0.01	Instant	-[b]	0.15	--	0.0015
			Roasted	-[b]	0.15	--	0.0015

[a] Processing factor for the sum of trifloxystrobin and CGA321113

[b] No processing factors for instant or roasted coffee could be derived for residues of trifloxystrobin since no quantifiable residues were observed in the RAC (green beans), despite the use of exaggerated application rates (approximately 2.5× the maximum label rate).

RESIDUES IN ANIMAL COMMODITIES

Farm animal feeding studies

Farm animal feeding studies (lactating dairy cow and laying hen) are reported in the evaluation of the 2004 JMPR.

Farm animal dietary burden

The Meeting has added feed items (pea vines and linseed meal) and their associated residues to the dietary burden calculations used by the 2015 JMPR. Cotton meal, cotton hulls and cotton gin-by-products from the 2017 JMPR were also included. Dietary burden calculations are provided in Annex 6; the dietary burden estimates are summarized below.

The Australian maximum beef and dairy dietary burdens exceeded the maximum dose in the feeding study. The Meeting refined the dietary burden to exclude pea vines from the Australian livestock diets as trifloxystrobin is not registered for use on peas in Australia and forages are generally not traded internationally.

Table 2 Estimated maximum and mean dietary burdens of farm animals

Animal	Dietary burden estimates, ppm							
	Canada-US		European Union		Australia		Japan	
	Maximum	Mean	Maximum	Mean	Maximum	Mean	Maximum	Mean
Beef cattle	1.54	0.51	15.59 [a]	3.74 [c]	8.22	5.00	4.52	0.84
Dairy cattle	6.38	1.68	15.26 [b]	3.66 [d]	8.19	4.11	2.10	0.42
Broiler poultry	0.10	0.10	0.07	0.07	0.15	0.15	0.03	0.03
Laying hen	0.10	0.10	4.98 [e]	1.09 [f]	0.15	0.15	0.07	0.07

[a] Highest maximum beef cattle dietary burden suitable for MRL estimates for mammalian tissues.

[b] Highest maximum dairy cattle dietary burden suitable for MRL estimates for milk.

[c] Highest mean 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 and eggs.

[f] Highest mean poultry dietary burden suitable for STMR estimates for poultry tissues and eggs.

Animal commodity maximum residue levels

Based on the estimated dietary burden and the results of the animal feeding studies evaluated by the 2004 JMPR, the calculations used to estimate highest total residues for use in estimating maximum residue levels and STMR values are shown below.

Table 3 Anticipated residues in mammalian commodities

Trifloxystrobin feeding study	Feed level (ppm) for milk residues	Total Residues (mg/kg) in milk [a]	Feed level (ppm) for tissue residues	Total Residues [a] (mg/kg)			
				Muscle	Liver	Kidney	Fat

Maximum residue level beef or dairy cattle							
Feeding study [b]	5.90	-	5.90	-	< 0.04	< 0.04	0.04
	21.00	< 0.02	21.00	< 0.04	0.11	0.04	0.08
Dietary burden and high residue	15.26	< 0.012	15.59	< 0.026	0.085	< 0.04	0.066
STMR beef or dairy cattle							
Feeding study [c]	2.00	-	2.00	-	< 0.04	< 0.04	< 0.04
	5.90	-	5.90	-	< 0.04	< 0.04	< 0.04
	21.00	< 0.02	21.00	< 0.04	0.07	0.04	0.06
Dietary burden and residue estimate	3.66	< 0.005	3.74	< 0.01	< 0.04	< 0.04	< 0.04

[a] Total residues = trifloxystrobin and CGA321113.

[b] highest residues for tissues and mean residues for milk

[c] mean residues for tissues and mean residues for milk

The Meeting confirmed its previous recommendation of a maximum residue level of 0.02* mg/kg for milks.

The Meeting estimated maximum residue levels of 0.07 mg/kg for meat, based on fat (from mammals other than marine mammals) and mammalian fats (except milk fats) and 0.09 mg/kg for edible offal (mammalian). The Meeting estimated STMRs of 0.01 mg/kg for meat (muscle), 0.04 mg/kg for mammalian fat, 0.04 mg/kg for liver, 0.04 mg/kg for kidney and 0.005 mg/kg in milks. These recommendations are intended to replace all previous recommendations for all ruminant matrices.

In the feeding study in laying hens, no trifloxystrobin or CGA321113 residues (total residue, < 0.04 mg/kg) were detected in eggs, tissues or organs from hens at the highest feeding level of 15 ppm. As the maximum and mean dietary burdens of 4.98 mg/kg and 1.09 mg/kg, respectively, were much lower, the Meeting estimated a maximum residue level of 0.04(*) mg/kg for poultry fats, confirmed its previous recommendations of maximum residue levels of 0.04(*) mg/kg for eggs, poultry meat (fat) and edible offal of poultry and recommended that the STMR values should be 0.0046 mg/kg in eggs, poultry meat, edible offal and fat.

RECOMMENDATION

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

Definition of residue for compliance with the MRL for plant commodities: *trifloxystrobin*

Definition of residue for compliance with the MRL for animal commodities and dietary risk assessment for plant and animal commodities : *sum of trifloxystrobin and (E,E)-methoxyimino-{2-[1-(3-trifluoromethyl-phenyl) ethylidene-aminooxymethyl]-phenyl}acetic acid = CGA321113) (expressed as trifloxystrobin equivalents).*

The residue is fat-soluble.

Table 4 Recommendations for residues of trifloxystrobin from the 2021 Extra JMPR

CCN	Crop/Commodity	Recommended Maximum residue level (mg/kg) New	Previous	STMR or STMR-P, mg/kg	HR or HR-P, mg/kg
VP 2060	Beans with pods, Subgroup of	0.5		0.09	

CCN	Crop/Commodity	Recommended Maximum residue level (mg/kg) New	Previous	STMR or STMR-P, mg/kg	HR or HR-P, mg/kg
FB 0261	Bilberry	3		0.33	
FB 0263	Bilberry, red	3		0.33	
FB 0020	Blueberries	3		0.33	
FB 2005	Cane berries, Subgroup of	3		0.56	
FC 0001	Citrus fruits	W	0.5		
AB 0001	Citrus pulp, dry	W	1		
SB 0716	Coffee beans	0.015		0.01	
FB 0021	Currant, black, red, white	3		0.33	
VL 0470	Corn salad	15		3.3	
MO 0105	Edible offal (mammalian)	0.09		Kidney 0.04 Liver 0.04	
PE 0112	Eggs	0.04(*)	0.04(*)	0.0046	
FB 0268	Gooseberry	3		0.33	
MO 0098	Kidney of cattle, goats, pig and sheep	W	0.04(*)		
VL 0483	Lettuce, leaf	15		3.3	
SO 0693	Linseed	0.4		0.015	
MO 0099	Liver of cattle, goats, pigs & sheep	W	0.05		
MF 0100	Mammalian fats (except milk fats)	0.07		0.04	
MM 0095	Meat (from mammals other than marine mammals)	0.07 (fat)	0.05 (fat)	Fat 0.04 Muscle 0.007	
ML 0106	Milks	0.02(*)	0.02(*)	0.003	
VP 2061	Peas with pods, Subgroup of	1.5		0.073	
PF 0111	Poultry fats	0.04(*)		0.0046	
PM 0110	Poultry meat	0.04(*) (fat)	0.04(*)(fat)	0.0046	
PO 0111	Poultry, edible offal of	0.04(*)	0.04(*)	0.0046	
FB 0273	Rose hips	3		0.33	
	Coffee, instant			0.0015	
SM 0716	Coffee beans, roasted			0.0015	
	Linseed oil, refined			0.012	

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for trifloxystrobin is 0–0.04 mg/kg bw. The International Estimated Daily Intakes (IEDIs) for trifloxystrobin were estimated for the 17 GEMS/Food Consumption Cluster Diets using the STMR or STMR-P values estimated by the JMPR. The results are shown in Annex 3 of the 2021 Extra JMPR Report.

The IEDIs ranged from 1–9% of the maximum ADI. The Meeting concluded that long-term dietary exposure to residues of trifloxystrobin from uses considered by the JMPR is unlikely to present a public health concern.

Acute dietary exposure

The 2004 JMPR decided that an ARfD for trifloxystrobin was unnecessary. The Meeting therefore concluded that the acute dietary exposure to residues of trifloxystrobin resulting from uses that have been considered by the JMPR is unlikely to present a public health concern.

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TRINEXAPAC-ETHYL (271)

First draft prepared by Dr G Ye, Ministry of Agriculture and Rural Affairs, China

EXPLANATION

Trinexapac-ethyl is a synthetic plant growth regulator used for growth management of crops. It was first evaluated by JMPR in 2013 (T, R). An ADI of 0–0.3 mg/kg bw, expressed as trinexapac acid equivalents, was established by the 2013 JMPR, and it was concluded that an ARfD for trinexapac-ethyl was unnecessary.

The 2013 JMPR Meeting concluded the following residue definitions:

Definition of the residue for compliance with the MRL for plant and animal commodities and dietary risk assessment for animal commodities: T*rinexapac acid*.

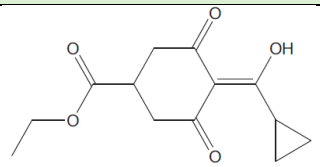
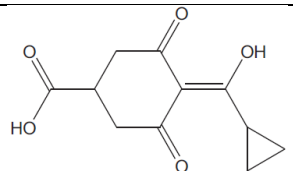
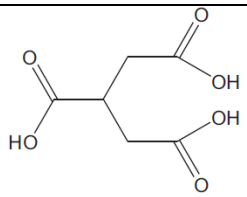
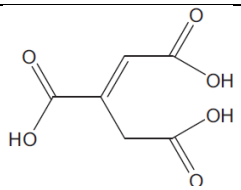
Definition of the residue for dietary risk assessment for plant commodities: *Trinexapac acid and its conjugates, expressed as trinexapac acid.*

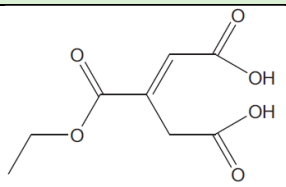
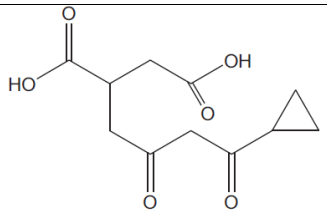
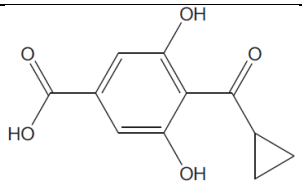
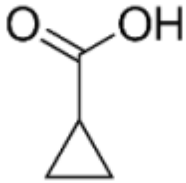
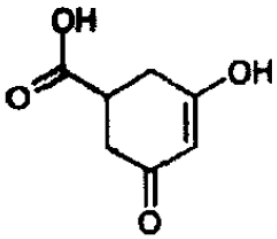
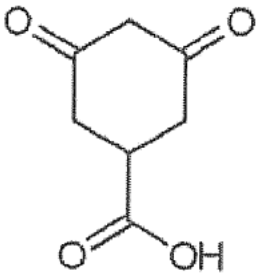
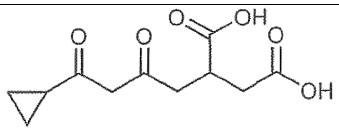
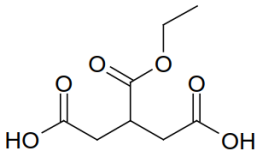
The residue is not fat-soluble.

It was scheduled at the Fifty-first Session of the CCPR for the evaluation of additional uses by the 2020 JMPR, which was postponed to the 2021 Extra JMPR.

The Meeting received information on metabolism in wheat and oilseed rape, methods of residue analysis, freezer storage stability, GAP information, supervised residue trials on rice, as well as industrial processing studies.

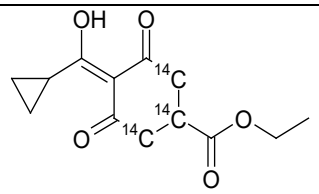
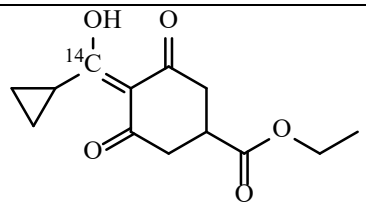
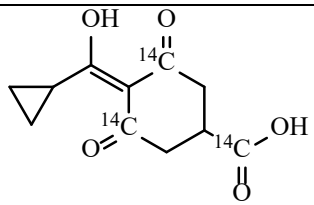
The structures and abbreviations of trinexapac-ethyl and its key metabolites discussed, are shown below

Compound	Structure
Trinexapac-ethyl CGA163935	
Trinexapac acid CGA179500	
Tricarballylic acid CGA275537	
Trans aconitic acid CGA312753	

Compound	Structure
Mono-ethyl ester of trans aconitic acid	
CGA313458	
CGA329773	
CGA224439	
CGA113745	
Cyclodion acid	
3-carboxyl-7-cyclopropyl-5,7-dioxoheptanoic acid	
CGA300405	

METABOLISM AND ENVIRONMENTAL FATE

|The position of the label for test substances used in the metabolism and processing studies are presented below.

 [3,5-Cyclohexadione-1,2,6-^{14}C]-trinexapac-ethyl	 [hydroxymethylene-^{14}C] trinexapac--ethyl	 [1, 6-cyclohexyl and hydroxymethylene-^{14}C] trinexapac acid

Plant Metabolism

Oilseed Rape

Metabolism of trinexapac-ethyl in spring oilseed rape was investigated (Piskorski R. 2015, CGA163935_10561) using [3,5-cyclohexadione-1,2,6-^{14}C]-trinexapac-ethyl (Specific Activity: 2.468 MBq/mg (66.7 µCi/mg), Radiochemical Purity >99.9%). Oilseed rape seeds (variety Jumbo) were sown directly into the containers filled with sandy loam soil to approximately 7 cm from the top at a planting density of 5 kg/ha. The soil was treated with Dursban 750 WG (chlorpyrifos) at a rate of 3.0 g/m^2 four days before sowing to prevent infestation with wireworms. The oilseed rape plants were treated at rate of 393.8 g ai/ha with [^{14}C]-trinexapac-ethyl formulated as a microemulsion at BBCH 55 by foliar spray application, and grown in a greenhouse under controlled climatic conditions. The temperature ranged from 6.4 to 37.9 °C with a mean value of 22.3 °C, the relative humidity ranged from 15.3 to 97.0% with a mean value of 45.2%. Foliage samples were collected 21 days after application (BBCH 57–67). Fully ripe plants were collected between 67 and 91 days after application (BBCH 89) and left to dry in the greenhouse. Only the seeds were analysed. Seeds were homogenized using a food processor and stored at approximately -20 °C.

Samples were extracted twice with acetonitrile: water: hexane 16:4:10 (v/v), followed by twice with acetonitrile: water 50:50 (v/v) in room temperature. The acetonitrile: water fraction was then partitioned between diethyl ether: hexane (8:2). The radioactivity contained in the extracts was measured directly by LSC. Radioactivity remaining in the extracted debris was measured by LSC after combustion of appropriate aliquots. Polar residues present in the aqueous phases were hydrolysed using mild base conditions (0.05M NaOH). Residues present in partitioned/hydrolysed fractions were subject to TLC analysis to enable the quantification of radio components and the identification/characterisation of residues by their comparison with authentic reference standards of parent and its metabolites. Results obtained by TLC were confirmed by HPLC. The identity of additional unassigned radio components was achieved by HPLC-MS/MS.

Residues remaining in solids after solvent extraction were subjected to SDS (sodium dodecysulphate) extraction, mineral acid extraction under mild conditions and harsh mineral acid extraction under reflux conditions followed by dabsyl chloride derivatization were undertaken to extract/release protein associated residues and/or residues physically entrapped within the seed matrix, to hydrolyse acid labile conjugated metabolites from the seed matrix, and to hydrolyse endogenous macromolecules to their basic chemical monomer subunits and then, in the case of proteins, chemically convert the resulting amino acids to their dabsyl derivatives in preparation for co-chromatography with appropriate dabsyl amino acid standards.

Initial analysis of the oilseed rape sampled seeds combined extracts took place within 6 months of harvest. These extracts were then re-analysed 16 months later upon study completion. Comparison of initial and final radio component profiles showed that no significant changes in the profiles had occurred during the interim period of storage.

The total radioactive residues and extractability and the identified components for each sample are summarized in Table 1 and Table 2. Total radioactive residues in the seed commodity were determined to be 0.394 mg eq/kg. Values without parentheses are the sum of both the free and conjugated forms. The values within parentheses indicate the amount of the TRR that is in the conjugated forms.

Table 1 Summary of total radioactive residues and extractability in oilseed rape treated with [^{14}C]-trinexapac-ethyl

Crop Commodity	Extracted Radioactivity		Non-extracted Radioactivity		TRR
	% TRR	mg eq/kg	% TRR	mg eq/kg	mg eq/kg
Seed	67.4	0.266	32.6	0.129	0.394

Table 2 Summary of identification and characterization of residues in oilseed rape treated with [^{14}C]-trinexapac-ethyl

TRR by summation mg eq/kg		0.394 [a]	
TRR by direct quantification mg eq/kg		0.427 [b]	
Percentage of TRR for chromatography, %		67.9 [c]	
	Component	**% TRR**	**Residue (mg eq/kg)**
Solvent extracted [c]		**67.4**	**0.266**
	Trinexapac acid (CGA179500)	*21.8 (2.8)*	*0.086 (0.011)*
	Tricarballylic acid (CGA275537)	*1.0 (1.0)*	*0.004 (0.004)*
	Oleic acid	*22.7*	*0.090*
	Unassigned [d]	*15.1*	*0.059*
	Baseline [e]	*7.3*	*0.029*
	Other fractions [f]	*1.4*	*0.006*
	Losses/gains on fractionation [g]	*1.9 (Gains)*	*0.008 (Gains)*
Unextracted [h]		**32.6**	**0.128**
Total		**100.0**	**0.394**

[a] TRR determined by summation of radioactivity present in the extracts and debris following solvent extraction

[b] The radioactive residue determined by direct quantification employing combustion/LSC

[c] The components of the TRR that were derived from chromatographic analysis

[d] Unassigned radio components which chromatographed away from origin in (i) 2D-TLC SSA (solvent system A) comprising at least 12 discrete components, none of which >1.7% TRR (>0.007 mg eq/kg) (acetonitrile:water derived fractions) or (ii) 1D-TLC SS4 (solvent system 4) comprising at least 2 components, none of which >3.6% TRR (>0.014 mg/kg) (hexane derived fractions)

[e] Polar material on origin of radiochromatogram of acetonitrile:water derived fractions using 2D-TLC SSA (6.7% TRR; 0.027 mg eq/kg) and of hexane derived fraction using 1D-TLC SS4 (0.6% TRR; 0.002 mg eq/kg)

[f] Extractable residues in 1 fraction produced during processing that was not analysed due to low residue levels

[g] The net cumulative incremental losses or gains during analysis. Calculated as 100% - sum of all components

[h] Radioactivity remaining in debris after extraction with aqueous acetonitrile/hexane mixtures. The nature of the residues was characterized further by reflux extraction with 6 M HCl, 1 M HCl and SDS extractions

Extraction of ^{14}C residues in seed with acetonitrile:water/hexane was 67.4% TRR (0.266 mg eq/kg). No parent trinexapac-ethyl was detected in seeds. The trinexapac acid (CGA179500) was identified as the major metabolite, accounting for 21.8% TRR (0.086 mg/kg; found in both the free and conjugated forms of the metabolite). Another major metabolite was also identified as oleic acid (22.7% TRR; 0.090 mg/kg, detected following saponification of radioactivity associated with hexane-extracted endogenous oils). The tricarboxylic acid (CGA275537) was the minor metabolite detected (1.0% TRR; 0004 mg/kg, found exclusively in the conjugated form). The levels of other individual unassigned radio components from extracted residues never exceeded 6.7% TRR (0.027 mg/kg).

The characterisation of the unextracted radioactive residues with SDS was conducted, only a small proportion of the residue (5.8% TRR, 0.023 mg eq/kg) was associated with proteinaceous material. Due to low residue levels released and the high levels of endogenous co-extractives present, no chromatographic analysis was undertaken.

A further 4.0% TRR (0.016 mg eq/kg) and 15.2% TRR (0.060 mg eq/kg) was rendered water soluble respectively after extraction of the unextracted radioactive residues with 1 M HCl at 60 °C and 6 M HCl at 140 °C (reflux), No chromatographic analysis was undertaken on 1M HCl extract due to low residue levels released and the high levels of endogenous co-extractives present. The HCl reflux extract was subjected to treatment with dabsyl chloride to derivatise amino acids present in the sample followed by TLC co-chromatography with (i) dabsyl derivative of glutamate; and (ii) [^{14}C]glucose. Although there was some evidence of natural incorporation of ^{14}C into glucose, it was still not possible to conclude on the presence of natural incorporation into amino acids. The 16.2% TRR (0.064 mg eq/kg) remained as unextracted residues after the harsh extraction conditions.

Metabolism of parent trinexapac-ethyl was extensive and complete (parent was not detected). Trinexapac acid (CGA179500) was the principal metabolite identified (21.8% TRR; 0.086 mg eq/kg) and was detected in both free and conjugated forms. The tricarboxylic acid metabolite (CGA275537) was also identified but at much lower levels (1.0% TRR; 0.004 mg eq/kg) and was detected in the conjugated form exclusively; Natural incorporation of ^{14}C into crop endogenous constituents was observed (quantified at a level of at least 22.7% TRR; 0.090 mg eq/kg). The pathway of metabolism was via de-esterification of the parent ethyl ester, followed by the ring opening and the elimination of cyclopropylhydroxyethylene moiety with the oxidation of carbonyl groups.

Spring wheat

Metabolism of trinexapac-ethyl in spring wheat was investigated (Piskorski R. 2015, CGA163935_10644) using [3,5-cyclohexadione-1,2,6-^{14}C]-trinexapac-ethyl (Specific Activity:

2.468 MBq/mg (66.7 µCi/mg), Radiochemical Purity >99.9%).

Spring wheat (Monsoon) was sown directly into the containers filled with sandy loam soil. Spring wheat plants were treated once at rate of 211 g ai/ha with [^{14}C]-trinexapac-ethyl as a microemulsion at BBCH 37 by foliar spray application. Samples were collected on three occasions, as an immature crop at a forage growth stage (BBCH 43, 7 days after application), at a hay growth stage (BBCH 77, 34 days after application) and at maturity (BBCH 89; as grain and straw, 62 days after application). The forage, hay, straw and grain samples were homogenised using a commercial food processor. Following homogenisation the samples were analysed by combustion/LSC of sub-samples, to determine the level of radioactive residue.

Samples were extracted sequentially with acetonitrile/water mixtures (three times with acetonitrile / water (4:1, v/v) and once with acetonitrile / water (1:1, v/v) at room temperature). Aliquots of the extracts were radioassayed by LSC. Aliquots of the post extraction solid (PES) were radioassayed by combustion analysis/LSC. Extracts containing significant quantities of radioactivity were combined and concentrated prior to TLC/HPLC analysis. Sub-samples of extracts were subject to acid and/or base hydrolysis to effect the release of metabolites from their conjugated forms. Unextracted residues in the post extraction debris from hay, straw and grain were further characterised using the clean fraction procedure to separate the residue into lignin, hemicellulose and cellulose containing fractions. Additionally, unextracted residues in the post extraction debris from grain were subject to enzyme hydrolysis to release metabolites from their conjugated forms and cleave ^{14}C starch to ^{14}C glucose.

Residues present in the principal residue containing fractions derived from both pre- and post-hydrolysis procedures were subject to thin layer chromatography (TLC)/bioimage analysis for quantification and identification by co-chromatography with authentic reference standards of parent trinexapac-ethyl and its postulated metabolites. Results obtained by TLC were confirmed by HPLC. Additionally, LC-MS/MS analysis was undertaken to confirm the presence of radio components and to identity an additional radio component for which no reference material was available.

Initial radio component profiles of the principal fractions from forage, hay, straw and grain analyses were obtained within 6 months of harvest. The original aqueous and organic phases of the partitioned grain extract were then re-analysed 34 months later. Comparison of the initial and final radio component profiles obtained showed no significant change in the profiles had occurred during the interim period of storage.

Total radioactive residues and extractability, characterization and identification of residues are summarised in Tables 3, 4, 5, 6 and 7. Values without parentheses are the sum of both the free and conjugated forms. The values within parentheses indicate the amount of the TRR that is in the conjugated forms. The total radioactive residues (TRR) for harvested commodities were 1.801 mg/kg (forage), 2.002 mg/kg (hay), 1.366 mg/kg (straw) and 1.444 mg/kg (grain). Good extractability with acetonitrile/water was achieved in forage, grain and hay (≥84.1% TRR) with lower extractability in straw (64.8% TRR).

Table 3 Summary of total radioactive residues and extractability in spring wheat treated with [^{14}C]-trinexapac-ethyl

Crop Commodity	Extracted Radioactivity		Non-extracted Radioactivity		TRR
	% TRR	mg eq/kg	% TRR	mg eq/kg	mg eq/kg
Forage	94.8	1.708	5.1	0.092	1.801
Hay	88.8	1.778	11.2	0.224	2.002
Grain	84.1	1.215	15.9	0.230	1.444
Straw	64.8	0.886	35.2	0.481	1.366

Table 4 Summary of identification and characterisation of residues in spring wheat grain samples treated with [^{14}C]-trinexapac-ethyl

TRR by summation mg eq/kg		1.444[a]	
TRR by direct quantification mg eq/kg		1.515 [b]	
Percentage of TRR for chromatography, %		76.6	
Origin of component	**Component**	**% TRR**	**Residue (mg eq/kg)**
Solvent extracted [d]	*Trinexapac-ethyl CGA163935* *Trinexapac acid CGA179500*	*nd* *40.0 (12.2)*	*nd* *0.577 (0.176)*

TRR by summation mg eq/kg		1.444[a]	
TRR by direct quantification mg eq/kg		1.515 [b]	
Percentage of TRR for chromatography, %		76.6	
Origin of component	**Component**	**% TRR**	**Residue (mg eq/kg)**
	CGA300405	*0.8 (0.2)*	*0.012 (0.002)*
	Tricarballylic acid CGA275537	*2.0 (0.3)*	*0.03 (0.004)*
	Hydroxylated trinexapac acid SYN548584[e]	*12.1 (0.3)*	*0.175 (0.004)*
	Unassigned in pre-hydrolysis organosoluble fraction [f]	*9.5*	*0.137*
	Unassigned in post hydrolysis fraction [g]	*7.8*	*0.113*
	Baseline components in pre- and post-hydrolysis fraction [h]	*4.3*	*0.063*
	Losses/gains on fractionation [i]	*7.6 (Loss)*	*0.107 (Loss)*
Unextracted [j]		**15.9**	**0.230**
Total		**100.0**	**1.444**

nd Not detected

[a] TRR determined by summation of radioactivity present in extracts and debris following solvent extraction.

[b] The radioactive residues determined by direct quantification by combustion/LSC.

[c] The components of the TRR derived from chromatographic analysis.

[d] Percentage of TRR for chromatography determined by summation of percentage of TRR present in Organic C and Hydrolysate D.

[e] A metabolite for which no reference standard was available. LC-MS/MS analysis indicates a hydroxylated form of CGA179500 with the position of the hydroxyl functional group in two possible locations on the cyclohexanedione ring.

[f] Unassigned radio components which chromatographed away from origin in 2D-TLC SSA comprising at least 9 discrete components, none of which >1.8% TRR (>0.026 mg eq/kg) in Organic C.

[g] Unassigned radio components which chromatographed away from origin in 2D-TLC SSA comprising at least 8 discrete components, none of which >2.3% TRR (>0.033 mg eq/kg) in Hydrolysate D.

[h] Polar material on origin of radiochromatogram using 2D-TLC SSA. Evidence of an element of natural incorporation in grain debris analysis indicates this radioactivity is likely to be similar in nature to that found in unextracted material.

[i] The net cumulative incremental losses or gains during analysis. Calculated as 100% - sum of all components.

[j] Radioactivity remaining in debris after extraction with acetonitrile and aqueous acetonitrile. The nature of the residues was characterised further by clean fractionation technique and by enzymatic hydrolysis.

Table 5 Summary of identification and characterisation of residues in spring wheat forage samples treated with [^{14}C]-trinexapac-ethyl

TRR by summation mg eq/kg		1.801 [a]	
TRR by direct quantification mg eq/kg		1.846 [b]	
Percentage of TRR for chromatography, %		93.9 [c]	
Origin of component	**Component**	**% TRR**	**Residue (mg eq/kg)**
Solvent extracted [d]		**94.9**	**1.709**
	Trinexapac-ethyl CGA163935	*0.3 (nd*	*0.006 (nd)*
	Trinexapac acid CGA179500	*22.1 (1.7)*	*0.399 (0.030)*
	CGA300405	*20.7 (3.6)*	*0.374 (0.065)*
	Tricarballylic acid CGA275537	*7.8 (5.0)*	*0.141 (0.091)*
	CGA329773	*0.7 (nd)*	*0.012 (nd)*
	Hydroxylated trinexapac acid SYN548584	*3.3 (nd)*	*0.060 (nd)*
	Unassigned in pre-hydrolysis organosoluble fraction [e]	*7.7*	*0.140*
	Unassigned in post hydrolysis fraction [f]	*24.0*	*0.431*

TRR by summation mg eq/kg		1.801 [a]	
TRR by direct quantification mg eq/kg		1.846 [b]	
Percentage of TRR for chromatography, %		93.9 [c]	
Origin of component	**Component**	**% TRR**	**Residue (mg eq/kg)**
	Baseline components in pre- and post-hydrolysis fractions [g]	*7.2*	*0.129*
	Losses/gains on fractionation [h]	*1.2 (Loss)*	*0.017 (Loss)*
Unextracted [i]		**5.1**	**0.092**
Total		**100.0**	**1.801**

nd: Not detected

[a] TRR determined by summation of radioactivity present in extracts and debris following solvent extraction.

[b] The radioactive residues determined by direct quantification by combustion/LSC.

[c] Percentage of TRR for chromatography.

[d] The components of the TRR derived from chromatographic analysis.

[e] Unassigned radio components chromatographed by 2D-TLC comprising at least 10 discrete components, none of which >1.4% TRR (>0.025 mg eq/kg).

[f] Unassigned radio components chromatographed by 2D-TLC comprising at least 4 discrete components, none of which >12.7% TRR (>0.228 mg eq/kg). Further investigation by HPLC and 1D-TLC indicates presence of citric acid and components similar in nature or more polar than citric acid, suggesting they are likely to be part of an overall pathway to natural products.

[g] Polar material on origin of radiochromatogram using 2D-TLC. Characterization of radioactivity using TLC staining techniques (iodine and bromocresol green) indicates residue is associated with endogenous components and acidic in nature. More forcing chromatographic conditions confirm radioactivity remains at the origin. This, alongside evidence of natural incorporation from the feed commodity debris analysis, characterises this radioactivity as similar in nature to that found in unextracted material.

[h] The net cumulative incremental losses or gains during analysis. Calculated as 100% - sum of all components.

[i] Radioactivity remaining in debris after extraction with acetonitrile:water.

Table 6 Summary of identification and characterisation of residues in spring wheat hay samples treated with [^{14}C]-trinexapac-ethyl

TRR by summation mg eq/kg		2.002 [a]	
TRR by direct quantification mg eq/kg		1.967 [b]	
Percentage of TRR for chromatography, %		88.0 [c]	
Origin of component	**Component**	**% TRR**	**Residue (mg eq/kg)**
Solvent extracted [d]		**88.8**	**1.778**
	Trinexapac acid (CGA179500)	*22.6 (2.0)*	*0.453 (0.041)*
	CGA300405	*8.0 (1.4)*	*0.161 (0.027)*
	Tricarballylic acid (CGA275537)	*10.3 (4.6)*	*0.206 (0.092)*
	CGA329773	*1.4 (nd)*	*0.027 (nd)*
	Hydroxylated trinexapac acid (SYN548584)	*5.1 (nd)*	*0.102 (nd)*
	Unassigned in pre-hydrolysis organosoluble fraction [e]	*8.3*	*0.167*
	Unassigned in post hydrolysis fraction [f]	*11.1*	*0.222*
	Baseline components in pre- and post-hydrolysis fractions [g]	*21.2*	*0.425*
	Losses/gains on fractionation [h]	*0.8 (Loss)*	*0.015 (Loss)*
Unextracted [i]		**11.2**	**0.224**
Total		**100.0**	**2.002**

nd: Not detected

[a] TRR determined by summation of radioactivity present in extracts and debris following solvent extraction.

[b] The radioactive residues determined by direct quantification by combustion/LSC.

[c] Percentage of TRR for chromatography.

[d] The components of the TRR derived from chromatographic analysis.

[e] Unassigned radio components chromatographed by 2D-TLC comprising at least 9 discrete components, none of which >1.9% TRR (>0.038 mg eq/kg).

[f] Unassigned radio components chromatographed by 2D-TLC and comprising at least 4 discrete components, none of which >6.7% TRR (>0.135 mg eq/kg). Further investigation by HPLC and 1D-TLC indicates presence of citric acid and components similar in nature or more polar than citric acid, suggesting they are likely to be part of an overall pathway to natural products.

[g] Polar material on origin of radiochromatogram using 2D-TLC. Characterisation of radioactivity using TLC staining techniques (iodine and bromocresol green) indicates residue is associated with endogenous components and acidic in nature. More forcing chromatographic conditions confirm radioactivity remains at the origin and also demonstrated that no single component exceeds 10.8% TRR (0.216 mg eq/kg). This, alongside evidence of natural incorporation from the feed commodity debris analysis, characterises this radioactivity as similar in nature to that found in unextracted material.

[h] The net cumulative incremental losses or gains during analysis. Calculated as 100% - sum of all components.

[i] Radioactivity remaining in debris after extraction with acetonitrile:water. The nature of the residues was characterised further by the clean fractionation technique.

Table 7 Summary of identification and characterisation of residues in spring wheat straw samples treated with [^{14}C]-trinexapac-ethyl

TRR by summation mg/kg		1.366 [a]	
TRR by direct quantification mg/kg		1.378 [b]	
Percentage of TRR for chromatography, %		60.2 [c]	
Origin of component	**Component**	**% TRR**	**Residue (mg eq/kg)**
Solvent extracted [d]		**64.8**	**0.885**
	Trinexapac acid CGA179500	*5.5 (2.0)*	*0.075 (0.027)*
	CGA300405	*9.6 (1.7)*	*0.131 (0.024)*
	Tricarballylic acid CGA275537	*8.1 (2.2)*	*0.111 (0.030)*
	CGA329773	*0.1 (nd)*	*0.002 (nd)*
	Hydroxylated trinexapac acid SYN548584	*1.9 (nd)*	*0.026 (nd)*
	Unassigned in pre-hydrolysis organosoluble fraction [e]	*2.0*	*0.027*
	Citric Acid	*4.2*	*0.057*
	Unassigned in post hydrolysis fraction [f]	*19.9*	*0.272*
	Baseline components in pre- and post-hydrolysis fractions [g]	*8.8*	*0.121*
	Losses/gains on fractionation [h]	*4.7 (Loss)*	*0.063 (Loss)*
Unextracted [i]		**35.2**	**0.481**
Total		**100.0**	**1.366**

nd: Not detected

[a] TRR determined by summation of radioactivity present in extracts and debris following solvent extraction.

[b] The radioactive residues determined by direct quantification by combustion/LSC.

[c] Percentage of TRR for chromatography.

[d] The components of the TRR derived from chromatographic analysis.

[e] Unassigned radio components chromatographed by 2D-TLC comprising at least 9 discrete components, none of which >1.3% TRR (>0.017 mg eq/kg).

[f] Unassigned radio components chromatographed by 2D-TLC. Further investigation by HPLC and 1D-TLC identified citric acid and demonstrated components similar in nature or more polar than citric acid suggesting they are likely to be part of an overall pathway to natural products. Comprises at least 5 components, none of which > 5.2% TRR (>0.071 mg eq/kg).g

[g] Polar material on origin of radiochromatogram using 2D-TLC. Characterization of radioactivity using TLC staining techniques (iodine and bromocresol green) indicates residue is associated with endogenous components and acidic in nature. More forcing chromatographic conditions confirm radioactivity remains at the origin. This, alongside evidence of natural incorporation from the feed commodity debris analysis, characterizes this radioactivity as similar in nature to that found in unextracted material.

[h] The net cumulative incremental losses or gains during analysis. Calculated as 100% - sum of all components.

[i] Radioactivity remaining in debris after extraction with acetonitrile:water. The nature of the residue was characterized further by the clean fractionation technique.

Metabolism of parent trinexapac-ethyl was extensive, the parent was only detected in forage with a low residue level of 0.3% TRR, 0.006 mg eq/kg. The principal metabolites identified in grain were trinexapac acid (40% TRR, 0.577 mg eq/kg), the minor metabolites identified in grain were tricarboxylic acid ethyl ester metabolite CGA300405 (0.8% TRR; 0.012 mg eq/kg), tricarboxylic acid metabolite CGA275537 (2.0% TRR; 0.030 mg eq/kg) and a hydroxylated metabolite of CGA179500 (12.1% TRR, 0.175 mg eq/kg).

The principal metabolites identified in forage, hay and straw were trinexapac acid (5.5–22.6% TRR; 0.075–0.453 mg eq/kg), the tricarboxylic acid ethyl ester metabolite (CGA300405, 8.0–20.7% TRR, 0.131–0.374 mg eq/kg) and tricarballylic acid (CGA275537, 7.8–10.3% TRR, 0.111–0.141 mg eq/kg). Formation of citric acid and subsequent incorporation of small ^{14}C containing moieties into the broader pool of natural biosynthetic products (characterised by the presence of ^{14}C-glucose in hay, straw and grain). All metabolites with the exception of CGA329773 and citric acid were found in their free and conjugated forms.

The characterisation of the unextracted radioactive residues in the hay, grain and straw samples were conducted with a clean fractionation technique, released additional small amounts of the same metabolites as observed in the extracted fractions. CGA300405 was identified as the largest residue in all samples analysed (0.3–0.6% TRR; 0.004–0.013 mg eq/kg). Acid hydrolysis of a hemicellulose fraction (11.8% TRR; 0.161 mg eq/kg) derived from straw showed the majority of this fraction to comprise [^{14}C]-glucose, demonstrating extensive natural incorporation into endogenous components. This is consistent with the detection of both citric acid (a component of the citric acid cycle) and CGA275537 and is highly indicative of incorporation of small ^{14}C containing moieties into the broader pool of natural biosynthetic products.

The metabolic Pathway was proposed as de-esterification of the parent ethyl ester, following oxidation of the cyclohexanedione ring, dehydration of hydroxylated trinexapac acid, ring opening and the elimination of the cyclopropylhydroxyethylene moiety, de-esterification to form tricarballylic acid, a naturally occurring component in grasses and hydroxylation of tricarballylic acid to form citric acid and subsequent incorporation into the carbon pool. In addition, conjugation of all metabolites (with the exception of the aromatic diol acid metabolite) to endogenous crop components was also observed.

In summary, the metabolic profile of trinexapac-ethyl in rapeseed and spring wheat was in line with the plant metabolism evaluated by the 2013 JMPR.

RESIDUE ANALYSIS

Analytical methods

The Meeting received descriptions and validation data for analytical methods for residues of trinexapac acid in plant and animal matrices. The methods are suitable for analysis of trinexapac acid in plant and

animal matrices. The analytical methods for determination of three metabolites CGA313458, CGA113745 and CGA224439 were also received.

Table 7 Overview of the new methods for determination of trinexapac acid in crops and animal matrices

		Method GRM020.05A	Method GRM020.09A	Method QuEChERS (EN 15662:2009-02)
Extraction and clean-up	Analytes	Trinexapac acid	Trinexapac acid and its conjugates	Trinexapac acid
	Matrix	Barley grain, barley hay, barley straw, tomato, apple and sunflower seed	Cereal grain and straw	Crops (lettuce, whole orange, wheat grain, dried broad bean, oilseed rape seed) and animal matrices (whole milk and egg, bovine muscle and liver, and animal fat)
	Extraction	Samples are extracted by homogenisation with methanol/water/phosphate buffer (pH 7) (30:56:14 v/v/v). Extracts are centrifuged and aliquots are subsequently diluted with ultra-pure water. SPE procedure is then carried out to facilitate sample clean up. Final determination is by high performance liquid chromatography with triple quadrupole mass spectrometric detection (LC-MS/MS)	Samples of grain (10 g) or straw (5 g) are extracted by sequential homogenisation with acetonitrile/water (80/20 v/v) and acetonitrile/water (50/50 v/v). Conjugated residues of trinexapac acid are hydrolysed under mild basic conditions with sodium hydroxide (0.05M at room temperature overnight). The extracted samples are acidified with hydrochloric acid and trinexapac acid residues are partitioned into ethyl acetate then filtered through a silica solid phase extraction (SPE) cartridge. The eluates are evaporated and dissolved in 0.1 M hydrochloric acid. Further clean-up is by an Oasis HLB SPE procedure. Final determination is by high performance liquid chromatography with triple quadrupole mass spectrometric detection (LC-MS/MS)	Water was added to samples to adjust water content to about 100% and samples were then extracted by shaking with acetonitrile containing 1% acetic acid. After the addition of a mixture of magnesium sulphate, sodium chloride, and buffering citrate salts, the extracts were shaken and then centrifuged. For oilseed rape seed, extracts were transferred to freezer to freeze out fat. After centrifugation, all extracts were first diluted with water containing 1% formic acid and then with acetonitrile/water (20/80; v/v) containing 1% formic acid. Final determination was performed with high-performance liquid chromatography with triple quadrupole mass-spectrometric detection (LC-MS/MS)
Chromatography	Type	LC	LC	LC
	Analytical column	ACE 5-C18	Phenomenex Luna C18(2)	Phenomenex Luna C18
	Dimensions	100× 3.0 mm	50×4.6 mm id	50×2 mm id
	Particle size	5 µm	3 µm	5 µm
	Injection volume	10 µL	50 µL	20 µL
	Mobile phase	Solvent 1: Acetonitrile Solvent 2: 0.2%, v/v Acetic acid	Solvent 1: 0.1% v/v formic acid Solvent 2: Acetonitrile	Solvent A: water + 0.5 % of acetic acid Solvent B: acetonitrile + 0.5 % of acetic acid
	Flow rate	1 mL/min	0.8 mL/min	0.5 mL/min
	Instrument	Agilent 1100 with Applied Biosystems API 4000 triple quadrupole mass spectrometer	Agilent 1290 with AB Sciex API6500 triple quadrupole mass spectrometer with Analyst™ software version 1.6.2.	Agilent Infinity 1290 with Applied Biosystems MDS Sciex API 5500 triple quadrupole mass spectrometer.

		Method GRM020.05A	Method GRM020.09A	Method QuEChERS (EN 15662:2009-02)
		with Analyst™ software version 1.4.1		
Detection	Quantitative detection	MS Primary transition: m/z 223→135 Confirmatory transition m/z = 223→179, with alternative possibility of m/z = 223→83	MS The primary transition: m/z = 223→135 The confirmatory transition: m/z = 223→83.	MS primary transition: m/z 223 → 83, confirmatory transition: m/z 223 → 135.
	LOQ	0.01 mg/kg	0.01 mg/kg for cereal grain 0.05 mg/kg for cereal straw	0.01 mg/kg
	Whole method linearity (r^2)	0.0005-0.01 µg/mL, r^2≥0.99	0.23-15 ng/mL, r≥0.99	0.1-20 ng/mL, r^2≥0.99

Table 8 Overview of the new methods for determination the metabolites CGA313458, CGA113745 and CGA224439 of trinexapac-ethyl in crop matrices

		Method GRM020.13A	Method GRM020.14A	Method GRM020.15A
Extraction and clean-up	Analytes	CGA313458 (2-((Z)-4-cyclopropyl-4-hydroxy-2-oxo-but-3-enyl)-succinic acid)	CGA113745 (3-hydroxy-5-oxo-3-cyclohexene-1-carboxylic acid), cyclodion acid	CGA224439 (Cyclopropanecarboxylic Acid)
	Matrix	Cereal grain and processed fractions	Brewing and baking commodities (wheat grain, flour, bran, beer and bread)	Brewing and baking matrices
	Extraction	samples of brewing or baking fractions (10 g) are extracted by sequential homogenization with acetonitrile/water (80/20; v/v) and acetonitrile/water (50/50; v/v). After evaporation of acetonitrile, the sample is diluted with ultra-pure water and the pH adjusted to pH 7-9 with dilute ammonium hydroxide solution. Samples are partitioned twice with ethyl acetate, the aqueous samples are filtered through an Oasis HLB SPE cartridge. Alternatively, samples may be analysed directly from the primary extracts without any further sample clean-up where there is sufficient instrument sensitivity. Final determination is by LC-MS/MS.	For liquid commodities, 1.0 g sub samples of liquid brewing fractions are filtered through a Chromabond (EC) C18 SPE cartridge before being diluted with ultra-pure water. For non-liquid commodities, 10 g sub samples are extracted twice by homogenisation with 0.2% ammonia in ultra-pure water. A 4 mL aliquot of the combined sample is acidified, mixed and centrifuged and 2 mL of the supernatant (equivalent to 0.2 g matrix) is taken through an Oasis WCX SPE cartridge procedure to remove endogenous co-extractives. Final determination is by LC-MS/MS	samples of brewing and baking matrices (4 g) are extracted by maceration with an acetonitrile/acidified water mixture. The contents of a Supel QuE Citrate (EN) tube i.e. magnesium sulphate, sodium chloride, sodium citrate dibasic sesquihydrate and sodium citrate tribasic dehydrate are added to an aliquot of the extract to partition the organic and aqueous phases. An aliquot of the organic phase is taken and derivatized with a mixture of 2-hydrazinoquinoline, triphenylphosphine and 2,2'-dipyridyl disulphide in acetonitrile by incubation at 60°C for 1 hour. After solvent removal, residues are reconstituted in deionized water. Final determination is by LC-MS/MS.

		Method GRM020.13A	Method GRM020.14A	Method GRM020.15A
Chromatography	Type	LC	LC	LC
	Analytical column	Develosil Aqueous RP-3, 150×3mm, 3 µm	For grain, bread and flour: Ultracore Super Phenylhexyl 50×2.1 mm, 2.5 µm For bran only: X-Select CSH C18, 50×3.0 mm, 2.5 µm	Develosil RPAqueous, 50×4.6 mm, 3µm
	Injection volume	20 µL	30 µL	20 µL
	Mobile phase	Solvent 1: acetonitrile Solvent 2: 0.1% v/v formic acid	Solvent 1: acetonitrile Solvent 2: 0.05 % v/v acetic acid	Solvent 1: 10 mM ammonium acetate in Water Solvent 2: Acetonitrile
	Flow rate	0.4 mL/min	0.4 mL/min	1.0 mL/min
	Instrument	Agilent 1260 with AB Sciex Triple Quad 5500 Q-Trap with Analyst™ software version 1.6.2	Agilent 1260 with AB Sciex Triple Quad 5500 Q-Trap with Analyst™ software version 1.6.2	Agilent 1100 Series with AB Sciex 4000 with Analyst™ software version 1.4.2
Detection	Quantitative detection	MS Primary transition: m/z 241→69 Confirmatory transition m/z = 241→83, if necessary, 241→113	MS primary *m/z* = 155→69, confirmatory *m/z* = 155→57	MS primary transition: m/z 228 → 160, confirmatory transition: m/z 228 → 69.
	LOQ	0.01 mg/kg	0.01 mg/kg	0.01 mg/kg
	Whole method linearity (r^2)	0.02-20 ng/mL, r^2≥0.99	0.2-20 ng/mL, r≥0.99	0.3-12.5 ng/mL, r^2≥0.99

Method GRM020.01A

Method GRM020.01A was evaluated by the 2013 JMPR. The meeting received new validation data for method GRM020.01A for determining residues (free and conjugated trinexapac acid) in/on rice (Smith N., 2014, A7725M_50010). Recovery and repeatability data for the determination of trinexapac acid residues in crops are presented in Table 9. Average recoveries ranged from 84 to 122%. The LOQ was 0.01 mg/kg. The %RSDs ranged from 0.69 to 12. Calibration curves over the range 0.01–1.0 mg/kg demonstrated linearity with r≥0.99. The method is suitable to measure free and conjugated trinexapac acid in rice grain and straw.

Table 9 Recovery data of trinexapac acid from rice grain and straw using method GRM020.01A (Smith N. 2014, A7725M_50010)

Commodity	Compound	Fortification level (mg/kg)	No.	Range of Recovery (%)	Mean recovery (%)	RSD (%)
Rice (grain)	Trinexapac acid (m/z = 223 → 135)	0.01	3	109-111	110	1.1
		1.0	3	97-122	108	12
		Overall	6	97-122	109	7.6
Rice (straw)	Trinexapac acid (m/z = 223 → 135)	0.01	3	100-111	106	5.5
		0.20	3	84-86	85	0.69
		Overall	6	84-111	96	13

Method GRM020.05A

Method GRM020.05A was developed and validated for determination of residues of free trinexapac acid in crop samples using an external standardisation procedure (Hargreaves S.L. 2008, CGA179500_10978 and Mayer L, 2008, No.466872). Samples are extracted by homogenisation with methanol/water/phosphate buffer (pH7) (30:56:14 v/v/v). Extracts are centrifuged and aliquots are subsequently diluted with ultra-pure water. An OasisTM HLB solid phase extraction (SPE) procedure is then carried out to facilitate sample clean up. Final determination is by high performance liquid chromatography with triple quadrupole mass spectrometric detection (LC-MS/MS) by monitoring for the primary transition (m/z = 223→135) and the confirmatory transition (m/z = 223→179; with alternative possibility of m/z = 223→83). The method was validated in barley grain, barley straw and hay, tomato, sunflower seed and apple, at a LOQ of 0.01 mg/kg, with average recoveries of 73–107%, and the RSDs of 1.9–6.9%. The extraction efficiency was shown to be satisfactory by reference to the metabolism study on spring wheat (2013 JMPR evaluation). The method is suitable to measure free trinexapac acid in the above commodities.

Table 10 Recoveries of trinexapac acid using method GRM020.05A (Hargreaves S.L. 2008, CGA179500_10978 and Mayer L, 2008, No.466872)

Commodity	Compound	Fortification level (mg/kg)	No.	Range of Recovery (%)	Mean recovery (%)	RSD (%)
Barley (grain)	Trinexapac acid (m/z = 223 → 135)	0.01	5	85-90	88	2.1
		0.1	5	87-90	89	1.5
		Overall	10	85-90	88	1.9
	Trinexapac acid (m/z = 223 → 179)	0.01	5	96-97	97	0.5
		0.1	5	89-97	92	3.2
		Overall	10	89-97	94	3.4
Barley (hay)	Trinexapac acid (m/z = 223 → 135)	0.01	5	80-86	82	2.7
		0.1	5	81-101	89	8.5
		Overall	10	80-101	86	7.4
	Trinexapac acid (m/z = 223 → 179)	0.01	5	90-103	98	5.6
		0.1	5	85-106	94	8.2
		Overall	10	85-106	96	6.9
Barley (straw)	Trinexapac acid (m/z = 223 → 135)	0.01	5	81-85	83	1.9
		0.1	5	84-89	86	2.7
		Overall	10	81-89	84	3.1
	Trinexapac acid (m/z = 223 → 179)	0.01	5	102-107	105	2.4
		0.1	5	90-96	93	3.0
		Overall	10	90-107	99	6.6
Tomato	Trinexapac acid (m/z = 223 → 135)	0.01	5	90-97	94	3.1
		0.1	5	89-97	95	3.6
		Overall	10	89-97	94	3.2
	Trinexapac acid (m/z = 223 → 179)	0.01	5	88-98	94	3.9
		0.1	5	90-99	95	3.3
		Overall	10	88-99	94	3.4
Apple	Trinexapac acid (m/z = 223 → 135)	0.01	5	85-96	90	4.8
		0.1	5	79-83	82	2.1
		Overall	10	79-96	86	6.3
	Trinexapac acid (m/z = 223 → 179)	0.01	5	85-93	90	3.5
		0.1	5	80-84	82	2.2
		Overall	10	80-93	86	5.6
		0.01	5	80-89	83	4.3

Commodity	Compound	Fortification level (mg/kg)	No.	Range of Recovery (%)	Mean recovery (%)	RSD (%)
Sunflower (seed)	Trinexapac acid (m/z = 223 → 135)	0.1	5	78-90	83	6.9
		Overall	10	78-90	83	5.5
	Trinexapac acid (m/z = 223 → 179)	0.01	5	73-84	80	5.7
		0.1	5	75-86	79	6.5
		Overall	10	73-86	80	5.8

Method GRM020.09A

The method was developed and validated for the determination of total residues of trinexapac acid free and conjugated in cereal grain and straw using matrix-matched standards (Braid S.L., Tsui G. (2016), CGA179500_10997 and Tsui G. (2015), CGA179500_10991). Grain (10 g) or straw (5 g) are extracted by sequential homogenisation with acetonitrile/water (80/20 v/v) and acetonitrile/water (50/50 v/v). Conjugated residues of trinexapac acid are hydrolysed under mild basic conditions with sodium hydroxide (0.05M at room temperature overnight). Following evaporation of the acetonitrile, samples are acidified with hydrochloric acid and trinexapac acid residues are partitioned into ethyl acetate then filtered through a silica solid phase extraction (SPE) cartridge. The eluates are evaporated and dissolved in 0.1 M hydrochloric acid. Further clean-up is by an Oasis HLB SPE procedure. Final determination is by high performance liquid chromatography with triple quadrupole mass spectrometric detection, LC-MS/MS, monitoring for the primary (m/z = 223→125) and the confirmatory mass transition (m/z = 223→83). The method was validated for cereal grain and cereal straw. Average recoveries ranged from 70 to 78% for cereal grain and 66 to 88% for barley straw. The LOQ was 0.01 for cereal grain and 0.05 mg/kg for barley straw. The %RSDs ranged from 2.4–8.9. Significant matrix effects (suppression or enhancement, below ±10%) were observed during method validation, therefore matrix matched linearity standards (i.e. solvent standards in control matrix extracts) were used for quantification. The extraction and hydrolysis of trinexapac acid were shown to be satisfactory by reference to the metabolism study on spring wheat. The method is suitable to measure free and conjugated trinexapac acid in cereal grain and straw with matrix-matched standards.

Table 11 Recoveries of trinexapac acid using method GRM020.09A

Commodity	Compound	Fortification level (mg/kg)	No.	Range of Recovery (%)	Mean recovery (%)	RSD (%)
Cereal (grain)	Trinexapac acid (m/z = 223 → 125)	0.01	5	70-75	73	2.4
		0.1	5	70-75	73	3.2
		Overall	10	70-75	73	2.7
	Trinexapac acid (m/z = 223 → 83)	0.01	5	71-78	74	4.8
		0.1	5	73-77	74	2.7
		Overall	10	71-78	74	3.7
Barley (straw)	Trinexapac acid (m/z = 223 → 125)	0.05	5	66-74	73	2.4
		0.5	5	71-85	78	7.9
		Overall	10	66-85	74	8.3
	Trinexapac acid (m/z = 223 → 83)	0.05	5	66-79	73	7.0
		0.5	5	73-88	81	7.8
		Overall	10	66-88	77	8.9

Method GRM020.13A

The method was developed and validated for the determination of residues of 2-((Z)-4-cyclopropyl-4-hydroxy-2-oxo-but-3-enyl)-succinic acid (CGA313458) in cereal grain and processed fractions with matrix-matched standards (Braid S. and Langridge G., 2016, CGA313458_10008; Langridge G., 2016, CGA313458_10000).

Samples of brewing or baking fractions (10 g) are extracted by sequential homogenization with acetonitrile/water (80/20; v/v) and acetonitrile/water (50/50; v/v). An aliquot of the combined extracts equivalent to 0.2 g (2 mL) is evaporated to remove the acetonitrile. The sample is diluted with ultra-pure water and the pH adjusted to pH 7–9 with dilute ammonium hydroxide solution. Samples are partitioned twice with ethyl acetate to remove co-extractives then the aqueous samples are filtered through an Oasis HLB SPE cartridge. Alternatively, samples may be analysed directly from the primary extracts without any further sample clean-up where there is sufficient instrument sensitivity. Final determination is by high performance liquid chromatography with triple quadrupole mass spectrometric detection (LC-MS/MS). The LOQ of the method is 0.01 mg/kg. Significant enhancement or suppression of detector response was observed for CGA313458 during validation and therefore matrix-matched standards were used for these analyses. The method is suitable to measure CGA313458 in cereal grain and processed fractions with matrix-matched standards.

Table 12 Recoveries of 2-((Z)-4-cyclopropyl-4-hydroxy-2-oxo-but-3-enyl)-succinic acid (CGA313458) from brewing and baking matrices using method GRM020.13A

Commodity	Compound	Fortification level (mg/kg)	No.	Range of Recovery (%)	Mean recovery (%)	RSD (%)
Cereal (grain)	CGA313458 (m/z = 241 → 69)	0.01	5	94-107	101	6.3
		0.1	5	100-109	106	3.3
		Overall	10	94-109	103	5.4
	CGA313458 (m/z = 241 → 83)	0.01	5	113-118	115	1.9
		0.1	5	100-109	104	3.4
		Overall	10	100-118	110	5.9
Cereal (flour)	CGA313458 (m/z = 241 → 69)	0.01	5	94 - 101	98	2.8
		0.1	5	78 - 83	80	2.4
		Overall	10	78 - 101	89	10.8
	CGA313458 (m/z = 241 → 83)	0.01	5	82 - 87	85	3.3
		0.1	5	76 - 81	79	2.4
		Overall	10	76 - 87	82	4.6
Cereal (bran)	CGA313458 (m/z = 241 → 69)	0.01	5	61-82	74	10.7
		0.1	5	75-107	93	13.6
		Overall	10	61-107	84	16.8
	CGA313458 (m/z = 241 → 83)	0.01	5	92-108	103	7.2
		0.1	5	69-88	75	10.5
		Overall	10	69-108	89	18.6
Bread	CGA313458 (m/z = 241 → 69)	0.01	5	95-110	103	5.9
		0.1	5	76-82	79	2.9
		Overall	10	76-110	91	14.7
	CGA313458 (m/z = 241 → 83)	0.01	5	71-77	74	3.0
		0.1	5	72-87	79	6.9
		Overall	10	71-87	76	6.1
Beer	CGA313458 (m/z = 241 → 69)	0.01	5	100-106	103	2.2
		0.1	5	97-105	100	3.2
		Overall	10	97-106	102	2.9
	CGA313458 (m/z = 241 → 83)	0.01	5	93-99	97	2.6
		0.1	5	96-102	99	2.8
		Overall	10	93-102	98	2.7

Method GRM020.14A

Method GRM020.14A was developed and validated for the determination of residues of 3-hydroxy-5-oxo-3-cyclohexene-1-carboxylic acid (cyclodion acid or CGA113745) in cereal processed fractions with matrix-matched standards (Braid S., Brookes S., Langridge G., 2016, CGA113745_10002; Langridge G. (2016, CGA313458_10000).

For liquid commodities, 1.0 g samples of liquid brewing fractions are filtered through a C18 SPE cartridge before being diluted with ultra-pure water. For non-liquid commodities, 10 g samples are extracted twice by homogenisation with 0.2% ammonia in ultra-pure water. A 4 mL aliquot of the combined sample is acidified, mixed and centrifuged and 2 mL of the supernatant (equivalent to 0.2 g matrix) is taken through an Oasis WCX SPE cartridge procedure to remove endogenous co-extractives. Final determination is by LC-MS/MS monitoring two mass transitions (primary *m/z* = 155→69, confirmatory *m/z* = 155→57). The LOQ of the method is 0.01 mg/kg. Significant enhancement or suppression of detector response was observed for CGA113745 during validation and therefore matrix-matched standards were used for these analyses.

The method is suitable to measure CGA113745 in cereal processed fractions with matrix-matched standards.

Table 13 Recoveries of 3-hydroxy-5-oxo-3-cyclohexene-1-carboxylic acid (CGA113745) from brewing and baking matrices using method GRM020.14A

Commodity	Compound	Fortification level (mg/kg)	No.	Range of Recovery (%)	Mean recovery (%)	RSD (%)
Cereal (grain)	CGA113745 (m/z = 155 → 69)	0.01	5	70-78	73	4.3
		0.1	5	70-78	75	4.0
		Overall	10	70-78	74	4.1
	CGA113745 (m/z = 155 → 57)	0.01	5	68-77	73	4.6
		0.1	5	67-78	76	6.4
		Overall	10	67-78	74	5.7
Cereal (flour)	CGA113745 (m/z = 155 → 69)	0.01	5	61-78	73	9.4
		0.1	5	86-89	88	1.3
		Overall	10	61-89	80	11.2
	CGA113745 (m/z = 155 → 57)	0.01	5	64-75	67	6.9
		0.1	5	85-94	88	4.0
		Overall	10	64-94	78	15.2
Cereal (bran)	CGA113745 (m/z = 155 → 69)	0.01	5	73-79	76	3.0
		0.1	5	66-98	79	14.8
		Overall	10	66-98	77	10.5
	CGA113745 (m/z = 155 → 57)	0.01	5	74-91	82	7.9
		0.1	5	70-107	83	17.8
		Overall	10	70-107	82	13.0
Bread	CGA113745 (m/z = 155 → 69)	0.01	5	83-93	89	4.4
		0.1	5	90-93	92	1.4
		Overall	10	83-93	90	3.5
	CGA113745 (m/z = 155 → 57)	0.01	5	89-97	91	3.7
		0.1	5	90-93	91	1.2
		Overall	10	89-97	91	2.6
Beer	CGA113745 (m/z = 155 → 69)	0.01	5	101-107	104	2.2
		0.1	5	80-84	82	1.8
		Overall	10	80-107	93	12.6
	CGA113745 (m/z = 155 → 57)	0.01	5	70-101	81	15.8
		0.1	5	76-83	80	3.3
		Overall	10	70-101	80	10.8

Method GRM020.15A

The method was developed and validated for the determination of residues of cyclopropane carboxylic acid (CPCA or CGA224439) in cereal grain and processed fraction (Watson G., 2016, CA876_10002 and CGA179500_11015). Samples of brewing and baking matrices (4 g) are extracted by maceration with an acetonitrile/acidified water mixture. The contents of a Supel QuE Citrate (EN) tube i.e. magnesium sulphate, sodium chloride, sodium citrate dibasic sesquihydrate and sodium citrate tribasic dehydrate are added to an aliquot of the extract to partition the organic and aqueous phases. An aliquot of the organic phase is taken and derivatized with a mixture of 2-hydrazinoquinoline, triphenylphosphine and 2,2'-dipyridyl disulphide in acetonitrile by incubation at 60 °C for 1 hour. After solvent removal, residues are reconstituted in deionized water. Final determination is by high performance liquid chromatography with triple quadrupole mass spectrometric detection (LC-MS/MS). The LOQ of the method is 0.01 mg/kg, recoveries of 61–99%, the %RSDs of 2.9–15.2. Significant matrix effects (i.e. suppression > 20%) were observed in

some crop matrices tested during method validation, therefore matrix matched linearity standards were used for quantification. The method is suitable to measure CGA224439 in cereal grain and processed fraction with matrix-matched standards.

Table 14 Recoveries data of cyclopropane carboxylic acid (CPCA) from brewing and baking matrices using method GRM020.15A

Commodity	Compound	Fortification level (mg/kg)	No.	Range of Recovery (%)	Mean recovery (%)	RSD (%)
Cereal (grain)	Cyclopropane carboxylic acid (CPCA) (m/z = 228 → 160)	0.01	5	73-91	82	9.3
		0.1	5	71-99	88	11.5
		Overall	10	71-99	85	10.7
	Cyclopropane carboxylic acid (CPCA) (m/z = 228 → 69)	0.01	5	73-94	84	10.9
		0.1	5	70-98	87	12.1
		Overall	10	70-98	86	11.1
Cereal (flour)	Cyclopropane carboxylic acid (CPCA) (m/z = 228 → 160)	0.01	5	81-92	85	5.7
		0.1	5	79-99	89	8.4
		Overall	10	79-99	87	7.3
	Cyclopropane carboxylic acid (CPCA) (m/z = 228 → 69)	0.01	5	81-93	84	5.9
		0.1	5	79-98	88	8.0
		Overall	10	79-98	86	7.1
Cereal (bran)	Cyclopropane carboxylic acid (CPCA) (m/z = 228 → 160)	0.01	5	70-86	76	8.6
		0.1	5	77-86	81	4.4
		Overall	10	70-86	79	7.1
	Cyclopropane carboxylic acid (CPCA) (m/z = 228 → 69)	0.01	5	61-93	79	15.2
		0.1	5	77-86	81	4.4
		Overall	10	61-93	80	10.5
Bread	Cyclopropane carboxylic acid (CPCA) (m/z = 228 → 160)	0.01	5	61-70	66	5.9
		0.1	5	71-80	76	6.0
		Overall	10	61-80	71	9.3
	Cyclopropane carboxylic acid (CPCA) (m/z = 228 → 69)	0.01	5	63-74	68	6.7
		0.1	5	71-81	76	5.7
		Overall	10	63-81	72	8.0
Beer	Cyclopropane carboxylic acid (CPCA) (m/z = 228 → 160)	0.01	5	82-97	87	6.7
		0.1	5	88-96	92	3.1
		Overall	10	82-97	89	5.6
	Cyclopropane carboxylic acid (CPCA) (m/z = 228 → 69)	0.01	5	82-98	87	7.4
		0.1	5	87-95	91	2.9
		Overall	10	82-98	89	5.6

Multi-residue method – QuEChERS

The Meeting received validation and independent laboratory validation of QuEChERS method for determination of trinexapac acid in crops and animal matrices. The analytical method is based on the QuEChERS (EN 15662:2009-02) multi-residue method.

Crop matrices

In the validation (Richter S., 2015CGA179500_10993) and independent laboratory validation (Brown D., 2015, CGA179500_11005) for crop matrices, water was added to the samples (lettuce, whole orange, wheat grain, dried broad bean, oilseed rape seed) to adjust water content to about 100% and samples were then extracted by shaking with acetonitrile containing 1% acetic acid. After the addition of a mixture of

magnesium sulphate, sodium chloride, and buffering citrate salts, the extracts were shaken and then centrifuged. For oilseed rape seed, extracts were transferred to freezer to freeze out fat. After centrifugation, all extracts were first diluted with water containing 1% formic acid and then with acetonitrile/water (20/80; v/v) containing 1% formic acid. Final determination was performed with high-performance liquid chromatography with triple quadrupole mass-spectrometric detection (LC-MS/MS), monitoring the following transitions: primary transition = *m/z* 223 → 83, confirmatory transition = *m/z* 223 → 135. The LOQ of the method is 0.01 mg/kg, the recovery of 68–101%, 69–102%, RSD(%) of 1–9.3, 1–9.5. Significant matrix effects (suppression) were observed for whole orange (-18 to -38%) and oilseed rape seed (-49 to -61%). The calibration standards in matrix were used for calibration and quantification of the analyte in all matrices. The method is suitable to measure free trinexapac acid in high water content, high acid content and high water content, high oil content and intermediate water content, and high starch content matrices with matrix-matched standards.

Table 15 Recoveries of trinexapac acid from lettuce, orange, wheat grain, dried broad bean and oilseed rape seed using the QuEChERS multi-residue method (method validation)

Commodity	Compound	Fortification level (mg/kg)	No.	Range of Recovery (%)	Mean recovery (%)	RSD (%)
Lettuce (leaves)	Trinexapac acid (m/z = 223 → 83; primary transition)	0.01	5	84–89	87	3
		0.1	5	87–91	89	2
		Overall	10	84–91	88	2
	Trinexapac acid (m/z = 223 → 135; confirmatory transition)	0.01	5	86–96	89	5
		0.1	5	87–91	89	2
		Overall	10	86–96	89	3
Orange (whole fruit)	Trinexapac acid (m/z = 223 → 83; primary transition)	0.01	5	78–90	86	5
		0.1	5	92–97	95	2
		Overall	10	78–97	90	6
	Trinexapac acid (m/z = 223 → 135; confirmatory transition)	0.01	5	83–91	88	3
		0.1	5	92–97	95	2
		Overall	10	83–97	91	5
Wheat (grain)	Trinexapac acid (m/z = 223 → 83; primary transition)	0.01	5	77–84	79	4
		0.1	5	87–90	89	2
		Overall	10	77–90	84	6
	Trinexapac acid (m/z = 223 → 135; confirmatory transition)	0.01	5	83–88	85	2
		0.1	5	87–89	88	1
		Overall	10	83–89	86	2
Broad bean (dry seeds)	Trinexapac acid (m/z = 223 → 83; primary transition)	0.01	5	70–77	74	4
		0.1	5	74–80	77	4
		Overall	10	70–80	75	4
	Trinexapac acid (m/z = 223 → 135; confirmatory transition)	0.01	5	72–81	77	4
		0.1	5	74–81	77	4
		Overall	10	72–81	77	4
Oilseed Rape Seed	Trinexapac acid (m/z = 223 → 83; primary transition)	0.01	5	72 - 84	78	6
		0.1	5	72–75	73	2
		Overall	10	72–84	76	5
	Trinexapac acid (m/z = 223 → 135; confirmatory transition)	0.01	5	72–87	82	7
		0.1	5	72–75	74	2
		Overall	10	72–87	78	7

Table 16 Recoveries of trinexapac acid from lettuce, orange, wheat grain, dried broad bean and oilseed rape seed using the QuEChERS multi-residue method (independent laboratory validation)

Commodity	Compound	Fortification level (mg/kg)	No.	Range of Recovery (%)	Mean recovery (%)	RSD (%)
Lettuce (leaves)	Trinexapac acid (m/z = 223 → 83; primary transition)	0.01	5	80–90	83	5.1
		0.1	5	94–101	98	3.1
		Overall	10	80–101	91	9.3
	Trinexapac acid (m/z = 223 → 135; confirmatory transition)	0.01	5	84–90	86	2.7
		0.1	5	96–101	98	2.3
		Overall	10	84–101	92	7.2
Orange (whole fruit)	Trinexapac acid (m/z = 223 → 83; primary transition)	0.01	5	83–99	91	6.3
		0.1	5	94–99	97	2.2
		Overall	10	83–99	94	5.3
	Trinexapac acid (m/z = 223 → 135; confirmatory transition)	0.01	5	93–106	98	5.1
		0.1	5	95–101	98	2.3
		Overall	10	93–106	98	3.7
Wheat (grain)	Trinexapac acid (m/z = 223 → 83; primary transition)	0.01	5	78–91	84	5.6
		0.1	5	89–99	94	3.7
		Overall	10	78–99	89	7.3
	Trinexapac acid (m/z = 223 → 135; confirmatory transition)	0.01	5	80–89	82	4.9
		0.1	5	92–98	94	2.1
		Overall	10	80–98	88	7.9
Broad bean (dry seeds)	Trinexapac acid (m/z = 223 → 83; primary transition)	0.01	5	76–87	83	5.1
		0.1	5	81–90	86	3.9
		Overall	10	76–90	84	4.6
	Trinexapac acid (m/z = 223 → 135; confirmatory transition)	0.01	5	81–90	86	3.9
		0.1	5	83–91	87	3.4
		Overall	10	81–91	86	3.5
Oilseed Rape Seed	Trinexapac acid (m/z = 223 → 83; primary transition)	0.01	5	71–78	74	4.1
		0.1	5	76–89	82	5.6
		Overall	10	71–89	78	7.1
	Trinexapac acid (m/z = 223 → 135; confirmatory transition)	0.01	5	68–77	73	5.2
		0.1	5	75–87	82	5.4
		Overall	10	68–87	78	7.9

In validation (Richter S., 2015CGA179500_10995) and independent laboratory validation (Brown D., 2015, CGA179500_11006), water was added to the sample material (whole milk and egg, bovine muscle and liver, and animal fat) to adjust water content to about 100% and samples were then extracted by shaking with acetonitrile containing 1% acetic acid. After the addition of a mixture of magnesium sulphate, sodium chloride, and buffering citrate salts, the extracts were shaken and then centrifuged. For fat, sample extracts were transferred to freezer to freeze out the fat. After centrifugation, all extracts were diluted first with water containing 1% formic acid and then with acetonitrile/water (20/80, v/v) containing 1% formic acid. Final determination was performed with high-performance liquid chromatography with triple quadrupole mass-spectrometric detection (LC-MS/MS), monitoring the following transitions: primary transition = *m/z* 223→135, confirmatory transition = *m/z* 223→83. The LOQ of the method is 0.01 mg/kg, the recovery of 69–102%, RSD (%) of 1–9.5. As significant matrix effects (enhancement) above 20% were observed for milk (18.2-23 %) in the independent laboratory validation, matrix matched standards were recommended for calibration and quantification of the analyte in all matrices. The method is suitable to measure free trinexapac acid in animal matrices with matrix-matched standards.

Table 17 Recoveries of trinexapac acid from animal commodities using the QuEChERS multi-residue method (method validation)

Commodity	Compound	Fortification level (mg/kg)	No.	Range of Recovery (%)	Mean recovery (%)	RSD (%)
Muscle (bovine)	Trinexapac acid (*m/z* = 223 → 135; primary transition)	0.01	5	70–72	71	1
		0.1	5	70–78	74	4
		Overall	10	70–78	72	4
	Trinexapac acid (*m/z* = 223 → 83; confirmatory transition)	0.01	5	69–78	72	5
		0.1	5	69–76	72	4
		Overall	10	69–78	72	4
Liver (bovine)	Trinexapac acid (*m/z* = 223 → 135; primary transition)	0.01	5	74–78	76	2
		0.1	5	82–84	83	1
		Overall	10	74–84	79	5
	Trinexapac acid (*m/z* = 223 → 83; confirmatory transition)	0.01	5	71–80	77	5
		0.1	5	81–84	82	1
		Overall	10	71–84	80	4
Fat (bovine)	Trinexapac acid (*m/z* = 223 → 135; primary transition)	0.01	5	94–100	96	3
		0.1	5	92–94	93	1
		Overall	10	92–100	94	2
	Trinexapac acid (*m/z* = 223 → 83; confirmatory transition)	0.01	5	91–96	94	2
		0.1	5	91–94	92	1
		Overall	10	91–96	93	2
Whole Milk (cow)	Trinexapac acid (*m/z* = 223 → 135; primary transition)	0.01	5	84–90	86	3
		0.1	5	91–94	93	2
		Overall	10	84–94	89	5
	Trinexapac acid (*m/z* = 223 → 83; confirmatory transition)	0.01	5	82–87	84	3
		0.1	5	90–94	93	2
		Overall	10	82–94	88	6
Egg (hen)	Trinexapac acid (*m/z* = 223 → 135; primary transition)	0.01	5	81–84	82	2
		0.1	5	85–89	87	2
		Overall	10	81–89	85	3
	Trinexapac acid (*m/z* = 223 → 83; confirmatory transition)	0.01	5	79–83	81	2
		0.1	5	85–89	87	2
		Overall	10	79–89	84	5

Table 18 Recoveries of trinexapac acid from animal commodities using the QuEChERS multi-residue method (independent laboratory validation)

Commodity	Compound	Fortification level (mg/kg)	No.	Range of Recovery (%)	Mean recovery (%)	RSD (%)
Muscle (bovine)	Trinexapac acid (*m/z* = 223 → 135; primary transition)	0.01	5	85–91	88	3.2
		0.1	5	98–102	99	1.7
		Overall	10	85–102	93	6.8
	Trinexapac acid (*m/z* = 223 → 83; confirmatory transition)	0.01	5	77–98	91	9.5
		0.1	5	98–100	99	0.8
		Overall	10	77–100	95	7.7
Liver (bovine)	Trinexapac acid (*m/z* = 223 → 135; primary transition)	0.01	5	78–89	86	5.5
		0.1	5	101–111	106	3.4
		Overall	10	78–111	96	11.7
		0.01	5	79–84	82	2.7
		0.1	5	101–110	105	3.4

Commodity	Compound	Fortification level (mg/kg)	No.	Range of Recovery (%)	Mean recovery (%)	RSD (%)
	Trinexapac acid (*m/z* = 223 → 83; confirmatory transition)	Overall	10	79–110	93	13.4
Fat (bovine)	Trinexapac acid (*m/z* = 223 → 135; primary transition)	0.01	5	76–89	82	6.5
		0.1	5	92–104	95	5.0
		Overall	10	76–104	88	9.8
	Trinexapac acid (*m/z* = 223 → 83; confirmatory transition)	0.01	5	78–89	84	5.3
		0.1	5	93–102	97	3.7
		Overall	10	78–102	90	8.7
Whole Milk (cow)	Trinexapac acid (*m/z* = 223 → 135; primary transition)	0.01	5	75–87	79	6.3
		0.1	5	94–99	97	2.0
		Overall	10	75–99	88	11.3
	Trinexapac acid (*m/z* = 223 → 83; confirmatory transition)	0.01	5	84–93	87	4.3
		0.1	5	92–99	96	3.2
		Overall	10	84–99	91	6.0
Egg (hen)	Trinexapac acid (*m/z* = 223 → 135; primary transition)	0.01	5	74–80	77	3.3
		0.1	5	88–91	89	1.4
		Overall	10	74–91	83	8.5
	Trinexapac acid (*m/z* = 223 → 83; confirmatory transition)	0.01	5	80–83	81	1.3
		0.1	5	89–91	90	0.6
		Overall	10	80–91	86	5.4

Stability of residues in stored analytical samples

Studies of the stability cyclopropane carboxylic acid in plant commodities following freezer storage of samples were made available to the Meeting (Watson G., 2017, CGA876_10009). Samples of cereal grain, flour, bran, bread and beer were fortified with cyclopropane carboxylic acid (CGA224439) at a nominal rate of 0.1 mg/kg. Five sub-samples were immediately taken and analysed for residues of the fortified material. The remaining samples were stored deep frozen at approximately <-18 °C for up to 12 months with duplicate sub-samples being taken at intervals and analysed. Residues of cyclopropane carboxylic acid were analysed according to Analytical Method GRM020.15A.

The analytical method was validated by running a set of concurrent recoveries during analysis of stored samples. The recovery data of cyclopropane carboxylic acid recovered in the stored samples after the storage interval of up to 12 months are presented in table 19 below.

The tables summarize the average % recovery at each interval. Average uncorrected recoveries were in the range of 71–109% with two exceptions for bran at storage intervals of ~6 months, where average recoveries were in the range of 55–67%. However, residues of cyclopropane carboxylic acid were considered to be stable in cereal bran, since the average recovery was acceptable at the 12-month storage interval indicating a methodical problem at the 6-month interval.

Table 19 Stability of cyclopropane carboxylic acid in cereal grain and processed commodities at -18 °C

Matrix	Fortification Level (mg/kg)	Storage Interval (days/months)	Individual Recovered Residues (mg/kg)	Individual Recoveries (%) [a]	Procedural Recoveries (%) [a]
Cereal (grain)	0.10	0/0	0.08322, 0.09152, 0.08848	83, 92, 88 (88)	94, 92 (93)
		30/1	0.09677, 0.10072	97, 101 (99)	104, 105 (105)
		104/3	0.10181, 0.10669	102, 107 (104)	109, 104 (107)
		183/6	0.09805, 0.09222	98, 92 (95)	114, 91 (102)
		365/12	0.07807, 0.08049	78, 80 (79)	112, 108 (110)
Cereal (flour)	0.10	0/0	0.11268, 0.10229, 0.09467	113, 102, 95 (103)	103, 105 (104)
		37/1	0.07600, 0.07561	76, 76 (76)	77, 78 (77)
		104/3	0.07828, 0.07308	78, 73 (76)	83, 79 (81)
		183/6	0.08133, 0.08845	81, 88 (85)	97, 86 (91)
		364/12	0.08641, 0.07936	86, 79 (83)	85, 95 (90)
Bran	0.10	0/0	0.06722, 0.07502, 0.07512	67, 75, 75 (72)	75, 91 (83)
		30/1	0.09783, 0.09662	98, 97 (97)	101, 105 (103)
		105/3	0.06886, 0.07808	69, 78 (73)	93, 101 (97)
		184/6	0.07283, 0.06128	73, 61 (67)	93, 108 (101)
		196/6	0.05626, 0.05416	56, 54 (55)	84, 76 (80)
		364/12	0.09306, 0.07669	93, 77 (85)	109, 117 (113)
Bread	0.10	0/0	0.09550, 0.08740, 0.09883	96, 87, 99 (94)	87, 94 (90)
		30/1	0.09592, 0.11597	96, 116 (106)	91, 94 (93)
		104/3	0.10625, 0.09554	106, 96 (101)	103, 96 (100)
		195/6	0.07062, 0.07057	71, 71 (71)	72, 73 (73)
		363/12	0.11006, 0.10814	110, 108 (109)	102, 117 (110)
Beer	0.10	0/0	0.09871, 0.10130, 0.09409	99, 101, 94 (98)	96, 97 (97)
		30/1	0.09223, 0.09028	92, 90 (91)	101, 98 (99)
		104/3	0.09221, 0.08461	92, 85 (88)	86, 81 (83)
		183/6	0.09601, 0.08039	96, 80 (88)	87, 84 (86)
		364/12	0.09758, 0.09229	98, 92 (95)	104, 102 (103)

[a] Mean recoveries are given in brackets

Residues of cyclopropane carboxylic acid have been shown to be stable in cereal grain, flour, bran, bread and beer when stored deep frozen at <-18 °C for at least 12 months. The demonstrated stability intervals on frozen storage encompass the duration of sample storage in the residue trials evaluated by the Meeting.

USE PATTERN

The meeting received GAP information for the use of trinexapac-ethyl on rice and rye.

Table 20 Registered uses of trinexapac-ethyl on rice and rye

Crop	Country	Formulation	Application		Spray				PHI (days)
			Method	Max (g ai/ha)	Max (g ai/hL)	L/ha	Max No. (g ai/ha/ season)	Growth stage	
020 Cereal grains									
Rye	USA	2EC	Foliar	123	not defined	≥94	1 (123)	29–32 BBCH	45
Rice	USA	2EC	Foliar	50	not defined		1 (50)	29–32 BBCH	50

RESULTS OF SUPERVISED RESIDUE TRIALS ON CROPS

The Meeting received information on supervised field trials for trinexapac-ethyl on rice. 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. The residue data are recorded unadjusted for recovery. In many trials, especially those conducted in the USA, duplicate or multiple field samples from replicate plots were taken at each sampling period and were analysed separately. Each value is reported in the tables.

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.

Rice

Sixteen supervised residue trials were conducted on rice in the USA in 2012 (Smith N., 2014, A7725M_50010). In all trials an EC formulation containing trinexapac-ethyl at 250 g/L was applied once to rice by foliar spray at crop growing stage BBCH 30–32 and a nominal rate of 50 g ai/ha. In two trials an additional plot was established at an exaggerated rate (3×) using a nominal application rate of 150 g ai/ha to generate samples for processing. Samples of grain and straw were taken at harvest, 51–100 days after application. In two trials, designed as decline trials, two additional sampling events each were established before and after sampling of the harvest sample. Harvest samples were taken in duplicate whereas additional decline samples were taken once for analytical determination.

In all trials, samples of rice grain and straw were analysed for total residues of trinexapac acid (free and conjugated) using the validated residue analytical method GRM020.01A (HPLC-MS/MS). Mean recoveries of trinexapac acid in rice grain and straw were within the acceptable range of 70–120%. Concurrent determination of residues in untreated samples gave residues below the LOQ.

Samples of rice (grain and straw) were stored deep-frozen for a maximum of 12 months (365 days). Summaries of the trial results are given in Table 21.

Table 21 Residues of trinexapac acid (free and conjugated) on rice from trials conducted in the USA

Location Country, Year Trial No. (Variety)	Application Rate (g ai/ha)	Growth Stage at Application	PHI (days)	Crop Part	Residue (mg/kg) Total trinexapac acid (free and conjugated)	Reference
Proctor, Arkansas, USA, 2012 Trial: TK0044924-01 (Roy J)	50.4	BBCH 30-32	74	grain	0.061, 0.052 (0.057)	Report: TK0044924
Pollard, Arkansas, USA, 2012 Trial: TK0044924-02 (RiceTec XL729)	50.4	BBCH 30-32	85	grain	0.054, 0.045 (<u>0.050</u>)	

Location Country, Year Trial No. (Variety)	Application Rate (g ai/ha)	Growth Stage at Application	PHI (days)	Crop Part	Residue (mg/kg) Total trinexapac acid (free and conjugated)	Reference
Cheneyville, Louisiana, USA, 2012 Trial: TK0044924-03 (CL 111)	51.5	BBCH 30-32	53	grain	0.053, 0.055 (0.054)	
Ville Platte, Louisiana, USA, 2012 Trial: TK0044924-04 (XL 729)	51.5	BBCH 30-32	51	grain	0.023, 0.022 (0.023)	
Washington, Louisiana, USA, 2012 Trial: TK0044924-05 (CL 151)	49.3	BBCH 30-32	60	grain	0.16, 0.18 (0.17)	
Proctor, Arkansas, USA, 2012 Trial: TK0044924-06 (CL 261)	50.4	BBCH 31-32	68	grain	0.021, 0.021 (0.021)	
Washington, Louisiana, USA, 2012 Trial: TK0044924-07 (Cocodrie)	49.3	BBCH 30.32	60	grain	0.24, 0.21 (0.23)	
Washington, Louisiana, USA, 2012 Trial: TK0044924-08 (Cypress)	51..5	BBCH 30-32	60	grain	0.17, 0.18 (0.18)	
Proctor, Arkansas, USA, 2012 Trial: TK0044924-09 (Wells)	50.4	BBCH 31-32	65 72 79 85 94	grain	0.038 0.047 0.078, 0.067 (0.073) 0.054 0.062	
Washington, Louisiana, USA, 2012 Trial: TK0044924-10 (Cypress)	51.5	BBCH 30-32	54	grain	0.17, 0.18 (0.18)	
	150.1	BBCH 30-32	54	grain polished hulls bran	0.77, 0.70, 0.22 (0.56) 0.92 0.33 2.5	
Fisk, Missouri, USA, 2012 Trial: TK0044924-11 (RiceTec XP 753)	50.4	BBCH 30-32	91	grain	0.034, 0.032 (0.033)	
	151.2	BBCH 30-32	91	grain polished hulls bran	0.10, 0.094, 0.097 (0.097) 0.10 0.022 0.56	

Location Country, Year Trial No. (Variety)	Application Rate (g ai/ha)	Growth Stage at Application	PHI (days)	Crop Part	Residue (mg/kg) Total trinexapac acid (free and conjugated)	Reference
Fisk, Missouri, USA, 2012 Trial: TK0044924-12 (Francis)	50.4	BBCH 30-32	85	grain	0.033, 0.036 (0.035)	
East Bernard, Texas, USA, 2012 Trial: TK0044924-13 (Presidio)	49.3	BBCH 31-32	90	grain	0.10, 0.10 (0.10)	
East Bernard, Texas, USA, 2012 Trial: TK0044924-14 (Cheniere)	45.9	BBCH 30-31	75	grain	0.017, 0.017 (0.017)	
Porterville, California, USA. 2012 Trial: TK0044924-15 (Koshihikari)	50.4	BBCH 30-32	85 92 99 106 113	grain	<0.01 <0.01 <0.01, <0.01 (<0.01) <0.01 <0.01	
Porterville, California, USA, 2012 Trial: TK0044924-16 (Koshihikari)	51.5	BBCH 30-32	100	grain	0.082, 0.065 (0.074)	

Animal feed commodities

Rice straws

The Meeting received supervised residue trials on rice straw.

Table 22

: Residues of trinexapac acid (free and conjugated) on rice from trials conducted in the USA

Location Country, Year Trial No. (Variety)	Application Rate (g ai/ha)	Growth Stage at Application	PHI (days)	Crop Part	Residue (mg/kg) Total trinexapac acid (free and conjugated)	Reference
Proctor, Arkansas, USA, 2012 Trial: TK0044924-01 (Roy J)	50.4	BBCH 30-32	74	straw	0.012, 0.017 (0.015)	Report: TK0044924
Pollard, Arkansas, USA, 2012 Trial: TK0044924-02 (RiceTec XL729)	50.4	BBCH 30-32	85	straw	<0.01, <0.01 (<0.01)	

Location Country, Year Trial No. (Variety)	Application Rate (g ai/ha)	Growth Stage at Application	PHI (days)	Crop Part	Residue (mg/kg) Total trinexapac acid (free and conjugated)	Reference
Cheneyville, Louisiana, USA, 2012 Trial: TK0044924-03 (CL 111)	51.5	BBCH 30-32	53	straw	0.018, 0.013 (0.016)	
Ville Platte, Louisiana, USA, 2012 Trial: TK0044924-04 (XL 729)	51.5	BBCH 30-32	51	straw	<0.01, <0.01 (<0.01)	
Washington, Louisiana, USA, 2012 Trial: TK0044924-05 (CL 151)	49.3	BBCH 30-32	60	straw	0.045, 0.034 (0.040)	
Proctor, Arkansas, USA, 2012 Trial: TK0044924-06 (CL 261)	50.4	BBCH 31-32	68	grain straw	0.021, 0.021 (0.021) <0.01, <0.01 (<0.01)	
Washington, Louisiana, USA, 2012 Trial: TK0044924-07 (Cocodrie)	49.3	BBCH 30.32	60	straw	0.036, 0.035 (0.036)	
Washington, Louisiana, USA, 2012 Trial: TK0044924-08 (Cypress)	51..5	BBCH 30-32	60	straw	0.027, 0.024 (0.026)	
Proctor, Arkansas, USA, 2012 Trial: TK0044924-09 (Wells)	50.4	BBCH 31-32	65 72 79 85 94	straw	<0.01 <0.01 0.013, <0.01 (0.012) <0.01 <0.01	
Washington, Louisiana, USA, 2012 Trial: TK0044924-10 (Cypress)	51.5	BBCH 30-32	54	straw	0.044, 0.045 (0.045)	
Fisk, Missouri, USA, 2012 Trial: TK0044924-11 (RiceTec XP 753)	50.4	BBCH 30-32	91	straw	<0.01, <0.01 (<0.01)	
Fisk, Missouri, USA, 2012 Trial: TK0044924-12 (Francis)	50.4	BBCH 30-32	85	straw	0.010, <0.01 (0.010)	
East Bernard, Texas, USA, 2012 Trial: TK0044924-13 (Presidio)	49.3	BBCH 31-32	90	straw	0.017, 0.023 (0.020)	
East Bernard, Texas, USA, 2012 Trial: TK0044924-14 (Cheniere)	45.9	BBCH 30-31	75	straw	<0.01, <0.01 (<0.01)	

Location Country, Year Trial No. (Variety)	Application Rate (g ai/ha)	Growth Stage at Application	PHI (days)	Crop Part	Residue (mg/kg) Total trinexapac acid (free and conjugated)	Reference
Porterville, California, USA. 2012 Trial: TK0044924-15 (Koshihikari)	50.4	BBCH 30-32	85 92 99 106 113	straw	<0.01 <0.01 <0.01, <0.01 (<0.01) <0.01 <0.01	
Porterville, California, USA, 2012 Trial: TK0044924-16 (Koshihikari)	51.5	BBCH 30-32	100	straw	<0.01, 0.024 (0.017)	

FATE OF RESIDUES IN STORAGE AND PROCESSING

The Meeting received new information on high temperature hydrolysis of trinexapac acid and trials on the processing of barley, wheat and rice.

Hydrolysis

The 2013 JMPR concluded that trinexapac-ethyl was not degraded during the simulation of pasteurisation (pH 4, 90 °C, 20 minutes), baking, boiling and brewing (pH 5, 100 °C, 60 minutes) and sterilisation (pH 6, 120 °C, 20 minutes). The 2013 JMPR also concluded that trinexapac acid undergoes limited degradation during the simulation of pasteurisation (pH 4, 90 °C, 25 minutes), baking, boiling and brewing (pH 5, 100 °C, 60 minutes) and sterilisation (pH 6, 120 °C, 20 minutes), with the major degradates being CGA113745 (10–12%) and CGA313458 (16–21%).

The hydrolytic stability of [^{14}C]trinexapac acid was investigated in aqueous buffer solutions at pH 4, 5 and 6 at temperatures of 90 °C (20 minutes), 100 °C (60 minutes) and 120 °C (20 minutes), to represent the processes of pasteurization, baking/ brewing/ boiling and sterilization, respectively (Scullion P., 2012, CGA179500_11002). The test item [hydroxymethylene-^{14}C]trinexapac acid was generated at the test site by hydrolysis of the [^{14}C]trinexapac-ethyl. 17.3 mg of [^{14}C]trinexapac-ethyl was dissolved in 20 mL water and placed in an ultrasonic bath for 5 minutes. The solution was adjusted to pH 9 by addition of 3 drops of ammonium hydroxide (25% v/v). The solution was heated for 48 hours at 40 C and was used without further modification for application of the test item. Buffered solutions (15 mL) of [^{14}C]trinexapac acid (1 mg/L) were incubated in duplicates in high pressure glass flasks immersed in an oil bath for the specific durations and temperatures.

At time 0 and after incubation (20 or 60 minutes) the samples were taken, measured for total radioactivity and analysed for the nature of degradates. The total radioactivity in the sample solutions was determined in triplicate by LSC, the concentration of test item and degradation products in the samples were determined by HPLC-UV at an LOQ of 0.005 mg/L. Selected samples were analysed by one-dimensional TLC in order to confirm the results obtained by HPLC-UV. Identification of hydrolysis products was performed by HPLC-MS/MS. For characterization and identification of the hydrolysis product M5 also NMR-spectroscopy was used.

The mean recoveries of radioactivity for the test item were 97.9 ± 0.3% (pH 4; 90 °C), 98.4 ± 0.1% (pH5; 100 °C) and 98.8 ± 0.3% (pH 6; 120 C).

At pH4 and 90 °C (20 minutes, simulating pasteurisation), [^{14}C]trinexapac acid was hydrolysed to 85.8% of the applied radioactivity. The pH 4 control sample kept at ambient temperature also showed a small amount of degradation with [^{14}C]trinexapac acid corresponding to 91.8% of applied radioactivity.

At pH 5 and 100 °C (60 minutes, simulating baking/ brewing/ boiling) the test item was hydrolysed to 63.2% of applied radioactivity. Two main hydrolysis products M5 and M6 were detected at levels of 16.3% and 17.7% of applied radioactivity, respectively. M6 was identified as R3A (2-((Z)-4-cyclopropyl-4-hydroxy-2-oxo-but-3-enyl)-succinic acid CGA313458) but M5 did not correspond to any of the available reference items. A subsequent study was therefore performed to identify the transformation product M5. Following re-incubation of the compound at a higher concentration, M5 was fractionated and concentrated before analysis by NMR. M5 was shown to be cyclopropane carboxylic acid.

At pH 6 and 120 °C (20 minutes, simulating the process of sterilisation) the test item corresponded to 82.1% of applied radioactivity.

Table 23 Distribution of radioactivity of [^{14}C]trinexapac acid in the buffer solutions before and after incubation at different temperatures

Pattern (mean % of applied)	Process					
	pasteurization pH 4 (90 °C)		baking/brewing/boiling pH 5 (100 °C)		sterilization pH 6 (120 °C)	
	0 min	20 min	0 min	60 min	0 min	20 min
Trinexapac acid	91.8	85.8	98.5	63.2	98.6	82.1
M1	nd	nd	nd	1.0	nd	0.9
M2	nd	0.3	nd	nd	nd	nd
M3	nd	0.3	nd	nd	nd	nd
M4	nd	nd	nd	nd	nd	4.0
M5 (cyclopropane carboxylic acid)	nd	5.4	nd	16.3	nd	3.8
M6 (CGA313458)	nd	4.7	nd	17.7	nd	8.4
M7	nd	1.0	nd	nd	nd	nd
M8	5.9	0.7	nd	nd	nd	nd

nd: Not detected

In summary, trinexapac acid is hydrolytically stable under conditions representative of pasteurisation and sterilisation but degrades in significant amounts to cyclopropane carboxylic acid (CGA224439) and CGA313458 under conditions representative of baking, brewing and boiling.

In another study (Flörchinger M., 2008, CGA179500_11004), the hydrolytic stability of [1, 6-cyclohexyl and hydroxymethylene-^{14}C]trinexapac acid was investigated in aqueous buffer solutions. 50 mL of citrate buffer (pH 4, and 6) or acetate buffer (pH 5) was added to the test vials followed by 10 µL of the radioactive standard (10 µCi/10 µL in acetone) and 90 µL of cold standard (2.31 g/L in acetone) to obtain a concentration of 5 mg/L trinexapac acid and an overall radioactivity of 10 µCi per vial. Aliquots of each buffer solution were stabilised with 1/10 volume acidified acetonitrile, the total radioactivity determined by LSC and characterised using TLC to give the pre-processing values. Duplicate preparations of each treatment buffer solution were weighed and treated according to pH 4 for 20 minutes at 90 °C (Pasteurisation), pH 5 for 60 minutes at 100 °C (Baking, brewing) and pH 6 for 20 minutes at 120 °C (Sterilisation). All samples were kept in the dark to avoid prospective degradation as a result of photolysis. After equilibration of the samples at ambient temperature, the test and control samples were weighed and then stabilised by addition of 1/10 volume acidified acetonitrile before being taken for quantification by LSC and characterisation by TLC.

No significant loss of weight during processing was observed. The post-hydrolysis quantification results based on the actual amount of radioactivity applied to the solutions shows recoveries ranging from 96.6 to 101.5% for the test samples and 98.5% to 103.6% for the control samples.

The majority of the recovered radioactivity was [^{14}C]-trinexapac acid. The TLC results show that no metabolites are formed during processing under pH 4, 90 °C, 20 min and pH 6, 100 °C, 60 min. The degradates of 3-carboxyl-7-cyclopropyl-5,7-dioxoheptanoic acid (CGA313458, 3.5%) and cyclodion acid (CGA 113745, 3.4%) were observed under pH 5, 100 °C, 60 min. No other hydrolysis products were formed during incubations. The levels of [^{14}C]trinexapac acid in the test samples are summarised in Table 24.

Table 24 Distribution of radioactivity of [^{14}C]trinexapac acid in the buffer solutions before and after incubation at different temperatures

Pattern (mean % of applied)	Process					
	pasteurization pH 4 (90 °C)		baking/brewing/boiling pH 5 (100 °C)		sterilization pH 6 (120 °C)	
	0 min	20 min	0 min	60 min	0 min	20 min
Trinexapac acid	96.1	95.2	96.3	93.1	98.9	97.7
3-carboxyl-7-cyclopropyl-5,7-dioxoheptanoic acid	2.7	2.7	2.0	3.5	1.1	2.3
Cyclodion acid	1.7	2.2	1.7	3.4	-	-
Pattern (% of applied)	pasteurization pH 4 (unheated)		baking/brewing/boiling pH 5 (unheated)		sterilization pH 6 (unheated)	
	0 min	20 min	0 min	60 min	0 min	20 min
Trinexapac acid	96.6	96.8	96.2	97.5	99.2	98.8
3-carboxyl-7-cyclopropyl-5,7-dioxoheptanoic acid	1.9	1.6	2.5	1.8	0.8	1.2
Cyclodion acid	1.5	1.6	1.4	0.7	-	-

Trinexapac acid is hydrolytically stable under conditions representative of pasteurisation, baking, brewing, boiling and sterilisation. The two minor metabolites were cyclodion acid and 3-carboxyl-7-cyclopropyl-5,7-dioxoheptanoic acid. So, the hydrolysis profiles of trinexapac acid observed were aligned to the conclusions of 2013 JMPR.

Barley

Two residue field trials were conducted on barley in Italy and North Spain during 2015 (MacDougall J., 2016, Syngenta file no. A8587F_10526; Langridge G., 2016, Syngenta file no. CGA313458_10010; Watson G., 2016, Syngenta file no. CA876_10004). Trinexapac-ethyl was applied to field barley as micro-emulsion (ME) formulation containing trinexapac-ethyl at 250 g/L. One application was made at a target rate of 400 g ai/ha for trinexapac-ethyl. Treated and control samples were collected at normal commercial harvest (NCH) for processing and for residue analysis. Samples were shipped frozen to the analytical facility for residue analysis and at ambient temperature to the processing facility. Each field trial generated a treated and an untreated field sample of grain. The untreated and treated grain samples were put through the relevant processes. The treated grain from each trial was split into 2 portions (T1 and T2) with both being taken through the procedures.

Barley grain was processed into pot barley, pearl barley, flour, bran, brewing malt, malt sprouts, brewer's grain (dried), brewer's yeast and beer. Relevant industrial practices and standardized procedures were applied to simulate the common processes used by industry.

Pot and pearl barley: grain samples were cleaned and an optimal moisture content of barley grain of ca 14% was achieved. The samples were then hulled until the stipulated abrasion for pot barley (20–25%) and pearl barley (30–35%) was reached.

Flour and bran: Grain samples were hulled until the stipulated abrasion of 30–35% was reached. Abrasion was then sieved to bran and flour. The hulled grain was milled to flour. Afterwards the flour of the sieved abrasion and the flour of the milled hulled grain were then mixed.

Brewing Malt and Malt Sprouts: Grain samples were cleaned and sieved. Following sieving, a combined wet and dry steeping was conducted until a degree of steeping between 42–45% was achieved. Germination was conducted and samples were placed into a kiln for drying. Following kiln-drying the germs were removed mechanically using a trimmer and the malt sprouts were sampled. The malt was stored at room temperature until brewing to produce brewing malt. Brewing malt samples were collected directly before brewing began.

Brewer's grain (dried): Samples of brewing malt were taken and mashed to allow enzyme degradation. The brewer's malt was milled and then mixed with brew water. Mashing was then started in a heatable tun. After mash boiling, the wort was separate from the insoluble malt components (brewer's grain). The extract remaining in the brewer's grain was then extracted by washing with hot water. The wort separation was done using a refining vat. After separation, brewer's grain was dried at 50 °C until a dry matter content of <10% was reached and sampled as "brewer's grain dried".

Brewer's yeast and beer: During production of brewer's yeast, hop pellets were added and the separated wort boiled to deactivate the enzymes of the malt, sterilise the wort, extract and isomerise the essential components of the hops, precipitate high molecular proteins and expel unwanted aromatic substances. After boiling, the flocs (hops draff) were separated in a whirlpool causing the sludge to deposit on the bottom in the shape of a cone. An intra-plant circulation was used for cooling and ventilating. Oxygen was added to prepare the conditions for the start of fermentation. The pure culture yeast fermented sugar of the wort to alcohol and CO_2 as well as unwanted byproducts (diacetyl, higher alcohols and others). Primary fermentation was carried out in bottom fermentation containers. As soon as the extract content of the fermented young beer was 2% higher than the final attenuation, storage began. Before maturation the young beer was cooled down. During the main fermentation the yeast was deposited on the tank bottom. At the beginning of maturation the young beer was stored at room temperature (warm maturation to break down the diacetyl) in casks. The young beer was then stored under pressure (approximately 0.7-1.2 bar) at ca 0–2 °C (cold maturation) for 4 weeks. During this time the remaining extract was fermented. Unwanted flavour and odorous substances were decomposed or expelled. The rack beer was filtered using a special filter combination. During filtration, all organisms harming the beer (bacteria and yeast) were removed and sludge particles were separated. The final product beer was then sampled.

Samples of barley grain and processed fractions were analysed for residues of free trinexapac acid, total trinexapac acid (free and conjugated) using the residue analytical methods GRM020.05A, GRM020.009A, respectively. The LOQ for all analytes in all matrices was 0.01 mg/kg except for trinexapac acid in straw (0.05 mg/kg). Samples were stored frozen for up to 12 months from sampling to analysis.

Residues of trinexapac acid (free and total) in barley grain samples prior to processing and after processing into pot barley, pearl barley, flour, bran, brewing malt, malt sprouts, brewer's grain (dried), brewer's yeast and beer are presented in Table 25.

Table 25 The residues of trinexapac acid (free and total) in barley grain samples prior to processing and after processing at its processing factor

Trial / Commodity		Residues (mg/kg)		Processing Factor	
		Trinexapac acid (free)	Trinexapac acid (total)	Trinexapac acid (free)	Trinexapac acid (total)
Trial T1A	Grain (RAC)	0.23 [a]	1.75 [a]	–	–
	Pot Barley	0.13	0.39	0.57	0.22
	Pearled Barley	0.12	0.30	0.52	0.17
	Grain (RAC)	0.21 [a]	1.80 [a]	–	–
	Barley Bran	0.39	0.67	1.86	0.37
	Barley Flour	0.16	0.89	0.76	0.49
	Grain (RAC)	0.22 [a]	1.80 [a]	–	–
	Brewing Malt	0.19	0.99	0.88	0.55
	Malt Sprouts	0.25	0.22	1.16	0.12
	Brewer's Grain dry	0.07	0.24	0.33	0.13
	Brewer's Yeast	0.40	0.42	1.86	0.23
	Beer	0.02	0.11	0.09	0.06
Trial T1B	Grain (RAC)	0.23 [a]	1.87 [a]	–	–
	Pot Barley	0.14	0.45	0.61	0.24
	Pearled Barley	0.13	0.33	0.57	0.18
	Grain (RAC)	0.27 [a]	1.82 [a]	–	–
	Barley Bran	0.46	0.81	1.74	0.45
	Barley Flour	0.25	0.97	0.94	0.53
	Grain (RAC)	0.22 [a]	1.58 [a]	–	–
	Brewing Malt	0.11	0.99	0.51	0.63
	Malt Sprouts	0.24	0.18	1.12	0.11
	Brewer's Grain dry	0.07	0.19	0.33	0.12
	Brewer's Yeast	0.41	0.14	1.91	0.09
	Beer	0.04	0.11	0.19	0.07
Trial T2A	Grain (RAC)	0.16 [1)]	1.58 [a]	–	–
	Pot Barley	0.13	0.23	0.84	0.15
	Pearled Barley	0.12	0.27	0.77	0.17
	Grain (RAC)	0.21 [a]	1.64 [a]	–	–
	Barley Bran	0.17	0.20	0.81	0.12
	Barley Flour	0.17	0.58	0.81	0.35
	Grain (RAC)	0.23 [a]	1.64 [a]	–	–
	Brewing Malt	0.20	0.70	0.87	0.43
	Malt Sprouts	0.16	0.11	0.70	0.07
	Brewer's Grain dry	0.10	0.16	0.43	0.10
	Brewer's Yeast	0.36	0.30	1.57	0.18
	Beer	0.04	0.09	0.17	0.06
Trial T2B	Grain (RAC)	0.24 [a]	1.60 [a]	–	–
	Pot Barley	0.14	0.28	0.58	0.18
	Pearled Barley	0.13	0.28	0.54	0.18
	Grain (RAC)	0.22 [a]	1.64 [a]	–	–
	Barley Bran	0.16	0.28	0.74	0.17
	Barley Flour	0.19	0.60	0.88	0.37
	Grain (RAC)	0.26 [a]	1.61 [a]	–	–
	Brewing Malt	0.20	0.64	0.78	0.40
	Malt Sprouts	0.14	0.12	0.55	0.07
	Brewer's Grain dry	0.10	0.16	0.39	0.10
	Brewer's Yeast	0.39	0.27	1.53	0.17
	Beer	0.03	0.08	0.12	0.05

[a] Mean of two analyses

Wheat

The Meeting received two new studies on the effect of processing on residues of trinexapac-ethyl in wheat.

In the studies conducted in France and Spain (MacDougall J.; 2016, A8587F_10524), trinexapac-ethyl was applied to field wheat as micro-emulsion (ME) formulation containing trinexapac-ethyl at 250 g/L. One application was made at a target rate of 400 g ai/ha for trinexapac-ethyl. Treated and control samples were collected at normal commercial harvest (NCH) for processing and for residue analysis. Samples were shipped frozen to the analytical facility for residue analysis and at ambient temperature to the processing facility. Each field trial generated a treated and an untreated field sample of grain. The untreated and treated grain samples were put through the relevant processes. The treated grain from each trial was split into 2 portions (T1 and T2) with both being taken through the procedures.

Wheat grain was processed into cleaned grain, waste (offal), white flour, total bran, shorts, middlings, wholemeal flour, wholemeal bread, germ, dry gluten, dry starch and gluten feed meal. Relevant industrial practices and standardized procedures were applied to simulate the common processes used by industry.

White flour: Wheat grain specimens were cleaned using a single grading unit and a sample of cleaned grain was taken. The water content of the wheat grains was measured and adjusted. About 5 kg of wheat grains were conditioned in a kneading machine for a minimum of 5 hours to increase the water content to approximately 17%. Samples of shorts were taken. The remaining shorts were placed through a mill consisting of reduction rolls and screened. After the reduction stage, fine bran and reduction flour were recovered. After weighing, coarse bran and fine bran were combined to obtain total bran and milling flour and reduction flour were obtained to obtain white flour. White flour and total bran samples were taken. The fine brans were placed through sieves and screened. After division, middling samples were taken.

Whole-meal flour: Wheat grain specimens were cleaned with a single grading unit and a sample of cleaned grain was taken. The water content of the wheat grains was measured and adjusted. The wheat grains were conditioned in a kneading machine for a minimum of 5 hours to increase the water content to approximately 17%. Wheat grains were placed through a mill consisting of break rolls. After the break stage, coarse bran and milling flour were recovered. Shorts were placed through a mill consisting of reduction rolls and screened. After the reduction stage, fine bran and reduction flour were recovered. Coarse bran, fine bran, milling flour and reduction flour were combined to obtain whole-meal flour. A sample of whole-meal flour was taken.

Whole-meal bread: The whole-meal bread processing was made from the whole-meal flour obtained. Dry bakers yeasts were rehydrated with sugar and water. The flour, the water and the yeasts preparation were put in the kneading machine. Five minutes before the end of the kneading, salt was added. The obtained dough was placed in a pan at ambient temperature for 45 minutes. The dough was divided in several little portions and were covered and kept at ambient temperature for 15 minutes. Each portion was shaped in baguette. The baguettes were covered and kept at ambient temperature for a minimum of 2 hours. The baguette was baked in an oven at 250 °C for approximately 30 minutes. Whole-meal bread samples were taken.

Germ: Wheat grain specimens were cleaned with a single grading unit. The cleaned grains were weighed and placed in a container. The same quantity of water was added. The steeping lasted 12 h minimum. After straining, a portion of grains was set down on absorbing paper. Germs were removed from wheat grain with a cutter. A sample of germs was taken.

Gluten and starch: Wheat grain specimens were cleaned with a single grading unit. The water content of the wheat grains was measured and adjusted. The wheat grains were conditioned in a kneading

machine for a minimum of 5 hours to increase the water content to approximately 17%. Wheat grains were placed through a mill consisting of break rolls. After the break stage, coarse bran and milling flour were recovered. Shorts were placed through a mill consisting of reduction rolls and screened. After the reduction stage, fine bran and reduction flour were recovered. After weighing, milling flour and reduction flour were combined to obtain white flour. The gluten and starch separation processing was made with the obtained white flour. A dough was prepared and after rest, washed with water to separate starch milk and gluten. Wet gluten was dried in an oven regulated at 50 °C. Dry gluten samples were taken. After settling of starch milk in cold room, wet starch was dried in an oven regulated at 50 °C. Dry starch samples were taken. Dry gluten and dry starch were ground separately with a mill (hammer-type) and mixed (1/1). Gluten feed meal samples were taken.

Samples of wheat grain and processed fractions were analysed for residues of free trinexapac acid, total trinexapac acid (free and conjugated) using the residue analytical methods GRM020.05A, GRM020.009A, respectively. The LOQ for all analytes in all matrices was 0.01 mg/kg. Samples were stored for up to 15 months from sampling to analysis.

Residues of trinexapac acid (free and total) in wheat grain samples prior to processing and after processing into white flour, bran, shorts, middlings, wholemeal flour, wholemeal bread, germ, gluten (dry), starch (dry) and gluten feed meal are presented in Table 26.

Table 26: Summary of transfer factors into processed wheat products

Trial / Commodity		Residues (mg/kg)		Processing Factor	
		Trinexapac acid (free)	Trinexapac acid (total)	Trinexapac acid (free)	Trinexapac acid (total)
Trial T1A	Grain (RAC)	0.59 [a]	1.03 [a]	–	–
	Cleaned Grain	1.11	1.16	1.90	1.13
	Waste (offal)	0.31	0.97	0.53	0.94
	White Flour	0.31	0.39	0.53	0.38
	Total Bran	0.63	1.01	1.08	0.98
	Shorts	0.57	0.63	0.97	0.61
	Middlings	0.33	0.55	0.56	0.53
	Grain (RAC)	0.64 [a]	1.06 [a]	–	–
	Wholemeal Flour	0.59	0.71	0.92	0.67
	Wholemeal Bread	0.57	0.64	0.89	0.60
	Grain (RAC)	0.44 [a]	0.87 [a]	–	–
	Germ	0.42	0.34	0.97	0.39
	Grain (RAC)	0.58 [a]	1.01 [a]	–	–
	Dry Gluten	0.19	0.26	0.33	0.26
	Dry Starch	0.05	0.11	0.09	0.11
	Gluten Feed Meal	0.13	0.19	0.22	0.19
Trial T1B	Grain (RAC)	0.51 [a]	0.93 [a]	–	–
	Cleaned Grain	0.52	1.00	1.02	1.08
	Waste (offal)	0.26	0.69	0.51	0.74
	White Flour	0.31	0.43	0.61	0.46
	Total Bran	0.58	0.65	1.14	0.70
	Shorts	0.40	0.54	0.78	0.58
	Middlings	0.29	0.54	0.57	0.58
	Grain (RAC)	0.55 [a]	0.76 [a]	–	–
	Wholemeal Flour	0.50	0.76	0.91	1.01
	Wholemeal Bread	0.40	0.63	0.73	0.83
	Grain (RAC)	0.54 [a]	1.18 [a]	–	–
	Germ	0.37	0.33	0.69	0.28

Trial / Commodity		Residues (mg/kg)		Processing Factor	
		Trinexapac acid (free)	Trinexapac acid (total)	Trinexapac acid (free)	Trinexapac acid (total)
	Grain (RAC)	0.57 [a]	0.83 [a]	–	–
	Dry Gluten	0.17	0.26	0.30	0.31
	Dry Starch	0.05	0.09	0.09	0.11
	Gluten Feed Meal	0.13	0.16	0.23	0.19
Trial T2A	Grain (RAC)	0.95 [a]	2.16 [a]	–	–
	Cleaned Grain	1.10	2.44	1.16	1.13
	Waste (offal)	1.02	2.47	1.08	1.14
	White Flour	0.54	0.97	0.57	0.45
	Total Bran	1.04	2.07	1.10	0.96
	Shorts	0.88	1.04	0.93	0.48
	Middlings	0.47	1.00	0.50	0.46
	Grain (RAC)	0.82 [a]	2.35 [a]	–	–
	Wholemeal Flour	1.06	1.83	1.30	0.78
	Wholemeal Bread	0.88	1.49	1.08	0.63
	Grain (RAC)	0.92 [a]	2.34 [a]	–	–
	Germ	1.09	0.95	1.19	0.41
	Grain (RAC)	0.91 [a]	2.54 [a]	–	–
	Dry Gluten	0.25	0.52	0.27	0.20
	Dry Starch	0.08	0.13	0.09	0.05
	Gluten Feed Meal	0.15	0.42	0.16	0.17
Trial T2B	Grain (RAC)	0.94 [a]	2.49 [a]	–	–
	Cleaned Grain	0.99	2.34	1.05	0.94
	Waste (offal)	1.06	2.48	1.13	1.00
	White Flour	0.45	0.87	0.48	0.35
	Total Bran	1.01	2.00	1.07	0.80
	Shorts	0.87	1.17	0.93	0.47
	Middlings	0.48	0.92	0.51	0.37
	Grain (RAC)	1.14 [a]	2.52 [a]	–	–
	Wholemeal Flour	1.06	1.59	0.93	0.63
	Wholemeal Bread	0.87	1.44	0.76	0.57
	Grain (RAC)	1.00 [a]	2.49 [a]	–	–
	Germ	1.13	0.66	1.13	0.27
	Grain (RAC)	1.09 [a]	2.65 [a]	–	–
	Dry Gluten	0.30	0.50	0.28	0.19
	Dry Starch	0.09	0.13	0.08	0.05
	Gluten Feed Meal	0.20	0.38	0.18	0.14

[a] Mean of two analyses

Rice

New processing studies are performed on rice in parallel to the supervised residue field trials (Smith N., 2014, A7725M_50010). Rice grain samples from the field phase of supervised field trials TK0044924-10 and TK0044924-11 treated at an exaggerated (3×) rate were used for processing of rice grain into polished rice, hulls and bran.

Rice samples (RAC) were removed from the freezer and dried to a final moisture content of 11–14%. Following this, samples were cleaned by aspiration and screening. The samples were then screened in a cleaner to separate large and small foreign particles (screening) from the cleaned rice.

The cleaned rice was milled in a rice mill. During the milling process, the rice entered the hulling portion of the mill where hull was removed by rubber rolls rotating in opposite directions at different speeds. Hull material was separated from the brown rice by aspiration. Two passes through the hulling section was

required to remove the hull. After dehulling, brown rice entered the milling chamber where it was milled into white polished rice and bran by friction. Bran was separated from the polished rice by air injected into the milling chamber (slotted screen). Bran percentage can be increased by adding weights to the exit door of the milling chamber. After exiting the chamber, bran was sieved with a 24-mesh screen to remove broken pieces of brown and polished rice and small amounts of hull material from bran. This material was added to the unhulled seed exiting the mill. Requested fractions of hull material, polished rice and bran were sampled and stored frozen until analysis.

Samples were analysed for residues of trinexapac acid (free and conjugated) using method GRM020.01A.

Residues of trinexapac acid in rice samples prior to processing and after processing into polished rice, hulls and bran are presented in Table 27.

Table 27: Residues trinexapac acid in rice samples prior to processing and after processing into polished rice, hulls and bran, and the processing factors

Trial	Commodity	Residues of trinexapac acid (free and congregated) (mg/kg)	Processing Factor
TK0044924-10	Rice before processing	0.77, 0.70, 0.22 (0.56)	-
	Rice (polished)	0.92	1.6
	Rice hulls	0.33	0.59
	Rice bran	2.5	4.5
TK0044924-11	Rice before processing	0.10, 0.094, 0.097 (0.097)	-
	Rice (polished)	0.10	1.0
	Rice hulls	0.022	0.23
	Rice bran	0.56	5.8
Mean	Rice (polished)		**1.3**
	Rice hulls		**0.41**
	Rice bran		**5.1**

A summary of relevant trinexapac-ethyl processing factors is provided below (Table 28).

Table 28 Trinexapac-ethyl processing factors for barley, wheat and rice from processing studies considered by the current Meeting and the 2013 JMPR

Commodity		Processing Factors for free trinexapac acid		Processing Factors for total trinexapac acid	
		Individual	Median or Best estimate	Individual	Median or Best estimate
Barley	Pot Barley	0.57, 0.58, 0.61, 0.84	0.60	0.15, 0.18, 0.22, 0.24	0.20
	Pearled Barley	0.52, 0.54, 0.57, 0.77	0.55	0.17, 0.17, 0.18, 0.18, 0.86*, 1.2*, 1.4*, 1.5*	0.52
	Barley Bran	0.74, 0.81, 1.74, 1.86	1.3	0.12, 0.17, 0.37, 0.45, 1.6*, 1.9*, 1.8*, 2.0*	1.1
	Barley Flour	0.76, 0.81, 0.88, 0.94	0.85	0.25*, 0.28*, 0.35, 0.37, 0.49, 0.53, 0.57*, 0.63*	0.43
	Brewing Malt	0.51, 0.78, 0.87, 0.88	0.83	0.40, 0.43, 0.55, 0.63	0.49
	Malt Sprouts	0.55, 0.70, 1.12, 1.16	0.88	0.07, 0.07, 0.11, 0.12	0.09

Commodity		Processing Factors for free trinexapac acid		Processing Factors for total trinexapac acid	
		Individual	Median or Best estimate	Individual	Median or Best estimate
	Brewer's Grain dry	0.33, 0.33, 0.39, 0.43	0.37	0.10, 0.10, 0.12, 0.13	0.11
	Brewer's Yeast	1.53, 1.57, 1.86, 1.91	1.72	0.09, 0.17, 0.18, 0.23	0.18
	Beer	0.09, 0.12, 0.17, 0.19	0.14	0.05, 0.06, 0.06, 0.07	0.06
Wheat	Cleaned Grain	1.02, 1.05, 1.16, 1.9	1.1	0.94, 1.08, 1.13, 1.13	1.1
	Waste (offal)	0.51, 0.53, 1.08, 1.13	0.81	0.74, 0.94, 1.00, 1.14	0.97
	White Flour	0.24*, 0.31*, 0.31*, 0.32*, 0.48, 0.53, 0.57, 0.61	0.4	0.35, 0.38, 0.4*, 0.4*, 0.4*, 0.45, 0.46, 0.5*	0.4
	Bran	1.07, 1.08, 1.10, 1.14, 2.1*, 2.2*, 2.4*, 2.5*	1.6	0.70, 0.80, 0.96, 0.98, 1.5*, 1.6*, 2.1*, 2.2*	1.2
	Shorts	0.45*, 0.68*, 0.78, 0.93, 0.93, 0.97, 1.1*, 1.4*	0.93	0.03*, 0.47, 0.48, 0.58, 0.6*, 0.6*, 0.6*, 0.61	0.59
	Middlings	0.41*, 0.43*, 0.45*, 0.49*, 0.50, 0.51, 0.56, 0.57,	0.50	0.3*, 0.37, 0.46, 0.5*, 0.53, 0.58, 0.6*, 11.7*	0.52
	Wholemeal Flour	0.91, 0.92, 0.93, 1.30	0.925	0.63, 0.67, 0.78, 1.01	0.72
	Wholemeal Bread	0.73, 0.76, 0.89, 1.08	0.825	0.57, 0.60, 0.63, 0.83	0.62
	Germ	0.29*, 0.69, 0.85*, 0.97, 1.0*, 1.1*, 1.13, 1.19	0.985	0.27, 0.28, 0.39, 0.41, 0.9*, 0.9*, 1.1*, 1.4*	0.66
	Dry Gluten	0.27, 0.28, 0.30, 0.33	0.29	0.19, 0.20, 0.26, 0.31	0.24
	Dry Starch	0.08, 0.09, 0.09, 0.09	0.09	0.05, 0.05, 0.11, 0.11	0.08
	Gluten Feed Meal	0.16, 0.18, 0.22, 0.23	0.20	0.14, 0.17, 0.19, 0.19	0.18
Rice	Rice (polished)			1.0, 1.6	1.3
	Rice hulls			0.23, 0.59	0.41
	Rice bran			4.5, 5.8	5.1

* processing factors from studies evaluated by the 2013 JMPR

APPRAISAL

Trinexapac-ethyl is a synthetic plant growth regulator used for growth management of crops. It was first evaluated by JMPR in 2013 (T, R). An ADI of 0–0.3 mg/kg bw, expressed as trinexapac acid equivalents, was established in 2013 JMPR and the Meeting concluded that an ARfD for trinexapac-ethyl was unnecessary.

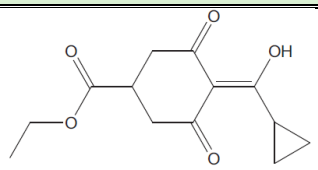
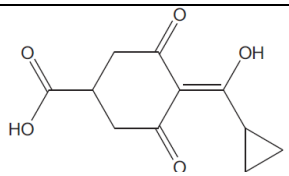
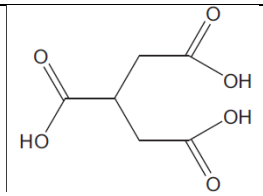
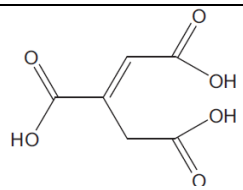
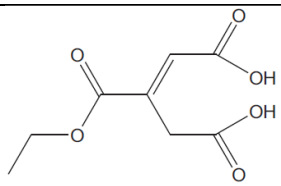
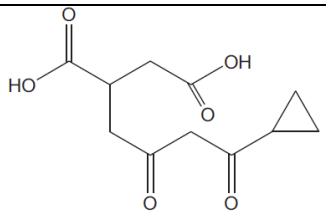
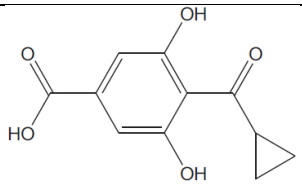
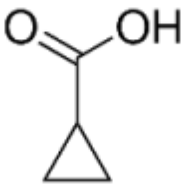
The definition of the residue for compliance with the MRL for plant and animal commodities and for dietary risk assessment for animal commodities is: *Trinexapac acid*.

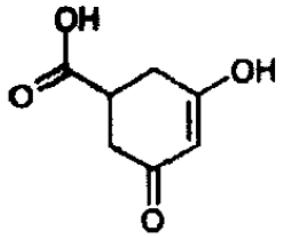
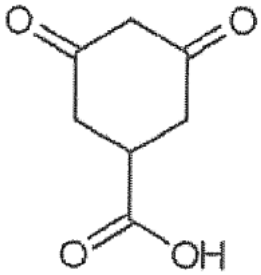
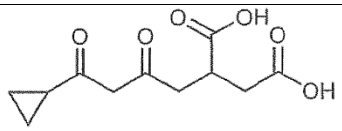
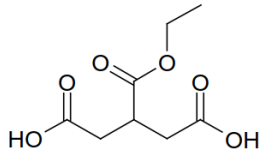
The definition of the residue for dietary risk assessment for plant commodities is: *Trinexapac acid and its conjugates, expressed as trinexapac acid*.

The residue is not fat-soluble.

It was scheduled at the Fifty-first Session of the CCPR for the evaluation of additional uses by the 2020 JMPR, which was postponed to the 2021 Extra JMPR. The Meeting received information on the metabolism in wheat and oilseed rape, methods of residue analysis, freezer storage stability, the GAP, supervised residue trials on rice, as well as industrial processing studies.

Table 1 The structures of trinexapac-ethyl and its key metabolites discussed are shown below

Compound	Structure
Trinexapac-ethyl	
Trinexapac acid CGA179500	
Tricarballylic acid CGA275537	
Trans aconitic acid CGA312753	
Mono-ethyl ester of trans aconitic acid	
CGA313458	
CGA329773	
CGA224439	

Compound	Structure
CGA113745	
Cyclodion acid	
3-carboxyl-7-cyclopropyl-5,7-dioxoheptanoic acid	
CGA300405	

Plant Metabolism

Oilseed rape

Oilseed rape plants were treated at a rate of 393.8 g ai/ha with [cyclohexadione-1,2,6-^{14}C]-trinexapac-ethyl formulated as a microemulsion at BBCH 55 by foliar spray application. Plants were collected (fully ripe) between 67 and 91 days after application (BBCH 89). Only the seeds were analysed. Total radioactive residue in the seeds was 0.39 mg eq/kg. The extraction of ^{14}C residues in seed (twice with acetonitrile:water:hexane 16:4:10 (v/v/v), followed by twice with acetonitrile:water 50:50 (v/v) at room temperature) was 67% TRR (0.27 mg eq/kg). No parent trinexapac-ethyl was detected in seeds. Trinexapac acid was identified as the major metabolite, accounting for 22% TRR, 0.086 mg eq/kg (19% TRR free and 2.8% TRR conjugated). The tricarballylic acid was the minor metabolite detected (1.0% TRR; 0.004 mg eq/kg, exclusively in the conjugated form). A major part of the radioactivity was identified as oleic acid (22.7% TRR; 0.09 mg eq/kg, detected following saponifaction of radioactivity associated with hexane-extracted endogenous oils). The individual levels of other unassigned radio components from extracted residues never exceeded 6.7% TRR, (0.027 mg eq/kg). The characterisation of the unextracted radioactive residues was conducted, however, no identification was achieved due to low residue levels released.

Spring wheat

Spring wheat plants were treated once at rate of 211 g ai/ha with [cyclohexadione-^{14}C]-trinexapac-ethyl as a microemulsion at BBCH 37 by foliar spray application. Samples were collected at forage growth stage (BBCH 43), at hay growth stage (BBCH 77) and at maturity (BBCH 89).

The total radioactive residues (TRR) for harvested commodities were 1.8 mg eq/kg in forage, 2.0 mg eq/kg in hay, 1.4 mg eq/kg in straw and 1.4 mg eq/kg in grain. Good extractability with acetonitrile/water (three times with 4:1 (v/v) and once with 1:1 (v/v) at room temperature) was achieved in forage, grain and hay (≥84% TRR) with lower extractability in straw (65% TRR). Metabolism of parent trinexapac-ethyl was extensive, the parent was only detected in forage with a low residue level of 0.3% TRR, 0.006 mg eq/kg. The principal metabolites identified in grain were trinexapac acid (40% TRR, 0.58 mg eq/kg; 28% TRR free and 12% TRR conjugated) in grain, the minor metabolites identified in grain were tricarballylic acid ethyl ester metabolite CGA300405 (0.8% TRR; 0.012 mg eq/kg), tricarballylic acid metabolite CGA275537 (2.0% TRR; 0.03 mg eq/kg) and a hydroxylated metabolite of trinexapac acid (12% TRR, 0.18 mg eq/kg).

The principal metabolites identified in forage, hay and straw were trinexapac acid (5.5–23% TRR; 0.075–0.45 mg eq/kg), the tricarballylic acid ethyl ester metabolite (CGA300405, 8.0–21% TRR, 0.13–0.37 mg eq/kg) and tricarballylic acid (CGA275537, 7.8–10% TRR, 0.11–0.14 mg eq/kg). Formation of citric acid and subsequent incorporation of small ^{14}C containing moieties into the broader pool of natural biosynthetic products was observed (characterised by the presence of ^{14}C-glucose in hay, straw and grain). All metabolites with the exception of CGA329773 and citric acid were found in their free and conjugated forms.

The characterisation of the unextracted radioactive residues in the hay, grain and straw samples was accomplished with a clean fractionation technique, which released additional small amounts of the same metabolites as observed in the extracted fractions. CGA300405 was identified as the highest residue in all samples analysed (0.3–0.6% TRR; 0.004–0.013 mg eq/kg). Acid hydrolysis of a hemicellulose fraction (12% TRR; 0.16 mg eq/kg) derived from straw showed the majority of this fraction to comprise [^{14}C]-glucose, demonstrating extensive incorporation into natural components.

The Meeting concluded that the metabolism profile of trinexapac-ethyl in rapeseed and spring wheat was comparable to that previously evaluated by the 2013 JMPR and the Meeting confirmed the current residue definition for plant commodities.

Methods of analysis

The Meeting received information on the description and validation of analytical methods for trinexapac acid in plant matrices, as well as a multi-residue method for trinexapac acid in plant and animal matrices.

The method GRM020.05A for free trinexapac acid involved extraction of the samples with methanol/water/phosphate buffer (pH7, v/v/v 30:56:14), clean-up with SPE and quantification with HPLC-MS/MS (monitoring for quantitation m/z=223→135 and for confirmation m/z = 223→179 or m/z = 223→83). An LOQ of 0.01 mg/kg was validated for free trinexapac acid in barley, tomato, apple and sunflower seed.

The method GRM020.09A for free and conjugated trinexapac acid involved sequential extraction with acetonitrile/water (80/20 v/v) and acetonitrile/water (50/50 v/v) for free trinexapac acid and hydrolysis under 0.05M sodium hydroxide at room temperature overnight (minimum 12 hours) for conjugated trinexapac acid, with quantification by HPLC-MS/MS (quantitation m/z=223→135, confirmatory m/z = 223→83 or m/z = 223→125). An LOQ of 0.01 mg/kg in cereal grain and 0.05 mg/kg in cereal straw was validated for trinexapac acid.

The extraction and hydrolysis were shown to be satisfactory by reference to the metabolism study on spring wheat. The Meeting concluded that the presented methods are suitable to measure either free or, free and conjugated trinexapac acid in plant commodities.

The multi-residues method of QuEChERS was newly validated for determination of free trinexapac acid in plant and animal matrices, with an LOQ of 0.01 mg/kg.

Stability of pesticide residues in stored analytical samples

The Meeting received additional information on storage stability of cyclopropane carboxylic acid in cereal grain and its processed commodities.

Storage stability studies showed that residues of cyclopropane carboxylic acid is stable for at least 12 months at <-18 °C for high starch commodities and processed commodities from cereal grain (flour, bran, bread and beer).

The samples of rice grain and straw in supervised trials available to the Meeting were stored deep frozen for a maximum of 12 months (365 days) and were covered by storage stability evaluated in 2013 JMPR.

Definition of the residue

The current Meeting received new plant metabolism data indicating that the metabolism profile of trinexapac-ethyl in rapeseed and spring wheat was comparable to that previously evaluated by the 2013 JMPR and the Meeting confirmed the current residue definition for plant commodities.

The current Meeting also received new information on the nature of residues during processing following high temperature hydrolysis. In combination with the information considered by the 2013 JMPR, it was confirmed that trinexapac acid shows limited stability under conditions representative of pasteurisation, baking, brewing, boiling and sterilisation. Major degradates were CGA113745 (10–12% AR), CGA313458 (16–21% AR) and cyclopropane carboxylic acid (CGA224439, 16% AR).

CGA113745 was also found in goat liver, kidney and fat up to 16% of the TRR. The residue ratio of CGA113745 to trinexapac acid was up to 0.25.

CGA313458 was also found in plant matrices. The residue ratios compared to trinexapac acid were 0.3 in rice grain and 0.03 in rape seeds.

The information on the toxicity for all three metabolites was limited and the Meeting decided to assess them using the thresholds of toxicological concern (TTC) approach in the following classes:

CGA113745 and CGA313458	Cramer Class III compounds
CGA224439	Cramer Class II compound

Results of supervised residue trials on crops

Supervised trials were available for the use of trinexapac-ethyl on rice. Product labels were available from the USA.

Rice

The critical GAP for trinexapac ethyl on rice in the USA is one application at 50 g ai/ha (up to panicle formation, panicle 1–2mm, BBCH32) with a PHI of 50 days.

In six trials conducted on rice in the USA approximating critical GAP, the residues of trinexapac acid (free and conjugated) in grains were (n = 6): 0.017, 0.023, 0.054, 0.073, 0.18 and 0.23 mg/kg. The free trinexapac acid was not separately analysed.

In four trials conducted in USA with longer days after last application (DALA, 85-100 days after application), the residues of trinexapac acid (free and conjugated) in grain were (n = 4): 0.035, 0.050, 0.074 and 0.10 mg/kg. The free trinexapac acid was not separately analysed.

Considering the ratios of % TRR for free compared to free and conjugated (total) trinexapac acid in rice grain from three metabolism studies, that were 0.86 (10% free/11.6% total), 0.82 (23% free/28.1% total) and 0.92 (20.2% free/24% total), the conjugated residues contributed less than 20% of the total. The Meeting agreed to recommend the maximum residue level based on the residues of total trinexapac acid.

Noting all trials were conducted at growth stage of BBCH 30–32 and the residue decline studies showed that longer sampling intervals had little or no impact on the residues of trinexapac acid in rice grain, the Meeting decided to use the combined data to make estimation. The combined data set was (n = 10): 0.017, 0.023, 0.035, 0.050, 0.054, 0.073, 0.074, 0.10, 0.18 and 0.23 mg/kg.

The Meeting estimated a maximum residue level of 0.5 mg/kg and an STMR of 0.064 mg/kg for trinexapac-ethyl in rice grain.

Rye

The critical GAP for trinexapac-ethyl on rye in the USA is one application at 123 g ai/ha with a PHI of 45 days. No data reflecting the critical GAP on rye were available.

In trials evaluated by the 2013 JMPR, the residues of free trinexapac acid in wheat grain were (n = 18): 0.10, 0.25, 0.32, 0.34, 0.35, 0.46, 0.49, 0.55, 0.55, 0.57, 0.77, 0.88, 0.91, 0.98, 0.99, 1.0, 1.4 and 2.0 mg/kg. The residues of free and conjugated trinexapac acid in wheat and barley grain were (n = 30): 0.03, 0.07, 0.08, 0.15, 0.27, 0.31, 0.32, 0.40, 0.44, 0.45, 0.47, 0.50, 0.52, 0.53, 0.53, 0.60, 0.72, 0.76, 0.77, 0.78, 0.82, 0.83, 0.85, 0.99, 1.0, 1.0, 1.1, 1.2, 1.6 and 3.3 mg/kg.

The current Meeting recognized that rye and wheat (spring wheat, winter wheat and durum wheat) have the same GAP and show comparable residues after early treatment. Application was before flowering. Therefore, the current Meeting agreed to extrapolate the maximum residue level of 3 mg/kg estimated for wheat grain to rye grain and to extrapolate the STMR of 0.57 mg/kg from wheat and barley to rye grain

Residues in animal feeds

Rice straw

The Meeting received supervised residue trials on rice. The critical GAP for trinexapac-ethyl on rice in the USA is one application at 50 g ai/ha (up to panicle formation (panicle 1–2mm, BBCH32) with a PHI of 50 days.

In six trials conducted on rice in the USA approximating critical GAP, the residues of trinexapac acid (free and conjugated) in straw were (n = 6): < 0.01(2), 0.012, 0.016, 0.036 and 0.045 mg/kg. The free trinexapac acid was not separately analysed.

In four trials conducted in the USA with longer days after last application (DALA, 85–100 days after application, more than 25%), the residues of trinexapac acid (free and conjugated) in straw were (n = 4): < 0.01, 0.01, 0.017 and 0.02 mg/kg. The free trinexapac acid was not separately analysed.

Considering the ratios of % TRR for free compared to total trinexapac acid in rice straw in three metabolism studies on rice, that were 0.86 (4.2% free/4.9% total), 0.97 (6.7% free/6.9% total) and 0.80 (6.7% free/8.4% total), the conjugated residues of trinexapac acid contributed less than 20% of the total. The Meeting agreed to recommend the maximum residue level based on the residues of total trinexapac acid.

Noting that all trials were conducted at a growth stage of BBCH 30–32 and the residue decline studies showed that longer sampling intervals had little or no impact on the residues of trinexapac acid in rice straw, the Meeting decided to use the combined data to make a maximum residue level estimation. The combined data set was (n = 10): <0.01(3), 0.01, 0.012, 0.016, 0.017, 0.02, 0.036 and 0.045 mg/kg.

The Meeting estimated a maximum residue level of 0.08 mg/kg (dw), a median residue of 0.014 mg/kg (as received) and a highest residue of 0.045 mg/kg (as received) for trinexapac-ethyl in rice straw.

Rye

The critical GAP for trinexapac-ethyl on rye in the USA is one application at 123 g ai/ha with a PHI of 45 days. No data reflecting the critical GAP on rye were available.

Rye forage

In trials evaluated by the 2013 JMPR, the resides of total trinexapac acid in wheat forage collected 30 days after application (dry weight) at the GAP application rate were (n = 18): 0.08, 0.12, 0.16, 0.24, 0.28, 0.32, 0.32, 0.36, 0.40, 0.40, 0.48, 0.68, 0.68, 0.88, 0.92, 1.32, 1.52 and 3.76 mg/kg.

The current Meeting recognized that rye and wheat (spring wheat, winter wheat and durum wheat) have the same GAP and show comparable residues after early treatment. Application was before flowering. The current Meeting agreed to extrapolate the median residue of 0.40 mg/kg and highest residue of 3.76 mg/kg for trinexapac acid in wheat forage to rye forage (dry).

Rye hay and straw

In the trials evaluated by the 2013 JMPR, the residues of free trinexapac acid in wheat hay collected 30 days after application (dry weight) at the GAP application rate were (n = 18): <0.06(3), 0.06, 0.07, 0.08, 0.08, 0.09, 0.09, 0.10, 0.13, 0.13, 0.15, 0.17, 0.20, 0.22, 0.22 and 0.85 mg/kg. The residues of total trinexapac acid in barley and wheat hay collected 30 days after application (dry weight) according to the GAP were (n = 29): <0.011, 0.03, 0.03, 0.05, 0.05, 0.06, 0.07, 0.10, 0.11, 0.13, 0.13, 0.15, 0.16, 0.17, 0.19, 0.20, 0.22, 0.27, 0.28, 0.34, 0.35, 0.38, 0.45, 0.47, 0.55, 0.57, 0.67, 0.89 and 1.34 mg/kg.

The current Meeting noted that rye and wheat (spring wheat, winter wheat and durum wheat) have the same GAP and normally show comparable residues after early treatment.

The current Meeting agreed to extrapolate the maximum residue level of 0.9 mg/kg for trinexapac-ethyl in wheat straw and fodder (dry) to rye straw and fodder (dry) and to extrapolate the median residue of 0.19 mg/kg and highest residue of 1.34 mg/kg for trinexapac-ethyl for wheat straw and fodder (dry) and barley straw and fodder (dry) to rye straw and fodder (dry).

Fate of residues during processing

The Meeting received two new studies on high temperature hydrolysis of residues of trinexapac acid

One study showed that trinexapac acid (hydroxymethylene-C^{14} labelled) underwent limited degradation under conditions representative of pasteurisation (pH 4, 90 °C, 20 minutes) and sterilisation (pH 6, 120 °C, 20 minutes) and degraded at significant amounts to cyclopropane carboxylic acid (16%) and CGA313458 (18%) under conditions representative of baking, brewing and boiling (pH 5, 100 °C, 60 minutes). Another study showed that trinexapac acid (1,6-cyclohexyl and hydroxymethylene-C^{14} labelled) is hydrolytically stable under conditions representative of pasteurisation, baking, brewing, boiling and sterilisation and two metabolites, cyclodion acid and 3-carboxyl-7-cyclopropyl-5,7-dioxoheptanoic acid

increased from 1.7 to 3.4% and 2 to 3.5%, respectively, under condition of baking, brewing, boiling (pH 5, 100 °C, 60 minutes).

The 2013 JMPR concluded that trinexapac acid (cyclohexyl C^{14} labelled) undergoes limited degradation during the simulation of pasteurisation (pH 4, 90 °C, 25 minutes), baking, boiling and brewing (pH 5, 100 °C, 60 minutes) and sterilisation (pH 6, 120 °C, 20 minutes). Trinexapac acid was the major component of the radioactive material at the end of hydrolysis reactions (51–59%) and the major degradates were CGA113745 (10–12%) and CGA313458 (16–21%).

The Meeting concluded that trinexapac acid might undergo limited hydrolysis under high temperature conditions, resulting in *> 60% of trinexapac acid* remaining at the end of each treatment.

The Meeting also received processing studies for barley, rice and wheat. Estimated processing factors for the commodities considered at this Meeting are summarised below.

Table 2 Processing factors for estimation of STMR-Ps (free and conjugated)

	Processed commodity	Median or best estimate processing factor	STMR-P =$STMR_{RAC}$ × PF (mg/kg)
Barley (STMR 0.57 mg/kg)	Pot Barley	0.20	0.11
	Pearled Barley	0.52	0.30
	Barley Bran	1.1	0.63
	Barley Flour	0.43	0.25
	Brewing Malt	0.49	0.28
	Beer	0.06	0.03
Wheat (STMR 0.57 mg/kg)	White Flour	0.4	0.23
	Bran	1.2	0.68
	Whole meal Flour	0.72	0.41
	Wholemeal Bread	0.62	0.35
	Germ	0.66	0.38
	Dry Gluten	0.24	0.14
	Dry Starch	0.08	0.046
	Gluten Feed Meal	0.18	0.10 (median residue)
Rice (STMR 0.064 mg/kg)	Rice (polished)	1.3	0.08
	Rice hulls	0.41	0.03
	Rice bran	5.1	0.33

Table 3 Processing factors for estimation of maximum residue levels in processed commodities (free acid)

	Processed commodity	**Mean or best estimate processing factor**	**Maximum residue level for processed commodity (mg/kg)**
Barley (maximum residue level 3 mg/kg)	Barley Bran	1.3	3.9
Rice (maximum residue level 0.5 mg/kg)	Rice (polished)	1.3	0.65
	Rice bran	5.1	2.55
Wheat (maximum residue level 3 mg/kg)	Wheat bran	1.6	4.8

Based on the processing factor of 1.3 for barley bran and the barley grain maximum residue level of 3 mg/kg, the calculated highest residues in barley bran are 3.9 mg/kg. The Meeting estimated a maximum residue level for trinexapac-ethyl in barley bran of 4 mg/kg, to replace the previous recommendation of 6 mg/kg.

Based on the processing factors of 1.3 for polished rice and 5.1 for rice bran and the rice grain maximum residue level of 0.5 mg/kg, the calculated highest residues in polished rice and rice bran are 0.65 and 2.55 mg/kg, respectively. The Meeting estimated a maximum residue level for trinexapac-ethyl in polished rice of 0.7 mg/kg, in rice bran of 3 mg/kg.

Based on the processing factor of 1.6 for wheat bran and the wheat grain maximum residue level of 3 mg/kg, the calculated highest residues in wheat bran are 4.8 mg/kg. The Meeting estimated a maximum residue level for trinexapac-ethyl in wheat bran of 5 mg/kg, to replace the previous recommendation of 8 mg/kg.

Residues in animal commodities

The current Meeting calculated dietary burdens based on residue estimates from previous meeting with updates to reflect feed commodities addressed by the current Meeting. Potential feed items include: wheat, barley, oat and triticale grain, straw, forage, hay and silage, wheat milled by-products(bran), wheat aspirated grain fraction, barley bran fractions, sugarcane molasses and bagasse and rape forage and rape seed meal, rice grain and straw, rye grain and straw. The dietary burdens estimated using the most recent version of the OECD livestock dietary burden calculator, are presented in Annex 6 and summarised below.

Table 4 Estimated maximum and mean dietary burdens of farm animals

	Animal dietary burden: Trinexapac acid, ppm of dry matter diet							
	US-Canada		EU		Australia		Japan	
	max	Mean	max	Mean	max	Mean	max	Mean
Beef cattle	1.14	0.741	1.743	0.726	3.969	0.752	0.791	0.791 (0.97) [a]
Dairy cattle	1.702	0.682	1.743	0.674	4.052 (3.76) [a]	0.729 (0.86) [a]	0.861	0.693
Poultry broiler	0.778	0.78 (0.94)[a]	0.635	0.635	0.698	0.6981	0.11	0.11
Poultry layer	0.778	0.778 (0.94)[a]	1.011 (1.08) [a]	0.7	0.697	0.697	0.273	0.273

[a] values in parenthesis from the 2013 evaluation

Despite the additional feed items, the Meeting noted that the dietary burdens changed within a range of -18% to 8% compared with those estimated by the 2013 JMPR to derive residue estimates for animal commodities. The Meeting confirmed its previous recommendations for animal commodities.

RECOMMENDATIONS

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

The definition of the residue for compliance with the MRL for plant and animal commodities and for dietary risk assessment for for animal commodities: *Trinexapac acid*.

The definition of the residue for dietary risk assessment for plant commodities: *Trinexapac acid and its conjugates, expressed as trinexapac acid.*

The residue is not fat-soluble.

Table 5 Recommendations for residues of trinexapac-ethyl from the 2021 Extra JMPR

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
CF 0640	Barley bran, processed	4	6	0.63	
GC 0649	Rice	0.5		0.064	
CM 1205	Rice polished	0.7		0.08	
	Rice bran	3		0.33	
AS 0649	Rice straw and fodder, dry	0.08 (dw)		Median: 0.014 (as)	Highest: 0.045 (as)
GC 0650	Rye	3		0.57	
AS 0650	Rye straw and fodder, dry	0.9 (dw)		Median: 0.19 (dw)	Highest: 1.34 (dw)
CM 0654	Wheat bran, unprocessed	5	8	0.68	
	Barley flour			0.25	
	Barley pearled			0.30	
	Barley malt			0.28	
	Barley beer			0.03	
	Rice hulls			Median: 0.03	
AF 0650	Rye forage (green)			Median: 0.40 (dw)	Highest: 3.76 (dw)
	Wheat, white flour			0.23	
CF 1210	Wheat germ			0.38	
	Wheat gluten			0.14	
	Wheat starch			0.046	
	Wheat whole meal bread			0.35	

(dw) – dry weight; (as) - as received

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for trinexapac-ethyl is 0–0.3 mg/kg bw (expressed as trinexapac acid equivalents). The International Estimated Daily Intakes (IEDIs) for trinexapac-ethyl were estimated for the 17 GEMS/Food Consumption Cluster Diets using the STMR or STMR-P values estimated by the JMPR. The results are shown in Annex 3 of the 2021 Extra JMPR Report.

The IEDIs were 0% of the maximum ADI. The Meeting concluded that long-term dietary exposure to residues of trinexapac-ethyl from uses considered by the JMPR is unlikely to present a public health concern.

Acute dietary exposure

The 2013 JMPR decided that an ARfD for trinexapac-ethyl was unnecessary. The Meeting therefore concluded that the acute dietary exposure to residues of trinexapac-ethyl from the uses considered is unlikely to present a public health concern.

Assessment of metabolites using the threshold of toxicological concern (TTC) approach

The metabolite CGA113745 could be assessed using the TTC approach (Cramer Class III threshold of 1.5 µg/kg bw per day).

The present Meeting noted that the ratio of residues for CGA113745 compared to trinexapac acid in animal commodities was up to 0.25. In addition, trinexapac acid could be converted into CGA113745 during high temperature hydrolysis by up to 12% of the AR. The Meeting applied a conservative ratio of residues of 0.37 (0.25+0.12) to the trinexapac acid IEDI to derive an upper limit estimate of dietary exposure to CGA113745.

Based on the highest IEDI for trinexapac acid of 1.5 µg/kg bw per day and the overall ratio of 0.37 for formation of CGA113745, an upper long-term dietary exposure of 0.56 µg/kg bw per day was estimated.

The Meeting concluded that the estimated dietary exposure to residues of CGA113745 from uses considered by the JMPR is below the TTC for Cramer Class III compounds and is unlikely to present a public health concern. Should further uses be considered in the future, these conclusions may need to be re-evaluated.

The metabolite CGA313458 could be assessed using the TTC approach (Cramer Class III threshold of 1.5 µg/kg bw per day).

The present Meeting noted that the ratio of residues for CGA313458 compared to trinexapac acid in plant commodities was up to 0.3 (rice grain). In addition, trinexapac acid could be converted during high temperature hydrolysis into CGA313458 by up to 21%. The Meeting applied a conservative ratio of residues of 0.51 (0.3+0.21) to the trinexapac acid IEDI to derive an upper limit estimate of dietary exposure to CGA313458.

Based on the highest IEDI for trinexapac acid of 1.5 µg/kg bw per day and the overall ratio of 0.51 for the formation of CGA313458, a long-term dietary exposure of 0.77 µg/kg bw per day was estimated.

The Meeting concluded that the estimated dietary exposure to residues of CGA313458 from uses considered by the JMPR is below the TTC for Cramer Class III compounds and is unlikely to present a public health concern. Should further uses be considered in the future, these conclusions may need to be re-evaluated.

The metabolite CGA224439 could be assessed using the TTC approach (Cramer Class II threshold of 9 µg/kg bw per day).

The present Meeting noted that CGA224439 is only formed during high temperature hydrolysis in limited amounts (up to 16% AR) and applied a conservative ratio of residues of 0.16 to the trinexapac acid IEDI to derive an upper limit estimate of the dietary exposure to CGA224439.

Based on the highest IEDI for trinexapac acid of 1.5 µg/kg bw per day and the ratio of 0.16 for the formation of CGA224439, a long-term dietary exposure of 0.24 µg/kg bw per day was estimated.

The Meeting concluded that the estimated dietary exposure to residues of CGA224439 from uses considered by the JMPR is below the TTC for Cramer Class II compounds and is unlikely to present a public health concern. Should further uses be considered in the future, these conclusions may need to be re-evaluated.

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An Extra 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 virtually over two sessions from 17 to 21 May and 7 to 11 June 2021. 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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