Food and Agriculture Organization of the United Nations

World Health Organization

EVALUATION 2019

PART I - RESIDUES

Pesticide Residues in Food

Extra Joint FAO/WHO Meeting on Pesticide Residues

Pesticide Residues in Food 2019

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Evaluation
Part I - 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
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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 2019
Evaluations
Part I: Residues

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List of participants

Dr Christos Anagnostopoulos, Laboratory of Pesticide Residues, Benaki Phytopathological Institute, 8 Stefanou Delta Street, Kifisia, Athens, 14561 Greece.

Professor Eloisa Dutra Caldas, Pharmaceutical Sciences Department, College of Health Sciences, University of Brasilia, Campus Universitário Darci Ribeiro, 70910-900 Brasília/DF, Brazil.

Dr Michael Doherty, Office of Pesticide Programs, Health Effects Division, Risk Assessment Branch II, United States Environmental Protection Agency, MS 7509C, Washington, DC 20460, USA.

Dr Ngan Chai Keong, Soil Science and Water Programme Soil Science, Water and Fertilizer Research Centre, Malaysian Agricultural Research and Development Institute (MARDI), P. O. Box 12301, General Post Office, 50774 Kuala Lumpur, Malaysia.

Dr Hidetaka Kobayashi, Food Safety and Consumer Affairs Bureau, Ministry of Agriculture, Forestry and Fisheries, 1-2-1 Kasumigaseki, Chiyoda-ku, Tokyo, 100-8950 Japan.

Mr David Lunn, Plants & Pathways Directorate, Ministry for Primary Industries, PO Box 2526, Wellington, New Zealand.

Mr Peter Rembischevski, Toxicology Division, Brazilian Health Regulatory Agency – Anvisa, SIA Trecho 5 Area Especial 57, 71205-050 Brasília/DF, Brazil.

Dr Rebecca Scrivens, Chemicals Regulation Division, Health & Safety Executive, Mallard House, Kings Pool, 3 Peasholme Green, York, YO1 7PX United Kingdom.

Mr Christian Sieke, Federal Institute for Risk Assessment, Unit Residues and Analytical Methods, Department Pesticide Safety, Max-Dohrn-Strasse 8–10, 10589 Berlin, Germany.

Dr Yukiko Yamada, Ministry of Agriculture, Forestry and Fisheries, 1-2-1 Kasumigaseki, Chiyoda-ku, Tokyo 100-8950, Japan.

Dr Guibiao Ye, Institute for the Control of Agrochemicals, Ministry of Agriculture and Rural Affairs, Maizidian 22, Chaoyang District, Beijing 100125, People's Republic of China.

Ms GuangYan Zhu, Residue Division, Institute for the Control of Agrochemicals, Ministry of Agriculture and Rural Affairs, Maizidian 22, Chaoyang District, Beijing 100125, People's Republic of China.

JMPR Secretariat

Mr Kevin Bodnaruk, West Pymble, NSW 2073, Australia *(FAO Editor)*

Dr Jeevan Khurana, Lyneham, ACT 2602, Australia *(FAO Editor)*

Mr Soren MADSEN, Department of Food Safety and Zoonoses (FOS), World Health Organization, 1211 Geneva 27, Switzerland *(WHO JMPR Secretariat)*

Ms Marla Sheffer, Orleans, Ontario, Canada K1E 2K5 *(WHO Editor)*

Ms Yong Zhen Yang, Plant Production and Protection Division, Food and Agriculture Organization of the United Nations, Viale delle Terme di Caracalla, 00153 Rome, Italy *(FAO JMPR Secretariat)*

Abbreviations

5-OH-dicamba	2,5-dichloro-3-hydroxy-6-methoxybenzoic acid
ADI	acceptable daily intake
AMBA	2-amino-4-methylsulfonylbenzoic acid
AR	applied radioactivity
ARfD	acute reference dose
BBCH	Biologische Bundesanstalt, Bundessortenamt Und Chemische Industrie
bw	body weight
CAS	Chemical Abstracts Service
CCPR	Codex Committee on Pesticide Residues
cGAP	critical GAP
DALA	days after last application
DAT	days after treatment
DCGA	3,6-dichlorogentisic acid
DCSA	3,6-dichlorosalicylic acid
DM	dry matter
equiv	equivalent(s)
EU	European Union
FAO	Food and Agriculture Organization of the United Nations
GAP	good agricultural practice
GC-ECD	gas chromatography – electron capture detector
GEMS	Global Environment Monitoring System – Food Contamination Monitoring and Assessment Programme
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
IUPAC	International Union of Pure and Applied Chemistry
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

LSC	liquid/solid chromatography
MNBA	2-nitro-4-methylsulfonylbenzoic acid
MRL	maximum residue limit
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
RAC	raw agricultural commodity
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
TLC	thin layer chromatography
T_{max}	time to reach maximum concentration
TRR	total radioactive residues
TTC	threshold of toxicological concern
UK	United Kingdom
USA	United States of America
WHO	World Health Organization
Xg	relative centrifugal force

Use of JMPR reports and evaluations by registration authorities

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

Introduction

The 2019 Extra Joint FAO/WHO Meeting on Pesticide Residues (JMPR) was held in Gatineau/Ottawa, Canada from 7 to 17 May. The meeting was opened by Mr Brent Wilson, Deputy Director of Technical Trade Policy, Department of Agriculture and Agri-Food.

Mr Wilson welcomed the participants of the first Extra JMPR Meeting to Canada and indicated that Canada is a strong supporter of the system of international standards, including those established by Codex, because they help to facilitate the production and trade of safe foods. He highlighted the fact that international food trade relied heavily on a predictable trade environment, in which decisions taken are based on scientific justification. As a result he believed the scientific advice provided by the JMPR played an important role in facilitating trade, as well as being used by many governments in their pesticide registration process to set standards when managing imports.

However, due to resource limitations and increasing submissions the timeframes for the scheduling of compounds for JMPR evaluation have been extended.

From that perspective he considered the hosting of the 2019 Extra JMPR by the Canadian government to be an important initiative in expediting the international standard setting process. Mr Wilson also noted that Canada's proposal and funding of the Extra JMPR Meeting opens the door for other countries to contribute to such a meeting.

The JMPR Secretariats expressed their appreciation to the Canadian government for hosting this meeting and as well as the training for the new JMPR experts in 2017, noting that half of the FAO experts participating in the current Meeting were the result of a previous training organized jointly by the Canadian government and FAO.

During the meeting, the FAO Panel of Experts on Pesticide Residues in Food and the Environment was responsible for reviewing residue and analytical aspects of the pesticides under consideration, including data on their metabolism, fate in the environment and use patterns, and for estimating the maximum levels of residues that might occur as a result of use of the pesticides according to good agricultural practice. The methodologies are described in detail in the FAO Manual on the submission and evaluation of pesticide residue data for the estimation of maximum residue levels in food and feed (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 19 pesticides for toxicity or residues, or both. 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 order to facilitate the decision-making process by the CCPR.

ACETOCHLOR (280)

First draft prepared by Mr P Rembischevski, Brazilian Health Regulatory Agency, Brasilia, Brazil

EXPLANATION

Acetochlor is a selective herbicide from the chloroacetanilide class used against grasses and broadleaf weeds in a variety of crops. It inhibits protein synthesis in shoot meristems and root tips. It was first and last evaluated by JMPR in 2015 (T, R), when an ADI of 0–0.01 mg/kg bw and an ARfD of 1 mg/kg bw were established. The residue definition for compliance with the MRL and for estimation of dietary exposure (for animal and plant commodities) is the sum of compounds hydrolysable with base to 2-ethyl-6-methylaniline (EMA) and 2-(1-hydroxyethyl)-6-methylaniline (HEMA), expressed in terms of acetochlor. The residue is not fat soluble.

Acetochlor was scheduled at the Fiftieth Session of the CCPR for the evaluation of additional uses by the 2019 Extra JMPR. The Meeting received new information on soya bean metabolism study, analytical method data and residue trials on soya bean and alfalfa (forage and hay).

Plant metabolism

A plant metabolism study conducted with acetochlor on soya beans after pre-plant (PP-T) or post-emergence (POE-T) application was reviewed by JMPR 2015, when metabolite identification were conducted only on forage and hay extracts, which contained the highest residues. The new study submitted to the present Meeting involves the characterization and identification of the metabolites in soya bean seed extracts from the original study (Kurtzweil *et al*., 2016). Metabolites associated with the peaks of interest of the POE-T and PP-T extract profiles were isolated and purified by liquid chromatography, and characterized chromatographically by acid pressure hydrolysis and by LC/MS. These metabolites were acetochlor *tert*-sulfinylacetic acid, acetochlor *tert*-sulfinyllactic acid and acetochlor 1-hydroxyethyl *sec*-oxanilic acid, along with another metabolite that was not identified, but likely belongs to the same hydroxyethylmethylaniline-forming class of chemistry as the 1-hydroxyethyl *sec*-oxanilic acid based on results from acid pressure hydrolysis. The metabolite associated with another PP-T peak was characterized as a natural product, possibly a carbohydrate. The metabolites or conjugate identified result from pathways that have been previously proposed for the metabolism of acetochlor in crops (JMPR 2015).

The three metabolites identified in this study are accounted for by the current analytical residue methodology. The *tert*-sulfinylacetic acid and *tert*-sulfinyllactic acid are both converted to 2-methyl-6-ethylaniline (EMA) and the 1-hydroxyethyl *sec*-oxanilic acid is converted to 2-(1-hydroxyethyl)-6-methylaniline (HEMA) in the method. A summary of the identified or characterized metabolites is provided in Table 1.

Table 1 Identified or characterized metabolites in following pre-plant (PP-T) or post-emergence (POE-T) soya bean seed

Metabolite	Treatment	Ret. Time (min)	Ret. Time (min)	mg/kg eq	% TRR
acetochlor *tert*-sulfinylacetic acid	POE-T	46.7	51.75	0.029	15.1
acetochlor *tert*-sulfinyllactic acid	POE-T	41.8	46.25	0.016	8.3
acetochlor 1-hydroxyethyl *sec*-oxanilic acid	POE-T	8.3	9.25	0.019	9.9
acetochlor 1-hydroxyethyl *sec*-oxanilic acid + Unknown *m/z* 149	PP-T	8.2	9.25	0.013	7.4
Unknown natural product	PP-T	5.8	5.75	0.12	6.9

RESIDUE ANALYSIS

Analytical methods

Method ME-1738-03 (Huang, 2016), which was used to analyse the soya bean seed and alfalfa (forage and hay) is a modification of the method ME-1215 (evaluated by the 2015 JMPR), in which sample size was decreased to 125 mg and the extraction solvent was changed from acetonitrile:water to methanol:water. Crop matrices are freezer-milled, weighed into 96-well format tubes, methanol/water added; the samples are capped and agitated on a high-speed shaker for extraction, then centrifuged. An aliquot of the extract is hydrolysed in aqueous sodium hydroxide, quenched with aqueous sulfuric acid, and an aliquot of the hydrolysate is mixed with isotopically labelled EMA and HEMA internal standards (IS), and then processed through an Oasis MCX SPE plate if additional selectivity is needed. Alternatively, the IS can be added along with the extraction solution. Representative compounds that generate EMA (*tert*-sulfonic acid) and HEMA (1-hydroxyethyl-tert-oxanilic acid) on base hydrolysis are used as reference materials for fortification and method validation. The eluate is analysed by LC-MS/MS. The quantitation and confirmation transitions for EMA are m/z 136→91 and m/z 136→119, respectively. For HEMA the transitions are m/z 136→119 and m/z 136→91, respectively. The LOQ of the method is either 0.010 or 0.025 mg/kg depending on the matrix and analyte. The residue is calculated as the sum of HEMA and EMA, and expressed as acetochlor. Recoveries are shown in Table for pre-study validation and Table 3 for in-study validation.

Table 2 Recovery results for EMA and HEMA from pre-study validation (n=6)

		Quantitation Transition		Confirmation Transition	
Analyte	Level (mg/kg)	Mean recovery (%)	RSD (%)	Mean recovery (%)	RSD (%)
Cotton Seed					
EMA	0.01	108	3.8	109	6.6
	0.25	103	8.7	103	7.5
	4	107	1.7	108	2.1
HEMA	0.01	105	2.2	105	7.6
	0.25	95	6.6	94	7.4
	4	99	1.2	99	1.1
Soya bean seed					
EMA	0.01	103	9.9	105	10.9
	0.25	105	7.2	107	8.7
	4	112	1.5	113	0.9
HEMA	0.01	96	6.3	96	7.0
	0.25	100	0.9	102	1.8
	4	101	2.0	101	1.8
Corn Grain					
HEMA	0.01	97	5.0	99	5.5
	0.25	92	2.0	91	1.6
	4	94	2.4	94	2.9

Table 3 Recovery results for EMA and HEMA from in-study validations (quantification transition)

Analyte	Level (mg/kg)	No. of samples (n)	Mean recovery, %	RSD (%)
Cotton seed				
EMA	0.01	14	110	5.4
	0.25	6	97	2.2
	4	6	98	3.9
HEMA	0.01	14	105	5.3
	0.25	6	98	1.6
	4	6	102	1.8

Analyte	Level (mg/kg)	No. of samples (n)	Mean recovery, %	RSD (%)
Soya bean seed				
EMA	0.01	16	101	7.4
	0.25	6	105	4.9
	4	6	105	5.0
HEMA	0.01	16	111	4.4
	0.25	6	104	1.6
	4	6	110	3.2
Soya bean seed				
EMA	0.024	6	101	3.7
	0.24	6	114	2.1
	3.88	6	118	2.7
HEMA	0.024	6	87	8.0
	0.24	6	99	6.6
	3.86	6	90	10.7
Corn grain				
EMA	0.025	6	78	6.2
	0.05	6	94	4.8
	0.09	6	87	4.3
	0.25	6	97	10.0
HEMA	0.01	16	111	4.7
	0.025	6	108	2.8
	0.05	6	101	0.8
	0.09	6	103	3.1
	0.25	6	99	2.4
Alfalfa forage				
EMA	0.025	5	85.0	11.3
	4	5	103	8.9
HEMA	0.025	5	83.7	9.0
	4	5	86.1	5.6
Alfalfa hay				
EMA	0.025	5	86.2	10.3
	4	5	85.2	9.1
HEMA	0.025	5	80.7	9.7
	4	5	89.1	9.5

The calibration curves for both transitions of EMA and HEMA were linear with coefficient of determination (R^2) values of > 0.990. A linear fit with 1/× weighting was used. No significant interferences (> 30% of the LOQ) were observed within the retention window of any analyte in any matrix using the quantitation ion transition (precursor-to-product transition) except EMA in corn grain with an LOQ at 0.01 mg/kg. The LOQ and LOD for each EMA and HEMA quantitation transition for all the matrices are summarized in Table4.

Table 4 LOD Values for EMA and HEMA from ME-1738-03

Matrix	Analyte (Precursor Ion/Product Ion) (amu)	LOQ (mg/kg)	LOD (mg/kg)
Cotton Seed[1]	EMA Primary (136/91)	0.01	0.0013
	EMA Secondary (136/119)	0.01	0.0023
	HEMA Primary (134/119)	0.01	0.0007
	HEMA Secondary (134/91)	0.01	0.0025
Corn Grain [b]	EMA Primary (136/91)	0.025	0.0041
	EMA Secondary (136/119)	NA	NA

Matrix	Analyte (Precursor Ion/Product Ion) (amu)	LOQ (mg/kg)	LOD (mg/kg)
	HEMA Primary (134/119)	0.01	0.0015
	HEMA Secondary (134/91)	0.01	0.0017
Soya bean seed [a]	EMA Primary (136/91)	0.01	0.0032
	EMA Secondary (136/119)	0.01	0.0036
	HEMA Primary (134/119)	0.01	0.0019
	HEMA Secondary (134/91)	0.01	0.0021
Soya bean seed [b]	EMA Primary (136/91)	0.025	0.0030
	HEMA Primary (134/119)	0.025	0.0056
Alfalfa forage [b]	EMA Primary (136/91)	0.025	0.0111
	EMA Secondary (136/119)	0.025	0.015
	HEMA Primary (134/119)	0.025	0.0086
	HEMA Secondary (134/91)	0.025	0.011
Alfalfa hay [b]	EMA Primary (136/91)	0.025	0.010
	EMA Secondary (136/119)	0.025	0.012
	HEMA Primary (134/119)	0.025	0.009
	HEMA Secondary (134/91)	0.025	0.0087

[a] Pre-study data;

[b] In-study data.

Method ME-2024 (Vogl, 2017) involves extraction of canola and soya bean matrices with 80% methanol in water, centrifugation and an aliquot of extract transferred to a vial containing EMA and HEMA internal standards and 50% sodium hydroxide solution. The vials are placed in a forced-air oven for at least 1 hour at approximately 95 °C to hydrolyse acetochlor residues, cooled to room temperature, and cold 50% formic acid added to quench the base. After vortexing the vials, a portion of the hydrolysed extract is filtered, centrifuged and submitted to quantification of EMA and HEMA by LC-MS/MS. The recovery results are shown in Table 5.

Table 5 Validation recovery results for EMA and HEMA in canola and soya bean seed (Method ME-2024)

			Quantitation Transition			Confirmation Transition		
Analyte	Fort. level (mg/kg)	No. of samples (n)	Recoveries (%)	Overall mean % ± std Dev	RSD (%)	Recoveries (%)	Overall mean % ± std Dev	RSD (%)
Canola Seed								
EMA	0.025	5	106, 94, 103, 100, 99	100 ± 4.5	4.5	108, 100, 97, 96, 108	102 ± 5.8	5.7
	0.25	5	98, 97, 93, 93, 94	95 ± 2.3	2.5	102, 97, 96, 95, 97	97 ± 2.7	2.8
HEMA	0.025	5	91, 90, 86, 91, 94	90 ± 2.9	3.2	85, 86, 87, 87, 82	85 ± 2.1	2.4
	0.25	5	95, 85, 83, 82, 83	86 ± 5.4	6.3	86, 85, 88, 91, 89	88 ± 2.4	2.7
Canola Meal								
EMA	0.025	5	85, 90, 85, 91, 80	86 ± 4.4	5.1	100, 87, 83, 86, 90	89 ± 6.5	7.3
	0.25	5	88, 88, 95, 87, 83	88 ± 4.3	4.9	89, 88, 93, 84, 79	87 ± 5.3	6.1
HEMA	0.025	5	83, 82, 83, 83, 87	84 ± 1.9	2.3	82, 87, 84, 89, 76	84 ± 5.0	6.0
	0.25	5	85, 82, 85, 81, 82	83 ± 1.9	2.3	85, 81, 78, 81, 85	82 ± 3.0	3.7
Canola Oil								
EMA	0.025	5	87, 86, 83, 84, 79	84 ± 3.1	3.7	92, 87, 90, 81, 83	87 ± 4.6	5.3
	0.25	5	102, 89, 88, 95, 98	94 ± 5.9	6.3	105, 87, 88, 94, 97	94 ± 7.3	7.8
HEMA	0.025	5	88, 95, 92, 90, 90	91 ± 2.6	2.9	84, 90, 87, 84, 87	86 ± 2.5	2.9
	0.25	5	92, 86, 82, 88, 89	87 ± 3.7	4.3	95, 85, 82, 88, 89	88 ± 4.9	5.5
Soya bean Seed								
EMA	0.025	5	80, 75, 85, 92, 92	85	8.8	88, 98, 93, 88, 92	92	4.5

Analyte	Fort. level (mg/kg)	No. of samples (n)	Quantitation Transition			Confirmation Transition		
			Recoveries (%)	Overall mean % ± std Dev	RSD (%)	Recoveries (%)	Overall mean % ± std Dev	RSD (%)
HEMA	0.25	5	84, 83, 82, 82, 92	85	5.0	84, 82, 83, 82, 94	85	6.0
	0.025	5	84, 92, 91, 93, 90	90	3.9	76, 87, 98, 91, 87	88	9.1
	0.25	5	77, 78, 79, 81, 84	80	3.5	78, 80, 81, 85, 84	82	3.5

All calibration curves (0.0075 to 4.0 mg/kg) used linear regression with 1/x weighting and had coefficients of determination (R^2) higher than 0.99. In general, no significant interferences (> 30% of the LOQ) were observed for EMA or HEMA for all matrices across all transitions, and no significant biases from matrix effects were observed. The LOQ for both EMA and HEMA was determined to be 0.025 mg/kg and the LOD was calculated as the standard deviation multiplied by the one-tailed t-test at 99% confidence for n-1 degrees of freedom, where n is equal to the number of replicates, as shown in Table 6.

Table 6 LOD calculations for EMA and HEMA

Matrix	Canola Seed		Canola Meal		Canola Oil		Soya bean Seed	
	Std Dev (s)	LOD ($s \times t_{0.99}$)	Std Dev (s)	LOD ($s \times t_{0.99}$)	Std Dev (s)	LOD ($s \times t_{0.99}$)	Std Dev (s)	LOD ($s \times t_{0.99}$)
EMA m/z 136/91	0.00114	0.00427	0.000532	0.00199	0.000774	0.00290	0.00275	0.0103
EMA m/z 136/77	0.00146	0.00546	NA	NA	0.00112	0.00420	0.00200	0.00748
HEMA m/z 134/119	0.000740	0.00277	0.000697	0.00261	0.000663	0.00248	0.000671	0.00251
HEMA m/z 134/115	0.000546	0.00205	0.00129	0.00485	0.000563	0.00211	0.00132	0.00496
EMA m/z 136/91	0.00181	0.00679	0.00107	0.00402	0.000911	0.00341	0.00190	0.00713
EMA m/z 136/77	0.000983	0.00368	0.00167	0.00624	0.00146	0.00547	0.00106	0.00398
HEMA m/z 134/119	0.000780	0.00292	0.000457	0.00171	0.000537	0.00201	0.000923	0.00346
HEMA m/z 134/115	0.00193	0.00724	0.00127	0.00476	0.00105	0.00395	0.00199	0.00746

Data from an independent laboratory validation (ILV) of method ME-2024 was also submitted (Bending & Przybylek, 2018), confirming the satisfactory performance and LOQ of 0.025 mg/kg for both EMA and HEMA (expressed as acetochlor equivalents).

Stability of pesticide residues in stored analytical samples

The stability of acetochlor incurred residues in the soya bean samples after more than eight years of frozen storage was estimated based on the analysis conducted when the study was performed (2007) and in 2016. The results are shown in Table 7.

Table 7 Comparison of incurred residues in treated soya bean seed obtained in the 2016 study (MSL0029938) with those obtained in the 2007 study (MSL0020719)

Sample site	EMA			HEMA			EMA + HEMA		
	2007	2016	% remaining	2007	2016	% remaining	2007	2016	% remaining
AR-1	0.193	0.221	115	0.056	0.082	146	0.249	0.303	122
IA-4	0.212	0.289	136	0.072	0.098	136	0.284	0.387	136
IL-2	0.157	0.191	122	0.058	0.094	162	0.215	0.285	133
IL-3	0.192	0.193	100	0.083	0.125	151	0.275	0.318	116
LA	0.283	0.331	117	0.099	0.126	127	0.381	0.457	120
MN-2	0.117	0.161	138	0.047	0.063	134	0.163	0.224	137
MO	0.177	0.228	129	0.061	0.090	148	0.239	0.318	133
NC	0.101	0.124	123	0.038	0.063	166	0.138	0.187	136

	Average	122	Average	146	Average	129

USE PATTERN

Acetochlor is registered for uses in a variety of food crops in various countries. GAP information relevant for this evaluation is shown in Table 8.

Table 8 Summary of Good Agricultural Practices for acetochlor in the USA using ground broadcast spray of micro-encapsulated formulation (359 g/L)

Crop	Application		Application rate per treatment			PHI	Remarks
	growth stage (j)	number	kg ai/hL	water L/ha	kg ai/ ha	(days)	
Soya bean	Apply pre-plant, at-planting, or pre-emergence, and post-emergence, optimally at growth stage V2–V3, before reaching stage R2.	1–2	1.12 to 1.80	≥93.6	1.05 to 1.68	—	Do not exceed 3.36 kg ai/ha per year.
Alfalfa	Apply pre-plant, at-planting, or pre-emergence, and post-emergence (up to or at the 4th-trifoliate stage [new stands], or following spring green-up [fall-planted or established stands], or between cuttings).	1–3	1.12 to 1.80	≥93.6	1.05 to 1.68	≥20	Do not exceed 3.36 kg ai/ha per year.

RESULTS OF SUPERVISED RESIDUE TRIALS ON CROPS

Soya bean

A study was conducted in 2007 with soya bean at 21 sites in the United States, stored under frozen conditions and analysed in 2016. The residues were demonstrated to be stable over the storage period. The results are shown in Table 9.

Table 9 Residues of acetochlor in soya bean seed from trials conducted in the USA in 2007 using 2 post-emergence applications of a microencapsulated formulation (Report MSL0029938). Residues are reported as acetochlor equivalents. Residues reported as < LOQ were considered at LOQ for the calculation of the total residues

Site code State Location	Crop variety	Application rate			Growth stage	Residues, mg/kg			DAT (days)
		kg ai/ ha	water (L/ha)	kg ai/ hL		EMA	HEMA	Total	
AR-1 Arkansas Crittenden	AG4403RR	1.69 1.67	130 131	1.30 1.28	V5 / R1-R2	0.061, 0.081 (0.071)	0.048, 0.058 (0.053)	0.109, 0.138 (0.12)	90
AR-2 Arkansas Jackson	JG55R505C	1.67 1.71	186 191	0.90 0.90	R2	0.147, 0.118, (0.133)	0.115, 0.101 (0.108)	0.262, 0.219 (0.24)	83
IA-1 Iowa Jefferson	Asgrow 3101	1.66 1.69	163 146	1.02 1.16	R1	0.026, <0.025 (0.025)	<0.025, <0.025 (<0.025)	0.051, <0.050 (0.05)	90
IA-2 Iowa Wapello	AG 3802	1.65 1.71	149 149	1.11 1.15	R1	<0.025, <0.025 (<0.025)	<0.025, <0.025 (<0.025)	<0.050 <0.050 (<0.05)	97
IA-3 Iowa Dickinson	NK S19-L7	1.66 1.70	145 191	1.14 0.89	R1-R2	0.067, 0.086 (0.076)	0.030, 0.035 (0.032)	0.096 0.121 (0.11)	100
IA-4	92M52	1.68	180	0.94	R2	0.097,	0.076,	0.173	83

Site code State Location	Crop variety	Application rate			Growth stage	Residues, mg/kg			DAT (days)
		kg ai/ ha	water (L/ha)	kg ai/ hL		EMA	HEMA	Total	
Iowa Guthrie		1.67	178	0.94		0.084 (0.091)	0.076 (0.076)	0.161 (0.17)	
IL-1 Illinois Clinton	5N382 RR	1.68 1.68	183 132	0.92 1.27	R1-R2	0.591, 0.495 (0.543)	0.377, 0.355 (0.366)	0.968 0.850 (0.91)	80
IL-2 Illinois Clinton	NK 37N4	1.67 1.71	110 133	1.51 1.29	R1-R2	0.108, 0.120 (0.114)	0.078, 0.084 (0.081)	0.186 0.204 (0.20)	91
IL-3 Illinois Effingham	Trisler T-3463 RR	1.69 1.69	144 148	1.18 1.15	R2	0.105, 0.099 (0.102)	0.111, 0.097 (0.104)	0.216 0.196 (0.21)	73
IL-4 Illinois Stark	AG3101	1.73 1.69	153 146	1.13 1.16	R1-R2	0.124, 0.080 (0.102)	0.088, 0.077 (0.083)	0.212 0.158 (0.19)	78
IN-1 Indiana Parke	T-3463RR	1.70 1.71	146 143	1.17 1.20	R1	0.054, 0.097 (0.076)	0.057, 0.069 (0.063)	0.111 0.166 (0.14)	90
IN-2 Indiana Montgomery	T-3463RR	1.69 1.73	146 145	1.16 1.19	R1	0.048, 0.050 (0.049)	0.051, 0.060 (0.056)	0.099 0.110 (0.10)	93
LA Louisiana St. Landry	AG 5905	1.70 1.69	174 140	0.98 1.21	R2	0.237, 0.181 (0.209)	0.071, 0.087 (0.079)	0.308 0.268 (0.29)	77
MN-1 Minnesota Stearns	90M60-N201	1.69 1.65	152 148	1.12 1.11	R2	<0.025, <0.025 (<0.025)	<0.025, <0.025 (<0.025)	<0.050 <0.050 (<0.05)	86
MN-2 Minnesota Freeborn	Pioneer 91M30	1.68 1.66	151 142	1.12 1.17	R2	0.048, 0.061 (0.055)	0.062, 0.060 (0.061)	0.110 0.122 (0.12)	82
MO Missouri Adair	Asgrow AG3802	1.65 1.68	142 154	1.16 1.09	R1-R2	0.141, 0.163 (0.152)	0.062, 0.084 (0.073)	0.203 0.247 (0.23)	96
NC N. Carolina Wayne	NK 565-M3	1.68 1.68	172 120	0.98 1.40	R1, beginning to flower	0.195, 0.104 (0.150)	0.119, 0.081 (0.100)	0.314 0.185 (0.25)	103
NE Nebraska York	WW152201	1.33 1.69	140 187	0.95 0.90	BBCH 61, R-1	0.069, 0.073 (0.071)	0.125, 0.124 (0.125)	0.194 0.197 (0.20)	87
OH-1 Ohio Fayette	Crop Plan RC 3935	1.68 1.64	146 146	1.15 1.12	R1-R2	0.074, 0.077 (0.076)	0.071, 0.076 (0.074)	0.146 0.153 (0.15)	78
OH-2 Ohio Pickaway	Crows 3518 R	1.71 1.65	149 147	1.15 1.12	R1-R2	0.120, 0.081 (0.100)	0.142, 0.102 (0.122)	0.262 0.183 (0.22)	78
SC S. Carolina Barnwell	97M50	1.67 1.69	137 140	1.22 1.21	R2	0.232, 0.221 (0.227)	0.282, 0.278 (0.280)	0.514 0.499 (0.51)	99

Alfalfa

Residue trials were conducted in 2013 and 2014 at 14 major growing regions in the United States. Hay samples were cut at the same time as forage samples, but were air-dried to a moisture level of 10-20% before taken from the field. Acetochlor residues in alfalfa forage were reported on the samples as collected, on fresh weight basis. Treatments involved either a 1.7 kg ai/ha pre-emergence application followed by a 1.7 kg ai/ha application at the 4th trifoliate stage, or a post-harvest application between cuttings. The results are showing in Table 10.

Table 10 Acetochlor residues in alfalfa forage and hay after a pre-emergence and/or post-emergence application in the United States in 2013/2014 (Study report MSL0027578) using microencapsulated formulation. Residues are reported as acetochlor equivalents.

Site code State, variety, Treatment	Application rate			Growth stage	Commodity analysed	Residues (mg/kg)			DAT (days)
	kg ai/ ha	water (L/ha)	kg ai/ hL			EMA	HEMA	Total	
01PA Pennsylvania DKA44-16RR	1.68 1.70	172.4 175.5	0.975 0.970	1) BBCH 00 bare soil pre-emergence 2) BBCH 14 4th trifoliate expanded	Forage 1 post app.	0.404, 0.368 (0.386)	0.136, 0.121 (0.129)	0.540, 0.489 (0.52)	49
					Forage 2 1st regrowth	0.110, 0.182 (0.146)	0.088, 0.093 (0.091)	0.198 0.275 (0.24)	81
					Forage 3 2nd regrowth	0.112, 0.112 (0.112)	0.127, 0.112 (0.120)	0.239, 0.224 (0.23)	123
					Forage 4 3rd regrowth	0.117, 0.107 (0.112)	0.119, 0.115 (0.117)	0.236, 0.222 (0.23)	158
	1.68 1.70	172.4 175.5	0.975 0.970	1) BBCH 00 bare soil pre-emergence 2) BBCH 14 4th trifoliate expanded	Hay 1 post app.	0.944, 0.931 (0.938)	0.342, 0.316 (0.329)	1.29, 1.25 (1.3)	51
					Hay 2 1st regrowth	0.319, 0.327 (0.323)	0.281, 0.334 (0.308)	0.600, 0.661 (0.63)	84
					Hay 3 2nd regrowth	0.358, 0.366 (0.362)	0.414, 0.376 (0.395)	0.772, 0.742 (0.76)	124
					Hay 4 3rd regrowth	0.196, 0.239 (0.218)	0.267, 0.285 (0.276)	0.463, 0.524 (0.49)	160
01PA Pennsylvania DKA44-16RR	1.72	177.3	0.973	BBCH 14 4th trifoliate expanded	Forage 1	0.480, 0.441 (0.461)	0.197, 0.144 (0.171)	0.677, 0.585 (0.63)	49
	1.72	177.3	0.973	BBCH 14 4th trifoliate expanded	Hay 1 Post app	0.920, 0.870 (0.895)	0.307, 0.275 (0.291)	1.23, 1.15 (1.2)	51
	1.70	175.1	0.972	2) BBCH 11-first trifoliate	Forage 2 Post app	0.536, 0.524 (0.530)	0.203, 0.187 (0.195)	0.739, 0.711 (0.73)	29
					Forage 3 1st regr.[4]	0.206, 0.247 (0.227)	0.180, 0.178 (0.179)	0.386, 0.425 (0.41)	71
					Forage 4 2nd regr.	0.176, 0.155 (0.166)	0.162, 0.124 (0.143)	0.338, 0.279 (0.31)	106
	1.70	175.1	0.972	2) BBCH 11-first trifoliate	Hay 2 Post app	1.33, 1.23 (1.28)	0.587, 0.562 (0.575)	1.92, 1.79 (1.9)	32
					Hay 3 1st regr.	0.661, 0.616 (0.639)	0.587, 0.516 (0.552)	1.25, 1.13 (1.2)	72
					Hay 4 2nd regr.	0.417, 0.364 (0.391)	0.395, 0.357 (0.376)	0.812, 0.721 (0.77)	106
2NJ New Jersey DKA44-16RR	1.76 1.76	191.1 171.8	0.920 1.02	1) Bare soil, pre-emergence 2) 4th trifoliate	Forage 1 post app.	0.134, 0.122 (0.128)	0.081, 0.099 (0.090)	0.215, 0.221 (0.22)	49
					Forage 2 1st regrowth	0.109, 0.120 (0.115)	0.089, 0.081 (0.085)	0.198, 0.201 (0.20)	85
					Forage 3 2nd regrowth	0.055, 0.064 (0.059)	0.060, 0.063 (0.061)	0.114, 0.127 (0.12)	125

Site code State, variety, Treatment	Application rate			Growth stage	Commodity analysed	Residues (mg/kg)			DAT (days)
	kg ai/ ha	water (L/ha)	kg ai/ hL			EMA	HEMA	Total	
					Forage 4 3rd regrowth	0.103, 0.106 (0.105)	0.126, 0.139 (0.133)	0.229, 0.245 (0.24)	165
	1.76 1.76	191.1 171.8	0.920 1.02	1) Bare soil, pre-emergence 2) 4th trifoliate	Hay 1 post app.	0.306, 0.292 (0.299)	0.189, 0.203 (0.196)	0.495, 0.495 (0.50)	52
					Hay 2 1st regrowth	0.306, 0.292 (0.299)	0.312, 0.280 (0.296)	0.618, 0.572 (0.60)	90
					Hay 3 2nd regrowth	0.116, 0.135 (0.126)	0.160, 0.155 (0.158)	0.276, 0.290 (0.28)	129
					Hay 4 3rd regrowth	0.255, 0.220 (0.238)	0.303, 0.318 (0.311)	0.558, 0.538 (0.55)	171
2NJ New Jersey DKA44-16RR	1.77	172.9	1.02	4th trifoliate	Forage 1	0.106, 0.100 (0.103)	0.077, 0.062 (0.069)	0.183, 0.162 (0.17)	49
					Forage 2 (1st regr)	0.079, 0.086 (0.082)	0.086, 0.079 (0.082)	0.165, 0.165 (0.17)	85
	1.77	172.9	1.02	4th trifoliate	Hay 1 Post app	0.247, 0.271 (0.259)	0.147, 0.159 (0.153)	0.394, 0.430 (0.41)	52
					Hay 2 (1st regrowth)	0.281, 0.248 (0.265)	0.289, 0.271 (0.280)	0.570, 0.519 (0.55)	90
	1.78	197.9	0.900	2" stubble/ cut 7/5/14	Forage 3 Post	0.233, 0.225, (0.229)	0.212, 0.221, (0.217)	0.445, 0.446 (0.45)	37
					Forage 4 1st regrowth	0.238, 0.214 (0.226)	0.264, 0.257 (0.261)	0.502, 0.471 (0.49)	77
	1.78	197.9	0.900	2" stubble/ cut 7/5/14	Hay 3 Post app	0.646, 0.677 (0.662)	0.609, 0.547 (0.578)	1.26, 1.22 (1.2)	41
					Hay 4 1st regrowth	0.596, 0.562 (0.579)	0.663, 0.652 (0.658)	1.26, 1.21 (1.2)	83
03IL Illinois DKA44-16RR	1.70 1.71	182.2 105.8	0.934 1.62	1) BBCH 00 - bare soil pre-emergence 2) BBCH 13	Forage 1 post app.	0.526, 0.665, 0.582, 0.589 (0.591)	0.084, 0.157, 0.122, 0.162 (0.131)	0.610, 0.822, 0.704, 0.751 (0.72)	38
					Forage 2 1st regrowth	0.092, 0.101 (0.096)	0.124, 0.120 (0.122)	0.216, 0.221 (0.22)	73
					Forage 3 2nd regrowth	0.052, 0.075 (0.064)	0.084, 0.105 (0.094)	0.136, 0.180 (0.16)	117
					Forage 4 3rd regrowth	0.050, 0.056 (0.053)	0.059, 0.067 (0.063)	0.109, 0.122 (0.12)	146
				1) BBCH 00 - bare soil pre-emergence 2) BBCH 13	Hay 1 post app.	0.813, 0.827 (0.820)	0.174, 0.223 (0.199)	0.987, 1.05 (1.0)	40
					Hay 2 1st regrowth	0.142, 0.193 (0.168)	0.187, 0.246 (0.217)	0.329, 0.439 (0.38)	75
					Hay 3	0.114,	0.177,	0.291,	122

Site code State, variety, Treatment	Application rate			Growth stage	Commodity analysed	Residues (mg/kg)			DAT (days)
	kg ai/ ha	water (L/ha)	kg ai/ hL			EMA	HEMA	Total	
					2nd regrowth	0.099 (0.106)	0.164 (0.171)	0.263 (0.28)	
					Hay 4 3rd regrowth	0.108, 0.046 (0.077)	0.157, 0.078 (0.117)	0.265, 0.124 (0.19)	150
03IL Illinois DKA44-16RR	1.75	107.9	1.62	BBCH 13	Forage 1	0.191, 0.230 (0.211)	0.089, 0.083 (0.086)	0.280, 0.313 (0.30)	38
					Hay 1 Post app	0.363, 0.540 (0.452)	0.160, 0.190 (0.175)	0.523, 0.730 (0.63)	40
	1.64	113.5	1.44	BBCH 29	Forage 2 Post app	2.59, 1.81 (2.20)	0.848, 0.615 (0.732)	3.44, 2.43 (2.9)	20
				BBCH 29	Forage 3 1st regrowth	0.142, 0.157 (0.150)	0.184, 0.178 (0.181)	0.326, 0.335 (0.33)	64
				BBCH 29	Forage 4 2nd regrowth	0.106, 0.139 (0.123)	0.120, 0.115 (0.118)	0.226, 0.265 (0.24)	93
				BBCH 29	Hay 2 Post app	3.84, 2.87 (3.36)	1.13, 0.909 (1.02)	4.97, 3.78 (4.4)	22
				BBCH 29	Hay 3 1st regrowth	0.386, 0.357 (0.372)	0.387, 0.370 (0.379)	0.773, 0.727 (0.75)	69
				BBCH 29	Hay 4 2nd regrowth	0.151, 0.169 (0.160)	0.218, 0.210 (0.214)	0.369, 0.379 (0.37)	97
04WI Wisconsin DKA44-16RR	1.67 1.68	179.8 185.7	0.928 0.905	1) Premerger. 2) 4th trifoliate	Forage 1 post app.	0.143, 0.305, 0.095, 0.101 (0.161)	0.058, 0.071 (0.065)	0.177, 0.274 (0.23)	50
					Forage 2 1st regrowth	0.148, 0.177 (0.163)	0.098, 0.110 (0.104)	0.246, 0.287 (0.27)	84
					Forage 3 2nd regrowth	0.190, 0.179 (0.185)	0.092, 0.103 (0.098)	0.282, 0.282 (0.28)	119
					Forage 4 3rd regrowth	0.140, 0.133 (0.137)	0.139, 0.133 (0.136)	0.279, 0.266 (0.27)	172
	1.67 1.68	179.8 185.7	0.928 0.905	1) Premerger. 2) 4th trifoliate	Hay 1 post app. (2 apps.)	0.463, 0.318 (0.391)	0.216, 0.143 (0.180)	0.679, 0.461 (0.57)	57
					Hay 2 1st regrowth	0.302, 0.290 (0.296)	0.247, 0.245 (0.246)	0.549, 0.535 (0.54)	92
					Hay 3 2nd regrowth	0.332, 0.421 (0.377)	0.238, 0.309 (0.274)	0.570, 0.730 (0.65)	125
					Hay 4 3rd regrowth	0.317, 0.301 (0.309)	0.309, 0.302 (0.306)	0.626, 0.603 (0.62)	179
4WI Wisconsin DKA44-16RR	1.68	185.7	0.905	4th trifoliate	Forage 1	0.110, 0.116 (0.113)	0.060, 0.049 (0.055)	0.170, 0.165 (0.17)	50
	1.68	185.7	0.905	4th trifoliate	Hay 1	0.261,	0.134,	0.395,	57

Site code State, variety, Treatment	Application rate			Growth stage	Commodity analysed	Residues (mg/kg)			DAT (days)
	kg ai/ ha	water (L/ha)	kg ai/ hL			EMA	HEMA	Total	
					Post app	0.226 (0.244)	0.122 (0.128)	0.348 (0.37)	
	1.68 1.67	185.7 184.5	0.905 0.905	7 days after 1st cutting	Forage 2 Post app	0.283, 0.283 (0.283)	0.118, 0.147 (0.133)	0.401, 0.430 (0.42)	27
					Forage 3 1st regrowth	0.208, 0.195 (0.202)	0.108, 0.109 (0.109)	0.316, 0.304 (0.31)	62
					Forage 4 2nd regrowth	0.243, 0.210 (0.227)	0.242, 0.215 (0.229)	0.485, 0.425 (0.46)	115
	1.68 1.67	185.7 184.5	0.905 0.905	7 days after 1st cutting	Hay 2 Post app	0.917, 1.24 (1.08)	0.410, 0.398 (0.404)	1.33, 1.64 (1.5)	35
					Hay 3 1st regrowth	0.530, 0.398 (0.464)	0.296, 0.331 (0.314)	0.826, 0.729 (0.78)	68
					Hay 4 2nd regrowth	0.423, 0.374 (0.399)	0.343, 0.298 (0.321)	0.766, 0.672 (0.72)	122
05IA Iowa DKA44-16RR	1.68 1.68	174.1 160.9	0.965 1.04	1) BBCH 00 2) BBCH 14	Forage 1 post app.	0.150, 0.159 (0.155)	0.129, 0.135 (0.132)	0.279, 0.294 (0.29)	51
					Forage 2 1st regrowth	0.077, 0.070 (0.074)	0.052, 0.037 (0.045)	0.129, 0.108 (0.12)	84
					Forage 3 2nd regrowth	0.152, 0.133 (0.143)	0.107, 0.099 (0.103)	0.259, 0.232 (0.25)	123
					Forage 4 3rd regrowth	0.179, 0.198 (0.189)	0.145, 0.160 (0.153)	0.324, 0.358 (0.34)	162
					Hay 1 post app.	0.611, 0.540 (0.576)	0.430, 0.409 (0.420)	1.04, 0.949 (0.99)	55
					Hay 2 1st regrowth	0.098, 0.212 (0.155)	0.074, 0.147 (0.111)	0.173, 0.359 (0.27)	86
					Hay 3 2nd regrowth	0.388, 0.409 (0.399)	0.282 0.261 (0.272)	0.670, 0.670 (0.67)	126
					Hay 4 3rd regrowth	0.440, 0.490 (0.465)	0.355, 0.411 (0.383)	0.795, 0.901 (0.85)	164
05IA Iowa DKA44-16RR	1.68	160.3	1.05	BBCH 14	Forage 1	0.125, 0.126 (0.126)	0.079, 0.084 (0.082)	0.204, 0.210 (0.21)	51
				BBCH 14	Forage 2 (1st regrowth)	0.146, 0.091 (0.118)	0.078, 0.076 (0.077)	0.224, 0.166 (0.19)	84
	1.68	177.1	0.949	BBCH 14	Hay 1 Post app	0.528, 0.526 (0.527)	0.264, 0.282 (0.273)	0.792, 0.808 (0.80)	55
					Hay 2 (1st regrowth)	0.393, 0.338 (0.366)	0.281, 0.245 (0.263)	0.674, 0.583 (0.63)	86
	1.68	177.1	0.949	BBCH 13	Forage 3 Post app	0.391, 0.460 (0.426)	0.146, 0.161 (0.154)	0.537, 0.621 (0.58)	28
					Forage 4	0.294,	0.193,	0.487,	67

Site code State, variety, Treatment	Application rate			Growth stage	Commodity analysed	Residues (mg/kg)			DAT (days)
	kg ai/ ha	water (L/ha)	kg ai/ hL			EMA	HEMA	Total	
					1st regrowth	0.239 (0.267)	0.184 (0.189)	0.423 (0.46)	
	1.68	177.1	0.949	BBCH 13	Hay 3 Post app	1.46, 1.47 (1.47)	0.551, 0.644 (0.598)	2.01, 2.11 (2.1)	31
					Hay 4 1st regrowth	0.758, 0.748, 0.645, 0.585, 0.895 (0.731)	0.563, 0.419, 0.507 (0.499)	1.24, 1.22 (1.2)	69
06MN Minnesota DKA44-16RR	1.70 1.66	157.6 131.2	1.08 1.26	1) 0	Forage 1 post app.	0.674, 0.891 (0.783)	0.285, 0.302 (0.294)	0.959, 1.19 (1.1)	24
					Forage 2 1st regrowth	0.182, 0.197 (0.190)	0.128, 0.152 (0.140)	0.310, 0.349 (0.33)	69
					Forage 3 2nd regrowth	0.072, 0.071 (0.071)	0.090, 0.071 (0.081)	0.162, 0.142 (0.15)	127
				2) 4th trifoliate	Hay 1 post app.	1.39, 1.54 (1.47)	0.508, 0.548 (0.528)	1.90, 2.09 (2.0)	28
					Hay 2 1st regrowth	0.368, 0.424 (0.396)	0.299, 0.331 (0.315)	0.667, 0.755 (0.71)	75
					Hay 3 2nd regrowth	0.081, 0.078 (0.080)	0.089, 0.089 (0.089)	0.171, 0.167 (0.17)	133
06MN Minnesota DKA44-16RR	1.69	133.2	1.26	4th trifoliate	Forage 1	0.577, 0.534 (0.556)	0.206, 0.198 (0.202)	0.783, 0.732 (0.76)	24
	1.69	133.2	1.26	4th trifoliate	Hay 1 Post app (1 app)	1.31, 1.29 (1.30)	0.455, 0.442 (0.449)	1.77, 1.73 (1.8)	28
06MN Minnesota DKA44-16RR	1.69	138.3	1.22	2) regrowth (1-3 inches)	Forage 2 Post app	0.472, 0.401 (0.437)	0.219, 0.187 (0.203)	0.691, 0.588 (0.64)	39
				2) regrowth (1-3 inches)	Forage 3 1st regrowth	0.109, 0.093 (0.101)	0.145, 0.153 (0.149)	0.254, 0.246 (0.25)	97
	1.69	138.3	1.22	2) regrowth (1-3 inches)	Hay 2 Post app (2 apps)	0.928, 0.693 (0.811)	0.422, 0.328 (0.375)	1.35, 1.02 (1.2)	45
				2) regrowth (1-3 inches)	Hay 3 1st regrowth	0.143, 0.148 (0.146)	0.208, 0.200 (0.204)	0.351, 0.348 (0.35)	103
07MO Missouri DKA44-16RR	1.69	175	0.965	1) Seeded - pre-emergence 2) BBCH 14	Forage 1 post app.	0.108, 0.075 (0.092)	0.096, 0.065 (0.080)	0.204, 0.140 (0.17)	49
					Forage 2 1st regrowth	0.134, 0.153 (0.144)	0.102, 0.125 (0.114)	0.236, 0.278 (0.26)	80
					Forage 3 2nd regrowth	0.127, 0.133 (0.130)	0.152, 0.161 (0.157)	0.279, 0.294 (0.29)	119
					Forage 4 3rd regrowth	0.062, 0.060 (0.061)	0.044, 0.054 (0.049)	0.106, 0.113 (0.11)	161
	1.69	175	0.965		Hay 1	0.315,	0.268,	0.583,	53

Site code State, variety, Treatment	Application rate			Growth stage	Commodity analysed	Residues (mg/kg)			DAT (days)
	kg ai/ ha	water (L/ha)	kg ai/ hL			EMA	HEMA	Total	
				1) Seeded - pre-emergence 2) BBCH 14	post app.	0.246 (0.281)	0.218 (0.243)	0.464 (0.52)	
					Hay 2 1st regrowth	0.347, 0.291 (0.319)	0.310, 0.260 (0.285)	0.657, 0.551 (0.60)	82
					Hay 3 2nd regrowth	0.322, 0.286 (0.304)	0.420, 0.353 (0.387)	0.742, 0.639 (0.69)	120
					Hay 4 3rd regrowth	0.161, 0.153 (0.157)	0.160, 0.158 (0.159)	0.321, 0.311 (0.32)	164
07MO Missouri DKA44-16RR	1.69	176.0	0.961	BBCH 14	Forage 1	0.115, 0.091 (0.103)	0.070, 0.057 (0.063)	0.185, 0.148 (0.17)	49
					Forage 2 (1st regrowth)	0.141, 0.130 (0.136)	0.111, 0.103 (0.107)	0.252, 0.233 (0.24)	80
					Hay 1 Post app	0.257, 0.174 (0.216)	0.137, 0.123 (0.130)	0.394, 0.297 (0.35)	53
					Hay 2 (1st regrowth)	0.344, 0.374 (0.359)	0.329, 0.345 (0.337)	0.673, 0.719 (0.70)	82
	1.68	176.6	0.951	BBCH 22	Forage 3 Post app	3.76, 4.56 (4.16)	1.45, 1.78 (1.62)	5.21, 6.34 (5.8)	24
					Forage 4 1st regrowth	0.257, 0.213 (0.235)	0.179, 0.161 (0.170)	0.436, 0.374 (0.41)	66
	1.68	176.6	0.951	BBCH 22	Hay 3 Post app	9.81, 8.67 (9.24)	3.38, 4.04 (3.71)	13.2, 12.7 (13.0)	25
					Hay 4 1st regr.	0.926, 0.891 (0.909)	0.645, 0.636 (0.641)	1.57, 1.53 (1.6)	69
08NE Nebraska DKA44-16RR	1.67 1.69	192.4 177.7	0.867 0.952	1) BBCH 0 2) BBCH 14	Forage 1 post app.	0.056, 0.122 (0.089)	0.049, 0.127 (0.088)	0.105, 0.249 (0.18)	57
					Forage 2 1st regrowth	0.266, 0.196 (0.231)	0.174, 0.142 (0.158)	0.440, 0.338 (0.39)	91
					Forage 3 2nd regrowth	0.249, 0.284 (0.267)	0.160, 0.205 (0.183)	0.409, 0.489 (0.45)	122
					Forage 4 3rd regrowth	0.071, 0.050 (0.060)	0.068, 0.058 (0.063)	0.139, 0.109 (0.12)	156
					Hay 1 post app.	0.560, 0.662 (0.611)	0.418, 0.473 (0.446)	0.978, 1.14 (1.1)	62
					Hay 2 1st regrowth	0.560, 0.554 (0.557)	0.513, 0.438 (0.476)	1.07, 0.992 (1.0)	97
					Hay 3 2nd regrowth	0.763, 0.784 (0.774)	0.740, 0.812 (0.776)	1.50, 1.60 (1.6)	132
					Hay 4 3rd regrowth	0.281, 0.219 (0.250)	0.330, 0.260 (0.295)	0.611, 0.479 (0.55)	167
08NE	1.69	178.1	0.950	BBCH 14	Forage 1	0.077,	0.065,	0.143,	57

Site code State, variety, Treatment	Application rate			Growth stage	Commodity analysed	Residues (mg/kg)			DAT (days)
	kg ai/ ha	water (L/ha)	kg ai/ hL			EMA	HEMA	Total	
Nebraska DKA44-16RR						0.064 (0.071)	0.059 (0.062)	0.123 (0.13)	
					Forage 2 (1st regrowth)	0.110, 0.112 (0.111)	0.090, 0.091 (0.090)	0.200, 0.203 (0.20)	91
					Hay 1 Post app	0.306, 0.340 (0.323)	0.185, 0.229 (0.207)	0.491, 0.569 (0.53)	62
					Hay 2 (1st regrowth)	0.382, 0.358 (0.370)	0.318, 0.326 (0.322)	0.700, 0.684 (0.69)	97
	1.68	177.9	0.944	2) BBCH 24	Forage 3 Post app	0.579, 0.556 (0.568)	0.269, 0.232 (0.251)	0.848, 0.788 (0.82)	24
					Forage 4 1st regrowth	0.092, 0.094 (0.093)	0.109, 0.085 (0.097)	0.201, 0.179 (0.19)	58
					Hay 3 Post app	1.83, 1.58 (1.71)	1.15, 1.09 (1.12)	2.98, 2.67 (2.8)	34
				1) BBCH 14 2) BBCH 24	Hay 4 1st regrowth	0.379, 0.391 (0.385)	0.434, 0.401 (0.418)	0.813, 0.792 (0.80)	69
09IN Indiana DKA44-16RR	1.72 1.69	160.2 151.8	1.08 1.11	1) BBCH 0 2) BBCH 09	Forage 1 post app.	0.251, 0.173, 0.276, 0.239 (0.235)	0.208, 0.151, 0.207, 0.182 (0.187)	0.459, 0.324, 0.483, 0.421 (0.42)	42
					Forage 2 1st regrowth	0.248, 0.227 (0.238)	0.243, 0.225 (0.234)	0.491, 0.452 (0.47)	83
					Forage 3 2nd regrowth	0.126, 0.110 (0.118)	0.175, 0.161 (0.168)	0.301, 0.271 (0.29)	176
					Hay 1 post app.	0.656, 0.755 (0.706)	0.533, 0.559 (0.546)	1.19, 1.31 (1.3)	46
					Hay 2 1st regrowth	0.687, 0.754 (0.721)	0.750, 0.771 (0.761)	1.44, 1.53 (1.5)	86
					Hay 3 2nd regrowth	0.224, 0.252 (0.238)	0.359, 0.385 (0.372)	0.583, 0.637 (0.61)	180
09IN Indiana DKA44-16RR	1.70	152.8	1.114	BBCH 09	Forage 1	0.178, 0.179 (0.179)	0.091, 0.105 (0.098)	0.269, 0.284 (0.28)	42
					Forage 2 (1st regrowth)	0.179, 0.162 (0.171)	0.165, 0.140 (0.153)	0.344, 0.302 (0.32)	83
				BBCH 09	Hay 1 Post app	0.735, 0.549 (0.642)	0.257, 0.224 (0.241)	0.992, 0.773 (0.88)	46
					Hay 2 (1st regr.)	0.458, 0.552 (0.505)	0.394, 0.579 (0.487)	0.852, 1.13 (0.99)	86
	1.68	176.3	0.953	2) BBCH 09	Forage 3 Post app	0.249, 0.313 (0.281)	0.190, 0.203 (0.197)	0.439, 0.516 (0.48)	92
	1.70	152.8	0.11	1) BBCH 09	Hay 3 Post app	0.887, 0.876	0.477, 0.504	1.36, 1.38	96

Site code State, variety, Treatment	Application rate			Growth stage	Commodity analysed	Residues (mg/kg)			DAT (days)
	kg ai/ ha	water (L/ha)	kg ai/ hL			EMA	HEMA	Total	
					(2 apps)	(0.882)	(0.491)	(1.4)	
10SD South Dakota DKA44-16RR	1.69 1.77	118.5 136.8	1.43 1.2	1) Pre-emerg. 2) BBCH 13	Forage 1 post app.	3.45, 3.40 (3.43)	0.514, 0.558 (0.536)	3.96, 3.96 (4.0)	23
					Forage 2 1st regrowth	0.348, 0.462 (0.405)	0.078, 0.079 (0.079)	0.426, 0.541 (0.48)	50
					Forage 3 2nd regrowth	0.374, 0.324 (0.349)	0.103, 0.095 (0.099)	0.477, 0.419 (0.45)	74
					Forage 4 3rd regrowth	0.159, 0.144 (0.152)	0.130, 0.106 (0.118)	0.289, 0.250 (0.27)	112
	1.69 1.77	118.5 136.8	1.43 1.2	1) Pre-emerg. 2) BBCH 13	Hay 1 post app.	4.17, 5.82 (5.00)	1.93, 1.81 (1.87)	5.98, 7.75 (6.9)	25
					Hay 2 1st regrowth	1.38, 1.67 (1.53)	0.339, 0.414 (0.377)	1.72, 2.08 (1.9)	53
					Hay 3 2nd regrowth	0.456, 0.397 (0.427)	0.347, 0.307 (0.327)	0.803, 0.704 (0.75)	77
					Hay 4 3rd regrowth	0.339, 0.436 (0.388)	0.415, 0.471 (0.443)	0.754, 0.907 (0.83)	116
10SD South Dakota DKA44-16RR	1.74	134.4	1.29	1) BBCH 13	Forage 1	3.72, 4.20 (3.96)	0.583, 0.799 (0.691)	4.30, 5.00 (4.7)	23
	1.74	134.4	1.29	1) BBCH 13	Hay 1 Post app	4.38, 3.74, 4.80 (4.43)	1.18, 1.24 (1.21)	5.30, 5.98 (5.6)	25
	1.74	134.4	1.29	2) BBCH 11	Forage 2 Post app	1.40, 1.65 (1.53)	0.303, 0.352 (0.328)	1.70, 2.00 (1.9)	21
					Forage 3 1st regrowth	0.495, 0.447 (0.471)	0.129, 0.120 (0.125)	0.624, 0.567 (0.60)	45
					Forage 4 2nd regrowth	0.171, 0.203 (0.187)	0.141, 0.154 (0.148)	0.312, 0.357 (0.34)	83
					Hay 2 Post app	1.86, 2.27 (2.07)	0.690, 0.809 (0.750)	2.55, 3.08 (2.8)	24
	1.74	134.4	1.29	2) BBCH 11	Hay 3 1st regrowth	0.976, 0.951 (0.964)	0.526, 0.534 (0.530)	1.50, 1.49 (1.5)	48
					Hay 4 2nd regrowth	0.535, 0.747 (0.641)	0.476, 0.590 (0.533)	1.01, 1.34 (1.2)	87
11NE Nebraska DKA44-16RR	1.68 1.66	177.4 174.6	0.95 0.95	1) 0 2) BBCH 14	Forage 1 post app.	0.457, 0.587 (0.522)	0.406, 0.454 (0.430)	0.863, 1.04 (0.95)	58
					Forage 2 1st regrowth	0.225, 0.180 (0.203)	0.147, 0.109 (0.128)	0.372, 0.289 (0.33)	86
					Forage 3 2nd regrowth	0.204, 0.228	0.093, 0.115	0.297, 0.343	112

Site code State, variety, Treatment	Application rate			Growth stage	Commodity analysed	Residues (mg/kg)			DAT (days)
	kg ai/ ha	water (L/ha)	kg ai/ hL			EMA	HEMA	Total	
						(0.216)	(0.104)	(0.32)	
					Forage 4 3rd regrowth	0.127, 0.117 (0.122)	0.063, 0.073 (0.068)	0.190, 0.190 0.190	142
	1.68 1.66	177.4 174.6	0.95 0.95	1) 0 2) BBCH 14	Hay 1 post app. (2 apps.)	1.26, 1.37 (1.32)	0.853, 0.911 (0.882)	2.11, 2.28 (2.2)	62
					Hay 2 1st regrowth	0.523, 0.574 (0.549)	0.711, 0.628 (0.670)	1.23, 1.20 (1.2)	93
					Hay 3 2nd regrowth	0.650, 0.556 (0.603)	0.485, 0.438 (0.462)	1.14, 0.994 (1.1)	115
					Hay 4 3rd regrowth	0.444, 0.470 (0.457)	0.399, 0.353 (0.376)	0.843, 0.823 (0.83)	150
11NE Nebraska DKA44-16RR	1.69	178.1	0.95	BBCH 14	Forage 1	0.417, 0.389 (0.403)	0.314, 0.313 (0.314)	0.731, 0.702 (0.72)	58
					Forage 2 (1st regrowth)	0.195, 0.177 (0.186)	0.141, 0.136 (0.139)	0.336, 0.313 (0.33)	86
	1.69	178.1	0.95	BBCH 14	Hay 1 Post app (1 app)	0.709, 0.857 (0.783)	0.451, 0.495 (0.473)	1.16, 1.35 (1.3)	62
					Hay 2 (1st regrowth)	0.386, 0.335 (0.361)	0.514, 0.499 (0.507)	0.900, 0.834 (0.87)	93
	1.68	141. 3	1.19	2) BBCH 25	Forage 3 Post app	2.02, 1.74 (1.88)	0.560, 0.584 (0.572)	2.58, 2.32 (2.5)	19
					Forage 4 1st regrowth	0.296, 0.254 (0.280)	0.189, 0.168 (0.179)	0.485, 0.422 (0.45)	49
					Hay 3 Post app	3.11, 3.38 (3.25)	1.31, 1.50 (1.41)	4.42, 4.88 (4.7)	22
					Hay 4 1st regrowth	0.854, 0.880 (0.867)	0.671, 0.836 (0.754)	1.53, 1.72 (1.6)	57
12UT Utah DKA44-16RR	1.67 1.66	171.3 175.1	0.974 0.947	1) BBCH 0 2) BBCH 14	Forage 1 post app. (2 apps.)	0.135, 0.135 (0.135)	0.067, 0.071 (0.069)	0.202, 0.206 (0.20)	38
					Forage 2 1st regrowth	0.176, 0.062 (0.119)	<0.025, <0.025 (<0.025)	0.201, 0.087 (0.14)	72
					Forage 3 2nd regrowth	<0.025, <0.025 (<0.025)	<0.025, <0.025 (<0.025)	<0.050, <0.050 (<0.05)	108
					Hay 1 post app. (2 apps.)	0.398, 0.396 (0.397)	0.303, 0.289 (0.296)	0.701, 0.685 (0.69)	44
					Hay 2 1st regrowth	0.194, 0.152 (0.173)	0.140, 0.136 (0.138)	0.334, 0.288 (0.31)	79
					Hay 3 2nd regrowth	0.076, 0.068 (0.072)	0.078, 0.074 (0.076)	0.153, 0.143 (0.15)	114
	1.70	178.5	0.954	BBCH 14	Forage 1	0.071, 0.079	0.028, 0.029	0.099, 0.108	38

Site code State, variety, Treatment	Application rate			Growth stage	Commodity analysed	Residues (mg/kg)			DAT (days)
	kg ai/ ha	water (L/ha)	kg ai/ hL			EMA	HEMA	Total	
						(0.075)	(0.028)	(0.10)	
	1.70	178.5	0.954	BBCH 14	Hay 1 Post app (1 app)	0.218, 0.270 (0.244)	0.135, 0.146 (0.141)	0.353, 0.416 (0.39)	44
12UT Utah DKA44-16RR	1.70 1.69	178.5 173.3	0.954 0.976	1) BBCH 14 2) BBCH 12	Forage 2 Post app (2 apps)	0.114, 0.145 (0.130)	0.036, 0.054 (0.045)	0.150, 0.199 (0.17)	31
					Forage 3 1st regrowth	0.027, 0.034 (0.031)	<0.025, <0.025 (<0.025)	0.050, 0.055 (0.05)	67
					Hay 2 Post app	0.331, 0.418 (0.370)	0.234, 0.336 (0.285)	0.565, 0.744 (0.66)	38
					Hay 3 1st regrowth	0.133, 0.123 (0.128)	0.136, 0.139 (0.138)	0.269, 0.262 (0.27)	73
13CA California RR841	1.70 1.67	148.4 140.5	1.15 1.19	1)Pre-emergence 2) Mid-vegetative	Forage 1 post app.	0.463, 1.01, 0.415, 1.08 (0.742)	0.139, 0.336, 0.180, 0.433 (0.272)	0.602, 1.35, 0.595, 1.51 (1.0)	39
					Forage 2 1st regrowth	0.071, 0.267, 0.078, 0.228 (0.161)	0.043, 0.243, 0.045, 0.259 (0.147)	0.119, 0.499, 0.124, 0.487 (0.31)	82
					Forage 3 2nd regrowth	0.222, 0.279 (0.251)	0.133, 0.153 (0.143)	0.355, 0.432 (0.39)	116
	1.70 1.67	148.4 140.5	1.15 1.19	1)Pre-emergence 2) Mid-vegetative	Hay 1 post app.	3.17, 0.62, 2.31, 0.499 (1.65)	0.912, 0.358, 0.878, 0.355 (0.626)	3.64, 0.916 (2.3)	48
					Hay 2 1st regrowth	0.647, 0.498 (0.573)	0.528, 0.384 (0.456)	1.18, 0.882 (1.0)	90
					Hay 3 2nd regrowth	0.269, 0.385 (0.327)	0.327, 0.362 (0.345)	0.596, 0.747 (0.67)	125
13CA California RR841	1.68	141.9	1.18	Mid-vegetative	Forage 1	0.366, 0.744, 0.361, 0.907 (0.595)	0.218, 0.278, 0.235, 0.372 (0.276)	0.584, 1.022, 0.596, 1.279 (0.87)	39
				Mid-vegetative	Forage 2 (1st regrowth)	0.302, 0.303 (0.303)	0.240, 0.224 (0.232)	0.542, 0.527 (0.54)	82
	1.68	141.9	1.18	Mid-vegetative	Hay 1 Post app	1.88, 4.49, 1.65, 4.23 (3.06)	0.644, 1.21, 0.693, 1.38 (0.982)	2.52, 5.70, 2.34, 5.61 (4.0)	48
				Mid-vegetative	Hay 2 (1st regrowth)	0.635, 0.232, 0.754, 0.277 (0.540)	0.828, 0.295, 0.670, 0.245 (0.510)	1.46, 0.527, 1.42, 0.522 (0.99)	90
13CA California	1.68 1.68	141.9 148.8	1.18 1.13	1) Mid-vegetative	Forage 3 Post app	0.506, 1.07,	0.166, 0.256,	0.672, 1.33,	25

Site code State, variety, Treatment	Application rate			Growth stage	Commodity analysed	Residues (mg/kg)			DAT (days)
	kg ai/ ha	water (L/ha)	kg ai/ hL			EMA	HEMA	Total	
RR841				2) Mid-vegetative	(2 apps)	0.401, 0.874 (0.713)	0.169, 0.229 (0.205)	0.570, 1.10 (0.92)	
				1) Mid-vegetative 2) Mid-vegetative	Forage 4 1st regrowth	0.048, 0.042 (0.045)	0.055, 0.033 (0.044)	0.103, 0.074 (0.09)	67
	1.68 1.68	141.9 148.8	1.18 1.13	1) Mid-vegetative 2) Mid-vegetative	Hay 3 Post app (2 apps)	3.46, 4.29 (3.88)	1.03, 1.23 (1.13)	4.49, 5.52 (5.0)	34
14WA Washington DKA44-16RR	1.68 1.68	140.8 140.5	1.19 1.20	1) Pre-emerg. 2) BBCH 14	Forage 1 post app. (2 apps.)	0.374, 0.391 (0.383)	0.396, 0.451 (0.424)	0.770, 0.842 (0.81)	70
					Forage 2 1st regrowth	0.161, 0.198 (0.180)	0.163, 0.166 (0.165)	0.324, 0.364 (0.34)	112
					Forage 3 2nd regrowth	0.159, 0.155 (0.157)	0.108, 0.101 (0.105)	0.267, 0.256 (0.26)	145
					Forage 4 3rd regrowth	0.258, 0.244 (0.251)	0.199, 0.212 (0.206)	0.457, 0.456 (0.46)	201
	1.68 1.68	140.8 140.5	1.19 1.20	1)Pre-emergence 2) BBCH 14	Hay 1 post app.	1.67, 1.32 (1.50)	2.09, 1.52 (1.81)	3.76, 2.84 (3.3)	79
					Hay 2 1st regrowth	0.421, 0.333 (0.377)	0.603, 0.428 (0.516)	1.02, 0.761 (0.89)	117
					Hay 3 2nd regrowth	0.608, 0.513 (0.561)	0.779, 0.595 (0.687)	1.39, 1.11 (1.2)	153
					Hay 4 3rd regrowth	0.544, 0.510 (0.527)	0.619, 0.561 (0.590)	1.16, 1.07 (1.1)	210
14WA Washington DKA44-16RR	1.68	140.4	1.20	BBCH 14	Forage 1	0.258, 0.322 (0.290)	0.336, 0.359 (0.348)	0.594, 0.681 (0.64)	70
	1.68	140.4	1.20	BBCH 14	Hay 1 Post app	0.886, 0.855 (0.871)	1.11, 1.24 (1.18)	2.00, 2.10 (2.1)	79
	1.69	140.9	1.20	BBCH 15	Forage 2 Post app	0.513, 0.544 (0.529)	0.214, 0.259 (0.237)	0.727, 0.803 (0.77)	29
					Forage 3 1st regrowth	0.324, 0.330 (0.327)	0.186, 0.209 (0.198)	0.510, 0.539 (0.53)	62
					Forage 4 2nd regrowth	0.323, 0.378 (0.351)	0.339, 0.325 (0.332)	0.662, 0.703 (0.68)	118
					Hay 2 Post app	4.16, 2.78 (3.47)	1.26, 0.977 (1.12)	5.42, 3.76 (4.6)	34
					Hay 3 1st regrowth	0.880, 1.11 (0.995)	0.953, 1.45 (1.20)	1.83, 2.56 (2.2)	70
					Hay 4 2nd regrowth	1.01, 0.881 (0.946)	1.12, 1.11 (1.12)	2.13, 1.99 (2.1)	127

APPRAISAL

Acetochlor is a selective herbicide belonging to the chloroacetanilide class that was first and last evaluated for residues and toxicological aspects by the 2015 JMPR, when an ADI of 0–0.01 mg/kg bw and an ARfD of 1 mg/kg bw were established. The residue definition for compliance with the MRL and for dietary risk assessment (for animal and plant commodities) is the sum of compounds hydrolysable with base to 2-ethyl-6-methylaniline (EMA) and 2-(1-hydroxyethyl)-6-methylaniline (HEMA), expressed in terms of acetochlor. The residue is not fat soluble.

Acetochlor was scheduled at the Fiftieth Session of the CCPR for the evaluation of additional uses by the 2019 Extra JMPR. The Meeting received new information on metabolism in soya bean, analytical method data, and residue trials on soya bean and alfalfa (forage and hay).

Metabolism in plants

The present Meeting received information on the identification of metabolites in soya bean seed extracts from a metabolism study on soya beans after pre-plant or post-emergence applications that had been previously evaluated by the Meeting. The identified acetochlor metabolites were its *tert*-sulfinylacetic acid, *tert*-sulfinyllactic acid and 1-hydroxyethyl *sec*-oxanilic acid, which were also previously identified in soya bean feed commodities. These metabolites are covered by the current definition of the residue based on the common moieties EMA and HEMA.

Methods of analysis

The methods developed to quantify residues of acetochlor in plant and animal matrices involve hydrolytic conversion of metabolites to the EMA or HEMA chemophores, which are quantified and expressed as total acetochlor residues. They involve extraction with methanol/water mixture, followed by hydrolysis of residues with aqueous hydroxide solution. The main differences between the previous and the new methods are the clean-up conditions, sample sizes and instrumentation for quantification (LC-MS/MS in more recent versions). LOQs are typically 0.025 mg/kg each for EMA and HEMA. Representative compounds that generate EMA (*tert*-sulfonic acid) and HEMA (1-hydroxyethyl-tert-oxanilic acid) on base hydrolysis are used as reference materials for fortification and method validation. The methods are suitable for analysis of acetochlor and related metabolites in plant and animal matrices.

Stability of residues in stored analytical samples

The stability of incurred residues analysed as EMA and HEMA in the soya bean samples after more than eight years of frozen storage was estimated based on the analysis conducted when the study was performed (2007/8) and when the samples were again analysed in 2016. The results were submitted to the present Meeting. On average (n=8), the percent remaining was 122% for EMA and 149% for HEMA, probably due to modifications in the LC-MS/MS analytical method used in the original study. The Meeting concluded that acetochlor residues in soya bean seeds are stable for at least 8 years.

In 2015, JMPR concluded that acetochlor residues were also stable in several plant matrices including alfalfa forage and clover hay for at least 330 days under freezer storage conditions (-20 ºC).

Results of supervised residue trials on crops

Soya bean, dry

The critical GAP for acetochlor on soya bean in the USA is pre-plant/pre-emergence, and post-emergence (before the R2 growth stage, full flowering) at up to 1.7 kg ai/ha and not exceeding a maximum rate per year of 3.4 kg ai/ha. Supervised trials were conducted in the USA in 2007. In 13 independent trials conducted according to GAP, total residues in soya bean seeds were < 0.05, 0.05, 0.10, 0.11, 0.12, 0.14, 0.15, 0.19, 0.20, 0.22, 0.23, 0.25 and 0.91 mg/kg.

The Meeting estimated a maximum residue level of 1.5 mg/kg and a STMR of 0.15 mg/kg for soya bean, dry.

Alfalfa

The critical GAP for acetochlor in alfalfa in the USA is pre-plant/at-planting/pre-emergence and post-emergence (up to or at the 4th-trifoliate stage - new stands - or following spring green-up - fall-planted or established stands - or between cuttings), with a maximum rate of 3.4 kg ai/ha per year and a PHI of at least 20 days. Supervised trials were conducted in the USA in 2013 and 2014. In eight trials conducted according to GAP, total residues in alfalfa forage were 0.82, 0.92, 1.1, 1.9, 2.5, 2.9, 4.0 and 5.8 mg/kg, and in alfalfa hay were 2.0, 2.8 (2), 4.4, 4.7, 5.0, 6.9 and 13.0 mg/kg (fresh weight basis).

The Meeting estimated a maximum residue level of 30 mg/kg (dry basis) for alfalfa hay.

The Meeting withdrew the previous recommendation for legume animal feed of 3 mg/kg and recommended a maximum residue level of 3 mg/kg for legume animal feed, except alfalfa hay.

The Meeting also estimated a median residue of 4.55 mg/kg and a highest residue of 13 mg/kg for alfalfa hay (fresh weight basis), a median residue of 2.2 mg/kg and a highest residue of 5.8 mg/kg for alfalfa forage.

Fate of residues during processing

The processing factors for soya bean oil, meal and hulls estimated by the 2015 JMPR are 0.11, 1.2 and 0.72, respectively. Therefore, considering a STMR of 0.15 mg/kg for soya bean seeds, the Meeting estimated a STMR-P of 0.016 mg//kg for soya bean oil, a median residue of 0.18 mg/kg for soya bean meal and of 0.108 mg/kg for soya bean hulls.

Animal feedstuffs

Estimation of livestock dietary burdens

Dietary burden calculations for beef cattle, dairy cattle and poultry are provided below. The dietary burdens were estimated using the OECD diets listed in Appendix IX of the 3rd edition (2016) of the FAO Manual. Considering the items estimated by the 2015 and present JMPR, livestock dietary burdens were estimated for cattle and poultry.

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

	US-Canada		EU		Australia		Japan	
Commodity	**Max**	Mean	**Max**	Mean	**Max**	Mean	**Max**	Mean
Beef cattle	2.33	0.87	12.27	4.47	16.57[a]	6.29[c]	1.63	0.68
Dairy cattle	4.28	1.43	7.77	2.65	10.75[b]	3.87[d]	3.82	1.44
Poultry-broiler	0.11	0.11	0.16	0.16	0.10	0.10	0.08	0.08
Poultry-layer	0.11	0.11	0.61[e]	0.18[f]	0.10	0.10	0.07	0.07

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

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

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

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

[e] Highest maximum poultry dietary burden suitable for maximum residue level estimated for poultry tissues and eggs.

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

Animal commodity maximum residue levels

Based on the estimated dietary burden and the results of farm animal feeding studies evaluated by the 2015 JMPR, the calculations used to estimate highest total residues for use in estimating maximum residue levels, STMR and HR values are shown below.

	Feed level	Residues	Feed level	Residues (mg/kg) in			
	(ppm) for milk residues	(mg/kg) in milk	(ppm) for tissue residues	Muscle	Liver	Kidney	Fat
MRL beef or dairy cattle							
Feeding study [a]	-		15	-	< 0.02	0.04	-
	50	< 0.02	50	< 0.02	0.02	0.09	<0.02
Dietary burden and high residue	10.75	< 0.0043	16.57	<0.0007	0.02	0.0418	<0.02
STMR beef or dairy cattle							
Feeding study[b]			5	-	-	<0.02	-
			15	-	<0.02	0.03	-
	50	< 0.02	50	<0.02	0.02	0.07	<0.02
Dietary burden and median residue estimate	3.87	< 0.0015	6.29	<0.0025	0.02	0.0213	<0.0025

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

[b] Mean residues for tissues and mean residues for milk

The Meeting estimated a maximum residue level of 0.05 mg/kg, a STMR of 0.0213 mg/kg and a HR of 0.0418 mg/kg for edible offal (mammalian) to replace the previous recommendation.

The Meeting confirmed its previous recommendations for meat (mammalian except marine mammals), mammalian fat (except milk fat) and milks.

No residues were observed in eggs and poultry tissues on dosing laying hens at up to 50 ppm in the diet for 28 days. Considering the poultry dietary burden of 0.61 ppm (highest maximum) and 0.18 ppm (highest mean), the Meeting confirmed its previous recommendation 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 maximum residue limits and for IEDI and IESTI assessment.

Residue definition for compliance with the MRL and for dietary risk assessment for animal and plant commodities: *Sum of compounds hydrolysable with base to 2-ethyl-6-methylaniline (EMA) and 2-(1-hydroxyethyl)-6-methylaniline (HEMA), expressed in terms of acetochlor*.

The residue is not fat soluble

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
AL 1020	Alfalfa hay	30 (dw)	-	Median: 4.55 (as)	Highest: 13 (as)
AL 0157	Legume animal feed	W	3 (dw)		
AL 0157	Legume animal feed, except alfalfa hay	3 (dw)	-		
VD 0541	Soya bean (dry)	1.5	-	0.15	
MO 0105	Edible offal (mammalian)	0.05	0.02*	0.0213	0.0418
OR 0541	Soya bean oil, Refined			0.016	

Additional recommendations for livestock dietary burden

	Median residue (mg/kg, fresh basis)	Highest residue (mg/kg, fresh, basis)
Alfalfa forage	2.2	5.8
Soya bean hulls	0.108	
Soya bean meal	0.18	

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for acetochlor is 0–0.01 mg/kg bw. The International Estimated Daily Intakes (IEDIs) for acetochlor 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 2019 Extra JMPR Report.

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

Acute dietary exposure

The ARfD for acetochlor is 1 mg/kg bw. The International Estimate of Short Term Intakes (IESTIs) for acetochlor were calculated for the food commodities for which HRs/HR-Ps or STMRs/STMR-Ps were estimated by the present Meeting. The results are shown in Annex 4 of the 2019 Extra JMPR Report.

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

Threshold of toxicological concern (TTC) approach for metabolites

Acetochlor *tert*-sulfinyllactic acid and acetochlor 1-hydroxyethyl *sec*-oxanilic acid are unlikely to be genotoxic, and could be assessed using the TTC Cramer Class III of 1.5 µg/kg bw per day.

The metabolites acetochlor tert-sulfinyllactic acid and acetochlor 1-hydroxyethyl sec-oxanilic acid were identified in metabolism studies, found in maize grain, soya bean seed and poultry commodities (<10% TRR). They belong to the group of metabolites that are hydrolysed in the analytical methods for plant and animal commodities to form EMA and HEMA.

The maximum IEDI calculated for acetochlor (based on total EMA and HEMA) from commodities considered by the JMPR (Annex 3) was 0.385 µg/kg bw. The Meeting concluded that dietary exposure to residues of acetochlor tert-sulfinyllactic acid and acetochlor 1-hydroxyethyl sec-oxanilic acid from the uses considered by the JMPR is unlikely to present a public health concern.

REFERENCES

Study No.	Author(s)	Year	Study Title
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N.A.	Huang, W.	2016	Determination of Acetochlor Residues in Raw Agricultural Commodities and Processed Fractions Using LC-MS/MS. Analytical Method ME-1738-03. Monsanto Company, St. Louis, MO. Unpublished.
MSL0027760	Kurtzweil M.L., Mierkowski M.J., Adio, A.M.	2016	Characterization and Identification of Metabolites in Seed from the Acetochlor Soybean Metabolism Study. Monsanto Company, St. Louis, MO. Unpublished.
MSL0029938	Maher, D.L.	2018	Amended from MSL0027634, Determination of the Residues in Treatment 3 Soybean Seed Samples from the 2007 Acetochlor Soybean Residue Study. Monsanto Company, St. Louis, MO. Unpublished.
84383	Vogl, E.	2017	Method Validation of Acetochlor Metabolites in Canola Seed, Canola Meal, Canola Oil, and Soybean Seed. EAG, Inc. Columbia, MO.. Unpublished.
MSL0029922	Bending, P.; Przybylek, A.	2018	Independent Laboratory Validation of Residue Enforcement Method ME-2024, Method for Determination of Acetochlor Residues in Crop Matrices Using LC-MS/MS, in Soybean Seed. EAG Laboratories GmbH, Ulm, Germany. Unpublished.
MSL0021112	Woodbury, S.; Quistad, G.; Baker, F.	2009	Metabolism of ^{14}C-Acetochlor in Soybean after Pre-plant or Post-emergence Applications. Monsanto Company, St. Louis, MO. Unpublished.
MSL0020719	Hay, J.D.; Moran, S.J.; Foster, J.E.	2008	Determination of the Residues in Soybean Raw Agricultural Commodities after Application of Acetochlor Herbicide Formulations. Monsanto Company, St. Louis, M. Unpublished.

AZOXYSTROBIN (229)

First draft prepared by Mr P Rembischevski, Brazilian Health Regulatory Agency, Brasilia, Brazil

EXPLANATION

Azoxystrobin is a broad spectrum fungicide with activity against several diseases on many edible crops and ornamental plants. It was first evaluated for toxicology and residues by JMPR in 2008. An ADI of 0–0.2 mg/kg bw was established and an ARfD was unnecessary. The residue definition for plant and animal commodities, for both compliance with the MRL and dietary risk assessment is azoxystrobin. The residue is fat soluble.

Azoxystrobin was scheduled at the Fiftieth session of the CCPR for the evaluation of additional uses by the 2019 Extra JMPR. The Meeting received new GAP information on guava in Egypt and coffee in Brazil, analytical methods, supervised residue trials for guava and coffee, and processing studies on coffee.

RESIDUE ANALYSIS

Analytical methods

Residue analytical method POPIT MET.068 was used for the analysis of azoxystrobin residues in the supervised residue trials (Oliveira, 2011a; Oliveira, 2011b) and processing studies for coffee (Casallanovo, 2012). Azoxystrobin was extracted from the treated samples with a solution of acetonitrile:water (9:1) and quantified by LC-MS/MS after re-dissolving the extract in methanol:water (1:1). The linearity of the detector was shown in the range of 0.0002-0.0064 µg/mL, with coefficients of determination (R^2) > 0.99. No interfering peaks around the retention time of azoxystrobin were found in any of the control samples at levels above 30% of the limit of quantification. Two transitions were monitored m/z 404→372 (quantification) and m/z 404→344 (confirmation). Mean recoveries were within the acceptable range of 70–120% with relative standard deviations of < 20%. The results are given in Table 1. The LOQ for coffee beans and processed commodities was 0.01 mg/kg.

Table 1 Recovery of azoxystrobin residues in coffee beans and processed commodities

Matrix	**Fortification level** [mg/kg]	**Range of recoveries** [%]	**Mean recovery** [%]	**RSD** [%]	Reference
Coffee beans (RAC)	0.01 (n=7)	90-105	96	7	M11074 / M11085
	0.1 (n=5)	95-108	101	5	
Coffee beans (RAC)	0.01 (n=3)	98	98	0	M11173
	0.1 (n=3)	104-109	106	2	
Roasted beans	0.01 (n=7)	71-81	75	4	
	0.1 (n=5)	82-87	86	3	
Slurry	0.01 (n=7)	75-84	79	4	
	0.1 (n=5)	91-94	93	1	
Extract	0.01 (n=7)	93-102	96	3	
	0.1 (n=5)	96-101	99	2	
Concentrated coffee	0.01 (n=7)	81-87	83	3	
	0.1 (n=5)	87-91	90	2	
Instant coffee	0.01 (n=7)	83-92	86	3	
	0.1 (n=5)	96-99	97	2	

USE PATTERN

The GAP for the use of azoxystrobin on guava and coffee as a foliar spray are summarized in Table 2.

Table 2 Registered uses of azoxystrobin using foliar application

			Application				
Crop	Country	Formulation	Rate	Water, L/ha	No.	Interval, days	PHI, days
Guava	Egypt	SC	0.01 kg ai/hL	952-1428	3	7-14	7
Coffee	Brazil	WG	0.12 kg ai/ha	400	3	60	21

RESULTS OF SUPERVISED RESIDUE TRIALS ON CROPS

Guava

Six supervised trials with azoxystrobin on guava were conducted in Egypt during 2015/2016 and submitted to the 2017 JMPR but did not match to GAP, since no official label was available from Egypt at that time. The trials were then submitted again to the current Meeting. The results are shown in Table 3.

Table 3 Residues of azoxystrobin on guava (fruit) in Egypt in 2015/16 through foliar application using SC formulation (Report 11605.15-EGR01)

Region	Variety	Application rate (kg ai/hL)	Spray volume (L/ha)	DAT (days)	Residue (mg/kg)	Trial
Moshtohor	Etmany	0.01 0.01 0.01	1010 1036 999	7	0.045, 0.056 (<u>0.05</u>)	11605.15-EG01
Qalama-Qudiouhia	Ghoneimy	0.01 0.01 0.01	1002 996 1032	0 3 7 10 14	0.088, 0.124 (0.10) 0.039, 0.040 (0.04) 0.013, 0.014 (0.01) 0.019, 0.037 (<u>0.03</u>) 0.018, 0.027 (0.02)	11605.15-EG02
Salheya-Sharqueya	Etmany	0.01 0.01 0.01	986 1022 1005	7	0.056, 0.067 (<u>0.06</u>)	11605.15-EG03
Arab-Al Khanka	Balady	0.01 0.01 0.01	985 1057 1060	6	0.017, 0.041 (<u>0.03</u>)	11605.15-EG04
Qualiobia	Etmany	0.01 0.01 0.01	989 1022 1047	8	0.085, 0.107 (<u>0.1</u>)	11605.15-EG05
Al Manzala	Banaty	0.01 0.01 0.01	1033 1013 983	8	0.043, 0.158 (<u>0.1</u>)	11605.15-EG06

Coffee

Twelve supervised residue trials (in two pairs of six independent trials) with azoxystrobin on coffee, from Brazil in 2010/2011, were previously evaluated by the 2011 JMPR, along with ten trials (seven independent) conducted in earlier years. Two additional trials (M11173-JJB and M11173-RWC) were submitted to the present Meeting. Samples of coffee beans were frozen (-20 °C) and maintained in frozen storage for up to 9.3 months prior to extraction. The results are summarized in Table 4.

Table 4 Residues of azoxystrobin on coffee bean in Brazil through foliar application, using WG, EC or SC formulations.

Region	Variety	Application Rate (kg ai/ha)	Growth Stage	DAT (days)	Residue (mg/kg)	Report, trial, year
Taiuva, SP	Catuai Amarelo	0.05 (WG) 0.05 (WG) 0.10 (EC) 0.10 (EC) 0.10 (EC)	BBCH 67 BBCH 69 BBCH 73 BBCH 75 BBCH 77-81	21 28 35	<0.01 <0.01 <0.01	Report: M11074 Trial: AMA Year: 2010/2011
Taiuva, SP	Catuai Amarelo	0.05 (WG) 0.05 (WG) 0.12 (WG) 0.12 (WG) 0.12 (WG)	BBCH 67 BBCH 69 BBCH 73 BBCH 75 BBCH 81	21 28 35	<u><0.01</u> <0.01 <0.01	Report: M11085 Trial: AMA Year: 2010/2011
Indianopólis, MG	Mundo Novo	0.05 (WG) 0.05 (WG) 0.10 (EC) 0.10 (EC) 0.10 (EC)	BBCH 60 BBCH 72 BBCH 72 BBCH 76 BBCH 83	21 28 35	0.02 <0.01 0.02	Report: M11074 Trial: JJB1 Year: 2010/2011
Indianopólis, MG	Mundo Novo	0.05 (WG) 0.05 (WG) 0.12 (WG) 0.12 (WG) 0.12 (WG)	BBCH 60 BBCH 72 BBCH 72 BBCH 76 BBCH 83	21 28 35	<u>0.02</u> <0.01 <0.01	Report: M11085 Trial: JJB1 Year: 2010/2011
Araguari, MG	Mundo Novo	0.05 (WG) 0.05 (WG) 0.10 (EC) 0.10 (EC) 0.10 (EC)	BBCH 60 BBCH 72 BBCH 72 BBCH 76 BBCH 83	21 28 35	<0.01 <0.01 <0.01	Report: M11074 Trial: JJB2 Year: 2010/2011
Araguari, MG	Mundo Novo	0.05 (WG) 0.05 (WG) 0.12 (WG) 0.12 (WG) 0.12 (WG)	BBCH 60 BBCH 72 BBCH 72 BBCH 76 BBCH 83	21 28 35	<u><0.01</u> <0.01 <0.01	Report: M11085 Trial: JJB2 Year: 2010/2011
Sâo Gonçalo do Sapucai, MG	Mundo Novo	0.05 (WG) 0.05 (WG) 0.10 (EC) 0.10 (EC) 0.10 (EC)	BBCH 53 BBCH 67-71 BBCH 71-73 BBCH 75-77 BBCH 81	21 28 35	<0.01 <0.01 <0.01	Report: M11074 Trial: RWC1 Year: 2010/2011
Sâo Gonçalo do Sapucai, MG	Mundo Novo	0.05 (WG) 0.05 (WG) 0.12 (WG) 0.12 (WG) 0.12 (WG)	BBCH 53 BBCH 69-71 BBCH 71-73 BBCH 75-77 BBCH 81	21 28 35	<u><0.01</u> <0.01 <0.01	Report: M11085 Trial: RWC1 Year: 2010/2011
Campinas, SP	Catuai Vermelho IAC 144	0.05 (WG) 0.05 (WG) 0.10 (EC) 0.10 (EC) 0.10 (EC)	BBCH 67 BBCH 69-71 BBCH 73 BBCH 75-77 BBCH 77-79	21 28 35	<0.01 <0.01 <0.01	Report: M11074 Trial: RWC2 Year: 2010/2011
Campinas, SP	Catuai Vermelho IAC 144	0.05 (WG) 0.05 (WG) 0.12 (WG) 0.12 (WG) 0.12 (WG)	BBCH 67 BBCH 69-71 BBCH 73 BBCH 75-77 BBCH 77-79	21 28 35	<u><0.01</u> <0.01 <0.01	Report: M11085 Trial: RWC2 Year: 2010/2011
Linhares, ES	Conilon	0.05 (WG) 0.05 (WG) 0.10 (EC) 0.10 (EC) 0.10 (EC)	BBCH 67 BBCH 69 BBCH 70 BBCH 75 BBCH 78	21 28 35	<u>0.02</u> <0.01 0.02	Report: M11074 Trial: RWC3 Year: 2010/2011

Region	Variety	Application Rate (kg ai/ha)	Growth Stage	DAT (days)	Residue (mg/kg)	Report, trial, year
Linhares, ES	Conilon	0.05 (WG) 0.05 (WG) 0.12 (WG) 0.12 (WG) 0.12 (WG)	BBCH 67 BBCH 69 BBCH 70 BBCH 75 BBCH 78	21 28 35	<0.01 <0.01 <0.01	Report: M11085 Trial: RWC3 Year: 2010/2011
Monte Carmelo, MG	Catuai 144	0.5 (EC) 0.5 (EC) 0.5 (EC)	BBCH 79 BBCH 84 BBCH 88	21	<0.01	Report: M11173 Trial: JJB Year: 2011
Jaboti, PR	Mundo Novo	0.5 (EC) 0.5 (EC) 0.5 (EC)	BBCH 79 BBCH 79 BBCH 81	21	<0.01	Report: M11173 Trial: RWC Year: 2011
Cravinhos, SP	Catuai	0.10 (SC) 0.10 (SC) 0.10 (SC)	-	0 7 14 21 30	0.02 0.01 < 0.01 < 0.01 < 0.01	Report: M02037 Trial: BAB Year: 2002/03
Cravinhos, SP	Catuai	0.20 (SC) 0.20 (SC) 0.20 (SC)	-	0 7 14 21 30	0.02 0.02 0.02 < 0.01 0.01	Report: M02037 Trial: BAB Year: 2002/03
Patrocinio, MG	Catuai	0.10 (SC) 0.10 (SC) 0.10 (SC)	-	0 7 14 21 30	0.02 0.03 < 0.01 < 0.01 0.01	Report: M02037 Trial: JJB1 Year: 2002/03
Patrocinio, MG	Catuai	0.20 (SC) 0.20 (SC) 0.20 (SC)	-	0 7 14 21 30	0.03 0.04 0.01 < 0.01 0.01	Report: M02037 Trial: JJB1 Year: 2002/03
Araxa, MG	Catuai	0.10 (SC) 0.10 (SC) 0.10 (SC)	-	0 7 14 21 30	0.02 0.02 < 0.01 < 0.01 < 0.01	Report: M02037 Trial: JJB2 Year: 2002/03
Araxa, MG	Catuai	0.20 (SC) 0.20 (SC) 0.20 (SC)	-	0 7 14 21 30	0.03 0.04 0.01 0.01 0.01	Report: M02037 Trial: JJB2 Year: 2002/03
Monte Carmelo, MG	Mundo Novo	0.15 (SC) 0.15 (SC) 0.15 (SC)	-	14 21 30	< 0.01 < 0.01 < 0.01	Report: M06024 Trial: JJB1 Year: 2006/07
Indianopolis, MG	Mundo Novo	0.15 (SC) 0.15 (SC) 0.15 (SC)	-	14 21 30	< 0.01 < 0.01 < 0.01	Report: M06024 Trial: JJB2 Year: 2006/07
Araxa, MG	Catuai	0.15 (SC) 0.15 (SC) 0.15 (SC)	-	14 21 30	< 0.01 < 0.01 < 0.01	Report: M06024 Trial: JJB3 Year: 2006/07
Santa Amelia, PR	Iapar 59	0.15 (SC) 0.15 (SC) 0.15 (SC)	-	14 21 30	< 0.01 < 0.01 < 0.01	Report: M06024 Trial: LZF Year: 2007

FATE OF RESIDUES DURING PROCESSING

Samples from two trials conducted in coffee at exaggerated rates (M11173-JJB and M11173-RWC) were submitted to processing (Casallanovo, 2012). Coffee beans were roasted at 196 to 211.5 °C for 8 to 9 hours to produce roasted coffee. A roasted bean sample was extracted with water and lyophilized to produce instant coffee. The resultant processing samples were produced and analysed for residues of azoxystrobin: roasted beans, concentrated coffee and instant coffee. The results in unprocessed coffee beans and all processed commodities were <0.01 mg/kg from both treated plots in both trials, so processing factors from these data could not be calculated.

APPRAISAL

Azoxystrobin was first evaluated for toxicology and residues by the JMPR in 2008. It was evaluated for residues by the JMPR in 2011, 2012, 2013 and 2017. An ADI of 0–0.2 mg/kg bw was established and an ARfD was unnecessary. The residue definition for plant and animal commodities for both compliance with MRLs and dietary risk assessment is azoxystrobin. The residue is fat soluble.

Azoxystrobin was scheduled at the Fiftieth Session of the CCPR for the evaluation of additional uses by the 2019 Extra JMPR. The Meeting received new GAP information on guava in Egypt and coffee in Brazil, analytical methods, supervised residue trials for guava and coffee, and processing studies on coffee.

Methods of analysis

The Meeting received validation data on a new analytical method on coffee bean (green). Azoxystrobin residues were extracted with a solution of acetonitrile:water (9:1) and residues quantified by LC-MS/MS, with an LOQ of 0.01 mg/kg.

Stability of residues under storage

Previous studies submitted to the Meeting showed that residues of azoxystrobin stored at ≤ 20 ºC are stable for at least 24 months in a variety of crops, including grape, peanut, tomato, apple, banana, cucumber, peach, soya bean, corn, carrot, lettuce, wheat and orange.

Results of supervised residue trials on crops

Guava

The critical GAP for guava in Egypt is 3 × 0.01 kg ai/hL, with a 7±14 day application interval and a PHI of 7 days. Residues in the six independent trials submitted to the 2017 JMPR according to this GAP were 0.03 (2), 0.05, 0.06 and 0.10 (2) mg/kg.

The Meeting estimated a maximum residue level of 0.2 mg/kg and a STMR of 0.055 mg/kg for azoxystrobin in guava.

Coffee

The critical GAP for coffee in Brazil is 3 × 0.12 kg ai/ha, with a 60 day application interval and a 21-day PHI. Residues from 13 independent trials conducted approximating this cGAP, and evaluated by the 2011 JMPR, were < 0.01 (10), 0.01 and 0.02 (2) mg/kg. Two new trials conducted at four times the GAP rate gave residues < 0.01 mg/kg.

The Meeting confirmed the previous recommendations for azoxystrobin in coffee bean.

Fate of residues during processing

Two new processing studies on coffee conducted in Brazil at four times the GAP rate were submitted to the Meeting. Residues in unprocessed coffee beans and all processed commodities (roasted beans, concentrated coffee and instant coffee) were < 0.01 mg/kg. Thus, the processing factors recommended by the 2013 JMPR for coffee remained unchanged.

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.

Residue definition for compliance with the MRL and dietary risk assessment for plant and animal commodities: *azoxystrobin.*

The residue is fat soluble.

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR or STMR-P (mg/kg)	HR or HR-P mg/kg
		New	Previous		
FT 0336	Guava	0.2	-	0.055	-

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for azoxystrobin is 0–0.2 mg/kg bw. The International Estimated Daily Intakes (IEDIs) for azoxystrobin 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 2019 Extra JMPR Report.

The IEDIs accounted for 2 to 20% of the maximum ADI. The Meeting concluded that the long-term dietary exposure to residues of azoxystrobin 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 azoxystrobin was unnecessary. The Meeting therefore concluded that the acute dietary exposure to residues of azoxystrobin is unlikely to present a public health concern.

REFERENCES

Study No.	Author(s)	Year	Study Title
11605.15-EGR01	DeFrancesco, J.	2016	Difenoconazole + Azoxystrobin: Magnitude of the Residue on Guava. GLP, Unpublished. Syngenta File No A13703G_11493
M11074	de Oliviera, F.F.	2011	A17961 - Magnitude of residues of SYN545192 (Solatenol) and metabolites, azoxystrobin and R230310 in coffee beans - Brazil, 2010-11. Syngenta File No. A17961A_10051
M11085	de Oliviera, F.F.	2011	A18126 - Magnitude of residues of SYN545192 and metabolites, azoxystrobin and R230310 in coffee beans - Brazil, 2010-11. Syngenta File No. A18126B_10039
M11173	Casallanovo, F.	2012	A17961 - Magnitude of residues of SYN545192 and metabolites, azoxystrobin and R230310 in coffee beans and its processed derivatives - Brazil, 2010-11. Syngenta Proteção de Cultivos Ltda, São Paulo/SP. Brazil. Study dates: April 2011 - May 2012. Syngenta File No. A17961A_50005
M02037	Francisco, E.	2003	Priori Xtra - Magnitude de residuos de azoxystrobin, R230310 e cyproconazole em café - Brasil, 2003.. Resolution-RDC n. 44/2000 (Brasil). Syngenta. Unpublished.
M06024	Roncato, C.	2008	Priori Xtra - Magnitude de residuos de azoxystrobin, R230310 e cyproconazole em café - Brasil, 2006-07. Resolution-RDC n. 216/2006 (Brasil). Syngenta. Unpublished.

BOSCALID (221)

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

EXPLANATION

Boscalid is a systemic fungicide first evaluated by JMPR in 2006 for residues and toxicology as a new active substance. An ADI of 0–0.04 mg/kg bw was established for boscalid, while no ARfD was considered necessary.

The 2006 JMPR recommended the following residue definition for boscalid:

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

Definition of the residue for dietary risk assessment in animal commodities: *sum of boscalid, 2-chloro-N-(4'-chloro-5-hydroxybiphenyl-2-yl)nicotinamide (M510F01) including its conjugate, expressed as boscalid.*

The residue is fat-soluble.

In 2008 and 2010 additional uses (and in 2009 residues in follow crops) were reviewed for residues by the Meeting. Boscalid was scheduled at the Fiftieth Session of the CCPR for the evaluation of additional uses by the Extra 2019 JMPR Meeting. The current Meeting received new information on use patterns for boscalid in pome fruit, stone fruit, berry fruit, tropical fruit and tea supported by additional plant and animal metabolism studies, analytical methods and recovery data, supervised field trials and studies simulating typical processing conditions.

METABOLISM AND ENVIRONMENTAL FATE

Metabolism studies were conducted using [pyridin-3-^{14}C]-boscalid (pyridin-label) and [diphenyl-^{14}C]-boscalid (diphenyl-label). The position of the label for both substances is presented in the following figures:

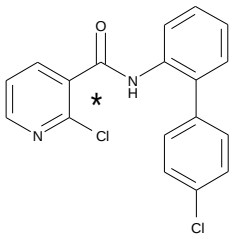


Figure 1 [pyridin-3-^{14}C]-boscalid

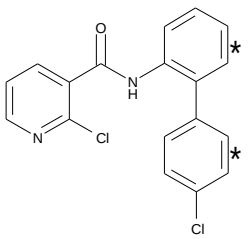
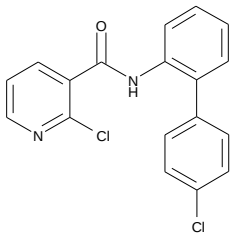
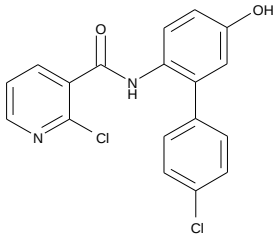
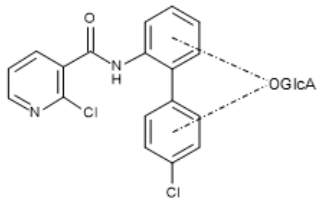


Figure 2 [diphenyl-^{14}C]-boscalid

Chemical names, structures and code names of metabolites and degradation products of boscalid discussed within this document are shown below. For a complete list of metabolites, please refer to the 2006 JMPR evaluation report.

Table 1 Metabolites of boscalid discussed within this document

Code Names	Structure	Where found
Boscalid BAS510F		Rat, plants, animals, rotational crops, soil
M510F01		Rat, animals
M510F65		Rat, animals

Plant metabolism

The Meeting received a new plant metabolism study with boscalid on green beans. In all samples, only unchanged boscalid was identified.

Green beans

The metabolic fate of ^{14}C-diphenyl-boscalid in beans was investigated by Schaffert D. (2017, BOSC19E_002). Beans were seeded in containers and treated by three foliar application at BBCH 61 (beginning of flowering), 11 days later and finally 13 days before harvest, each conducted at a rate of 0.52 kg ai/ha. Samples of plants and whole pods were collected 3 days before and 13 days after final treatment. Pods collected at harvest were also separated into hulls and seeds.

Total radioactive residues (TRR) were analysed following combustion by means of an oxidizer. For the quantitation of radioactive residues in liquid samples a liquid scintillation counter (LSC) was used. All samples were extracted three times with methanol and two times with water. After each extraction step, solid material was separated from the extract by centrifugation and filtration. The filtered supernatants (methanol extracts and water extracts) were pooled and aliquots thereof were radioassayed. The residue after solvent extraction was dried, homogenized, and aliquots thereof were radioassayed.

The combined extracts were diluted with methanol or directly analysed by HPLC-LSC. All samples were analysed within 6 months.

TRR levels found were highest in the plants, followed by pods/hulls and seeds (Table 2). The solvent extraction using methanol and water release more than 98% of the TRR for all matrices except seeds, for which only 70.2% TRR could be extracted.

Table 2 Total radioactivity in bean matrices following application of ^{14}C-diphenyl-boscalid (3 × 0.52 kg ai/ha)

Matrix	TRR measured by combustion in mg eq/kg	TRR calculated from extracts in mg eq/kg
Pods (DALA -3)	1.02	1.2
Plant (DALA -3)	28.8	29.2
Pods (DALA 13)	0.757	0.789
Hulls (DALA 13)	0.833	0.802
Seeds (DALA 13)	0.066	0.065
Plant (DALA 13)	49.4	52.1

The identification of the radioactivity revealed only unchanged parent boscalid in all plant matrices.

Table 3 Identification of radioactivity in bean matrices following application of ^{14}C-diphenyl-boscalid (3 × 0.52 kg ai/ha)

Compound	% TRR (mg eq/kg)					
	Pods (-3 DALA)	Plant (-3 DALA)	Pods (13 DALA)	Hulls (13 DALA)	Seeds (13 DALA)	Plant (13 DALA)
Methanol extract	98.9 (1.18)	99.0 (28.9)	98.0 (0.773)	97.8 (0.785)	65.1 (0.042)	98.7 (51.4)
Water extract	0.4 (0.005)	0.4 (0.114)	0.6 (0.005)	0.7 (0.005)	5.1 (0.003)	0.5 (0.275)
Total Extracted	99.3 (1.19)	99.3 (29.0)	98.6 (0.778)	98.5 (0.79)	70.2 (0.046)	99.2 (51.6)
Boscalid	99.3 (1.19)	102.3 (29.8)	96.5 (0.761)	98.3 (0.789)	17.3 (0.011)	101.4 (52.8)
Characterised	<0.1 (<0.001)	<0.1 (0.006)	2.1 (0.016)	0.1 (0.001)	52.8 (0.034)[a]	<0.1 (0.024)
Post-extraction solids	0.7 (0.009)	0.7 (0.191)	1.4 (0.011)	1.5 (0.012)	29.8 (0.019)	0.8 (0.4)
Total	100 (1.20)	103.0 (30.0)	100 (0.789)	100 (0.802)	99.9 (0.065)	102.2 (53.2)

[a] five peaks, two up to 0.011 mg eq/kg and 16.7% TRR, three up to 9.2% TRR and 0.006 mg eq/kg

Animal metabolism

The Meeting received a new metabolism study on laying hens using the ^{14}C-pyridin-labeled boscalid.

Laying hens

The metabolism of boscalid in laying hens was investigated by Thiaener J. (2017, BOSC19E_001). Ten laying hens received a dose of ^{14}C-pyridin-labelled boscalid equivalent to 12 ppm for 13 consecutive days via capsule administration. Animals were sacrificed approximately 6 hours after the final dosing. During the whole dosing period eggs and excreta were collected and analysed with pooled tissue samples for each group at the end of the study.

Total radioactive residues (TRR) were determined by combustion and direct liquid scintillation counting (LSC). Samples of tissues and eggs were each extracted with an appropriate solvent (acetonitrile or methanol). Aliquots of the residues after methanol extraction of liver and excreta were each extracted dichloromethane. The residues after this solvent extraction were extracted again with water. Aliquots of the residues after acetonitrile extraction of egg yolk and egg white were each extracted with water. Solubilization with enzymes (protease, pepsin and pancreatin) of the residue after solvent extraction was conducted for egg yolk, liver and muscle. Generally, identification of metabolites was based on analysis by HPLC MS/MS, on co chromatography as well as chromatographic comparison of retention times of reference substances. In addition, various HPLC peaks were characterized by their chromatographic properties. All samples were stored up to a maximum interval of 149 days between sampling and analysis.

In total, approximately 92.5% of the administered dose (AR) was recovered, primarily in the excreta (Table 4). In eggs, TRR levels plateaued after approximately 10 days both in egg white and egg yolk. The TRR levels found in eggs (white and yolk) and in tissues are presented in Table 5.

Table 4 Recovered radioactivity after oral administration of ^{14}C-pyridin-boscalid (12 ppm) for 13 consecutive days to laying hens

Matrix	% AR
Excreta	87.7
Cage wash	4.59
Egg yolk	0.086
Egg white	0.058
Liver	0.111
Fat	0.000
Muscle	0.026
Bile	0.005
Total	92.5

Table 5 Total radioactive residues in eggs and offal after oral administration of ^{14}C-pyridin-boscalid (12 ppm) for 13 consecutive days to laying hens

Matrix	TRR in mg eq/kg	
Eggs	Egg white	Egg yolk
Day 1	0.019	0.005
Day 2	0.020	0.009
Day 3	0.023	0.034
Day 4	0.027	0.062
Day 5	0.029	0.088
Day 6	0.031	0.105
Day 7	0.034	0.128
Day 8	0.034	0.142
Day 9	0.031	0.143
Day 10	0.028	0.137
Day 11	0.031	0.150
Day 12	0.032	0.146
Day 13	0.031	0.140
Liver	0.439	
Muscle	0.051	
Fat	0.095	

Subsequent solvent extraction released between 68.4% to 94.1% of the TRR, mostly in the acetonitrile or methanol extracts. With dichloromethane and water, an additional amount of up to 1.4% TRR and 9.9% TRR were extracted. Egg white and fat showed low unextracted residues of 8.0% TRR and 5.8% TRR, respectively. In egg yolk, liver and muscle unextracted TRR was higher (22.5–31.8% TRR) and these matrices were subsequently treated with enzymes to release additional radioactivity. Final unextracted residues were less than 10% for each matrix.

Table 6 Total radioactive residues in eggs and offal after oral administration of ^{14}C-pyridin-boscalid (12 ppm) for 13 consecutive days to laying hens

Extraction	% TRR (mg eq/kg)				
	Egg yolk	Egg white	Liver	Muscle	Fat
TRR	100 (0.123)	100 (0.03)	100 (0.439)	100 (0.051)	100 (0.095)
Solvent extraction					
Acetonitrile or methanol	62.5 (0.077)	85.4 (0.026)	65.1 (0.286)	77.5 (0.039)	94.1 (0.09)
Dichloromethane	NP	NP	1.4 (0.006)	NP	NP
Water	9.9 (0.012)	6.0 (0.002)	1.8 (0.008)	NP	NP
Subtotal solvent extraction	72.4 (0.089)	91.4 (0.028)	68.4 (0.30)	77.5 (0.039)	94.1 (0.09)
Post-extraction solids	28.3 (0.035)	8.0 (0.002)	31.8 (0.14)	22.5 (0.011)	5.8 (0.006)
Protease solubilizate	23.7 (0.029)	NP	21.6 (0.095)	35.1 (0.018)	NP

Extraction	% TRR (mg eq/kg)				
	Egg yolk	Egg white	Liver	Muscle	Fat
Pepsin solubilizate	NP	NP	2.0 (0.009)	NP	NP
Pancreatin solubilizate	NP	NP	1.7 (0.007)	NP	NP
Subtotal enzyme treatment	23.7 (0.029)	NP	25.3 (0.111)	35.1 (0.018)	NP
Unextracted	7.5 (0.009)	8.0 (0.002)	9.6 (0.042)	8.1 (0.004)	5.8 (0.006)

NP: not performed

In the following table the identification and characterisation of the radioactivity found is summarized.

Table 7 Composition of radioactivity in eggs and offal after oral administration of ^{14}C-pyridin-boscalid (12 ppm) for 13 consecutive days to laying hens

Compound	% TRR (mg eq/kg)				
	Egg yolk	Egg white	Liver	Muscle	Fat
TRR	100 (0.123)	100 (0.03)	100 (0.439)	100 (0.051)	100 (0.095)
Solvent extract					
Boscalid	34.0 (0.042)	34.3 (0.01)	1.8 (0.008)	29.4 (0.015)	84.9 (0.081)
M510F01	27.4 (0.034)	28.1 (0.008)	35.2 (0.155)	10.8 (0.005)	5.3 (0.005)
M510F65	8.4 (0.01)	16.4 (0.005)	18.2 (0.08)	-	-
Characterised as minor peaks	2.6 (0.003) [a]	12.6 (0.004) [a]	13.1 (0.058) [a]	37.2 (0.019) [a]	4.0 (0.004) [a]
Post-extraction solids					
M510F65 (characterized via RT)	23.7 (0.029)	NP	2.2 (0.01)	-	NP
Characterised as minor peaks	-	NP	19.4 (0.085) [1]	35.1 (0.018) [b]	NP
Unextracted	7.5 (0.009)	8.0 (0.002)	9.6 (0.042)	8.1 (0.004)	5.8 (0.006)
Grand total	103.6 (0.127)	99.4 (0.03)	103.2 (0.453)	120.7 (0.061)	100 (0.096)

[a] each minor analytical peak <10% TRR and <0.01 mg eq/kg

[b] two analytical peaks at 17.5% TRR each and 0.009 mg eq/kg

NP:not performed

The metabolic pathway of ^{14}C-pyridin-labelled boscalid in laying hens was limited. In the first step, hydroxylation at the diphenyl-ring was observed forming M510F01. In a second step, glucuronidation occurs into M510F65.

In laying hens transfer of radioactivity into eggs plateaued after approximately ten days. Highest TRR levels were found in liver, followed by egg, fat and muscle. Extraction showed that a significant part of the radioactivity in liver, egg yolk and muscle was only released after enzyme treatment. Identification revealed parent boscalid and its metabolites M510F01 and M510F65.

Environmental fate in soil

The Meeting received a large environmental fate data package in addition to the studies already evaluated by previous JMPRs. The Meeting decided to postpone the assessment of all new data received on fate and behaviour in soil, hydrolytic degradation in aquatic systems and photochemical degradation until the next periodic review of boscalid for a complete view of the data and its impact on residues in following crops.

The Meeting also received an additional field rotational crop study on boscalid on fruiting vegetables grown as follow crop after soil treatment. Since this type of study is directly linked to the estimation of maximum residue levels, the current Meeting decided to assess this study before the next periodic review.

Fate and behaviour in soil

The Meeting received the following studies on the fate and behaviour in soil, but decided to postpone their evaluation until the next periodic review of boscalid:

Paulick, R.C. (BOSC19E_003, 2002); Pape, L. (BOSC19E_004, 2014); Class, T. (BOSC19E_005, 2013); Heinz, N. (BOSC19E_006, 2014); Pape, L. (BOSC19E_007, 2014); Sachers, S. (BOSC19E_008, 2015); Schulz, H. (BOSC19E_009, 2002); Budde, E. (BOSC19E_010, 2014); Richter, T. (BOSC19E_011, 2013); Richter, T. (BOSC19E_012, 2013); Oliver, G.(BOSC19E_013, 2001); Jackson, S. (BOSC19E_014, 2001); Jackson, S. (BOSC19E_015, 2001); Jackson, S. (BOSC19E_016, 2001); Jackson, S. (BOSC19E_017, 2001); Jackson, S. (BOSC19E_018, 2003); Gooding, R. (BOSC19E_019, 2001); Gooding, R. (BOSC19E_020, 2003); Richter, T. (BOSC19E_021, 2017); Schriever, C. (BOSC19E_022, 2017); Corden, M. (BOSC19E_023, 2014); Corden, M. (BOSC19E_024, 2014)

Hydrolytic degradation in aquatic systems

The Meeting received the following studies on hydrolytic degradation in aquatic systems, but decided to postpone their evaluation until the next periodic review of boscalid:

Yeomans, P. (BOSC19E_025, 2015); Budde, E. (BOSC19E_026, 2015); Schriever, C. (BOSC19E_027, 2016); Schaefer, D. (BOSC19E_028, 2007)

Photochemical degradation

The Meeting received the following studies on photochemical degradation, but decided to postpone their evaluation until the next periodic review of boscalid:

Goetz, N. von (BOSC19E_029, 2002); Hassink, J. (BOSC19E_030, 2002)

Field rotational crop studies

The Meeting received a new field rotational crop study conducted by Martin, T. (BOSC19E_031, 2015). Four field trials were conducted with three rotational crops (cucumber or zucchini, tomato and seeded lettuce) in different representative growing areas in Northern and Southern Europe. Boscalid was applied once to bare soil approximately 30 days before seeding/planting at a rate of 2.1 kg ai/ha. Specimens of plant were collected at growth stages representative to commercial harvest and stored frozen at or below -18 °C until analysis (Method BASF 535/1, L0076/01, LC-MS/MS) for a maximum period of 100 days for plant material.

In the samples collected, no residues above the LOQ were found in zucchini and tomatoes. Lettuce plants contained boscalid residues above the LOQ for all samples, ranging from 0.014 mg/kg up to 0.12 mg/kg.

Table 8 Residues of boscalid in zucchini, tomatoes and lettuce grown as follow crop after application of 2.1 kg ai/ha to bare soil

Trial site	Application rate, Plantback interval	Commodity	DALA	Boscalid in mg/kg
Germany, Kleve	2.1 kg ai/ha (bare soil), 30 day PBI	Zucchini	73	<0.01
		Tomato	129	<0.01
		Lettuce	73	0.078
		Lettuce	86	0.055
The Netherlands, Limburg	2.1 kg ai/ha (bare soil), 30 day PBI	Zucchini	73	<0.01
		Tomato	129	<0.01
		Lettuce	73	0.018
		Lettuce	86	0.014
Italy, Bologna	2.1 kg ai/ha (bare soil), 30 day PBI	Zucchini	66	<0.01
		Tomato	129	<0.01
		Lettuce	104	0.036
		Lettuce	119	0.038
Spain, Sevilla	2.1 kg ai/ha (bare soil), 30 day PBI	Cucumber	80	<0.01
		Tomato	140	<0.01
		Lettuce	60	0.12
		Lettuce	87	0.022

RESIDUE ANALYSIS

Analytical methods

For the analysis of boscalid in various plant matrices additional analytical methods were submitted. In the following table an overview of these methods is presented.

Table 9 Overview of analytical methods for boscalid

Method	Matrix	Extraction	Clean-Up	Detection, LOQ
BASF 535/1	High water High oil High starch High acid	methanol, water and hydrochloric acid (70:25:5, v/v/v) Partitioning against cyclohexane	none	HPLC-MS/MS (ESI+) Boscalid m/z: 343→271 (detection) m/z: 343→307 (quantification) LOQ: 0.01 mg/kg
L0076/01	Hops Spices Herbal infusions	methanol, water and hydrochloric acid (70:25:5, v/v/v) Partitioning against cyclohexane	None	HPLC-MS/MS (ESI+) Boscalid m/z: 343→272 (detection) m/z 343→271 (detection and quantification) m/z 343→307 (detection) m/z: 343→140 (quantification) LOQ: 0.01 mg/kg
D9908	High water High oil	Acetonitrile Portioning with hexane	SPE	HPLC-MS/MS (ESI+) Boscalid m/z: 343→307 (detection) LOQ: 0.05 mg/kg
QuEChERS	High water High oil High starch High acid	Acetonitrile + buffer salts	SPE with primary secondary amine	HPLC-MS/MS (ESI+) Boscalid m/z: 343→307 (detection) m/z: 343→271 (quantification) LOQ: 0.01 mg/kg

The Meeting also received additional recovery data for the method 471/0 already evaluated by the 2006 JMPR, measuring residues of boscalid and M510F01 in animal commodities.

Additionally, multiple studies for the analysis of soil and water were submitted. The Meeting decided that the suitability of these methods will be assessed together with the corresponding environmental fate studies during the next periodic review of boscalid.

Plant materials

Method BASF 535/1 (Mackenroth, C., BOSC19E_032, 2007)

In method BASF 535/1 residues of boscalid are extracted using a mixture of methanol, water and hydrochloric acid (70:25:5, v/v/v). An aliquot of the extract is centrifuged and partitioned at alkaline conditions against cyclohexane. After evaporation of cyclohexane, the residues are dissolved in methanol/water (50/50, v/v). Detection was accomplished by electrospray ionization in positive mode at mass transition 343→271 for quantification and 343→307 for confirmation.

Table 10 Recovery data for method BASF 535/1 measuring boscalid in plant matrices

Matrix	Fortification level (mg/kg)	n	Recovery, mean (%)		RSD (%)	
			343→271	343→307	343→271	343→307
Wheat, plant	0.01	5	93	92	5.2	16.6
	0.1	5	83	84	5.6	7.7
Wheat, grain	0.01	5	84	100	7.3	5.2

Matrix	Fortification level (mg/kg)	n	Recovery, mean (%)		RSD (%)	
			343→271	343→307	343→271	343→307
	0.1	5	84	82	4.3	13.9
Wheat, straw	0.01	5	86	86	7.3	11.1
	0.1	5	87	82	11.6	9.4
Lemon, fruit	0.01	5	88	89	2.1	17.4
	0.1	5	82	77	8.7	14.8
Lettuce, head	0.01	5	82	92	6.0	5.3
	0.1	5	82	81	8.8	3.0
Rapeseeds	0.01	5	80	80	7.1	12.9
	0.1	5	84	86	6.5	18.2
Tomato, fruit	0.01	5	86	81	5.0	18.4
	0.1	5	86	82	3.2	9.2
Onion, bulb	0.01	5	88	83	7.0	10.3
	0.1	5	88	81	8.8	9.2

Method L0076/01 (Austin, R., BOSC19E_033, 2015)

Residues of boscalid are extracted from hops, spices and herbal infusions (green tea) with a mixture of methanol, water and hydrochloric acid (70:25:5, v/v/v). An aliquot of the extract was centrifuged and partitioned in alkaline conditions against cyclohexane, evaporated to dryness and dissolved in methanol/water (1:1, v/v) for analyses. The final determination is performed by LC-MS/MS monitoring selective ion mass transitions 343→272, 343→271, 343→307 and 343→140 using positive electrospray ionization. Table 11 below shows the transitions that were used for each matrix.

Table 11 Recovery data for method L0076/01 measuring boscalid in hops, spices and green tea

Matrix	Fortification level (mg/kg)	n	Recovery, mean (%)		RSD (%)	
			1st Transitions	2nd Transitions	1st Transitions	2nd Transitions
Hops, dry	m/z		343→272	343→140	343→272	343→140
Cones	0.01	5	83	80	6	4
	0.1	5	90	87	2	3
Spices	m/z		343→271	343→140	343→271	343→140
(pepper)	0.01	5	81	83	5	4
	0.1	5	77	77	2	2
Green tea,	m/z		343→307	343→271	343→307	343→271
dry leaves	0.01	5	83	85	2	3
	0.1	5	90	90	9	9

Method D9908 (Jones, J., BOSC19E_034, 2001)

In method D9908 residues of boscalid were extracted from almond with acetonitrile, cleaned by a liquid/liquid partition with hexane, and further purified. Residues in plum are extracted using a mixture of methanol, water and hydrochloric acid and further cleaned via C_{18}- and Silica Gel-SPE. Residues in onions are extracted using a mixture of methanol, water and hydrochloric acid and an aliquot was

cleaned by liquid/liquid partitioning using cyclohexane, followed by purification via C_{18}- and Silica Gel-SPE. Detection was accomplished by LC-MS/MS using electrospray ionization in positive mode at mass transition 343→307 for quantification.

Table 12 Recovery data for method D9908 measuring boscalid in plant matrices

Matrix	Fortification level (mg/kg)	n	Recoveries % (mean %)	RSD (%)
			343→307	
Almond, nutmeat	0.05	2	73, 84 (79)	-
	3.0	2	82, 85 (84)	-
Plum, fruit	0.05	2	97, 97 (97)	-
	3.0	2	94, 93 (94)	-
Onion, bulb	0.05	4	67, 67, 96, 113 (86)	23
	3.0	2	81, 88 (85)	-

Method QuEChERS (Schernikau, N., BOSC19E_035, 2015)

Samples of homogenized plant were extracted with acetonitrile after addition of water to the plant matrix. After addition of a buffer salt mixture, containing magnesium sulphate, sodium chloride and sodium citrate, the extract was shaken. After centrifugation, an aliquot of the acetonitrile phase was cleaned by dispersive solid phase extraction, using primary secondary amine (PSA). The samples were analysed using LC-MS/MS to quantify and to confirm boscalid using two mass transitions (343→307 m/z and 343→271 m/z) (ESI+).

Table 13 Recovery data for QuEChERS method measuring boscalid in plant matrices

Matrix	Fortification level (mg/kg)	n	Recovery, mean (%)		RSD (%)	
			343→307	343→271	343→307	343→271
Wheat, plant	0.01	5	109	109	3.7	3.8
	0.1	5	102	103	5.8	4.3
Wheat, grain	0.01	5	103	105	2.6	3.5
	0.1	5	101	101	1.5	2.2
Wheat, straw	0.01	5	94.4	97.3	2.8	1.6
	0.1	5	93.4	93.4	4.3	3.9
Lemon, fruit	0.01	5	107	106	3.6	3.2
	0.1	5	106	104	2.4	3.4
Onion, bulb	0.01	5	86.6	87.9	3.2	3.5
	0.1	5	83.8	83.7	6.1	5.6

Animal materials

Method 471/0 – additional recovery data

The general methodology was already evaluated by the 2006 JMPR: "A 25 g sample is extracted with methanol. An aliquot corresponding to a 5 g sample is taken for further work-up. The methanol extract is evaporated to dryness, redissolved in buffer solution and incubated with ß-glucuronidase / arylsulfatase to cleave the glucuronide M510F02 to M510F01. Then a liquid / liquid partition with ethyl acetate is carried out and the organic phase is purified on SPE C18 and if necessary on SPE silica gel

columns. The final determination of the analytes boscalid and M510F01 is performed by HPLC/MS/MS."

In the following table, newly submitted additional recovery data and independent laboratory validation recovery data are summarized.

Table 14 Additional Recovery data for method 471/0 measuring boscalid and M510F01 in animal matrices (Courtois J., 2015, BOSC19E_036)

Matrix	Fortification level (mg/kg)	n	Recovery, mean (%)		RSD (%)	
Boscalid, m/z:			343→307	343→140	343→307	343→140
Cow, milk	0.01	5	86.0	86.2	3.6	6.7
	0.1	5	88.7	87.6	7.7	11.9
Cow, cream	0.01	5	72.2	73.4	1.5	9.6
	0.1	5	89.9	83.5	4.7	8.7
Cow, muscle	0.025	5	86.4	107	4.0	3.4
	0.25	5	94.5	93.1	1.5	1.8
Cow, fat	0.025	5	80.0	79.9	5.4	5.8
	0.25	5	81.0	80.9	8.5	6.6
Cow, liver	0.025	5	86.7	74.2	6.3	8.7
	0.25	5	96.0	90.9	8.7	8.8
Hen, egg	0.01	5	82.5	88.2	3.8	4.9
	0.1	5	93.1	93.3	3.1	5.3
Cow, kidney			343→307	343→271	343→307	343→271
	0.025	5	83.3	84.5	1.9	27.7
	0.25	5	90.6	92.3	3.9	7.6
M510F01, m/z:			359→323	359→140	359→323	359→140
Cow, milk	0.01	5	88.4	83.3	5.8	5.6
	0.1	5	84.9	82.8	8.6	11.5
Cow, cream	0.01	5	89.5	83.9	1.7	3.2
	0.1	5	94.2	93.9	2.3	6.0
Cow, muscle	0.025	5	89.3	106	2.1	2.9
	0.25	5	86.3	88.6	1.4	4.0
Cow, fat	0.025	5	81.0	79.5	4.0	2.6
	0.25	5	82.6	82.3	7.4	5.5
Cow, kidney	0.025	5	81.6	73.2	2.5	7.0
	0.25	5	82.2	78.7	4.6	4.2
Cow, liver	0.025	5	90.9	91.9	10.3	11.6
	0.25	5	91.5	91.0	6.2	5.1
Hen, egg	0.01	5	82.7	82.5	6.1	4.2
	0.1	5	89.1	88.5	8.2	6.8

Table 15 Independent laboratory validation for method 471/0 measuring boscalid and M510F01 in animal matrices (Weber, H., 2015, BOSC19E_037 and Weber, H., 2015, BOSC19E_038)

Matrix	Fortification level (mg/kg)	n	Recovery, mean (%)		RSD (%)	
Boscalid, m/z:			343→307	343→271	343→307	343→271
Cow, muscle	0.01	5	83.3	80.7	4.8	5.1
	0.025	5	78.8	80.7	3.5	6.1
	0.25	5	80.0	80.2	4.7	4.7
Cow, kidney	0.01	5	77.7	77.5	4.6	8.3
	0.025	5	78.1	78.9	4.0	4.8
	0.25	5	74.0	72.5	2.9	3.2
Cow, liver	0.01	5	71.6	78.3	3.1	4.9
	0.025	5	70.8	73.4	8.2	11
	0.25	5	71.9	72.8	5.4	4.4
Cow, fat	0.01	5	87.6	87.5	8.2	8.0
	0.025	5	84.4	83.9	6.2	9.8
	0.25	5	80.9	80.4	7.3	9.5
Cow, cream	0.01	5	73.5	71.2	3.3	4.2
	0.10	5	76.2	78.2	5.8	6.3
Cow, milk	0.01	5	75.6	72.5	5.3	5.0
	0.10	5	85.9	86.8	2.6	5.6
Hen, egg	0.01	5	75.7	74.7	2.1	5.5
	0.10	5	89.9	89.1	3.0	2.4
M510F01, m/z:			359→323	359→140	359→323	359→140
Cow, muscle	0.01	5	78.3	76.4	6.3	7.0
	0.025	5	81.7	82.6	4.7	3.8
	0.25	5	82.6	83.0	4.8	4.1
Cow, kidney	0.01	5	83.1	81.9	2.1	6.7
	0.025	5	79.6	82.0	4.3	5.5
	0.25	5	73.8	75.3	2.2	3.0
Cow, liver	0.01	5	75.1	79.6	3.5	5.7
	0.025	5	74.7	75.4	5.6	6.3
	0.25	5	80.9	82.2	6.6	6.1
Cow, fat	0.01	5	84.5	82.1	6.6	6.4
	0.025	5	86.8	84.0	1.8	3.8
	0.25	5	80.6	80.9	3.3	4.2
Cow, cream	0.01	5	79.3	83.4	3.3	5.0
	0.10	5	86.7	87.0	6.6	6.8

Matrix	Fortification level (mg/kg)	n	Recovery, mean (%)		RSD (%)	
Cow, milk	0.01	5	85.4	80.8	3.2	5.3
	0.10	5	86.8	85.8	11	10
Hen, egg	0.01	5	81.7	79.3	6.0	2.2
	0.10	5	94.5	96.7	2.8	1.9

Soil and water

The Meeting received the following studies on analytical methods for soil and water but decided to postpone their evaluation until the next periodic review of boscalid:

Obermann, M. (BOSC19E_039, 2009);Obermann, M. (BOSC19E_040, 2015); Kreidler, D. (BOSC19E_041, 2013);Ertunc, T. (BOSC19E_042, 2015); Saha, M.G. (BOSC19E_043, 2001); Penning, H. (BOSC19E_044, 2009);Ertunc, T. (BOSC19E_045, 2015); Ertunc, T. (BOSC19E_046, 2015) and Goecer, M. (BOSC19E_047, 2016).

USE PATTERN

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

Table 1: List of uses of boscalid

Crop or crop group	Country	Rate	Number of treatments (minimum interval, days)	Pre-harvest interval (PHI), days
Pome fruits				
Apple	Australia	0.01 kg ai/hL	3 (7 day early growth stage, 10 day after petal fall)	14
	Austria	0.2 kg ai/ha (0.067 kg ai/m crown height/ha)	4 (8)	7
	Belarus	0.2 kg ai/ha	2 (NS)	72
	Belgium	0.2 kg ai/ha	4 (7)	7
	Bulgaria	0.2 kg ai/ha	3 (8)	7
	China	NS	3 (7)	7
	Croatia	0.2 kg ai/ha	3 (10)	7 (stored apples) 14 (directly sold apples)
	Finland	0.21 kg ai/ha	3 (10)	10
	Greece	0.08 kg ai/hL (0.3 kg ai/ha)	3 (10)	7
	Hungary	0.2 kg ai/ha	4 (8)	7
	Ireland	0.2 kg ai/ha	4 (10)	7
	Italy	0.2 kg ai/ha	3 (7)	7
	Japan	×2000 dilution rate (0.0068 kg ai/hL)	3 (NS)	1
	Kazakhstan	0.2 kg ai/ha	3 (NS)	7
	Korea, Republic of	0.32 kg ai/ha	5 (7)	30
	Macedonia	0.2 kg ai/ha	4 (10)	7 (stored apples) 14 (directly sold apples)
	Morocco	0.013 kg ai/hL	3 (14)	7
	Netherlands	0.2 kg ai/ha	4 (7)	7
	Peru	0.2 kg ai/ha	2 (14)	7
	Poland	0.2 kg ai/ha	2 (8)	7
	Portugal	0.2 kg ai/ha	3 (7)	7

Crop or crop group	Country	Rate	Number of treatments (minimum interval, days)	Pre-harvest interval (PHI), days
	Romania	0.2 kg ai/ha	4 (8)	7
	Russian Federation	0.2 kg ai/ha	4 (10)	10
	Serbia	0.2 kg ai/ha	3 (NS)	7
	Slovenia	0.2 kg ai/ha	3 (7)	7
	Spain	0.02 kg ai/hL (up to 0.2 kg ai/ha)	3 (30)	7
	Taiwan, Province of China	×1500 dilution rate (0.017 kg ai/hL)	NS (7)	21
	Turkey	0.1 kg ai/hL	NS	7
	Ukraine	0.2 kg ai/ha	3 (NS)	20
	United Kingdom	0.2 kg ai/ha	4 (10)	7
Pear	Australia	0.01 kg ai/hL	3 (7 days early growth stage, 10 days after petal fall)	14
	Belarus	0.2 kg ai/ha	2 (NS)	72
	Belgium	0.2 kg ai/ha	4 (7)	7
	Bulgaria	0.2 kg ai/ha	3 (8)	7
	Finland	0.21 kg ai/ha	3 (10)	10
	Greece	0.02 kg ai/hL (0.2 kg ai/ha)	3 (10)	7
	Hungary	0.2 kg ai/ha	4 (8)	7
	Ireland	0.2 kg ai/ha	4 (10)	7
	Italy	0.2 kg ai/ha	3 (7)	7
	Japan	×2000 dilution rate (0.0068 kg ai/hL)	3 (NS)	1
	Korea, Republic of	0.32 kg ai/ha	4 (10)	20
	Netherlands	0.2 kg ai/ha	4 (7)	7
	Poland	0.2 kg ai/ha	2 (8)	7
	Portugal	0.2 kg ai/ha	3 (7)	7
	Romania	0.2 kg ai/ha	4 (8)	7
	Russian Federation	0.2 kg ai/ha	4 (10)	10
	Serbia	0.2 kg ai/ha	3 (NS)	7
	Slovenia	0.2 kg ai/ha	3 (7)	7
	Spain	0.02 kg ai/hL (up to 0.2 kg ai/ha)	3 (30)	7
	Taiwan, Province of China	×1500 dilution rate (0.017 kg ai/hL)	4 (7)	15
	Turkey	0.01 kg ai/hL	NS	7
	United Kingdom	0.2 kg ai/ha	4 (10)	7
Quinces	Bulgaria	0.2 kg ai/ha	3 (8)	7
	Hungary	0.2 kg ai/ha	4 (8)	7
	Turkey	0.013 kg ai/hL	NS	7
Pome fruit group	Canada	0.3 kg ai/ha	4 (7)	5
	Czech Republic	0.2 kg ai/ha	4 (8)	7
	France	0.02 kg ai/hL	3 (8)	7
	Germany	0.013 kg ai/hL (0.067 kg ai in 500 L water per m crown height/ha)	4 (8)	7
	Slovakia	0.2 kg ai/ha	3 (NS)	15
	Switzerland	0.2 kg ai/ha	3 (NS)	21
	USA	0.33 kg ai/ha	4 (7)	0
Stone fruits				
Apricots	Spain	0.2 kg ai/ha	2 (7)	7
	Turkey	0.01 kg ai/hL	NS	14
	Ukraine	0.33 kg ai/ha	2 (10)	40
Cherries	Belgium	0.13 kg ai/ha	3 (7)	7
	Bulgaria	0.08 kg ai/ha	3 (10)	7
	Czech Republic	0.013 kg ai/hL (0.067 kg ai in 500 L water per m crown height/ha)	3 (10)	7

Crop or crop group	Country	Rate	Number of treatments (minimum interval, days)	Pre-harvest interval (PHI), days
	Japan	×x2000 dilution rate (0.0068 kg ai/hL)	3 (NS)	1
	Macedonia	0.27 kg ai/ha	2 (NS)	7
	Netherlands	0.013 kg ai/hL (max. 0.19 kg/ha)	3 (7)	7
	Norway	0.027 kg ai/hL	2 (5)	Covered by growth stage (during flowering)
	Poland	0.27 kg ai/ha	2 (5)	Covered by growth stage (during flowering)
	Slovakia	0.067 kg ai/ha	3 (10)	7
	Spain	0.2 kg ai/ha	2 (7)	7
	Sweden	0.2 kg ai/ha	3 (7)	3
	Turkey	0.04 kg ai/hL	NS	7
	Ukraine	0.33 kg ai/ha	2 (7)	30
Peaches and nectarines	Argentina	0.013 kg ai/hL	1	7
	Belgium	0.13 kg ai/ha	3 (10)	7
	Japan	×2000 dilution rate (0.0068 kg ai/hL)	3 (NS)	1
	Spain	0.2 kg ai/ha	2 (7)	7
	Turkey	0.027 kg ai/hL	NS	7
	Ukraine	0.33 kg ai/ha	2 (10)	40
	Uruguay	0.25 kg ai/ha	2	Covered by growth stage (up to petal fall)
Plums	Austria	0.19 kg ai/ha (0.063 kg ai/m crown height/ha)	3 (7)	7
	Belgium	0.13 kg ai/ha	3 (7)	7
	Netherlands	0.013 kg ai/hL (max. 0.19 kg/ha)	3 (7)	7
	Norway	0.027 kg ai/hL	2 (5)	Covered by growth stage (during flowering)
	Slovakia	0.067 kg ai/ha	3 (10)	7
	Spain	0.2 kg ai/ha	2 (7)	7
	Sweden	0.2 kg ai/ha	3 (7)	3
Stone fruit group	Austria	0.19 kg ai/ha (0.063 kg ai/m crown height/ha)	3 (10-14)	7
	Canada	0.26 kg ai/ha	5 (7)	0
	Chile	0.6 kg ai/ha	1	3
		0.25 kg ai/ha	2 (10)	0
	Germany	0.013 kg ai/hL (0.067 kg ai in 500 L water per m crown height/ha)	3 (10)	7
	Hungary	0.27 kg ai/ha	3 (7)	7
	Italy	0.2 kg ai/ha	3 (7)	3
	Malta	0.2 kg ai/ha	3 (7)	3
	Portugal	0.02 kg ai/hL (0.2 kg ai/ha)	3 (7)	7
	Slovenia	0.2 kg ai/ha	2 (10)	7
	USA	0.26 kg ai/ha	5 (7)	0
Small stone fruits (Japanese apricot, apricot, plum)	Japan	×2000 dilution rate (0.0068 kg ai/hL)	2 (NS)	7
Berries and other small fruits				
Blackberries	Austria	0.25 kg ai/ha (field and glasshouse)	3 (7-10)	7
	Belgium	0.13 kg ai/ha (field and glasshouse)	2 (7)	3 (field) 7 (glasshouse)
	Canada	0.4 kg ai/ha	4 (7)	0

Crop or crop group	Country	Rate	Number of treatments (minimum interval, days)	Pre-harvest interval (PHI), days
	Chile	0.6 kg ai/ha	2 (NS)	0
	Germany	0.25 kg ai/ha (field and glasshouse)	3 (7-10)	7
	Netherlands	0.4 kg ai/ha	2 (7)	3
	Spain	0.4 kg ai/ha	2 (7)	3
Blueberries	Argentina	0.25 kg ai/ha	1	7
	Bulgaria	0.4 kg ai/ha	2 (7)	3
	Canada	0.4 kg ai/ha	4 (7)	0
	Hungary	0.27 kg ai/ha	2 (7)	14
	Malta	0.4 kg ai/ha	2 (7)	3
	Morocco	0.13 kg ai/ha	1	14
	Peru	0.2 kg ai/ha	1	1
	Poland	0.45 kg ai/ha	2 (10)	Covered by growth stage (during flowering)
	Slovenia	0.27 kg ai/ha	2 (7)	7
	Spain	0.4 kg ai/ha	2 (7)	3
	Uruguay	0.05 kg ai/hL	2 (7)	7
Cranberries	Belgium	0.13 kg ai/ha (field and glasshouse)	2 (7)	3
Currants	Hungary	0.27 kg ai/ha	2 (7)	14
	Malta	0.4 kg ai/ha	2 (7)	3
	Netherlands	0.4 kg ai/ha	2 (7)	3
	Poland	0.45 kg ai/ha	2 (7)	
	Spain	0.4 kg ai/ha	2 (7)	3
Raspberries	Austria	0.25 kg ai/ha (field and glasshouse)	3 (7-10)	7
	Canada	0.39 kg ai/ha	4 (7)	0
	Chile	0.6 kg ai/ha	2 (NS)	0
	Germany	0.25 kg ai/ha (field and glasshouse)	3 (7-10)	7
	Netherlands	0.4 kg ai/ha	2 (7)	3
	Poland	0.48 kg ai/ha	2 (7)	Covered by growth stage (during flowering)
	Spain	0.4 kg ai/ha	2 (7)	3
Bush berries subgroup	Germany	0.25 kg ai/ha (field and glasshouse)	3 (7-10)	7
	Italy	0.4 kg ai/ha	2 (7)	3
	USA	0.4 kg ai/ha	4 (7)	0
Cane berries subgroup	Malta	0.4 kg ai/ha	2 (7)	3
	USA	0.4 kg ai/ha	4 (7)	0
Assorted tropical and subtropical fruits				
Avocado	Mexico	0.25 kg ai/ha	1	14
	Peru	0.25 kg ai/ha	2 (7)	NS
Mango	Brazil	0.024 kg ai/hL (0.24 kg ai/ha)	2 (15)	7
	Mexico	0.3 kg ai/ha	2 (7)	0
	Peru	0.2 kg ai/ha and 0.019 kg ai/hL	2 (7)	7
	Taiwan, Province of China	×2000 dilution rate (0.017 kg ai/hL)	2 (7)	6
Papaya	Belize	0.25 kg ai/ha	6 (7)	0
	Costa Rica	0.25 kg ai/ha	6 (7)	0
	Mexico	0.3 kg ai/ha	3 (7)	0
	Taiwan, Province of China	×1500 dilution rate (0.014 kg ai/hL)	4 (10)	12
Pomegranate	Turkey	0.0126 kg ai/hL	3 (bud formation up to final fruit size)	Not specified

Crop or crop group	Country	Rate	Number of treatments (minimum interval, days)	Pre-harvest interval (PHI), days
Tropical fruit group (Avocado, Black sapote, Canistel, Mamey Sapote, Mango, Papaya, Sapodilla, Star apple)	USA	0.33 kg ai/ha	2 (7)	0
Tea				
Tea	Japan	×2000 dilution rate (0.0068 kg ai/hL)	2 (NS)	7

NS: not stated or not defined

RESULTS OF SUPERVISED RESIDUE TRIALS ON CROPS

Residue levels were reported as measured. Application rates were always reported as boscalid 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. The residue values from trials conducted according to maximum GAP that have been used for the estimation of maximum residue levels, STMR and HR, are underlined.

Laboratory reports included method validation including batch recoveries with spiking at residue levels similar to those occurring in samples from the supervised trials. Dates of analyses or duration of residue sample storage were also provided. Field reports provided data on the sprayers used and their calibration, plot size, residue sample size and sampling date. Although trials included control plots, no control data are recorded in the tables except where residues in control samples exceeded the LOQ. Residue data are recorded unadjusted for percent recovery.

Boscalid - Supervised residue trials

Commodity	Indoor/Outdoor	Treatment	Countries	Table
Apples (new data)	Outdoor	Foliar	Argentina, Belgium, Canada, France, Italy, Netherlands, USA	Table 17
Apples (2006 data)	Outdoor	Foliar	Belgium, France, Germany, Italy, Netherlands	Table 18
Pear	Outdoor	Foliar	Argentina, Canada, France, Germany, Italy, Netherlands, Poland, Spain, United Kingdom, USA	Table 19
Cherries (new data)	Outdoor	Foliar	Austria, Canada , Denmark, France, Germany, Hungary, Italy, Netherlands, Poland, Sweden, USA	Table 20
Cherries (2006 data)	Outdoor	Foliar	USA	Table 21
Peaches (new data)	Outdoor	Foliar	Canada, France, Germany, Italy, USA	Table 22
Peaches (2006 data)	Outdoor	Foliar	USA	Table 23
Plums (new data)	Outdoor	Foliar	Canada, Denmark, France, Germany, Italy, Sweden, USA	Table 24
Plums (2006 data)	Outdoor	Foliar	USA	Table 25
Blueberries (new data)	Outdoor	Foliar	Canada, USA	Table 26
Blueberries (2006 data)	Outdoor	Foliar	Canada, USA	Table 27
Blueberries	Indoor	Foliar	Germany	Table 24
Currants	Indoor	Foliar	Germany	Table 25
Raspberries (2006 data)	Outdoor	Foliar	Canada, USA	Table 26
Avocado	Outdoor	Foliar	USA	Table 27
Mango	Outdoor	Foliar	Brazil	Table 28
Pomegranate	Outdoor	Foliar	Greece, Italy, Spain	Table 29
Tea	Outdoor	Foliar	China, India, Japan, Taiwan (Province of China)	Table 30

Table 17 Residues of boscalid in apples (submitted to the Extra 2019 JMPR Meeting)

Location, Year (variety)	Application				Residues, mg/kg			Report/Trial No., Reference, analytical method, validation data, storage period
	kg ai/ha	Inter-val days	kg ai/hL	Growth stage	Sample	DALA	Boscalid	
Southern America (cGAP: none)								
Argentina, Lujan de Cuyo 2002 (Gala)	2 × 0.23	NS	4 × 0.013	Up to BBCH 75	Whole fruit	1 10 20 45 89	0.34 0.17 0.08 <0.05 <0.05	2003/1026457-, BOSC19E_056 Method: 445/0 modified Storage period: 3 months
	2 × 0.45	NS	4 × 0.025	Up to BBCH 75	Whole fruit	1 10 20 45	0.94 0.43 0.24 0.10	
Argentina, Allen 2014/2015 (NS)	0.25	-	NS	NS	Whole fruit	1 7 15	0.16, 0.18, 0.12, 0.17 (0.16) 0.18, 0.21, 0.2, 0.27 (0.24) 0.12, 0.15, 0.16, 0.16 (0.15)	2016/3004409-G150156, BOSC19E_057 Method: L0076/09 Storage period: 12 months
Argentina, Tunuyán 2014/2015 (Chañar 34)	0.25	-	NS	NS	Whole fruit	1 7 15	0.2, 0.12, 0.2, 0.19 (0.18) 0.17, 0.16, 0.16, 0.14 (0.16) 0.058, 0.067, 0.046, 0.039 (0.052)	2016/3004409-G150157, BOSC19E_057 Method: L0076/09 Storage period: 12 months
(Red Chief)	0.25	-	NS	NS	Whole fruit	1 7 15	0.18, 0.2, 0.27, 0.27 (0.23) 0.2, 0.25, 0.18, 0.23 (0.22) 0.12, 0.16, 0.11, 0.081 (0.12)	2016/3004409-G150158
Argentina, Ingeniero Huergo 2014/2015 (NS)	0.25	-	NS	NS	Whole fruit	1 8 15	0.12, 0.13, 0.09, 0.14 (0.12) 0.14, 0.11, 0.14, 0.14 (0.13) 0.16, 0.19, 0.13, 0.12 (0.15)	2016/3004409-G150389, BOSC19E_057 Method: L0076/09 Storage period: 12 months
Argentina, Mainqué 2014/2015 (NS)	0.25	-	NS	NS	Whole fruit	1 8 15	0.15, 0.15, 0.12, 0.13 (0.14) 0.16, 0.12, 0.13, 0.16 (0.14) 0.12, 0.13, 0.13, 0.13 (0.13)	2016/3004409-G150390, BOSC19E_057 Method: L0076/09 Storage period: 12 months

Location, Year (variety)	Application				Residues, mg/kg			Report/Trial No., Reference, analytical method, validation data, storage period
	kg ai/ha	Inter-val days	kg ai/hL	Growth stage	Sample	DALA	Boscalid	
Argentina, Tupungato 2014/2015 (NS)	0.25	-	NS	NS	Whole fruit	1 8 15	0.29, 0.17, 0.13, 0.22 (0.2) 0.18, 0.16, 0.20, 0.21 (0.19) 0.11, 0.12, 0.11, 0.11 (0.11)	2016/3004409-G150391, BOSC19E_057 Method: L0076/09 Storage period: 12 months
Europe (cGAP: CZ, 4 × 0.2 kg ai/ha, 8 day interval, 7 d PHI)								
Belgium, Limburg 2003 (Decofta)	4 × 0.2 (SE)	NS	4 × 0.02	Up to BBCH 81	Whole fruit	0 21 28 35 42	0.27 0.23 0.19 0.26 0.22	2004/1000752-AGR/09/03, BOSC19E_053 Method: 445/0 Storage period: 3 months
France, Rottelsheim 2003 (Golden)	4 × 0.2 (SE)	NS	4 × 0.02	Up to BBCH 77	Whole fruit	0 20 28 35 42	0.33 0.21 0.13 0.14 0.11	2004/1000752-FAN/09/03, BOSC19E_053 Method: 445/0 Storage period: 3 months Final application: 04.08.2003
France, Bouloc 2003 (Star Krimson)	4 × 0.2 (SE)	NS	4 × 0.02	Up to BBCH 81	Whole fruit	0 21 28 35 42	0.87 0.56 0.62 0.40 0.55	2004/1000752-FTL/05/03, BOSC19E_053 Method: 445/0 Storage period: 3 months Final application: 14.08.2003
Italy, Montemarcino 2003 (Copper 4)	4 × 0.2 (SE)	NS	4 × 0.02	Up to BBCH 81	Whole fruit	0 21 29 35 42	0.61 0.54 0.46 0.32 0.34	2004/1000752-ITA/07/03, BOSC19E_053 Method: 445/0 Storage period: 3 months Final application: 14.08.2003
Netherlands, Groesbeek 2003 (Golden Delicious)	4 × 0.2 (SE)	NS	4 × 0.02	Up to BBCH 81	Whole fruit	0 20 27 34 41	0.82 0.77 0.51 0.60 0.41	2004/1000752-AGR/10/03, BOSC19E_053 Method: 445/0 Storage period: 3 months
Northern America (cGAP: CZ, 4 × 0.33 kg ai/ha, 7 d interval, 0 d PHI)								
USA, Hereford (PN) 2001	6 × 0.34-0.35	6-8	6 × 0.066-0.067	50-70mm to harvest	Whole fruit	0	1.1	2002/5002108-2001828, BOSC19E_048 Method: D9908
(Starkrimson Red Delicious)	6 × 0.34-0.35	6-8 d	6 × 0.009-0.011	50-70mm to harvest	Whole fruit	0	0.61	Storage period:7 months
USA, Dundee (NY) 2001	6 × 0.34	6-8	6 × 0.071-0.072	BBCH81 to Harvest	Whole fruit	0	1.2	2002/5002108-2001829, BOSC19E_048 Method: D9908
(Empire)	6 × 0.34	6-8	6 × 0.024	BBCH81 to Harvest	Whole fruit	0	1.1	Storage period:7 months
USA, Alton (NY)	6 × 0.34	6-8	6 × 0.045	50-75mm to harvest	Whole fruit	0	0.78	2002/5002108-2001830, BOSC19E_048

Location, Year (variety)	Application				Residues, mg/kg			Report/Trial No., Reference, analytical method, validation data, storage period
	kg ai/ha	Inter-val days	kg ai/hL	Growth stage	Sample	DALA	Boscalid	
2001 (Red Delicious)		+ postharvest spray at 2000 mg/L			Whole fruit	0	1.6	Method: D9908 Storage period:7 months
		+ postharvest dip at 2000 mg/L			Whole fruit	0	3.9	
		+ postharvest spray+dip at 2000 mg/L			Whole fruit	0	3.3	
		+ postharvest drench at 2000 mg/L			Whole fruit	0	1.8	
	6 × 0.34-0.35	6-8	6 × 0.024	50-75mm to harvest	Whole fruit	0	0.72	
		+ postharvest spray at 2000 mg/L			Whole fruit	0	2.0	
		+ postharvest dip at 2000 mg/L			Whole fruit	0	4.2	
		+ postharvest spray+dip at 2000 mg/L			Whole fruit	0	3.2	
		+ postharvest drench at 2000 mg/L			Whole fruit	0	1.8	
Canada, Berwick (Nova Scotia) 2001 (McIntosh)	0.34 0.34 0.35 0.29 0.34 0.35	7	0.082 0.084 0.083 0.072 0.085 0.084	75mm to harvest	Whole fruit	0	0.94	2002/5002108-2001832, BOSC19E_048 Method: D9908 Storage period:7 months
	0.34 0.34 0.35 0.28 0.34 0.35	7	0.033 0.034 0.034 0.029 0.035 0.035	75mm to harvest	Whole fruit	0	0.96	
USA, Covesville (VA) 2001 (Earligold)	6 × 0.34-0.35	7	6 × 0.065-0.07	BBCH73 to harvest	Whole fruit	0	0.42	2002/5002108-2001834, BOSC19E_048 Method: D9908 Storage period:7 months
	6 × 0.34-0.35	7	6 × 0.03-0.032	BBCH73 to harvest	Whole fruit	0	0.17	
USA, Conklin (MI) 2001 (Empire)	6 × 0.34	7	6 × 0.024	60mm to harvest	Whole fruit	0	0.49	2002/5002108-2001835, BOSC19E_048 Method: D9908 Storage period:7 months
	6 × 0.34	7	6 × 0.017-0.018	60mm to harvest	Whole fruit	0	0.34	
Canada, St. George (Ontario) 2001 (Spartan)	6 × 0.34-0.35	7	6 × 0.071-0.073	50mm to harvest	Whole fruit	0	1.3	2002/5002108-2001836, BOSC19E_048 Method: D9908 Storage period:7 months
	6 × 0.34-0.35	7	6 × 0.024-0.025	50mm to harvest	Whole fruit	0	0.6	

Location, Year (variety)	Application				Residues, mg/kg			Report/Trial No., Reference, analytical method, validation data, storage period
	kg ai/ha	Inter-val days	kg ai/hL	Growth stage	Sample	DALA	Boscalid	
Canada, St. Paul d'Abbotsford	6 × 0.33-0.35	6-7	6 × 0.083-0.085	Enlarged-harvest	Whole fruit	0	0.48	2002/5002108-2001840, BOSC19E_048 Method: D9908
(Quebec) 2001 (Vista Bella)	6 × 0.32-0.35	6-7	6 × 0.019-0.02	Enlarged-harvest	Whole fruit	0	0.46	Storage period:7 months
Canada, Granby (Quebec) 2001	6 × 0.32-0.34	6-8	6 × 0.076-0.077	Enlarged-harvest	Whole fruit	0	0.73	2002/5002108-2001841, BOSC19E_048 Method: D9908
(Spartan)	6 × 0.33-0.35	6-8	6 × 0.015-0.016	Enlarged-harvest	Whole fruit	0	0.43	Storage period:7 months
USA, Eckert (CO)	6 × 0.34	7	6 × 0.072	BBCH78-harvest	Whole fruit	0	0.82	2002/5002108-2001842, BOSC19E_048
2001 (Red Delicious)	6 × 0.34	7	6 × 0.024	BBCH78-harvest	Whole fruit	0	0.92	Method: D9908 Storage period:7 months
USA, Yuba City (CA) 2001	6 × 0.34-0.35	7	6 × 0.12	Fruit dev. up to harvest	Whole fruit	0	0.42	2002/5002108-2001938, BOSC19E_048 Method: D9908
(Light Red Fuji)	6 × 0.34	7	6 × 0.028	Fruit dev. up to harvest	Whole fruit	0	1.1	Storage period:7 months
USA, Porterville (CA) 2001	6 × 0.34	7	6 × 0.062-0.064	50mm to harvest	Whole fruit	0	1.1	2002/5002108-2001939, BOSC19E_048 Method: D9908
(Granny Smith)		+ postharvest spray at 2000 mg/L			Whole fruit	0	3.1	Storage period:7 months
		+ postharvest dip at 2000 mg/L			Whole fruit	0	2.7	
		+ postharvest spray+dip at 2000 mg/L			Whole fruit	0	4.6	
		+ postharvest drench at 2000 mg/L			Whole fruit	0	2.5	
	6 × 0.34	7	6 × 0.011	50mm to harvest	Whole fruit	0	0.84	
		+ postharvest spray at 2000 mg/L			Whole fruit	0	2.3	
		+ postharvest dip at 2000 mg/L			Whole fruit	0	3.7	
		+ postharvest spray+dip at 2000 mg/L			Whole fruit	0	4.1	
		+ postharvest drench at 2000 mg/L			Whole fruit	0	2.5	
USA, Ephrata (WA) 2001	6 × 0.34	7	6 × 0.071-0.072	BBCH86-89	Whole fruit	0	0.85	2002/5002108-2001942, BOSC19E_048 Method: D9908

Location, Year (variety)	Application				Residues, mg/kg			Report/Trial No., Reference, analytical method, validation data, storage period
	kg ai/ha	Inter-val days	kg ai/hL	Growth stage	Sample	DALA	Boscalid	
(Red Delicious)		+ postharvest spray at 2000 mg/L			Whole fruit	0	3.3	Storage period:7 months Final treatment: 27.09.2001
		+ postharvest dip at 2000 mg/L			Whole fruit	0	2.1	
		+ postharvest spray+dip at 2000 mg/L			Whole fruit	0	3.1	
		+ postharvest drench at 2000 mg/L			Whole fruit	0	2.6	
	6 × 0.34	7 d	6 × 0.018	BBCH86-89	Whole fruit	0	0.9	
		+ postharvest spray at 2000 mg/L			Whole fruit	0	2.4	
		+ postharvest dip at 2000 mg/L			Whole fruit	0	2.7	
		+ postharvest spray+dip at 2000 mg/L			Whole fruit	0	3.7	
		+ postharvest drench at 2000 mg/L			Whole fruit	0	2.0	
USA, Ephrata (WA) 2001	6 × 0.34	7	6 × 0.071-0.072	Up to harvest	Whole fruit	0	1.8	2002/5002108-2001943, BOSC19E_048 Method: D9908
(Gala)	6 × 0.34	7	6 × 0.009	Up to harvest	Whole fruit	0	0.68	Storage period:7 months Final treatment: 19.09.2001
USA, Hood River (OR) 2001	6 × 0.34-0.35	7	6 × 0.044-0.051	50mm to harvest	Whole fruit	0	0.62	2002/5002108-2001944, BOSC19E_048 Method: D9908
(Jonagold)	6 × 0.34-0.35	7	6 × 0.016-0.017	50mm to harvest	Whole fruit	0	0.62	Storage period:7 months
USA, Caldwell (ID) 2001	6 × 0.34-0.35	6-8	6 × 0.049-0.061	75mm to harvest	Whole fruit	0	0.31	2002/5002108-2001945, BOSC19E_048 Method: D9908
(Empire)	6 × 0.33-0.35	6-8	6 × 0.012-0.017	75mm to harvest	Whole fruit	0	0.24	Storage period:7 months

DALA: days after last application

NS: not stated

Table 18 Summary information on residues of boscalid in apples (reported in the 2006 JMPR Evaluation)

Location, Year (variety)	Application: kg ai/ha	Application: Interval days	Application: kg ai/hL	Application: Growth stage	Residues, mg/kg: Sample	Residues, mg/kg: DALA	Residues, mg/kg: Boscalid	Report/Trial No., Reference, analytical method, validation data, storage period
Europe (cGAP: CZ, 4 × 0.2 kg ai/ha, 8 day interval, 7 PHI)								
Belgium, Kortenaken 2001 (Jonagold)	4 × 0.2	NS	4 × 0.02	Up to BBCH 81	Whole fruit	0 6 13 22 27	0.39 <u>0.37</u> 0.26 0.26 0.16	2001/1015029-AGR/15/01
France, Cambrai 2000 (Jonagold)	4 × 0.2-0.22	8	4 × 0.02	BBCH 77-85	Whole fruit	0 6 13 21 28	0.55 <u>0.34</u> 0.31 0.17 0.15	2001/1000946-X006203
France, St. Loup Terrier 2000 (Jonagold)	4 × 0.2-0.21	7	4 × 0.02	BBCH 81-85	Whole fruit	0 7 15 22 28	0.56 <u>1.2</u> 0.42 0.52 0.43	2001/1000946-X006204
France, Buzet sur Baize 2000 (Canada)	4 × 0.2-0.21	7-9	4 × 0.02	BBCH 78-81	Whole fruit	0 7 14 21 28	0.38 <u>0.51</u> 0.39 0.28 0.22	2001/1000946-X006205
France, Le Beugnon 2000 (Golden)	4 × 0.2-0.21	8	4 × 0.02	BBCH 75-77	Whole fruit	0 7 14 21 28	0.51 <u>0.42</u> 0.42 0.18 0.075	2001/1000946-X006206
France, Chevire 2001 (Golden Smothee)	4 × 0.2	NS	4 × 0.02	Up to BBCH 85	Whole fruit	0 8 14 20 28	0.42 0.38 0.35 <u>0.39</u> 0.20	2001/1015029-FBM/02/01
France, Verquires 2001 (Ozar Gold)	4 × 0.2-0.21	8 d	4 × 0.02	BBCH 77-85	Whole fruit	0 7 14 21 28	0.73 <u>0.65</u> 0.41 0.43 0.47	2001/1015046-X0106208
France, Verquires 2001 (Golden Delicious)	4 × 0.19-0.21	8	4 × 0.02	BBCH 77-	Whole fruit	0 7 14 21 28	0.60 <u>0.53</u> 0.51 0.35 0.35	2001/1015046-X0106209
France, Rottelsheim 2003 (Golden)	4 × 0.2 (SE)	7-8	4 × 0.02	Up to BBCH 85	Whole fruit	0 8 15 22 29	0.24 <u>0.32</u> 0.23 0.25 0.20	2004/1001291-FAN/18/03
	4 × 0.2 (WG)	7-8	4 × 0.02	Up to BBCH 85	Whole fruit	0 8 15 22 29	0.42 0.24 0.19 0.20 0.15	

Location, Year (variety)	Application				Residues, mg/kg			Report/Trial No., Reference, analytical method, validation data, storage period
	kg ai/ha	Interval days	kg ai/hL	Growth stage	Sample	DALA	Boscalid	
France, Bouloc 2003 (Star Krimson)	4 × 0.2 (SE)	7-8	4 × 0.02	Up to BBCH 87	Whole fruit	0 7 14 21 28	0.92 <u>0.86</u> 0.43 0.51 0.70	2004/1001291-FLT/15/03
	4 × 0.2 (WG)	7-8	4 × 0.02	Up to BBCH 87	Whole fruit	0 7 14 21 28	0.85 0.49 0.38 0.30 0.29	
Germany, Vehlefanz 2000 (Pinova)	4 × 0.2-0.21	7-8	4 × 0.02	BBCH 76-78	Whole fruit	0 6 14 21 28	0.3 <u>0.15</u> 0.14 0.14 0.11	2001/1006135-ACK/06/00
Germany, Stetten a.H. 2000 (Jonagold)	4 × 0.2	- 7 8 10	4 × 0.02	BBCH 77-81	Whole fruit	0 7 14 21 28	0.35 <u>0.36</u> 0.27 0.24 0.16	2001/1006135-DU2/12/00
Germany, Eschbach 2000 (Braeburn)	4 × 0.2-0.21	- 7 8 10	4 × 0.02	BBCH 77-81	Whole fruit	0 7 14 21 28	0.38 <u>0.32</u> 0.28 0.19 0.19	2001/1006135-DU4/11/00
Germany, Stetten a.H. 2001 (Golden Delicious)	4 × 0.2	NS	4 × 0.02	Up to BBCH 85	Whole fruit	0 7 14 21 27	0.81 <u>0.55</u> 0.52 0.41 0.47	2001/1015029-DU2/07/01
Germany, Vehlefanz 2003 (Piros)	4 × 0.2 (SE)	7-8	4 × 0.02	Up to BBCH 77	Whole fruit	0 8 15 21 28	0.37 <u>0.29</u> 0.16 0.16 0.17	2003/1001291-ACK/11/03
	4 × 0.2 (WG)	7-8	4 × 0.02	Up to BBCH 77	Whole fruit	0 8 15 21 28	0.23 0.14 0.13 0.11 0.08	
Italy, Ferrara 2000 (Red Chief)	4 × 0.2	7-9	4 × 0.02	BBCH 78-85	Whole fruit	0 7 13 20 27	0.36 <u>0.30</u> 0.19 0.20 0.22	2001/1000946-0025R
Italy, Forli 2000 (Royal Gala)	4 × 0.19-0.22	7-8	4 × 0.02	BBCH 78-85	Whole fruit	0 8 14 22 28	0.36 <u>0.29</u> 0.24 0.14 0.12	2001/1000946-0026R
Italy, Ferrara 2001 (Red Chief)	4 × 0.2	8	4 × 0.02	BBCH 75-85	Whole fruit	0 7 14 21 27	0.13 <u>0.24</u> 0.13 0.16 0.15	2001/1015046-0148R

Location, Year (variety)	Application				Residues, mg/kg			Report/Trial No., Reference, analytical method, validation data, storage period
	kg ai/ha	Inter-val days	kg ai/hL	Growth stage	Sample	DALA	Boscalid	
Italy, Argenta 2001 (Golden Delicious)	4 × 0.2	7-9	4 × 0.02	BBCH 77-81	Whole fruit	0 7 14 21 28	0.27 <u>0.20</u> 0.18 0.20 0.18	2001/1015046-0149R
Italy, Cesena 2001 (Royal Gala)	4 × 0.2-0.21	8	4 × 0.02	BBCH 72-77	Whole fruit	0 6 13 20 27	0.22 <u>0.19</u> 0.13 0.11 0.09	2001/1015046-0150R
Italy, Montemarcino 2003 (Golden Delicious)	4 × 0.2 (SE)	7-8	4 × 0.02	Up to BBCH 78	Whole fruit	0 7 15 21 28	0.46 0.35 0.39 0.32 0.16	2004/1001291-ITA/09/03
	4 × 0.2 (WG)	7-8	4 × 0.02	Up to BBCH 78	Whole fruit	0 7 15 21 28	0.55 <u>0.43</u> 0.17 0.20 0.17	
Netherlands, Groesbeek 2001 (Elstar)	4 × 0.2	NS	4 × 0.02	Up to BBCH 81	Whole fruit	0 8 13 21 29	0.24 <u>0.42</u> 0.25 0.26 0.15	2001/1015029-AGR/16/01

DALA: days after last application

NS: not stated

Table 19 Residues of boscalid in pears

Location, Year (variety)	Application				Residues, mg/kg			Report/Trial No., Reference, analytical method, validation data, storage period
	kg ai/ha	Inter-val days	kg ai/hL	Growth stage	Sample	DALA	Boscalid	
Southern America (cGAP: none)								
Argentina, Allen 2014/2015 (NS)	0.25	-	NS	NS	Whole fruit	1 7 15	0.34, 0.28, 0.25, 0.37 (0.31) 0.23, 0.053, 0.34, 0.28 (0.23) 0.29, 0.17, 0.15, 0.23 (0.21)	2016/3004402-G150153, BOSC19E_058 Method: L0076/09 Storage period: 12 months
Argentina, Vista Flores 2014/2015 (NS)	0.25	-	NS	NS	Whole fruit	1 7 15	0.31, 0.31,.035, 0.28 (0.31) 0.24, 0.22, 0.17, 0.19 (0.21) 0.24, 0.14, 0.2, 0.21 (0.2)	2016/3004402-G150154, BOSC19E_058 Method: L0076/09 Storage period: 12 months
Argentina, Tunuyán 2014/2015 (NS)	0.25	-	NS	NS	Whole fruit	1 7 15	0.2, 0.16, 0.17, 0.21 (0.18) 0.15, 0.22, 0.17, 0.17 (0.18) 0.15, 0.18, 0.15, 0.13 (0.15)	2016/3004402-G150155, BOSC19E_058 Method: L0076/09 Storage period: 12 months

Location, Year (variety)	Application				Residues, mg/kg			Report/Trial No., Reference, analytical method, validation data, storage period
	kg ai/ha	Inter-val days	kg ai/hL	Growth stage	Sample	DALA	Boscalid	
Argentina, Villa Regina 2014/2015 (NS)	0.25	-	NS	NS	Whole fruit	1 8 15	0.074, 0.071, 0.1, 0.13 (0.094) 0.078, 0.08, 0.11, 0.099 (0.092) 0.096, 0.09, 0.11, 0.08 (0.094)	2016/3004402-G150392, BOSC19E_058 Method: L0076/09 Storage period: 12 months
Argentina, Mainqué 2014/2015 (NS)	0.25	-	NS	NS	Whole fruit	1 8 15	0.17, 0.19, 0.22, 0.17 (0.19) 0.18, 0.21, 0.13, 0.14 (0.16) 0.12, 0.12, 0.089, 0.11 (0.11)	2016/3004402-G150393, BOSC19E_058 Method: L0076/09 Storage period: 12 months
Argentina, Tupungato 2014/2015 (NS)	0.25	-	NS	NS	Whole fruit	1 8 15	0.081, 0.09, 0.1, 0.2 (0.12) 0.14, 0.11, 0.14, 0.16 (0.14) 0.12, 0.082, 0.098, 0.12 (0.1)	2016/3004402-G150394, BOSC19E_058 Method: L0076/09 Storage period: 12 months
Europe (cGAP: CZ, 4 × 0.2 kg ai/ha, 8 day interval, 7 PHI)								
France, Orange 2014 (Guyot)	4 × 0.2	6-8	4 × 0.02	BBCH 74-78	Whole fruit	0 8 15 22	0.30 0.084 <u>0.086</u> 0.068	2016/1041500-L140650, BOSC19E_048 Method: L0076/01 Storage period:16 months
Germany, Heidesheim 2014 (Gräfin von Paris)	4 × 0.2	7	4 × 0.02	BBCH 79-85	Whole fruit	0 8 14 21	0.13 <u>0.11</u> 0.072 0.089	2016/1041500-L140646, BOSC19E_048 Method: L0076/01 Storage period: 16 months
Greece, Arseni 2014 (Krystali)	4 × 0.2	7	4 × 0.02	BBCH 76-81	Whole fruit	0 7 14 22	0.35 <u>0.29</u> 0.22 0.16	2016/1041500-L140651, BOSC19E_048 Method: L0076/01 Storage period: 16 months
Italy, Volpedo 2014 (Santa Maria)	4 × 0.2	7	4 × 0.02	BBCH 75-77	Whole fruit	0 8 14 20	0.31 0.14 <u>0.16</u> 0.094	2016/1041500-L140652, BOSC19E_048 Method: L0076/01 Storage period: 16 months
Netherlands, Gelderland 2014 (Doyenné du Comice)	4 × 0.2	6-8	4 × 0.02	BBCH 77-83	Whole fruit	0 8 15 22	0.71 <u>0.33</u> 0.33 0.26	2016/1041500-L140647, BOSC19E_048 Method: L0076/01 Storage period: 16 months
Poland, Dmosin 2014 (Konjerencja)	4 × 0.2	6-8	4 × 0.02	BBCH 78-85	Whole fruit	0 7 14 22	0.34 <u>0.39</u> 0.35 0.27	2016/1041500-L14050, BOSC19E_048 Method: L0076/01 Storage period: 16 months
Spain, Llambiles 2014 (Conference)	4 × 0.2	6-8	4 × 0.02	BBCH 77-81	Whole fruit	0 7 14 22	0.58 <u>0.48</u> 0.40 0.35	2016/1041500-L14048, BOSC19E_048 Method: L0076/01 Storage period: 16 months

Location, Year (variety)	Application				Residues, mg/kg			Report/Trial No., Reference, analytical method, validation data, storage period
	kg ai/ha	Inter-val days	kg ai/hL	Growth stage	Sample	DALA	Boscalid	
United Kingdom, Winchcombe 2014 (Conference)	4 × 0.2	6-8	4 × 0.02	BBCH 76-79	Whole fruit	0 7 14 21	0.97 1.3 0.76 0.78	2016/1041500-L14049, BOSC19E_048 Method: L0076/01 Storage period: 16 months
Northern America (cGAP: CZ, 4 × 0.33 kg ai/ha, 7 day interval, 0 d PHI)								
USA, Alton (NY) 2001 (Bartlett)	6 × 0.34	7	6 × 0.045	45mm to harvest	Whole fruit	0	0.57	2002/5002108-2001831, BOSC19E_048 Method: D9908 Storage period:7 months
		+ postharvest spray at 2000 mg/L			Whole fruit	0	2.7	
		+ postharvest dip at 2000 mg/L			Whole fruit	0	4.7	
		+ postharvest spray+dip at 2000 mg/L			Whole fruit	0	5.2	
		+ postharvest drench at 2000 mg/L			Whole fruit	0	2.7	
	6 × 0.34	7	6 × 0.024	45mm to harvest	Whole fruit	0	0.74	
		+ postharvest spray at 2000 mg/L			Whole fruit	0	2.6	
		+ postharvest dip at 2000 mg/L			Whole fruit	0	4.8	
		+ postharvest spray+dip at 2000 mg/L			Whole fruit	0	6.6	
		+ postharvest drench at 2000 mg/L			Whole fruit	0	3.1	
Canada, Berwick (Nova Scotia)	6 × 0.34-0.35	6-7	6 × 0.069-0.078	40mm to harvest	Whole fruit	0	1.9	2002/5002108-2001833, BOSC19E_048 Method: D9908
2001 (Clapps)	6 × 0.34-0.35	6-7	6 × 0.029-0.031	45mm to harvest	Whole fruit	0	1.3	Storage period:7 months
USA, Conklin (MI) 2001	6 × 0.34	7-8	6 × 0.041-0.045	50mm to harvest	Whole fruit	0	1.2	2002/5002108-2001837, BOSC19E_048 Method: D9908
(Bartlett)	6 × 0.34	7-8	6 × 0.017-0.019	50mm to harvest	Whole fruit	0	0.74	Storage period:7 months Last application: 28.08.2001
USA, Conklin (MI) 2001	6 × 0.34	7-8	6 × 0.046-0.05	50mm to harvest	Whole fruit	0	0.78	2002/5002108-2001838, BOSC19E_048 Method: D9908
(Bartlett)	6 × 0.34	7-8	6 × 0.019-0.021	50mm to harvest	Whole fruit	0	0.56	Storage period:7 months Last application: 28.08.2001
Canada, St. George (ON) 2001	6 × 0.33-0.35	6-7	6 × 0..071-0.072	70mm to harvest	Whole fruit	0	2.3	2002/5002108-2001839, BOSC19E_048 Method: D9908

Location, Year (variety)	Application				Residues, mg/kg			Report/Trial No., Reference, analytical method, validation data, storage period
	kg ai/ha	Inter-val days	kg ai/hL	Growth stage	Sample	DALA	Boscalid	
(Bosc)	6 × 0.34-0.35	6-7	6 × 0..024-0.025	70mm to harvest	Whole fruit	0	0.85	Storage period:7 months
USA, Yuba City (CA) 2001	6 × 0.34	6	6 × 0.078-0.08	Fruit dev. to harvest	Whole fruit	0	0.69	2002/5002108-2001940, BOSC19E_048 Method: D9908
(Bosc)	6 × 0.34	6	6 × 0.022-0.023	Fruit dev. to harvest	Whole fruit	0	1.7	Storage period:7 months
USA, Porterville (CA) 2001	6 × 0.33-0.34	7	6 × 0.062-0.064	Fruit dev. to harvest	Whole fruit	0	0.89	2002/5002108-2001941, BOSC19E_048 Method: D9908
(Bosc)		+ postharvest spray at 2000 mg/L			Whole fruit	0	4.7	Storage period:7 months
		+ postharvest dip at 2000 mg/L			Whole fruit	0	5.1	
		+ postharvest spray+dip at 2000 mg/L			Whole fruit	0	6.5	
		+ postharvest drench at 2000 mg/L			Whole fruit	0	6.6	
	6 × 0.34	7	6 × 0.011	Fruit dev. to harvest	Whole fruit	0	0.85	
		+ postharvest spray at 2000 mg/L			Whole fruit	0	3.9	
		+ postharvest dip at 2000 mg/L			Whole fruit	0	5.5	
		+ postharvest spray+dip at 2000 mg/L			Whole fruit	0	7.1	
		+ postharvest drench at 2000 mg/L			Whole fruit	0	4.8	
USA, Soap Lake (WA)	6 × 0.34	7	6 × 0.047	Fruit dev. to harvest	Whole fruit	0	0.83	2002/5002108-2001946, BOSC19E_048
2001 (Bartlett)		+ postharvest spray at 2000 mg/L			Whole fruit	0	2.5	Method: D9908 Storage period:7 months
		+ postharvest dip at 2000 mg/L			Whole fruit	0	2.4	
		+ postharvest spray+dip at 2000 mg/L			Whole fruit	0	3.7	
		+ postharvest drench at 2000 mg/L			Whole fruit	0	2.6	
	6 × 0.34	7	6 × 0.018	Fruit dev. to harvest	Whole fruit	0	0.87	
		+ postharvest spray at 2000 mg/L			Whole fruit	0	2.8	

Location, Year (variety)	Application				Residues, mg/kg			Report/Trial No., Reference, analytical method, validation data, storage period
	kg ai/ha	Inter-val days	kg ai/hL	Growth stage	Sample	DALA	Boscalid	
		+ postharvest dip at 2000 mg/L			Whole fruit	0	2.3	
		+ postharvest spray+dip at 2000 mg/L			Whole fruit	0	3.6	
		+ postharvest drench at 2000 mg/L			Whole fruit	0	2.6	
USA, Hood River (OR) 2001 (Starkrimson)	6 × 0.34-0.35	6-7	6 × 0.049-0.05	45mm to harvest	Whole fruit	0	0.54	2002/5002108-2001947, BOSC19E_048 Method: D9908 Storage period:7 months
	6 × 0.34	6-7	6 × 0.015-0.018	45mm to harvest	Whole fruit	0	0.65	
USA, Greenleaf (ID) 2001 (Bartlett)	6 × 0.33-0.35	6-8	6 × 0.049-0.053	55mm to harvest	Whole fruit	0	0.37	2002/5002108-2001948, BOSC19E_048 Method: D9908 Storage period:7 months
	6 × 0.33-0.35	6-8	6 × 0.011-0.012	55mm to harvest	Whole fruit	0	0.38	

DALA: days after last application

NS: not stated

Table 20 Residues of boscalid in cherries

Location, Year (variety)	Application				Residues, mg/kg			Report/Trial No., Reference, analytical method, validation data, storage period
	kg ai/ha	Inter-val days	kg ai/hL	Growth stage	Sample	DALA	Boscalid	
Europe (cGAP: Austria, 3 × 0.19 kg ai/ha, 10 day interval, 7 d PHI)								
Austria, Scharten 2016 (Regina)	0.26 0.26	5	0.052	BBCH 85	Whole fruit	0 3 7 14	0.21 0.22 0.19 0.14	2017/1000803-L160240 BOSC19E_070 Method: L0076/01 Storage period: 9 months
Denmark, Nyberg 1999 (Kellerils 16)	5 × 0.2	20 56 14 14	5 × 0.02	BBCH 60-85	Whole fruit	0 3 8 15	1.4 0.70 0.54 0.54	2001/1006132-ALB/08/99 BOSC19E_063 Method: 445/0 Storage period: 11 months
Denmark, Arslev 1999 (Knuthenborg)	5 × 0.2	12 28 15 14	5 × 0.02	BBCH 61-85	Whole fruit	0 3 7 13	0.32 0.23 0.18 0.22	2001/1006132-ALC/09/99 BOSC19E_063 Method: 445/0 Storage period: 11 months
Denmark, Fuenen 2003 (Adriana)	3 × 0.2	14	3 × 0.02	Up to BBCH 87	Whole fruit	0 3 7	0.57 0.57 0.37	2004/1010551-ALB/19/03 BOSC19E_066 Method: 445/0 Storage period: 6 months
Denmark, Otterup 2004 (Stævnsbaer)	3 × 0.2	14	3 × 0.02	Up to BBCH 87	Whole fruit	0 3 7	0.54 0.21 0.16	2005/1004972-ALB/11/04 BOSC19E_067 Method: 445/0 Storage period: 3 months

Location, Year (variety)	Application				Residues, mg/kg			Report/Trial No., Reference, analytical method, validation data, storage period
	kg ai/ha	Inter-val days	kg ai/hL	Growth stage	Sample	DALA	Boscalid	
France, Marmande 1999 (Gros Gain)	4 × 0.2	28 15 13	4 × 0.018-0.022	BBCH 67-82	Whole fruit	0 3 6 13	0.48 0.32 0.25 0.31	2001/1000934-X996204 BOSC19E_062 Method: 445/0 Storage period: 12 months
France, La Berthonniére (Noire de Meched)	5 × 0.2	15 28 14 14	5 × 0.022	BBCH 60-85	Whole fruit	0 3 7 14	0.2 0.16 0.16 0.12	2001/1000934-X996205 BOSC19E_062 Method: 445/0 Storage period: 12 months Sampling size only 0.85 kg for DALA 3 and 7.
France, Coulanges la Vineuse 2000 (Belle de Juillet)	5 × 0.2-0.21	22 42 14 14	5 × 0.029	BBCH 60-87	Whole fruit	0 3 7 14	0.30 0.19 0.089 0.073	2001/1009061-BSF/620-1 BOSC19E_065 Method: 445/0 Storage period: 8 months
France, Jussy 2000 (Marmotte)	5 × 0.2-0.21	29 14 13 14	5 × 0.025	BBCH 62-85	Whole fruit	0 3 7 14	0.21 0.21 0.18 0.13	2001/1009061-BSF/620-2 BOSC19E_065 Method: 445/0 Storage period: 8 months
France, Beauvoison 2000 (Les Brunots)	5 × 0.2-0.21	18 7 17 11	5 × 0.04	BBCH 61-85	Whole fruit	0 3 7 14	0.13 0.069 0.1 0.087	2001/1009061-BSF/620-3 BOSC19E_065 Method: 445/0 Storage period: 8 months
France, Buis les Barronies 2000 (Burlat)	5 × 0.2-0.21	11 7 17 11	5 × 0.04	BBCH 65-85	Whole fruit	0 3 7 14	0.14 0.12 0.17 0.17	2001/1009061-BSF/620-4 BOSC19E_065 Method: 445/0 Storage period: 8 months
France, Verges de Souzay 2003 (Montmorency)	3 × 0.2	14	3 × 0.02	Up to BBCH 85	Whole fruit	0 3 7	0.5 0.29 0.47	2004/1010551-FBM/17/03 BOSC19E_066 Method: 445/0 Storage period: 6 months
France, La Bouscasse 2003 (Duroni)	3 × 0.2	14	3 × 0.02	Up to BBCH 89	Whole fruit	0 3 7	<0.05 0.09 0.14	2004/1010551-FTL/22/03 BOSC19E_066 Method: 445/0 Storage period: 6 months
France, Malijacs 2003 (Regnier)	3 × 0.2	14	3 × 0.02	Up to BBCH 85	Whole fruit	0 4 8	0.85 1.5 1.3	2004/1010551-FBD/16/03 BOSC19E_066 Method: 445/0 Storage period: 6 months
France, Traenheim 2004 (Regina)	3 × 0.2	14	3 × 0.02	Up to BBCH 85	Whole fruit	0 3 7	0.068 <0.05 <0.05	2005/1004972-FAN/16/04 BOSC19E_067 Method: 445/0 Storage period: 5 months
France, Lapalud 2004 (Régnier)	3 × 0.2	14	3 × 0.02	Up to BBCH 87	Whole fruit	0 4 7	0.19 0.18 0.22	2005/1004972-FBD/17/04 BOSC19E_067 Method: 445/0 Storage period: 6 months
France, La Bouscasse 2004 (Regina)	3 × 0.2	14	3 × 0.02	Up to BBCH 87	Whole fruit	0 4 8	0.095 0.072 0.052	2005/1004972-FTL/17/04 BOSC19E_067 Method: 445/0 Storage period: 5 months

Location, Year (variety)	Application				Residues, mg/kg			Report/Trial No., Reference, analytical method, validation data, storage period
	kg ai/ha	Inter-val days	kg ai/hL	Growth stage	Sample	DALA	Boscalid	
Germany, Wesendahl 1999 (Karina)	5 × 0.2	17 28 13 14	5 × 0.02	BBCH 60-87	Whole fruit	0 2 7 13	0.24 0.26 0.14 0.18	2001/1006132-ACK/04/99 BOSC19E_063 Method: 445/0 Storage period: 11 months
Germany, Vehlefanz 1999 (K27 Kellerils)	5 × 0.2	14 28 13 14	5 × 0.02	BBCH 60-87	Whole fruit	0 3 7 13	0.65 0.34 0.43 0.30	2001/1006132-ACK/05/99 BOSC19E_063 Method: 445/0 Storage period: 11 months
Germany, Rödersheim-Gronau 1999 (Scheiders Späte Knorbel)	5 × 0.2	28 7 8 20	5 × 0.02	BBCH 60-85	Whole fruit	0 2 6 12	0.37 0.42 0.24 0.32	2001/1006132-DU4/03/99 BOSC19E_063 Method: 445/0 Storage period: 11 months
Germany, Vehlefanz 2000 (Schattenmorelle)	5 × 0.2	7 1 13 14	5 × 0.02	BBCH 60-87	Whole fruit	0 3 7 14	0.75 0.56 0.53 0.39	2001/1006133-ACK/05/00 BOSC19E_064 Method: 445/0 Storage period: 7 months
Germany, Vehlefanz 2003 (K27 Kelores)	3 × 0.2	14	3 × 0.02	Up to BBCH 89	Whole fruit	0 3 7	1.1 0.63 0.7	2004/1010551-ACK/20/03 BOSC19E_066 Method: 445/0 Storage period: 6 months
Germany, Horrenberg 2004 (Geisenheimer Schwarz)	3 × 0.2	14	3 × 0.02	Up to BBCH 87	Whole fruit	0 3 7	0.16 0.15 0.088	2005/1004972-DU2/10/04 BOSC19E_067 Method: 445/0 Storage period: 5 months
Germany, Algesheim 2015 (Hedelfinger)	0.27 0.27	6	0.054	BBCH 85	Whole fruit	0 3 7 14	0.63 0.47 0.37 0.081	2016/1000745-L150126 BOSC19E_069 Method: L0076/01 Storage period: 3 months Last application: 23.06.2015
Germany, Algesheim 2015 (Schattenmorelle)	0.27 0.27	5	0.054	BBCH 85	Whole fruit	0 3 7 14	1.9 0.76 0.63 0.54	2016/1000745-L150128 BOSC19E_069 Method: L0076/01 Storage period: 4 months Last application: 07.07.2015
Germany, Algesheim 2016 (Schattenmorelle)	0.26 0.26	5	0.052	BBCH 85	Whole fruit	0 3 7 14	1.0 0.88 0.88 0.58	2017/1000803-L160242 BOSC19E_070 Method: L0076/01 Storage period: 9 months
Hungary, Lovasbereny 2016 (Úlfehétóifürtös)	0.26 0.26	5	0.052	BBCH 87	Whole fruit	0 3 7 13	1.0 0.99 0.54 0.49	2017/1000803-L160243 BOSC19E_070 Method: L0076/01 Storage period: 9 months
Italy, Modena-Vignola 1999 (Silvia)	5 × 0.2	12 28 13 14	5 × 0.022	BBCH 62-90	Whole fruit	0 3 7 13	0.54 0.25 0.37 0.23	2001/1000934-9936R BOSC19E_062 Method: 445/0 Storage period: 12 months
Italy, Modena-Savignano 1999 (Marasca di Vignola)	5 × 0.2	7 28 14 13	5 × 0.022	BBCH 60-88	Whole fruit	0 3 7 14	0.9 0.44 0.29 0.11	2001/1000934-9937R BOSC19E_062 Method: 445/0 Storage period: 12 months

Location, Year (variety)	Application				Residues, mg/kg			Report/Trial No., Reference, analytical method, validation data, storage period
	kg ai/ha	Interval days	kg ai/hL	Growth stage	Sample	DALA	Boscalid	
Italy, Nonantola 2000 (Montmercy)	5 × 0.2	7 d 28 14 13	5 × 0.027	BBCH 65-85	Whole fruit	0 3 7 14	0.48 0.59 0.32 0.36	2001/1009061-BSF/620-4 BOSC19E_065 Method: 445/0 Storage period: 8 months
Italy, Solignano 2000 (Pissei)	5 × 0.2	7 28 14 13	5 × 0.025	BBCH 65-89	Whole fruit	0 3 7 14	0.69 0.79 0.69 0.82	2001/1009061-BSF/620-5 BOSC19E_065 Method: 445/0 Storage period: 8 months
Italy, Pecetto Torinese 2003 (Amarisa)	3 × 0.2	14	3 × 0.02	Up to BBCH 87	Whole fruit	0 2 8	0.75 0.43 0.36	2004/1010551-ITA/16/03 BOSC19E_066 Method: 445/0 Storage period: 6 months
Italy, Garbagne 2003 (Sweet Heart)	3 × 0.2	14	3 × 0.02	Up to BBCH 81	Whole fruit	0 2 7	2.5 0.66 0.66	2004/1010551-ITA/17/03 BOSC19E_066 Method: 445/0 Storage period: 5 months
Italy, Pavis 2004 (Nero secondo)	3 × 0.2	14	3 × 0.02	Up to BBCH 87	Whole fruit	0 3 8	0.19 0.13 0.096	2005/1004972-ITA/16/04 BOSC19E_067 Method: 445/0 Storage period: 6 months
Netherlands, Ressen 2015 (Lapins)	0.27 0.27	5	0.054	BBCH 85	Whole fruit	0 3 7 14	0.34 0.17 0.073 0.08	2016/1000745-L150127 BOSC19E_069 Method: L0076/01 Storage period: 3 months
Netherlands, Ressen 2016 (Regina)	0.26 0.26	5	0.052	BBCH 87	Whole fruit	0 3 7 14	0.83 0.43 0.27 0.27	2017/1000803-L160241 BOSC19E_070 Method: L0076/01 Storage period: 9 months
Poland, Wronki 2015 (Lutowka)	0.27 0.27	5	0.054	BBCH 81	Whole fruit	0 3 8 15	0.35 0.27 0.21 0.10	2016/1000745-L150129 BOSC19E_069 Method: L0076/01 Storage period: 3 months
Sweden, Sjöbo 2000 (Regina)	5 × 0.2-0.22	14 28 15 15	5 × 0.02-0.022	BBCH 60-87	Whole fruit	0 4 7 14	0.36 0.23 0.2 0.18	2001/1006133-HUS/03/00 BOSC19E_064 Method: 445/0 Storage period: 7 months
Sweden, Sjöbo 2003 (Karina)	3 × 0.2	14	3 × 0.02	Up to BBCH 87	Whole fruit	0 3 7	0.84 0.32 0.39	2004/1010551-HUS/12/03 BOSC19E_066 Method: 445/0 Storage period: 6 months
Sweden, Orelund 2004 (Stevnsbar)	3 × 0.2	14	3 × 0.02	Up to BBCH 85	Whole fruit	0 3 7	0.18 0.21 0.14	2005/1004972-HUS/06/04 BOSC19E_067 Method: 445/0 Storage period: 3 months
Sweden, Malmoe 2004 (Van)	3 × 0.2	14	3 × 0.02	Up to BBCH 81	Whole fruit	0 4 8	<0.05 <0.05 <0.05	2005/1004972-HUS/07/04 BOSC19E_067 Method: 445/0 Storage period: 4 months
Northern America (cGAP: USA 5 × 0.26 kg ai/ha, 7 day interval, 0 d PHI)								

Location, Year (variety)	Application				Residues, mg/kg			Report/Trial No., Reference, analytical method, validation data, storage period
	kg ai/ha	Inter-val days	kg ai/hL	Growth stage	Sample	DALA	Boscalid	
USA, Conklin (MI) 2007 (Napoleon)	5 × 0.26	6-8	5 × 0.018-0.14	Up to BBCH 87	Whole fruit	0 1	1.6, 1.5 (1.6) 1.1, 0.98 (1.0)	2007/7013460-R070178, BOSC19E_061 Method: D9908 Storage period: 7 months Last application: 28.06.2007
USA, Conklin (MI) 2007 (Montmorency)	5 × 0.26	6-8	5 × 0.018-0.14	Up to BBCH 87	Whole fruit	0 1	1.3, 1.5 (1.4) 0.96, 1.4 (1.2)	2007/7013460-R070183, BOSC19E_061 Method: D9908 Storage period: 7 months Last application: 04.07.2007
USA, Plainview (CA) 2007 (Tulare)	5 × 0.26	6-9	5 × 0.018-0.14	Up to BBCH 85	Whole fruit	0 1	2.5, 2.8 (2.6) 2.2, 2.7 (2.4)	2007/7013460-R070179, BOSC19E_061 Method: D9908 Storage period: 7 months
USA, Marysville (CA) 2007 (Lapin)	5 × 0.26	6-7	5 × 0.018-0.14	Up to BBCH 85	Whole fruit	0 1 5 10	0.06, <0.05 (0.055) <0.05, <0.05 (<0.05) <0.05, <0.05 (<0.05) <0.05, <0.05 (<0.05)	2007/7013460-R070180, BOSC19E_061 Method: D9908 Storage period: 7 months
USA, Ephrata (WA) 2007 (Bing)	5 × 0.26	6-7	5 × 0.018-0.14	Up to BBCH 87	Whole fruit	0 1	1.4, 1.0 (1.2) 0.92, 0.93 (0.92)	2007/7013460-R070181, BOSC19E_061 Method: D9908 Storage period: 7 months
Canada, Pelham (ON) 2007 (Montmorency)	5 × 0.26	7	5 × 0.018-0.14	Up to BBCH 89	Whole fruit	0 1	2.3, 3.0 (2.6) 1.9, 2.6 (2.3)	2007/7013460-R070182, BOSC19E_061 Method: D9908 Storage period: 7 months
USA, Karman (CA) 2007 (Brooks)	5 × 0.26	6-7 d	5 × 0.018-0.14	Up to BBCH 87	Whole fruit	0 1	1.8, 1.2 (1.5) 1.1, 1.1 (1.1)	2007/7013460-R070184, BOSC19E_061 Method: D9908 Storage period: 7 months
USA, Fennville (MI) 2002 (Montmorency)	0.046 g/kg	-	-	-	post-harvest spray	0	3.4, 3.5 (3.4)	2005/7004639-02-MI38 BOSC19E_068 Method: D9908 Storage period: 4 months
USA, Stockton (CA) 2002 (Bing)	0.046 g/kg	-	-	-	post-harvest spray	0	4.1, 4.9 (4.5)	2005/7004639-02-CA119 BOSC19E_068 Method: D9908 Storage period: 4 months
	0.067 kg/l	-	-	-	Post-harvest dip with wax	0	3.6, 5.0 (4.3)	
USA, Prosser (WA) 2002 (Bing)	0.046 g/kg	-	-	-	post-harvest spray	0	6.6, 7.4 (7.0)	2005/7004639-02-WA47 BOSC19E_068 Method: D9908 Storage period: 4 months

DALA: days after last application

Table 21 Summary information on residues of boscalid in cherries (reported in the 2006 JMPR Evaluation)

Location, Year (variety)	Application				Residues, mg/kg			Report/Trial No., Reference, analytical method, validation data, storage period
	kg ai/ha	Inter-val days	kg ai/hL	Growth stage	Sample	DALA	Boscalid	
Northern America (cGAP: USA 5 × 0.26 kg ai/ha, 7 day interval, 0 d PHI)								
USA, North Rose (NY)	5 × 0.26	6-8	5 × 0.035	15mm to harvest	Whole fruit	0	1.6	2001/5000831-99101
1999 (Montmorency)	5 × 0.26	6-8	5 × 0.019	15mm to harvest	Whole fruit	0	1.4	
USA, Conklin (MI)	5 × 0.26	7	5 × 0.043	13mm to harvest	Whole fruit	0	1.3	2001/5000831-99102
1999 (Montmorency)	5 × 0.26	7	5 × 0.014	13mm to harvest	Whole fruit	0	1.5	
USA, Conklin (MI) 1999	5 × 0.26	7	5 × 0.037	30% red colour to harvest	Whole fruit	0	0.76	2001/5000831-99104
(Sommerset)	5 × 0.26	7	5 × 0.013	30% red colour to harvest	Whole fruit	0	0.74	
USA, Casnovia (MI)	5 × 0.26	7	5 × 0.043	13mm to harvest	Whole fruit	0	1.1	2001/5000831-99103
1999 (Montmorency)	5 × 0.26	7	5 × 0.014	13mm to harvest	Whole fruit	0	1.2	
USA, Poplar (CA) 1999	5 × 0.26	7	5 × 0.045	Fruit matur. to harvest	Whole fruit	0	0.64	2001/5000831-99105
(Brooks)	5 × 0.26	7	5 × 0.011	Fruit matur. to harvest	Whole fruit	0	1.0	
USA, Ephrata (WA)	5 × 0.26	7	5 × 0.055	10mm to harvest	Whole fruit	0	0.91	2001/5000831-99106
1999 (Bing)	5 × 0.26	7	5 × 0.013	10mm to harvest	Whole fruit	0	1.5	
USA, Ephrata (WA) 2004 (Bing)	6 × 0.26	7-8	5 × 0.033	50% final size to mature fruit	Whole fruit	0	1.7, 1.3 (1.5)	2005/5000024-RCN2004142

DALA: days after last application

Table 22 Residues of boscalid in peaches

Location, Year (variety)	Application				Residues, mg/kg			Report/Trial No., Reference, analytical method, validation data, storage period
	kg ai/ha	Inter-val days	kg ai/hL	Growth stage	Sample	DALA	Boscalid	
Europe (cGAP: Austria, 3 × 0.19 kg ai/ha, 10 d interval, 7 day PHI)								
France, Le Beugnon 1999 (Hale-Haven)	4 × 0.2	84 14 16	4 × 0.022	BBCH 69-81	Whole fruit	0 4 7 14	0.13 0.05 0.05 <0.05	2001/1000934-X996206 BOSC19E_062 Method: 445/0 Storage period: 12 months

Location, Year (variety)	Application				Residues, mg/kg			Report/Trial No., Reference, analytical method, validation data, storage period
	kg ai/ha	Inter-val days	kg ai/hL	Growth stage	Sample	DALA	Boscalid	
France, Equarrans 1999 (Katia)	4 × 0.2	56 14 14	4 × 0.022	BBCH 69-85	Whole fruit	0 3 7 14	0.32 0.32 0.17 0.13	2001/1000934-X996207 BOSC19E_062 Method: 445/0 Storage period: 12 months
Germany, Horrenberg 1999 (South Haven)	5 × 0.2	27 56 14 14	5 × 0.02	BBCH 60-78	Whole fruit	0 2 7 14	0.33 0.28 0.29 0.20	2001/1006132-DU2/08/99 BOSC19E_063 Method: 445/0 Storage period: 11 months
Germany, Durlach 1999 (Red Top)	5 × 0.2	20 56 14 14	5 × 0.02	BBCH 60-85	Whole fruit	0 4 7	0.46 0.23 0.15	2001/1006132-DU2/09/99 BOSC19E_063 Method: 445/0 Storage period: 11 months
Germany, Rödersheim-Gronau 1999 (Red Haven)	5 × 0.2	26 70 13 14	5 × 0.02	BBCH 60-85	Whole fruit	0 3 7 14	0.51 0.84 0.35 0.17	2001/1006132-DU4/02/99 BOSC19E_063 Method: 445/0 Storage period: 11 months
Italy, Ravenna 1999 (Fayette)	4 × 0.2	3 mo 14 d 13 d	4 × 0.022	BBCH 69-87	Whole fruit	0 3 6 13	0.29 0.49 0.21 0.12	2001/1000934-9931R BOSC19E_062 Method: 445/0 Storage period: 12 months
Italy, Ferrara 1999 (Duchessa dèste)	5 × 0.2	13 84 14 13	5 × 0.022	BBCH 60-86	Whole fruit	0 3 6 13	0.4 0.38 0.11 0.21	2001/1000934-9932R BOSC19E_062 Method: 445/0 Storage period: 12 months
Italy, Modena 1999 (Red Haven)	5 × 0.2	12 56 13 16	5 × 0.022	BBCH 61-79	Whole fruit	0 2 6 13	0.47 0.38 0.35 0.23	2001/1000934-9933R BOSC19E_062 Method: 445/0 Storage period: 12 months

Location, Year (variety)	Application				Residues, mg/kg			Report/Trial No., Reference, analytical method, validation data, storage period
	kg ai/ha	Inter-val days	kg ai/hL	Growth stage	Sample	DALA	Boscalid	
Northern America (cGAP: USA 5 × 0.26 kg ai/ha, 7 day interval, 0 d PHI)								
USA, Alton (NY) 2007 (Gold Nine)	5 × 0.26	6-8	5 × 0.018-0.14	Up to BBCH 89	Whole fruit	0 1	2.8, 3.8 (3.3) 3.9, 3.4 (<u>3.6</u>)	2007/7013460-R070186, BOSC19E_061 Method: D9908 Storage period: 7 months
USA, Chula (GO) 2007 (June Gold)	5 × 0.26	7	5 × 0.018-0.14	Up to BBCH 87	Whole fruit	0 1 5 10	0.9, 0.92 (0.91) 1.1, 1.0 (<u>1.0</u>) 0.66, 0.69 (0.68) 0.57, 0.4 (0.48)	2007/7013460-R070187, BOSC19E_061 Method: D9908 Storage period: 7 months
USA, Montezuma (GO) 2007 (MarQueen)	5 × 0.26	6-8	5 × 0.018-0.14	Up to BBCH 89	Whole fruit	0 1	0.58, 0.7 (<u>0.64</u>) 0.46, 0.55 (0.5)	2007/7013460-R070188, BOSC19E_061 Method: D9908 Storage period: 7 months
USA, Conklin (MI) 2007	5 × 0.26	7	5 × 0.018-0.14	Up to BBCH 85	Whole fruit	0 1	0.59, 0.73 (0.66) 0.68, 0.74 (<u>0.71</u>)	2007/7013460-R070190, BOSC19E_061 Method: D9908

Location, Year (variety)	Application				Residues, mg/kg			Report/Trial No., Reference, analytical method, validation data, storage period
	kg ai/ha	Inter-val days	kg ai/hL	Growth stage	Sample	DALA	Boscalid	
(Bellaire)								Storage period: 7 months
USA, Ada (OK) 2007 (Contender)	5 × 0.26	6-7	5 × 0.018-0.14	Up to BBCH 89	Whole fruit	0 1	0.64, 0.56 (0.6) 0.56, 0.5 (0.53)	2007/7013460-R070189, BOSC19E_061 Method: D9908 Storage period: 7 months
USA, Madera (CA) 2007 (Rayson)	5 × 0.26	7	5 × 0.018-0.14	Up to BBCH 87	Whole fruit	0 1	0.78, 0.8 (0.79) 0.75, 0.79 (0.77)	2007/7013460-R070191, BOSC19E_061 Method: D9908 Storage period: 7 months
USA, Jackson Springs (NC) 2002 (Contender)	0.002 8 g/kg	-	-	-	post-harvest spray	0	1.9, 2.1 (2.0)	2005/7004639-02-NC24 BOSC19E_068 Method: D9908 Storage period: 4 months
	0.067 kg/l	-	-	-	Post-harvest dip with wax	0	3.7, 3.5 (3.6)	
USA, Parlier (CA) 2002 (Elegant Lady)	0.002 8 g/kg	-	-	-	post-harvest spray (high vol.)	0	3.1, 2.5 (2.8)	2005/7004639-02-CA116 BOSC19E_068 Method: D9908 Storage period: 4 months
	0.002 8 g/kg	-	-	-	post-harvest spray (low vol.)	0	5.8, 6.5 (6.2)	
	0.067 kg/l	-	-	-	Post-harvest dip with wax	0	6.7, 7.7 (7.2)	
USA, Bridgeton (NJ) 2002 (Dine Red)	0.067 kg/l	-	-	-	Post-harvest dip with wax	0	5.5, 4.8, 4.8, 9.7, 7.1, 6.6 (6.4)	2005/7004639-02-NJ36 BOSC19E_068 Method: D9908 Storage period: 4 months

DALA: days after last application

Table 23 Summary information on residues of boscalid in peaches (reported in the 2006 JMPR Evaluation)

Location, Year (variety)	Application				Residues, mg/kg			Report/Trial No.
	kg ai/ha	Inter-val	kg ai/hL	Growth stage	Sample	DALA	Boscalid	
Northern America Northern America (cGAP: USA 5 × 0.26 kg ai/ha, 7 day interval, 0 d PHI)								
USA, Hereford (PA)	5 × 0.26	7	5 × 0.051	45mm to harvest	Whole fruit	0	0.66	2001/5000831-99107
1999 (Red Haven)	5 × 0.26	7	5 × 0.013	45mm to harvest	Whole fruit	0	0.75	
USA, Monetta (SC)	5 × 0.26	7-8	5 × 0.053	35mm to harvest	Whole fruit	0	0.16	2001/5000831-99108
1999 (Contender)	5 × 0.26	7-8	5 × 0.013	35mm to harvest	Whole fruit	0	0.19	

Location, Year (variety)	Application				Residues, mg/kg			Report/Trial No.
	kg ai/ha	Inter-val	kg ai/hL	Growth stage	Sample	DALA	Boscalid	
USA, Winterville (GA)	5 × 0.26	6-7	5 × 0.043	35mm to harvest	Whole fruit	0	0.4	2001/5000831-99109
1999 (Harmony)	5 × 0.26	6-7	5 × 0.021	35mm to harvest	Whole fruit	0	<u>0.42</u>	
USA, Tifton (GA) 1999 (June Gold)	5 × 0.26	7	5 × 0.051	25mm to harvest	Whole fruit	0 7 14 21 28	<u>0.49</u> 0.32 0.21 0.13 0.15	2001/5000831-99110
	5 × 0.26	7	5 × 0.01	25mm to harvest	Whole fruit	0 7 14 21 28	0.48 0.21 0.21 0.14 0.25	
USA, Conklin (MI)	5 × 0.26	7	5 × 0.041	45mm to harvest	Whole fruit	0	<u>0.4</u>	2001/5000831-99111
1999 (Red Haven)	5 × 0.26	7	5 × 0.014	45mm to harvest	Whole fruit	0	0.33	
USA, Vernon (TX)	5 × 0.26	7	5 × 0.051	50mm to harvest	Whole fruit	0	0.64	2001/5000831-99112
1999 (Lauring)	5 × 0.26	7	5 × 0.024	50mm to harvest	Whole fruit	0	<u>0.73</u>	
USA, Porterville (CA) 1999 (Red Sun)	5 × 0.26	- 7 2 11 7	5 × 0.037	50mm to harvest	Whole fruit	0	<u>0.52</u>	2001/5000831-99113
	5 × 0.26	- 7 2 11 7	5 × 0.01	50mm to harvest	Whole fruit	0	0.49	
USA, Selma (CA)	5 × 0.26	6-8	5 × 0.028	Full size to harvest	Whole fruit	0	<u>0.48</u>	2001/5000831-99114
1999 (September Sun)	5 × 0.26	6-8	5 × 0.014	Full size to harvest	Whole fruit	0	0.19	
USA, Gridley (CA)	5 × 0.26	7	5 × 0.05	Fruit matur. to harvest	Whole fruit	0	<u>0.32</u>	2001/5000831-99115
1999 (Loadel)	5 × 0.26	7	5 × 0.021	Fruit matur. to harvest	Whole fruit	0	0.32	
USA, Ephrata (WA) 2004 (Snow King)	5 × 0.26	7	5 × 0.036	60% final size to advanced coloring	Whole fruit	0	1.2, 1.2 (<u>1.2</u>)	2005/5000024-RCN2004134
USA, Carlyle (IL) 2004	5 × 0.26	6-7	5 × 0.033	60% final size to ripe	Whole fruit	0	0.51, 0.47 (<u>0.49</u>)	2005/5000024-RCN2004135

Location, Year (variety)	Application				Residues, mg/kg			Report/Trial No.
	kg ai/ha	Inter-val	kg ai/hL	Growth stage	Sample	DALA	Boscalid	
(Cresthaven)				for picking				
Canada, Branchton (ON) 2004 (Red Haven)	5 × 0.26	7	5 × 0.031	60% final size to ripe for picking	Whole fruit	0	0.51, 0.72 (0.60)	2005/5000024-RCN2004136
USA, Nodine (MN) 2004 (Bailey Hardy)	5 × 0.26	7	5 × 0.027	70% final size to ripe for picking	Whole fruit	0	0.82, 0.75 (0.78)	2005/5000024-RCN2004137

DALA: days after last application

Table 24 Residues of boscalid in plums

Location, Year (variety)	Application				Residues, mg/kg			Report/Trial No., Reference, analytical method, validation data, storage period
	kg ai/ha	Inter-val days	kg ai/hL	Growth stage	Sample	DALA	Boscalid	
Europe (cGAP: Austria, 3 × 0.19 kg ai/ha, 10 d interval, 7 day PHI)								
Denmark, Arslev 1999 (Oullins)	5 × 0.2	20 56 14 14	5 × 0.02	BBCH 60-85	Whole fruit	0 3 7 14	0.15 0.11 0.074 0.15	2001/1006132-ALB/17/99 BOSC19E_063 Method: 445/0 Storage period: 11 months
France, Le Puy 1999 (707 GF 81)	4 × 0.2	56 11 14	4 × 0.02	BBCH 69-81	Whole fruit	0 3 6 14	0.26 0.15 0.15 0.23	2001/1000934-X996203 BOSC19E_062 Method: 445/0 Storage period: 12 months
Germany, Perleberg 1999 (Späte Anna)	5 × 0.2	6 84 14 14	5 × 0.02	BBCH 61-85	Whole fruit	0 3 7 14	0.064 0.061 0.047 0.07	2001/1006132-ACK/07/99 BOSC19E_063 Method: 445/0 Storage period: 11 months
Germany, Horrenberg 1999 (Stanley)	5 × 0.2	20 84 13 15	5 × 0.02	BBCH 60-85	Whole fruit	0 3 7 14	0.34 0.31 0.45 0.23	2001/1006132-DU2/12/99 BOSC19E_063 Method: 445/0 Storage period: 11 months
Germany, Rödersheim-Gronau 1999 (St. Hubertus)	5 × 0.2	20 42 14 14	5 × 0.02	BBCH 60-85	Whole fruit	0 4 8	0.26 0.19 0.13	2001/1006132-DU4/09/99 BOSC19E_063 Method: 445/0 Storage period: 11 months
Germany, Limburgerhof 2000 (Stanley)	5 × 0.2-0.21	9 70 13 15	5 × 0.02	BBCH 60-85	Whole fruit	0 3 7 14	0.37 0.32 0.27 0.19	2001/1006133-DU2/08/00 BOSC19E_064 Method: 445/0 Storage period: 7 months
Germany, Limburgerhof 2000 (St. Hubertus)	5 × 0.2	19 28 14 13	5 × 0.02	BBCH 60-81	Whole fruit	0 3 7 14	0.092 0.074 0.057 0.053	2001/1006133-DU4/07/00 BOSC19E_064 Method: 445/0 Storage period: 7 months
Italy, Bologna 1999 (Empress)	5 × 0.2	12 84 14	5 × 0.022	BBCH 61-86	Whole fruit	0 3 6	0.22 0.56 0.18	2001/1000934-9934R BOSC19E_062 Method: 445/0

Location, Year (variety)	Application				Residues, mg/kg			Report/Trial No., Reference, analytical method, validation data, storage period
	kg ai/ha	Inter-val days	kg ai/hL	Growth stage	Sample	DALA	Boscalid	
		14				13	0.11	Storage period: 12 months
Italy, Ravenna 1999 (President)	5 × 0.2	12 56 15 13	5 × 0.022	BBCH 61-82	Whole fruit	0 2 7 15	0.14 0.11 0.08 0.06	2001/1000934-9935R BOSC19E_062 Method: 445/0 Storage period: 12 months
Sweden, Bjärred 2000 (Victoria)	5 × 0.2- 0.22	11 28 15 16	5 × 0.02	BBCH 60-85	Whole fruit	0 3 7 14	0.23 0.17 0.11 0.1	2001/1006133-HUS/04/00 BOSC19E_064 Method: 445/0 Storage period: 7 months
Northern America (cGAP: USA 5 × 0.26 kg ai/ha, 7 day interval, 0 d PHI)								
USA, Orland (CA) 2007 (French)	5 × 0.26	7-8	5 × 0.018- 0.14	Up to BBCH 87	Whole fruit	0 1	0.15, 0.1 (0.12) 0.1, 0.06 (0.08)	2007/7013460-R070193, BOSC19E_061 Method: D9908 Storage period: 7 months
USA, Lindsey (CA) 2007 (Angeleno)	5 × 0.26	7	5 × 0.018- 0.14	Up to BBCH 87	Whole fruit	0 1	0.62, 0.59 (0.6) 0.55, 0.57 (0.56)	2007/7013460-R070194, BOSC19E_061 Method: D9908 Storage period: 7 months
USA, Fresno (CA) 2007 (Howard Sun)	5 × 0.26	7	5 × 0.018- 0.14	Up to BBCH 87	Whole fruit	0 1	<0.05, <0.05 (<0.05) <0.05, <0.05 (<0.05)	2007/7013460-R070195, BOSC19E_061 Method: D9908 Storage period: 7 months Last application: 02.08.2007
USA, Fresno (CA) 2007 (Flavor Rich)	5 × 0.26	7	5 × 0.018- 0.14	Up to BBCH 87	Whole fruit	0 1 5 10	0.13, 0.13 (0.13) 0.11, 0.08 (0.095) 0.11, 0.08 (0.095) 0.06, 0.06 (0.06)	2007/7013460-R070196, BOSC19E_061 Method: D9908 Storage period: 7 months Last application: 11.07.2007
USA, Conklin (MI) 2007 (Stanley)	5 × 0.26	7-8	5 × 0.018- 0.14	Up to BBCH 87	Whole fruit	0 1	0.74, 0.79 (0.76) 0.48, 0.57 (0.52)	2007/7013460-R070197, BOSC19E_061 Method: D9908 Storage period: 7 months
USA, Parlier (CA) 2002 (Casselman)	0.003 g/kg	-	-	-	post-harvest spray (high vol.)	0	0.65, 0.67 (0.66)	2005/7004639-02-CA117 BOSC19E_068 Method: D9908 Storage period: 4 months
	0.0028 g/kg	-	-	-	post-harvest spray (low vol.)	0	2.8, 3.0 (2.9)	
	0.067 kg/l	-	-	-	Post-harvest dip with wax	0	0.97, 0.96 (0.96)	
USA, Parlier (CA) 2002	0.0028 g/kg	-	-	-	post-harvest spray (low vol.)	0	0.99, 0.85 (0.92)	2005/7004639-02-CA118 BOSC19E_068 Method: D9908 Storage period: 4 months

Location, Year (variety)	Application				Residues, mg/kg			Report/Trial No., Reference, analytical method, validation data, storage period
	kg ai/ha	Inter-val days	kg ai/hL	Growth stage	Sample	DALA	Boscalid	
	0.067 kg/l	-	-	-	Post-harvest dip with wax	0	0.84, 0.59 (0.72)	

DALA: days after last application

Table 25 Summary information on residues of boscalid in plums (reported in the 2006 JMPR Evaluation)

Location, Year (variety)	Application				Residues, mg/kg			Report/Trial No.
	kg ai/ha	Inter-val days	kg ai/hL	Growth stage	Sample	DALA	Boscalid	
Northern America (cGAP: USA 5 × 0.26 kg ai/ha, 7 day interval, 0 d PHI)								
USA, Conklin (MI) 1999 (Stanley)	5 × 0.26	6-8	5 × 0.037	35mm to early maturity	Whole fruit	0 7 14 21 28	<u>0.57</u> 0.55 0.4 0.29 0.23	2001/5000831-99116
	5 × 0.26	6-8	5 × 0.013	35mm to early maturity	Whole fruit	0 7 14 21 28	0.34 0.21 0.27 0.23 0.25	
USA, Porterville (CA), 1999	5 × 0.26	7	5 × 0.051	Fruit matur. to harvest	Whole fruit	0	0.14	2001/5000831-99117
(July Rosu´s)	5 × 0.26	7	5 × 0.013	Fruit matur. to harvest	Whole fruit	0	<u>0.15</u>	
USA, Porterville	5 × 0.26	7	5 × 0.046	60mm to harvest	Whole fruit	0	0.17	2001/5000831-99118
(CA), 1999 (Angelino)	5 × 0.26	7	5 × 0.012	60mm to harvest	Whole fruit	0	<u>0.32</u>	
USA, Chilo (CA)	5 × 0.26	7	5 × 0.031	30mm to harvest	Whole fruit	0	0.09	2001/5000831-99119
1999 (French Prune)	5 × 0.26	7	5 × 0.016	30mm to harvest	Whole fruit	0	<u>0.1</u>	
USA, Selma (CA)	5 × 0.26	6-8	5 × 0.028	Full size to harvest	Whole fruit	0	0.24	2001/5000831-99120
1999 (Howard Sun)	5 × 0.26	6-8	5 × 0.014	Full size to harvest	Whole fruit	0	<u>0.25</u>	
USA, Dallas (OR)	5 × 0.26	6-8	5 × 0.043	Coloring to harvest	Whole fruit	0	0.08	2001/5000831-99308
1999 (Parsons)	5 × 0.26	6-8	5 × 0.014	Coloring to harvest	Whole fruit	0	<u>0.11</u>	
USA, Payette (ID), 2004 (Empress)	5 × 0.26	6-7	5 × 0.027	Coloring to ripe for picking	Whole fruit	0	0.55, 0.54 (<u>0.54</u>)	2005/5000024-RCN2004138

Location, Year (variety)	Application				Residues, mg/kg			Report/Trial No.
	kg ai/ha	Inter-val days	kg ai/hL	Growth stage	Sample	DALA	Boscalid	
Canada, Bewick (NS) 2004, (Blufre)	5 × 0.26	6-8	5 × 0.041	50% final size to ripe for picking	Whole fruit	0	0.55, 0.85 (0.7)	2005/5000024-RCN2004139
Canada, Branchton (ON) 2004 (Yellow Plum)	5 × 0.26	6-7	5 × 0.029	50% final size to ripe for picking	Whole fruit	0	0.46, 0.46 (0.46)	2005/5000024-RCN2004140
USA, Nodine (MN) 2004, (Alderman)	5 × 0.26	7	5 × 0.028	60% final size to ripe for picking	Whole fruit	0	0.25, 0.088 (0.17)	2005/5000024-RCN2004141

DALA: days after last application

Table 26 Residues of boscalid in blueberries

Location, Year (variety)	Application				Residues, mg/kg			Report/Trial No., Reference, analytical method, validation data, storage period
	kg ai/ha	Inter-val days	kg ai/hL	Growth stage	Sample	DALA	Boscalid	
Northern America (cGAP: USA 4 × 0.26 kg ai/ha, 7 days interval, 0 d PHI)								
USA, Tift 2007 (Brightwell)	4 × 0.41	7	4 × 0.21	Mature	Fruits	0 1 5 10	2.0 1.9 1.9 1.6	2007/7013452-R070217 BOSC19E_072 Method: D9908 Storage period: 4 months
Canada, Lac St.Jean 2007 (Wild Lowbush)	4 × 0.41	7d	4 × 0.21	Mature	Fruits	0 1	5.4 3.7	2007/7013452-R070218 BOSC19E_072 Method: D9908 Storage period: 4 months

DALA: days after last application

Table 27 Summary information on residues of boscalid in blueberries (reported in the 2006 JMPR Evaluation)

Location, Year (variety)	Application				Residues, mg/kg			Report/Trial No., Reference, analytical method, validation data, storage period
	kg ai/ha	Inter-val days	kg ai/hL	Growth stage	Sample	DALA	Boscalid	
Northern America (cGAP: USA 4 × 0.26 kg ai/ha, 7 day interval, 0 d PHI)								
USA, Maiden Rock (WI), 1999, (Blue Chop, Highbush)	4 × 0.41	6-7	4 × 0.22	Mature	Fruits	0	1.4, 0.92 (1.2)	2000/5195-99278
USA, Corvallis (OR) 1999, (Blue Crop, Highbush)	4 × 0.41	6-8	4 × 0.11	Mature	Fruits	0	0.49, 1.2 (0.84)	2000/5195-99279
USA, Dundee (NY) 1999, (Blue Ray and Blue Crop, Highbush)	4 × 0.41	6-7	4 × 0.19	Mature	Fruits	0	1.1, 1.4 (1.2)	2000/5195-99328

Location, Year (variety)	Application				Residues, mg/kg			Report/Trial No., Reference, analytical method, validation data, storage period
	kg ai/ha	Interval days	kg ai/hL	Growth stage	Sample	DALA	Boscalid	
USA, Hixton (WI) 1999, (Berkley, Highbush)	4 × 0.41	6-9	4 × 0.22	Mature	Fruits	0	1.1, 1.5 (1.3)	2000/5195-99329
USA, Chula (GO) 1999 (Tift Blue, Highbush)	4 × 0.41	7	4 × 0.22	Mature	Fruits	0	1.4, 1.5 (1.4)	2000/5195-99330
USA, Pineboro (GO) 1999 (Climax, Highbush)	4 × 0.41	7	4 × 0.22	Mature	Fruits	0	2.2, 2.5 (2.4)	2000/5195-99331
Canada, Riverton 2004 (Wild Lowbush)	4 × 0.4-0.41	6	4 × 0.15	Mature	Fruits	0	4.3, 4.4 (4.4)	2005/5000144-RCN2004146
USA, Conklin (MI) 2004 (Blue Crop, Highbush)	4 × 0.41-0.42	6-7	4 × 0.06	Mature	Fruits	0	2.4, 2.8 (2.6)	2005/5000144-RCN2004149
	4 × 0.41-0.42	6-7	4 × 0.06	Mature	Fruits	0	2.7, 2.6 (2.6)	
USA, Arkansaw (WI) 2004 (Elliot, Highbush)	4 × 0.41-0.42	7	4 × 0.07	Mature	Fruits	0	3.6, 4.0 (3.8)	2005/5000144-RCN2004151
Canada, Berwick 2004 (Lowbush)	4 × 0.41-0.42	6-7	4 × 0.06	Mature	Fruits	0	6.6, 7.0 (6.8)	2005/5000144-RCN2004198
	4 × 0.4-0.42	6-7	4 × 0.06	Mature	Fruits	0	6.3, 7.4 (6.8)	

DALA: days after last application

Table 28 Residues of boscalid in blueberries grown indoors

Location, Year (variety)	Application				Residues, mg/kg			Report/Trial No., Reference, analytical method, validation data, storage period
	kg ai/ha	Interval days	kg ai/hL	Growth stage	Sample	DALA	Boscalid	
Europe (cGAP: Germany 3 × 0.25 kg ai/ha, 7 day interval, 7 d PHI)								
Germany, Gilten 2009 (Duke)	3 × 0.27	7	4 × 0.027	Mature	Fruits	0 7 10 14 21	16.6 3.1 4.2 0.37 0.83	2010/1224114-AK Lück 0929 BOSC19E_074 Method: L00.00-113 Storage period: 6 months Reduced sample size (25-180 g)

DALA: days after last application

Table 29 Residues of boscalid in currants grown indoors

Location, Year (variety)	Application				Residues, mg/kg			Report/Trial No., Reference, analytical method, validation data, storage period
	kg ai/ha	Inter-val days	kg ai/hL	Growth stage	Sample	DALA	Boscalid	
Europe (cGAP: Germany 3 × 0.25 kg ai/ha, 7 day interval, 7 d PHI)								
Germany, Köln 2009 (Ometa)	3 × 0.27	7	4 × 0.027	Mature	Fruits	0 7 10 14 21	5.3 4.8 3.6 3.7 2.1	2010/1224114- AK Lück 0927 BOSC19E_074 Method: L00.00-113 Storage period: 6 months Same location, different glasshouse
	3 × 0.27	7	4 × 0.027	Mature	Fruits	0 7 10 14 21	4.5 3.4 3.5 2.9 1.7	2010/1224114- AK Lück 0928 BOSC19E_074 Method: L00.00-113 Storage period: 6 months Same location, different glasshouse
Germany, Karlsruhe 2009 (Titania)	3 × 0.27	7-8	4 × 0.027	Mature	Fruits	14	2.6	2010/1224114- AK Lück 0930 BOSC19E_074 Method: L00.00-113 Storage period: 6 months

DALA: days after last application

Table 30 Residues of boscalid in raspberries

Location, Year (variety)	Application				Residues, mg/kg			Report/Trial No., Reference, analytical method, validation data, storage period
	kg ai/ha	Inter-val days	kg ai/hL	Growth stage	Sample	DALA	Boscalid	
Northern America (cGAP: USA 4 × 0.26 kg ai/ha, 7 day interval, 0 d PHI)								
USA, Penn Yau (NY) 1999 (Titau)	4 × 0.41	6	4 × 0.19	Mature	Fruits	0 2 4 6 8	3.3, 2.1 (2.7) 2.5, 2.1 (2.3) 2.3, 1.6 (2.0) 2.0, 1.1 (1.6) 0.96, 1.5 (1.2)	2000/5195-99277 BOSC19E_071 Method: D9908 Storage period: 3 months
USA, Sherwood (OR) 1999 (Meeker)	4 × 0.41	7	4 × 0.2	Mature	Fruits	0	1.6, 1.4 (1.5)	2000/5195-99280 BOSC19E_071 Method: D9908 Storage period: 3 months Last application: 07.07.1999
USA, Sherwood (OR) 1999 (Tulamene)	4 × 0.41	7	4 × 0.2	Mature	Fruits	0	2.4, 1.6 (2.0)	2000/5195-99281 BOSC19E_071 Method: D9908 Storage period: 3 months Last application: 07.07.1999
USA, Nodine (MN) 2004 (Nova)	4 × 0.41-0.43	7	4 × 0.08	Mature	Fruits	0	3.7, 3.3 (3.5)	2005/5000144-RCN2004143 BOSC19E_073 Method: D9908 Storage period: 6 months

Location, Year (variety)	Application				Residues, mg/kg			Report/Trial No., Reference, analytical method, validation data, storage period
	kg ai/ha	Interval days	kg ai/hL	Growth stage	Sample	DALA	Boscalid	
USA, Corvallis (OR) 2004 (Caroline)	4 × 0.41-0.42	7	4 × 0.06	Mature	Fruits	0	2.5, 2.4 (2.4)	2005/5000144-RCN2004144 BOSC19E_073 Method: D9908 Storage period: 6 months
Canada, Abbotsford 2004 (Kilarme)	4 × 0.38-0.41	7	4 × 0.06	Mature	Fruits	0	3.1, 4.4 (3.7)	2005/5000144-RCN2004145 BOSC19E_073 Method: D9908 Storage period: 6 months

DALT: days after last application

Table 31 Residues of boscalid in avocado

Location, Year (variety)	Application				Residues, mg/kg			Report/Trial No., Reference, analytical method, validation data, storage period
	kg ai/ha	Interval days	kg ai/hL	Growth stage	Sample	DALA	Boscalid	
Northern America (cGAP: USA 2 × 0.33 kg ai/ha, 7 day interval, 0 d PHI)								
USA, Homestead (FL) 2002 (Peterson)	4 × 0.41	6-8	4 × 0.06	Mature	Fruits without stone	0	0.14, 0.17 (0.16)	2006/1045610-02-FL44 BOSC19E_075 Method: D9908 Storage period: 6 months Last application: 10.07.2002
USA, Homestead (FL) 2002 (Booth 8)	4 × 0.41	7	4 × 0.07	Mature	Fruits without stone	0	0.18, 0.19 (0.18)	2006/1045610-02-FL45 BOSC19E_075 Method: D9908 Storage period: 6 months Last application: 27.08.2002
USA, Homestead (FL) 2002 (Peterson)	4 × 0.41	7-8	4 × 0.07	Mature	Fruits without stone	0	0.22, 0.27 (0.24)	2006/1045610-02-FL46 BOSC19E_075 Method: D9908 Storage period: 6 months Last application: 29.08.2002
USA, Woodland (CA) 2002 (Zutano)	4 × 0.41	7	4 × 0.06	Mature	Fruits without stone [a]	0	0.59, 0.42 (0.5)	2006/1045610-02-CA100 BOSC19E_075 Method: D9908 Storage period: 6 months
USA, Orosi (CA) 2002 (Hass)	4 × 0.41	6-7	4 × 0.07	Mature	Fruits without stone [a]	0	0.76, 1.3 (1.0)	2006/1045610-02-CA101 BOSC19E_075 Method: D9908 Storage period: 6 months
USA, Lindcove (CA) 2002 (Bacon)	4 × 0.41	7	4 × 0.07	Mature	Fruits without stone [a]	0	0.38, 0.31 (0.34)	2006/1045610-02-CA102 BOSC19E_075 Method: D9908 Storage period: 6 months
USA, Nipomo (CA) 2002 (Gwen and Bacon)	4 × 0.41	6-7	4 × 0.06	Mature	Fruits without stone [a]	0	0.47, 0.34 (0.4)	2006/1045610-02-CA103 BOSC19E_075 Method: D9908 Storage period: 6 months

DALA: days after last application

[a] fruit halves in the field and stone removed

Table 32 Residues of boscalid in mangoes

Location, Year (variety)	Application kg ai/ha	Inter-val days	kg ai/hL	Growth stage	Residues, mg/kg Sample	DALA	Boscalid	Report/Trial No., Reference, analytical method, validation data, storage period
Southern America (cGAP: Brazil 2 × 0.024 kg ai/hl, 15 day interval, 7 d PHI)								
Brazil, Londrina 2011 (Tommy Atkins)	2 × 0.45	14	2 × 0.022	BBCH 81	Whole fruits	0 7 14	0.38 <u>0.55</u> 0.13	2011/1226624-G100443 2011/1266277 2011/3008004 2011/3008003 BOSC19E_076, _077; _078 & _079 Method: L0076/01 Storage period: 8 months
Brazil, Anápolis 2011 (Tommy)	2 × 0.45	14	2 × 0.022	BBCH 85	Whole fruits	0 7 14	0.9 <u>0.68</u> 0.54	2011/1226624-G100444 2011/1266277 2011/3008004 2011/3008003 BOSC19E_076, _077; _078 & _079 Method: L0076/01 Storage period: 8 months
Brazil, Sto. Antônio de Posse 2011 (Palmer)	2 × 0.45	14	2 × 0.022	BBCH 85	Whole fruits	7	<u>0.25</u>	2011/1226624-G100445 2011/1266277 2011/3008004 2011/3008003 BOSC19E_076, _077; _078 & _079 Method: L0076/01 Storage period: 8 months
Brazil, Urai 2011 (Palmer)	2 × 0.45	14	2 × 0.022	BBCH 87	Whole fruits	7	<u>1.0</u>	2011/1226624-G100446 2011/1266277 2011/3008004 2011/3008003 BOSC19E_076, _077; _078 & _079 Method: L0076/01 Storage period: 8 months
Brazil, Rolândia 2014 (Tommy Atkins)	2 × 0.24	15	2 × 0.024	BBHCH 79-89	Whole fruit, calculated Control	0 7 14 0.018	<0.01 <0.01 0.017	2015/3002561-G140014 2015/3002961 BOSC19E_080 & _081 Method: L0076/01 Storage period: 9 months Note: fruits separated in peel and pulp in the field. Stone discarded. Trial not considered due to significant residues in control samples.
Brazil, Petrolina 2014 (Palmer)	2 × 0.24	15	2 × 0.024	BBHCH 78-88	Whole fruit, calculated	7	<u>0.1</u>	2015/3002561-G140015 2015/3002961 BOSC19E_080 & _081 Method: L0076/01 Storage period: 9 months Last application: 27.02.2014 Note: fruits separated in peel and pulp in the field. Stone discarded.

Location, Year (variety)	Application: kg ai/ha	Application: Interval days	Application: kg ai/hL	Application: Growth stage	Residues, mg/kg: Sample	Residues, mg/kg: DALA	Residues, mg/kg: Boscalid	Report/Trial No., Reference, analytical method, validation data, storage period
Brazil, Mogi Mirim 2014 (Choc Anao)	2 × 0.24	15	2 × 0.024	BBHCH 79-89	Whole fruit, calculated	7	0.22	2015/3002561-G140022 2015/3002961 BOSC19E_080 & _081 Method: L0076/01 Storage period: 9 months Note: fruits separated in peel and pulp in the field. Stone discarded.
Brazil, Petrolina 2014 (Tommy)	2 × 0.24	15	2 × 0.024	BBHCH 79-89	Whole fruit, calculated	0 7 14	0.54 0.26 0.16	2015/3002561-G140111 2015/3002961 BOSC19E_080 & _081 Method: L0076/01 Storage period: 9 months Last application: 09.07.2014 Note: fruits separated in peel and pulp in the field. Stone discarded.
Brazil, Urai 2014 (Palmer)	0.24	-	0.024	BBHCH 81	Whole fruit, calculated	0 7 14	<0.01 <0.01 <0.01	2015/3002561-G140257 2015/3002961 BOSC19E_080 & _081 Method: L0076/01
	2 × 0.24	15	2 × 0.024	BBHCH 81-85	Whole fruit, calculated	0 7 14	0.027 0.015 0.032	Storage period: 9 months Note: fruits separated in peel and pulp in the field. Stone discarded.

DALA: days after last application

Whole fruit calculation factor: pulp and peel → whole fruit with stone: 0.7-0.88

Table 33 Residues of boscalid in pomegranate

Location, Year (variety)	Application: kg ai/ha	Application: Interval days	Application: kg ai/hL	Application: Growth stage	Residues, mg/kg: Sample	Residues, mg/kg: DALA	Residues, mg/kg: Boscalid	Report/Trial No., Reference, analytical method, validation data, storage period
Northern America (cGAP: USA 2 × 0.33 kg ai/ha, 7 day interval, 0 d PHI)								
Greece, Nea Magnisia 2017 (Wonderful)	2 × 0.5	5	2 × 0.05	BBCH 87	Whole fruit Peel Seeds	0 3 7 14 7 14 7 14	0.67 0.42 0.37 0.24 1.0 0.63 <0.01 <0.01	2018/1013073-L170329 BOSC19E_082 Method: L0076/01 Storage period: 6 months
Greece, Apollonia 2017 (Wonderful)	2 × 0.5	5	2 × 0.05	BBCH 87	Whole fruit Peel Seeds	0 3 7 14 7 14 7 14	1.0 0.89 0.53 0.26 1.4 0.84 <0.01 <0.01	2018/1013073-L170330 BOSC19E_082 Method: L0076/01 Storage period: 6 months

Location, Year (variety)	Application				Residues, mg/kg			Report/Trial No., Reference, analytical method, validation data, storage period
	kg ai/ha	Inter-val days	kg ai/hL	Growth stage	Sample	DALA	Boscalid	
Italy, Grottaglie 2017 (Wonderful One)	2 × 0.5	5	2 × 0.05	BBCH 88	Whole fruit	0	1.1	2018/1013073-L140328 BOSC19E_082 Method: L0076/01 Storage period: 6 months
						3	0.94	
						7	0.82	
						14	0.78	
					Peel	7	2.3	
						14	2.1	
					Seeds	7	0.072	
						14	0.053	
Spain, Tocina 2017 (Acco)	2 × 0.5	5	2 × 0.05	BBCH 85	Whole fruit	0	0.8	2018/1013073-L170327 BOSC19E_082 Method: L0076/01 Storage period: 6 months
						3	0.8	
						7	0.45	
						14	0.46	
					Peel	7	0.63	
						14	0.6	
					Seeds	7	0.14	
						14	0.13	

DALA: days after last application

Tea

The Meeting received a supervised field trial study conducted by Lenz, C. (2017, BOSC19E_083) on tea. In this study fresh leaves and green tea, dried were sampled. The production of green tea, dried was conducted according to the local practice for each of the regions:

China: fresh leaves were taken through an indoor drying, panning/fixation (high heat exposure of a few minutes to stop further enzyme breakdown) and a second drying.

India: fresh leaves were taken through the steps of steaming (hot water bath), withering, CTC (crush, tear, and curl) and drying.

Japan: fresh leaves were taken through the steps of steaming, fan drying, roasting, and air cooling

Taiwan (Province of China): fresh leaves were taken through the steps of sun drying, steaming, pan frying, and cooling.

Table 34 Residues of boscalid in tea

Location, Year (variety)	Application				Residues, mg/kg			Report/Trial No., Reference, analytical method, validation data, storage period
	kg ai/ha	Inter-val days	kg ai/hL	Growth stage	Sample	DALA	Boscalid	
East Asia (cGAP: Japan 2 × Factor 2000 dilution ≙ 0.0068 kg ai/hl, unspecified interval, 7 day PHI)								
China, Huang Tang 2014 (Fuding Dahao #2)	2 × 0.27	7	2 × 0.009	BBCH 40-43	Fresh leaves	0	8.2	2015/1086962-L140320 BOSC19E_083 Method: L0076/01 Storage period: 12 months
						6	2.1	
						13	<0.01	
						21	0.21	
					Green tea, dried	13	4.1	
					Controls:			

Location, Year (variety)	Application				Residues, mg/kg			Report/Trial No., Reference, analytical method, validation data, storage period
	kg ai/ha	Inter-val days	kg ai/hL	Growth stage	Sample	DALA	Boscalid	
					Black tea, dried	13	0.042	
China, Zhongyong 2014 (Fuyun #6)	2 × 0.27	7	2 × 0.009	BBCH 40-43	Fresh leaves Green tea, dried Controls: Green tea, dried	0 8 14 22 14 14	8.5 0.83 <0.01 0.056 <u>1.7</u> 0.019	2015/1086962- L140321 BOSC19E_083 Method: L0076/01 Storage period: 12 months
India, Coimbatore 2015 (UPASI-3)	2 × 0.27	7	2 × 0.013	BBCH 40-43	Fresh leaves Green tea, dried	0 7 15 22 15	12 5.5 1.1 0.42 <u>6.3</u>	2015/1086962- L140322 BOSC19E_083 Method: L0076/01 Storage period: 12 months Last application: 18.02.2015
India, Coimbatore 2015 (UPASI-9)	2 × 0.27	7	2 × 0.012	BBCH 40-43	Fresh leaves Green tea, dried	0 7 15 22 15	17 11 1.4 0.62 <u>6.2</u>	2015/1086962- L140323 BOSC19E_083 Method: L0076/01 Storage period: 12 months Last application: 18.02.2015
Japan, Bungo-Ono Shi 2014 (Saemidori)	2 × 0.27	7	2 × 0.012	BBCH 40-43	Fresh leaves Green tea, dried	0 7 15 21 15	33 7.4 2.3 1.9 <u>5.6</u>	2015/1086962- L140324 BOSC19E_083 Method: L0076/01 Storage period: 12 months Last application: 27.08.2014
Japan, Bungo-Ono Shi 2014 (Yabukita)	2 × 0.27	7	2 × 0.012	BBCH 40-43	Fresh leaves Green tea, dried	0 7 15 21 15	37 4.9 2.2 1.9 <u>7.3</u>	2015/1086962- L140325 BOSC19E_083 Method: L0076/01 Storage period: 12 months Last application: 27.08.2014
Taiwan (Province of China), Chiayi 2014 (Ching Shin Oolong)	2 × 0.27	7	2 × 0.013	BBCH 40-43	Fresh leaves Green tea, dried Controls: Fresh leaves Green tea, dried	0 7 12 21 12 7 12	8.1 5.7 5.5 3.8 <u>19</u> 13 0.14	2015/1086962- L140326 BOSC19E_083 Method: L0076/01 Storage period: 12 months Last application: 16.10.2014
Taiwan (Province of China),Chiayi	2 × 0.27	7	2 × 0.01	BBCH 40-43	Fresh leaves	0 7 12	9.2 6.7 7.0	2015/1086962- L140327 BOSC19E_083 Method: L0076/01

Location, Year (variety)	Application				Residues, mg/kg			Report/Trial No., Reference, analytical method, validation data, storage period
	kg ai/ha	Inter-val days	kg ai/hL	Growth stage	Sample	DALA	Boscalid	
2014 (Taiwan No. 27)						21	2.3	Storage period: 12 months Last application: 14.11.2014
					Green tea, dried	12	16	
					Controls: Green tea, dried	12	0.039	

DALA: days after last application

FATE OF RESIDUES DURING PROCESSING

Residues after processing

The fate of boscalid during processing of raw agricultural commodity (RAC) was investigated in tea.

Tea

The transfer of residues of boscalid was investigated in tea by Lenz, C. (2017, BOSC19E_083) in three supervised field trial conducted in China and Taiwan Province of China. The trials were performed at rates of 2 × 0.54 kg ai/ha (7 day interval) 12–13 day before harvest. Tea leaves collected were processed into black tea following local practice. The black tea was shipped to the laboratory and processed into infusions, instant tea, tea extract, stepped leaves and cooked leaves.

Black tea preparation in China: fresh leaves were taken through the steps of air or fan drying, crushing/rolling (by machine), fermentation, and a second machine drying.

Black tea preparation in Taiwan Province of China: fresh leaves were taken through the steps of sun drying, indoor drying, crushing/rolling (by machine), fermentation, and second drying with a roaster.

The subsequent processing of black tea was conducted according to the following procedures:

Tea infusion: An amount of 1000 g of boiling water was added to 13 g of milled black tea leaves (1.3%). The tea remained in the water for three minutes and was then sieved. Samples were collected from the infusion and the steeped leaves.

Instant tea: An amount of 600 g of boiling water was added to 100 g of milled black tea leaves and cooked for 30 minutes. The cooked leaves were separated from the tea extract (dry matter content approx. 5%) by using a centrifuge and a sieve. A sample of cooked leaves (instant) was taken. The tea extract was concentrated using a rotary evaporator until a dry matter content of approx. 25% was reached (temperature 58 °C, vacuum 100 mbar in the beginning - was increased until 20 to 40 mbar at the end of the concentration). After concentration a sample of the tea extract (dry matter content approx. 25%) was taken. The following substances were added (the amounts varied in dependence on the reached dry matter content of the concentrated tea extract) and stirred for approx. 8 minutes:

50 g Concentrated tea extract (dry matter content approx. 25%)

12 g Silica gel (Becosorb 1000)

12 g Maltodextrin

1.25 g Citric acid

The mixture was added as a thin layer to a sheet metal (height of the layer: approx. 1 to 2 mm) and dried for 20 hours at 42 °C and afterwards for 4 hours at 50 °C. The dry intermediate (approx. 94% dry matter content) was milled and saccharose was added to get instant tea with a dry matter tea content

of approx. 5% (approx. 1 part dry intermediate and 5.5 parts saccharose were mixed). The instant tea was homogenized / milled and a sample was taken.

In the following table the residues of boscalid and the resulting processing factors for tea products are summarized:

Table 35 Summary of boscalid in tea and processed commodities (Lenz, C., 2017, BOSC19E_083) following treatment with 2 × 0.54 kg ai/ha (13 DALA)

Location, Year (Variety)	Matrix	Boscalid in mg/kg	PF
China, Huang Tang 2014 (Fuding Dahao #2)	Black tea (RAC)	0.41	-
	Infusion	<0.01	<0.02
	Steeped leaves (infusion)	0.084	0.2
	Instant tea	<0.01	<0.02
	Tea extract	0.021	0.05
	Cooked leaves (instant)	0.13	0.32
China, Zhongyong 2014 (Fuyun #6)	Black tea (RAC)	4.5	-
	Infusion	<0.01	<0.002
	Steeped leaves (infusion)	1.4	0.31
	Instant tea	0.031	0.007
	Tea extract	0.33	0.07
	Cooked leaves (instant)	2.4	0.53
Taiwan (Province of China), Chiayi 2014 (Ching Shin Oolong)	Black tea (RAC)	25	-
	Infusion	0.038	0.002
	Steeped leaves (infusion)	6.1	0.24
	Instant tea	0.13	0.005
	Tea extract	1.2	0.05
	Cooked leaves (instant)	9.7	0.39

RAC:raw agricultural commodity

In summary, the following processing factors were derived for processed tea:

Table 36 Summary of processing factors for boscalid in tea

Matrix	Individual PF	Median or best estimate
Infusion	<0.002, 0.002, <0.02	0.002
Steeped leaves (infusion)	0.2, 0.24, 0.31	0.24
Instant tea	0.005, 0.007, <0.02	0.007
Tea extract	0.05, 0.05, 0.07	0.05
Cooked leaves (instant)	0.32, 0.39, 0.53	0.39

APPRAISAL

Boscalid is a systemic fungicide first evaluated by JMPR in 2006 for residues and toxicology as a new active substance. An ADI of 0–0.04 mg/kg bw was established for boscalid, while no ARfD was considered necessary.

The 2006 JMPR recommended the following residue definition for boscalid:

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

Definition of the residue for dietary risk assessment in animal commodities: *sum of boscalid, 2-chloro-N-(4'-chloro-5-hydroxybiphenyl-2-yl)nicotinamide (M510F01) including its conjugate, expressed as boscalid.*

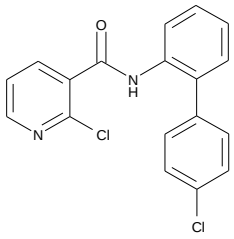
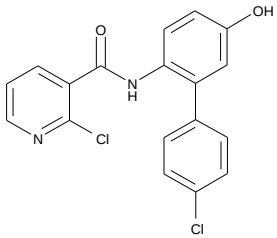
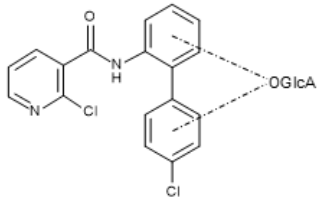
The residue is fat-soluble.

In 2008 and 2010 additional uses (and in 2009 residues in follow crops) were reviewed for residues by the Meeting. Boscalid was scheduled at the Fiftieth Session of the CCPR for the evaluation of additional uses for the Extra 2019 JMPR Meeting.

The current Meeting received new information on use patterns for boscalid in pome fruit, stone fruit, berry fruit, tropical fruit and tea supported by additional plant and animal metabolism studies, field rotational crop studies, analytical methods and recovery data, supervised field trials and studies simulating typical processing conditions.

The current Meeting also received additional data on environmental fate and on corresponding analytical methods in environmental matrices (see evaluation). The Meeting concluded that these data are not directly linked to the current consideration of additional uses on permanent crops and decided to postpone the assessment of the data until the next periodic review of boscalid.

The following abbreviations are used for the metabolites discussed below:

Code Names	Structure	Where found
Boscalid BAS510F		Rat, plants, animals, rotational crops, soil
M510F01		Rat, animals
M510F65		Rat, animals

Plant metabolism

The fate of boscalid in plants was evaluated by the 2006 Meeting following foliar spray application of ^{14}C-diphenyl- or ^{14}C–pyridine-radiolabelled substance to grapes, lettuce and green beans. A detailed assessment of these studies is presented in the 2006 JMPR Report. For the current Meeting, an additional plant metabolism study on green beans was submitted.

The metabolism of ^{14}C-diphenyl-boscalid in common beans was investigated under enclosed conditions by application of three foliar sprays at 0.52 kg ai/ha each. The treatments were performed at the beginning of flowering (BBCH 61, 33 days before harvest), 11 days later (22 days before harvest) and 13 days before harvest (BBCH 75–79). Samples of plants and whole pods were collected 3 days before and 13 days after final treatment. Pods collected at harvest were additionally separated into hulls and green seeds.

In all samples except green seeds, the extraction of radioactivity with methanol, followed by water, was nearly complete (>98% TRR). In green seeds 70% of the TRR was extracted by the solvents

used. TRR levels ranged from 29–52 mg eq/kg in plants, 0.79–1.2 mg eq/kg in whole pods, 0.80 mg eq/kg in hulls and 0.065 mg eq/kg in green seeds.

The identification of the radioactive residues revealed only unchanged boscalid in plants, pods and hulls, representing 97–102% of the TRR. In green seeds, only 17% of the TRR (0.011 mg eq/kg) was identified as boscalid. The majority of the extracted radioactivity (53% TRR) was characterised as five minor components, two of them present up to 0.011 mg eq/kg (up to 17% TRR) and three of them up to 0.006 mgeq/kg (up to 9% TRR).

Post-extraction solids were not investigated and represented 30% TRR in green seeds (0.019 mg eq/kg) and <2% TRR in all other matrices.

The Meeting concluded that parent boscalid is the predominant residue in all plant parts directly treated (plant, whole pods, hulls). In green seeds, it is also present as a major component by proportion, but absolute concentrations are much lower. No metabolites were identified in bean plants, pods or hulls. In green seeds, characterised metabolites were present in minor amounts.

Animal metabolism

The fate of boscalid in lactating goats and laying hens was evaluated by the 2006 Meeting following administration of ^{14}C-diphenyl-radiolabelled substance. A detailed assessment of these studies is presented in the 2006 JMPR Report. For the current Meeting, an additional metabolism study on laying hens was submitted.

For the investigation of the metabolism of boscalid in laying hens ten animals received a dose of ^{14}C-pyridin-labelled boscalid equivalent to 12 ppm for 13 consecutive days via capsule administration. Animals were sacrificed approximately 6 hrs after the final dosing. During the whole dosing period eggs and excreta were collected and analysed with pooled tissue samples for each group at the end of the study.

TRR levels found were highest in liver (0.44 mg eq/kg), followed by egg yolk (0.12 mg eq/kg), fat (0.095 mg eq/kg), muscle (0.051 mg eq/kg) and egg white (0.03 mg eq/kg).

Solvent extraction using acetonitrile or methanol released the majority of the residue from all matrices (63–94% TRR). In addition, 2–10% TRR could be released from liver and eggs with water extraction while only 1.4% TRR was additionally released with dichloromethane from liver. Post extraction solids ranged from 6–32% TRR. Their characterisation by enzymatic hydrolysis released most of the radioactivity with protease treatment (22–35% TRR). The pepsin and pancreatin solubilizate contained only minor radioactivity (≤2% TRR).

Parent boscalid was found as a major residue in the extracts of fat (85% TRR), egg white/yolk (34% TRR) and muscle (29% TRR). In liver, only 1.8% of the TRR (0.008 mg eq/kg) were identified as unchanged parent. The major residue in liver extracts was M510F01 representing 35% TRR (0.16 mg eq/kg), which was also present in major proportions in egg white/yolk (27–28% TRR, 0.008–0.034 mg eq/kg) but not in muscle or fat (5–11% TRR, 0.005 mg eq/kg). Additionally, M510F65 (glucuronides of M510F01) was found as a major metabolite, representing 16–32% TRR in egg white/yolk (0.005–0.039 mg eq/kg) and 20% TRR in liver (0.09 mg eq/kg). In egg yolk, the majority of the M510F65 was recovered after enzymatic hydrolysis of the post-extraction solids (24% TRR, 0.029 mg eq/kg).

The metabolic pathway of ^{14}C-pyridin-labelled boscalid in laying hens was limited. In the first step, hydroxylation at the diphenyl-ring was observed forming M510F01. In a second step, glucuronidation occurs into M510F65. All metabolites identified in laying hens were also found in the rat.

Environmental fate

The current Meeting received one additional field rotational crop study involving application of 2.1 kg ai/ha to bare soil at four sites in Europe. Zucchini, cucumbers, tomatoes and lettuce were planted as rotational crops 30 days after treatment. In all fruiting vegetables (cucumber, zucchini and tomato), no

residues above the LOQ of 0.01 mg/kg were found (66–140 days after treatment). Only lettuce contained quantifiable residues ranging from 0.014–0.12 mg/kg.

The Meeting noted that boscalid residues found in rotated lettuce (up to 0.12 mg/kg) surpass findings in rotated Brassica vegetables (up to 0.05 mg/kg). However, the Meeting confirmed its previous conclusion that residues taken up from soil add insignificantly compared to directly treated leafy vegetables (maximum residue level recommendation of the 2010 JMPR was 40 mg/kg for leafy vegetables).

Methods of analysis

The current Meeting received additional analytical methods for the determination of boscalid in plant commodities and additional concurrent recovery information for method 471/0 evaluated by the 2006 Meeting, measuring boscalid and M510F01 (incl. conjugates) in animal matrices.

For plant matrices, three new single residue analytical methods were provided involving initial extraction with methanol/water/hydrochloric acid (70:25:5) or acetonitrile, followed by partitioning against cyclohexane or hexane, respectively. The first solvent system does not require further clean-up while the acetonitrile/hexane system includes a C_{18}- and Silica Gel-solid-phase extraction step. All methods involve analysis by LC-MS/MS at LOQs of 0.01 mg/kg for high water, high starch and high acid content matrices as well as for hops, spices and herbal infusions. For high oil content matrices, a LOQ of 0.05 mg/kg was validated.

In addition, the QuEChERS-Multimethod was successfully tested in high water, high acid and high starch content matrices at a LOQ of 0.01 mg/kg for boscalid.

In animal matrices, additional concurrent recovery data were submitted for method 471/0. LOQs of 0.01 mg/kg were validated each for boscalid and M510F01 (incl. conjugates) in bovine tissues, milk, cream and eggs.

Definition of the residue

The current Meeting received new data on the metabolism of boscalid in green beans and laying hens.

Following foliar application to green beans, boscalid was the only residue identified. The Meeting therefore confirms its previous recommendation of boscalid for compliance with the MRL and for the estimation of the dietary exposure for plant commodities.

In laying hens parent boscalid was found as a major residue in fat (85% TRR), egg white/yolk (34% TRR) and muscle (29% TRR) and in lower proportions in the liver (1.8% TRR). The Meeting confirms its previous recommendation of boscalid for compliance with the MRL for animal commodities and also on the fat-solubility of the residue.

Besides boscalid, its hydroxylated metabolite M510F01 and glucoronides thereof (M510F65) were the only components identified in hen matrices. Therefore the Meeting confirmed its previous recommendation for the estimation of the dietary exposure to be the sum of boscalid and M510F01 (2-chloro-N-(4'-chloro-5-hydroxybiphenyl-2-yl)nicotinamide) including its conjugate, expressed as boscalid.

Based on new information submitted, the present Meeting assessed the toxicity of M510F49 and considered it to be covered by the ADI for the parent substance. Since this metabolite was exclusively found in hen liver hydrolysate representing 12% of the TRR, no inclusion into the residue definition for compliance with the MRL or for the estimation of the dietary exposure is required.

Results of supervised residue trials on crops

The Meeting received supervised trial data for applications of boscalid on pome fruit, stone fruit, bush berries, cane berries, avocado, mango, pomegranate and tea, respectively.

Pome fruit

For boscalid, the 2006 JMPR Meeting recommended a maximum residue level of 2 mg/kg and estimated an STMR value of 0.365 mg/kg for apples based on a GAP from the UK (4×0.2 kg ai/ha, 7 day PHI). The current Meeting received new GAP information with supporting supervised field trials on apples and pears.

Boscalid is registered in the USA for the use pome fruits with a critical GAP involving four foliar sprays of 0.33 kg ai/ha each (7 day interval) and a PHI of 0 days.

Supervised field trials conducted in the USA on apples and pears were submitted which matched the individual application rates, their interval and the PHI, but six instead of four treatments were conducted.

In absence of decline data from Northern America on pome fruits, the Meeting decided to use decline trials from Europe reported by the current and by the 2006 JMPR, which were conducted at growth stages comparable to the US GAP. In total, 31 trials on apples and eight trials on pears were identified with reported residues at 0 days and sampling intervals up to 29 days. Based on first-order kinetics, decline rates of k=-0.0197 for apples and k=-0.0307 for pears were estimated.

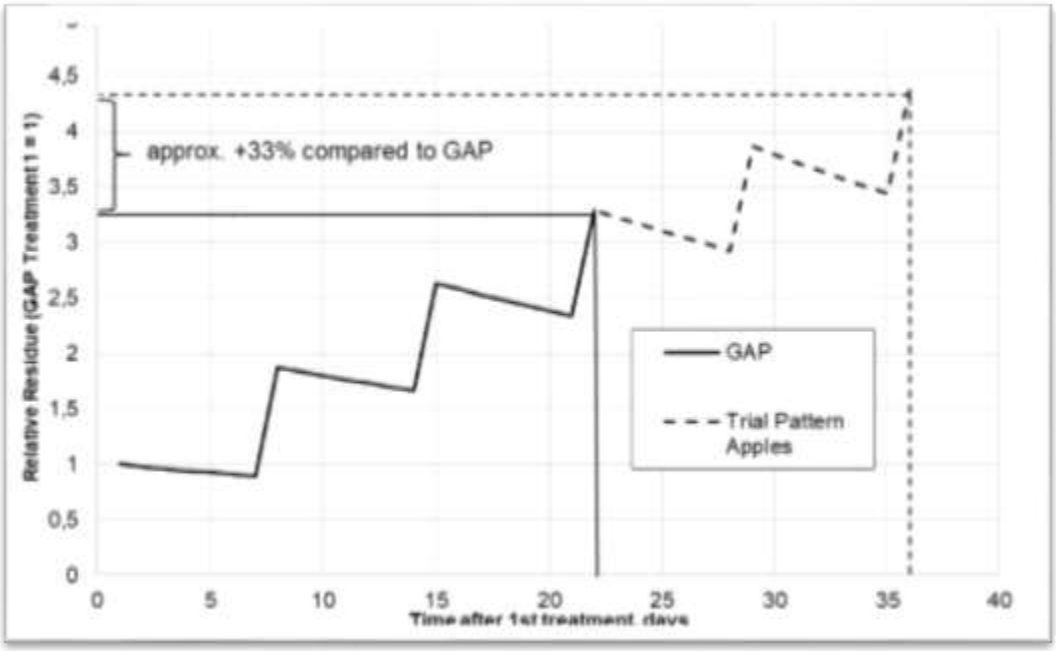


Figure 1 Anticipated residues at GAP vs Field trials (Boscalid – Apple)

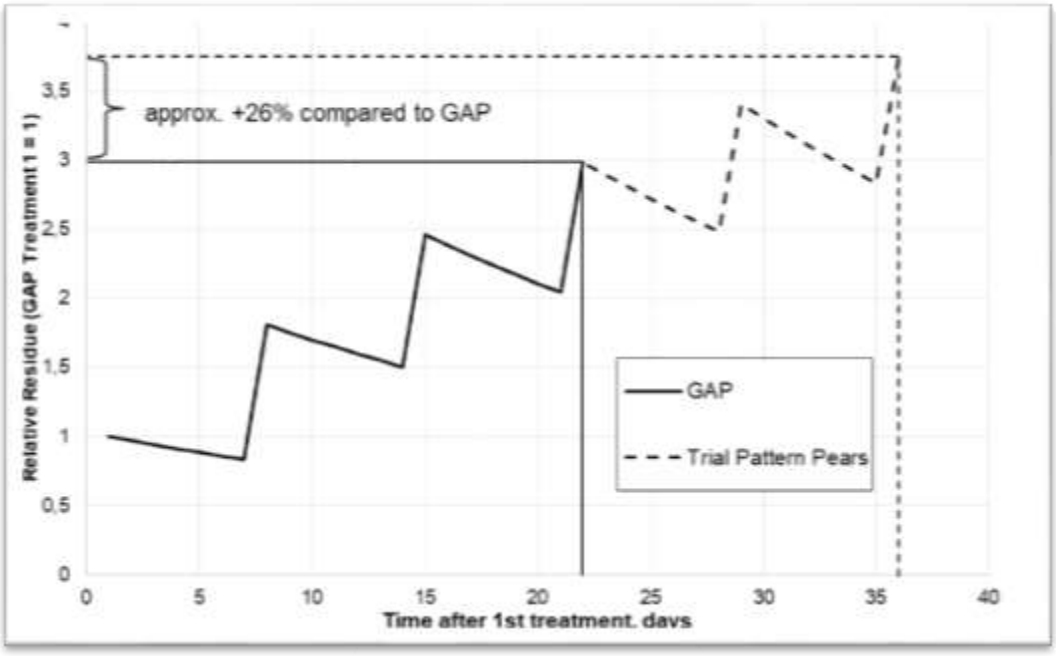


Figure 2 Anticipated residues at GAP vs Field trials (Boscalid – Pear)

The Meeting concluded that the supervised field trial data submitted for apples and pears from the USA overestimate the residue according to the US GAP by more than +25% and cannot be used to estimate maximum residue levels in pome fruits. The Meeting also concluded that proportional adjustment of these trials is inappropriate due to the deviating treatment regime compared to the critical GAP from the USA.

Boscalid is also registered in the Czech Republic for the use on pome fruits with a maximum GAP involving four foliar sprays of 0.2 kg ai/ha each (8 day interval) and a PHI of 7 days.

New supervised field trials conducted in Europe on pears approximating this GAP were submitted to the Meeting. In addition, residue data on apples assessed by the 2006 JMPR against a comparable GAP from the UK were considered.

Residues of boscalid in apples submitted to the 2006 JMPR were (n=22): 0.15, 0.19, 0.2, 0.24, 0.29, 0.29, 0.3, 0.32, 0.32, 0.34, 0.36, 0.37, 0.39, 0.42, 0.42, 0.43, 0.51, 0.53, 0.55, 0.65, 0.86, 1.2 mg/kg.

Residues of boscalid in pears were (n=8): 0.086, 0.11, 0.16, 0.29, 0.33, 0.39, 0.48, 1.3 mg/kg.

The Meeting noted that residues in apples and pears are not significantly different, which was confirmed by the Mann-Whitney-U Test, and decided to combine the datasets.

Residues of boscalid in apples and pears were (n=30): *0.086*, *0.11*, 0.15, 0.16, 0.19, 0.2, 0.24, *0.29*, 0.29, 0.29, 0.3, 0.32, 0.32, *0.33*, 0.34, 0.36, 0.37, *0.39*, 0.39, 0.42, 0.42, 0.43, *0.48*, 0.51, 0.53, 0.55, 0.65, 0.86, 1.2, *1.3* mg/kg (italic = pear residues).

Based on the combined dataset for apples and pears, the Meeting estimated a maximum residue level of 2 mg/kg and a STMR value of 0.35 mg/kg for boscalid in pome fruit.

The Meeting withdraws its previous recommendation of 2 mg/kg for boscalid in apples.

Stone fruit

The 2006 JMPR Meeting estimated a maximum residue level of 3 mg/kg and a STMR value of 1.21 mg/kg for boscalid in stone fruit based on a GAP from the USA (5×0.26 kg ai/ha, 0 day PHI). The current Meeting received new GAP information for stone fruit with supporting supervised field trials on cherries, peaches and plums.

Boscalid is registered in Austria for use on stone fruits with a maximum GAP involving three foliar sprays of 0.19 kg ai/ha each (10 day interval) and a PHI of 7 days.

Supervised field trials conducted in Europe on cherries were newly submitted approximating the GAP from Austria. Although treated at intervals slightly longer than the cGAP, the Meeting considered this deviation as insignificant since boscalid residues remain stable on treated fruits.

For peaches and plums, new supervised field trials from Europe were submitted involving four or five instead of three sprays at 0.2 kg ai/ha. However, the Meeting noted that the first sprays were conducted at flowering and/or beginning of fruit development, not contributing to the final residue at harvest. Therefore, the Meeting concluded that the treatment regime used in the submitted trials approximates the Austrian GAP and that the data can be used for an assessment.

Residues of boscalid in cherries were (n=16): <0.05, < 0.05, 0.052, 0.088, 0.096, 0.14, 0.14, 0.16, 0.22, 0.36, 0.37, 0.39, 0.47, 0.66, 0.7, 1.3 mg/kg.

Residues of boscalid in peaches were (n=8): 0.05, 0.15, 0.17, 0.21, 0.21, 0.29, 0.35, 0.35 mg/kg.

Residues of boscalid in plums were (n=10): 0.057, 0.07, 0.08, 0.11, 0.13, 0.15, 0.18, 0.23, 0.27, 0.45 mg/kg.

Boscalid is registered in the USA for use on stone fruits with a critical GAP involving five foliar sprays of 0.26 kg ai/ha each (7 day interval) and a PHI of 0 days.

New supervised field trials conducted in Canada and in the USA on cherries, peaches and plums approximating the GAP from the USA were submitted. In addition, the current Meeting considered residue data on stone fruit evaluated by the 2006 JMPR against the GAP from the USA.

Residues of boscalid in cherries were (n=14): 0.055, *0.76*, *1.0*, *1.2*, 1.2, 1.4, *1.5*, *1.5*, *1.*5, 1.5, *1.6*, 1.6, 2.6, 2.6 mg/kg.

Residues of boscalid in peaches were (n=19): *0.19*, *0.32*, *0.4*, *0.42*, *0.48*, *0.49*, *0.49*, *0.52*, 0.6, *0.60*, 0.64, 0.71, *0.73*, *0.75*, *0.78*, 0.79, 1.0, *1.2*, 3.6 mg/kg.

Residues of boscalid in plums were (n=15): <0.05, *0.1*, *0.11*, 0.12, 0.13, *0.15*, *0.17*, *0.25*, *0.32*, *0.46*, *0.54*, *0.57*, 0.6, *0.7*, 0.76 mg/kg.

(italic = 2006 residue data)

The Meeting noted that the US GAP for stone fruit results in higher residues than the Austrian GAP and decided to explore the possibility for a group recommendation based on it. However, median residues differ by more than a factor of 5, suggesting significant differences in residues between the three commodities investigated. Therefore, the Meeting decided to base its recommendation on the individual sub-groups of cherries, plums and peaches.

The Meeting estimated maximum residue levels and STMR values for boscalid of 5 mg/kg and 1.5 mg/kg for cherries (subgroup 003A) and of 4 mg/kg and 0.6 mg/kg for peaches (subgroup 003C), respectively.

The Meeting also estimated a maximum residue level of 1.5 mg/kg and a STMR value of 0.25 mg/kg for plums (subgroup 003B), because of the significantly lower residue population in plums compared to other members of the stone fruit group and due to the availability of a specific subgroup for plums.

The Meeting withdraws its previous recommendation of 3 mg/kg for boscalid in stone fruit.

Berries and other small fruits, except strawberries and grapes

For boscalid, the 2006 JMPR Meeting recommended a maximum residue level of 10 mg/kg and estimated a STMR value of 2.53 mg/kg for berries and other small fruits, except strawberries and grapes based on a US GAP (4×0.4 kg ai/ha, PHI 0 days). The current Meeting received new GAP information for bush berries and cane berries with supporting supervised field trials.

Boscalid is registered in the USA for use on bush berries and cane berries with a maximum GAP identical to the one considered by the 2006 Meeting involving four foliar sprays of 0.4 kg ai/ha each (7 day interval) and a PHI of 0 days.

Two new supervised field trials conducted in Canada and the USA on blueberries were submitted to the Meeting approximating the GAP from the USA. In addition, supervised field trials on blueberries and caneberries were evaluated by the 2006 Meeting against the same GAP.

Residues of boscalid in blueberries were (n=12): 0.84, 1.2, 1.2, 1.3, 1.4, *2.0*, 2.4, 2.6, 3.8, 4.4, *5.4*, 6.8 mg/kg (italic=new trial data).

Residues of boscalid in raspberries were (n=6): 1.5, 2.0, 2.4, 2.7, 3.5, 3.7 mg/kg.

The Meeting noted that residues in blueberries and raspberries were not significantly different (confirmed by Whitney-Mann-U Test) and decided to combine the data for a group recommendation.

Combined residues of boscalid in blueberries and raspberries were (n=18): 0.84, 1.2, 1.2, 1.3, 1.4, 1.5, 2.0, 2.0, 2.4, 2.4, 2.6, 2.7, 3.5, 3.7, 3.8, 4.4, 5.4, 6.8 mg/kg.

The Meeting noted that the OECD MRL Calculator result for the combined dataset is 10 mg/kg, which is covered by the previous recommendation. The Meeting confirmed its previous recommendation for boscalid in small fruits and berries, except strawberry and grapes.

Avocado

Boscalid is registered for use on tropical fruits (including avocado) in the USA with a maximum GAP involving two foliar sprays of 0.33 kg ai/ha each (7 day interval) and a PHI of 0 days.

Supervised field trials conducted in the USA on avocado were submitted involving four instead of two treatments (7 day interval) with higher individual rates per treatment than the GAP (0.41 kg ai/ha vs. 0.33 kg ai/ha).

The Meeting concluded that the supervised field trial data submitted was conducted at significantly more critical conditions (>+25%) than the US GAP and decided that the data is insufficient for a recommendation.

Mango

The critical GAP for boscalid in mangoes is from Mexico, involving two foliar sprays at 0.3 kg ai/ha each (7 day interval) with a PHI of 0 days. Two supervised field trials from Brazil approximating this GAP were submitted.

Residues of boscalid in mango (whole fruits, calculated) approximating the Mexican GAP were (n=2): 0.032 and 0.54 mg/kg.

The Meeting concluded that two trials are insufficient for a recommendation based on the Mexican GAP.

The critical GAP for boscalid on mango in Brazil is two foliar sprays of 0.024 kg ai/hl each (15 day interval) with a PHI of 7 days.

Supervised field trials conducted in Brazil were submitted approximating the GAP. In some trials, the stone was removed already in the field. Since metabolism information indicates that boscalid is stable both in primary plants and rotational crops, in freezer storage and during simulated hydrolysis, the Meeting decided that no significant impact on the residue in the remaining fruit has to be expected from the procedure in the field.

Residues of boscalid in mango (whole fruits, calculated) approximating Brazilian GAP were (n=8): 0.032, 0.1, 0.22, 0.25, 0.26, 0.55, 0.68, 1.0 mg/kg.

Based on the dataset for mango according to the Brazilian GAP, the Meeting estimated a maximum residue level of 2 mg/kg and a STMR value of 0.255 mg/kg for boscalid in mangoes.

Pomegranate

Boscalid is registered in Turkey for use on pomegranates with a maximum GAP involving three foliar sprays of 0.0126 kg ai/hl each (bud formation, end of flowering (loss of calix) and close to harvest) without specified PHI.

Supervised field trials on pomegranate from Europe were submitted, involving two applications directly before harvest at a 5 day interval.

The Meeting concluded that these trials do not match the GAP from Turkey.

Tea, green, black (black, fermented and dried)

Boscalid is registered in Japan for use on tea with a maximum GAP involving two foliar sprays of a factor 2000 diluted product (WG formulation, 13.6% boscalid, calculated: 0.0068 kg ai/hL) each corresponding to a maximum calculated rate of 0.27 kg ai/ha in combination with a PHI of 7 days.

The Meeting received eight supervised trials from China, India, Japan and Taiwan Province of China on tea approximating the highest calculated rate per hectare according to GAP.

Based on the calculated maximum treatment rate of 0.27 kg ai/ha the estimated residues in dried green tea were (n=8): 1.7, 4.1, 5.6, 6.2, 6.3, 7.3, 16, 19 mg/kg.

Based on the dataset for tea according to the Japanese GAP, the Meeting estimated a maximum residue level of 40 mg/kg and a STMR value of 6.25 mg/kg for tea, green, black (black, fermented and dried).

Fate of residues during processing

Processing factors for the commodities considered at this Meeting are summarized below based on the estimations of the 2006 JMPR.

Raw commodity	Processed commodity	Boscalid	
		Median or best estimate processing factor	STMR-P (mg/kg)
Apple	Wet apple pomace	6.06	2.121
(STMR:0.35 mg/kg)	Juice	0.08	0.028
Plums (STMR:0.25 mg/kg)	Dried prunes	2.8	0.7
	Puree	1.95	0.49
Tea, black	Infusion	<0.002, 0.002, <0.02	0.0125
(STMR=6.25 mg/kg)	Instant tea	0.005, 0.007, <0.02	0.044

Based on a maximum residue level of 1.5 mg/kg for plums the Meeting estimated a maximum residue level of 5 mg/kg for boscalid in prunes, dried to replace its previous recommendation of 10 mg/kg.

Residues in animal commodities

The only feed commodity affected by the current recommendations is dry apple pomace, which was already considered by all previous Meetings for boscalid residues. Since the new recommendation for boscalid in pome fruit is slightly lower than the previous recommendation for apples (2006: STMR 0.365 mg/kg for apples, 2019: 0.35 mg/kg for pome fruit), no re-calculation of the livestock animal dietary burden is necessary.

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 for plant and animal commodities and dietary risk assessment for plant commodities: *boscalid.*

Definition of the residue for dietary risk assessment for animal commodities: *sum of boscalid, 2-chloro-N-(4'-chloro-5-hydroxybiphenyl-2-yl)nicotinamide (M510F01) including its conjugate, expressed as boscalid.*

The residue is *fat-soluble*.

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg
		New	Previous	
FP 0226	Apple	W	2	-
003A	Cherries (subgroup)	5	-	1.5
FI 0345	Mango	2	-	0.255
003C	Peaches (subgroup)	4	-	0.6
003B	Plums (subgroup)	1.5	w	0.25
FP 0009	Pome fruit	2	-	0.35
DF 0014	Prunes, dried	5	10	0.7
FS 0012	Stone fruit	W	3	-
DT 1114	Tea, green, black (black, fermented and dried)	40	-	6.25
	Apple, juice			0.028
	Dried prunes			0.7
	Plum, puree			0.49
	Tea, infusion			0.0125
	Tea, instant tea			0.044

Additional values used in estimating livestock dietary burdens.

Codex classification	Commodity	Median residue (-P) (mg/kg)	Highest residue (-P) (mg/kg)
	Apple, wet pomace	2.121	-

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for boscalid is 0–0.04 mg/kg bw. The International Estimated Daily Intakes (IEDIs) for boscalid 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 2019 Extra JMPR Report.

The IEDIs ranged from 10–60% of the maximum ADI. The Meeting concluded that long-term dietary exposure to residues of boscalid 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 boscalid was unnecessary. The Meeting therefore concluded that the acute dietary exposure to residues of boscalid 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 M510F47 could be assessed using the TTC approach (Cramer Class III threshold of 1.5 µg/kg bw per day). Since this metabolite was not identified in food or feed commodities, the Meeting concluded that it is unlikely to present a public health concern.

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Code	Author	Year	Title, Institute, Report reference
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BOSC19E_029	Goetz N. von	2002	Photolysis of BAS 510 F in a natural water; BASF AG, Limburgerhof, Germany Fed.Rep.; 2002/1000211; GLP: yes; Unpublished
BOSC19E_030	Hassink J.	2002	Report amendment No. 1 to final report: Photolysis of BAS 510 F in a natural water; BASF AG, Limburgerhof, Germany Fed.Rep.; 2002/1005320; GLP: yes; Unpublished
BOSC19E_031	Martin T.	2015	BAS 510 F on rotational crops: Fruiting (cucumber, zucchini, tomato) and leafy vegetables (lettuce) after one application of BAS 510 01 F to bare soil 30 days before seeding/planting, field conditions in Germany Netherlands Italy Spain 2015; Agrologia SLU, Utrera, Spain; 2015/1117845; GLP: yes; Unpublished
BOSC19E_032	Mackenroth C.,Lehmann A.	2007	Validation of BASF method No. 535/1 in plant matrices; BASF AG, Limburgerhof, Germany Fed.Rep.; 2006/1039427; GLP: yes; Unpublished
BOSC19E_033	Austin R.	2015	Validation of BASF analytical method number L0076/01 for the determination of BAS 510 F (Boscalid) in hops, spices and herbal infusions; Battelle UK Ltd., Chelmsford Essex CM2 5LB, United Kingdom; 2015/1091103; GLP: yes; Unpublished
BOSC19E_034	Jones J.E.	2001	Method for determination of BAS 500 F, BF 500-3 and BAS 510 F residues in plant matrices using LC/MS/MS (Validation of Method D9908); BASF Agro

Code	Author	Year	Title, Institute, Report reference
			Research RTP, Research Triangle Park NC, United States of America; 2001/5001020; GLP: yes; Unpublished
BOSC19E_035	Schernikau N.	2015	Validation of the analytical method QuEChERS for the determination of BAS 510 F (Boscalid) in foodstuff of plant origin; Eurofins Agroscience Services Chem GmbH, Hamburg, Germany Fed.Rep.; 2015/1114667; GLP: yes; Unpublished
BOSC19E_036	Courtois J. *et al*.	2015	Report Amendment No.2: The validation of BASF method 471/0: The determination of BAS 510 F and the metabolite M510F01 in animal matrices; BASF AG, Limburgerhof, Germany Fed.Rep.; 2015/1174463; GLP: yes; Unpublished
BOSC19E_037	Weber H.,Zetzsch A.	2015	Independent laboratory validation of the BASF method L0041/01 (471/0) for the determination of BAS 510 F (Boscalid) and metabolite M510F01 in animal matrices; Eurofins Agroscience Services Chem GmbH, Hamburg, Germany Fed.Rep.; 2015/1114666; GLP: yes; Unpublished
BOSC19E_038	Weber H.	2015	Amendment No. 1 - Independent laboratory validation of the BASF method L0041/01 (471/0) for the determination of BAS 510 F (Boscalid) and metabolite M510F01 in animal matrices; Eurofins Agroscience Services Chem GmbH, Hamburg, Germany Fed.Rep.; 2015/1251211; GLP: yes; Unpublished
BOSC19E_039	Obermann M.	2009	Validation of analytical method L0096/01: Determination of Boscalid Reg.No. 300355 in soil using HPLC/MS-MS; BASF SE, Limburgerhof, Germany Fed.Rep.; 2008/1084832; GLP: yes; Unpublished
BOSC19E_040	Obermann M.,Studenroth S.	2015	Report amendment no. 1 - Validation of analytical method L0096/01: Determination of Boscalid Reg.No. 300355 in soil using HPLC/MS-MS; BASF SE, Limburgerhof, Germany Fed.Rep.; 2015/1174527; GLP: yes; Unpublished
BOSC19E_041	Kreidler D.	2013	Validation of analytical method for determination of residues of M510F47 and M510F49 in soil; Eurofins Agroscience Services EcoChem GmbH, Niefern-Oeschelbronn, Germany Fed.Rep.; 2013/1415720; GLP: yes; Unpublished
BOSC19E_042	Ertunc T.,Studenroth S.	2015	Comparative analysis of extraction procedures on Boscalid (BAS 510 F) originating from a field accumulation and dissipation study in Northern Italy; BASF SE, Limburgerhof, Germany Fed.Rep.; 2015/1109589; GLP: yes; Unpublished
BOSC19E_043	Saha M.G.	2001	Validation of BASF method no. D0004 and D0004 /1: The determination of residues of BAS 510 F and its metabolites in soil using LC-MS/MS; BASF Agro Research RTP, Research Triangle Park NC, United States of America; 2001/5000881; GLP: yes; Unpublished
BOSC19E_044	Penning H.	2009	Validation of analytical method L0127/1 for the determination of BAS 510 F (Boscalid) residues in surface water and groundwater; BASF SE, Limburgerhof, Germany Fed.Rep.; 2008/1086809; GLP: yes; Unpublished
BOSC19E_045	Ertunc T.,Studenroth S.	2015	Report Amendment No. 1 to final report: Validation of analytical method L0127/1 for the determination of BAS 510 F (Boscalid) residues in surface water and groundwater; BASF SE, Limburgerhof, Germany Fed.Rep.; 2015/1174526; GLP: yes; Unpublished
BOSC19E_046	Ertunc T.,Studenroth S.	2015	Validation of analytical method L0127/02 for the determination of M510F47 (Reg.No. 107371) and M510F49 (Reg.No. 391572) in surface and groundwater; BASF SE, Limburgerhof, Germany Fed.Rep.; 2015/1109588; GLP: yes; Unpublished
BOSC19E_047	Goecer M.	2016	Independent laboratory validation (ILV) of the BASF method L0127 for the determination of Boscalid (BAS 510 F) and two of its metabolites M510F47 (Reg.No. 107371) and M510F49 (Reg.No. 391572) in surface water and groundwater; Eurofins Agroscience Services EcoChem GmbH, Niefern-Oeschelbronn, Germany Fed.Rep.; 2016/1112645; GLP: yes; Unpublished
BOSC19E_048	Kukel C. *et al*.	2002	The magnitude of BAS 500 F and BAS 510 F residues in pome fruit; BASF Agro Research RTP, Research Triangle Park NC, United States of America; 2002/5002108; GLP: yes; Unpublished
BOSC19E_054	Schulz H.	2004	Study on the residue behaviour of BAS 216 F, BAS 500 F, BF 500-3 and BAS 510 F in apples after application of either BAS 584 GB F, BAS 216 03 F or BAS 516 01 F under field conditions in France, Belgium, Italy and the Netherlands, 2003; Institut Fresenius Chemische und Biologische Laboratorien AG, Taunusstein, Germany Fed.Rep.; 2004/1000752; GLP: yes; Unpublished

Code	Author	Year	Title, Institute, Report reference
BOSC19E_055	Schneider E.	2016	Study on the residue behaviour of Pyraclostrobin (BAS 500 F) and Boscalid (BAS 510 F) after treatment with BAS 516 04 F on pears under field conditions in Northern and Southern Europe, 2014; Anadiag SA, Haguenau, France; 2016/1041500; GLP: yes; Unpublished
BOSC19E_056	Borges Z.	2003	Study on the residues of BAS 500 F, BF 500-3 (metabolite) and BAS 510 F in apple (whole fruit) from Argentina; BASF SA, Resende, Brazil; 2003/1026457; GLP: yes; Unpublished
BOSC19E_057	Jose W.F.P. de	2016	Analysis of Pyraclostrobin and Boscalid residues in apple (whole fruit) after treatment with BAS 516 04 F under field non-GLP conditions in Argentina; BASF SA, Guaratingueta, Brazil; 2016/3004409; GLP: yes; Unpublished
BOSC19E_058	Jose W.F.P. de	2016 b	Analysis of Pyraclostrobin and Boscalid residues in pear (whole fruit) after treatment with BAS 516 04 F under field non-GLP conditions in Argentina; BASF SA, Guaratingueta, Brazil; 2016/3004402; GLP: yes; Unpublished
BOSC19E_061	White M.T.	2008	Magnitude of BAS 500 F and BAS 510 F residues in cherries, peaches and plums following applications of BAS 516 04 F; BASF Agro Research RTP, Research Triangle Park NC, United States of America; 2007/7013460; GLP: yes; Unpublished
BOSC19E_062	Schulz H.	2001	Determination of the residues of BAS 510 F and BAS 500 F in stone fruit following treatment with BAS 516 GA F under field conditions in Italy and France 1999; Institut Fresenius Chemische und Biologische Laboratorien GmbH, Taunusstein, Germany Fed. Rep.; 2001/1000934; GLP: yes; Unpublished
BOSC19E_063	Raunft E.,Funk H.	2001	Determination of the residues of BAS 500 F and BAS 510 F in stone fruit following treatment with BAS 516 GA F under field conditions in Denmark and Germany, 1999; BASF AG, Limburgerhof, Germany Fed.Rep.; 2001/1006132; GLP: yes; Unpublished
BOSC19E_064	Raunft E. *et al*.	2001	Study on the residue behavior of BAS 500 F and BAS 510 F in stone fruit after treatment with BAS 516 GA F under field conditions in Germany and Sweden, 2000; BASF AG, Limburgerhof, Germany Fed.Rep.; 2001/1006133; GLP: yes; Unpublished
BOSC19E_065	Blaschke U.	2001	Determination of the magnitude of the residue of BAS 516 GA F in/on cherry raw agricultural commodity specimens from supervised field trials in northern and southern Europe in 2000; Huntingdon Life Sciences Ltd., Huntingdon Cambridgeshire PE28 4HS, United Kingdom; 2001/1009061; GLP: yes; Unpublished
BOSC19E_066	Schulz H.	2004	Study on the residue behaviour of BAS 510 F and BAS 500 F in cherries after application of BAS 516 00 F under field conditions in Denmark, France (North and South), Germany, Italy and Sweden, 2003; Institut Fresenius Chemische und Biologische Laboratorien AG, Taunusstein, Germany Fed.Rep.; 2004/1010551; GLP: yes; Unpublished
BOSC19E_067	Reichert N.	2005	Study on the residue behaviour of BAS 500 F and BAS 510 F in cherries after treatment with BAS 516 00 F under field conditions in Denmark, Germany, Northern and Southern France, Italy and Sweden, 2004; SGS Institut Fresenius GmbH, Taunusstein, Germany Fed. Rep.; 2005/1004972; GLP: yes; Unpublished
BOSC19E_068	Starner V.R.	2005	BAS 516 F: Magnitude of the residue on fresh market stone fruits (peach, cherry, plum) following post-harvest applications; Rutgers State University of New Jersey, North Brunswick NJ, United States of America; 2005/7004639; GLP: yes; Unpublished
BOSC19E_069	Fleischer G.	2015	Residue of Boscalid (BAS 510 F) and Pyraclostrobin (BAS 500 F) on cherries (sweet and sour) after treatment with BAS 516 07 F under field conditions in Germany, Netherlands and Poland, 2015; BASF SE, Limburgerhof, Germany Fed.Rep.; 2016/1000745; GLP: yes; Unpublished
BOSC19E_070	Fleischer G.	2017	Study on the residue behaviour of Boscalid (BAS 510 F) and Pyraclostrobin (BAS 500 F) on cherries (sweet and sour) after treatment with BAS 516 07 F under field conditions in Austria, Netherlands, Germany and Hungary, 2016; BASF SE, Limburgerhof, Germany Fed.Rep.; 2017/1000803; GLP: yes; Unpublished
BOSC19E_072	White M.T.	2007	Magnitude of BAS 500 F and BAS 510 F residues in blueberries following applications of BAS 516 04 F; BASF Corp. Agricultural Products Center,

Code	Author	Year	Title, Institute, Report reference
			Research Triangle Park NC, United States of America; 2007/7013452; GLP: yes; Unpublished
BOSC19E_074	Goebel R. *et al*.	2011	Residue behaviour of Boscalid and Pyraclostrobin in/on currants and bilberry after indoor application of Signum, BAS 516 07 F (WG 33,4, 267 g/kg Boscalid, 67 g/kg Pyraclostrobin) in Germany, 2009; IVPT - Institut fuer Veterinaer-Pharmakologie und Toxikologie GmbH, Bernau, Germany Fed.Rep.; 2010/1224114; GLP: yes; Unpublished
BOSC19E_075	Carpenter D.H.	2006	Volume 5 - BAS 516 (BAS 500 + BAS 510): Magnitude of the residue on avocado; BASF Agro Research RTP, Research Triangle Park NC, United States of America; 2006/1045610; GLP: yes; Unpublished
BOSC19E_076	Silva M.A.D.,Alves M.	2011	Estudo de residuos de Boscalid e Kresoxim-methyl em manga (frutos), apos tratamento com Collis, em condicoes de campo no Brasil; BASF SA, Guaratingueta, Brazil; 2011/1226624; GLP: yes; Unpublished
BOSC19E_077	Duchen Silva M.A.	2011	Adendo 01 - Estudo de residuos de Boscalid e Kresoxim-methyl em manga (frutos), apos tratamento com Collis, em condicoes de campo no Brasil; BASF SA, Guaratingueta, Brazil; 2011/1266277; GLP: yes; Unpublished
BOSC19E_078	Silva M.A.D.,Alves M.	2011	Study of Boscalid and Kresoxim-methyl residue in mango (fruit) after treatment with Collis under field conditions in Brazil; BASF SA, Guaratingueta, Brazil; 2011/3008004; GLP: yes; Unpublished
BOSC19E_079	Silva M.A.D.	2011 a	Addendum 01 - Study of Boscalid and Kresoxim-methyl residue in mango (fruit) after treatment with Collis under field conditions in Brazil; BASF SA, Guaratingueta, Brazil; 2011/3008003; GLP: yes; Unpublished
BOSC19E_080	Silva M.A.D.	2015	Residue study of Boscalid and Kresoxim-methyl in mango (fruit) after treatment with Collis (BAS 517 01 F) under field conditions in Brazil; BASF SA, Guaratingueta, Brazil; 2015/3002561; GLP: yes; Unpublished
BOSC19E_081	Silva M.A.D.	2015	Amendment 01 to the final report - Residue study of Boscalid and Kresoxim-methyl in mango (fruit) after treatment with Collis® (BAS 517 01 F) under field conditions in Brazil; BASF SA, Guaratingueta, Brazil; 2015/3002961; GLP: yes; Unpublished
BOSC19E_082	Galvez O.	2018	Study on the residue behaviour of Boscalid (BAS 510 F) on pomegranate after treatment with BAS 510 01 F under field conditions in Southern Europe, season 2017; Agricultura y Ensayo SL, Alcala de Guadaira, Spain; 2018/1013073; GLP: yes; Unpublished
BOSC19E_083	Lenz C.A.	2017	Study on the residue behaviour of Pyraclostrobin and Boscalid and their metabolites in tea and tea processed products after treatment of BAS 516 05 F under field conditions 2014; SynTech Research Laboratory Services LLC, Stilwell KS, United States of America; 2015/1086962; GLP: yes; Unpublished

CHLORANTRANILIPROLE (230)

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

EXPLANATION

Chlorantraniliprole is an insecticide that operates by a highly specific biochemical mode of action. It was first evaluated for residues and toxicological aspects by the 2008 JMPR. The 2008 JMPR established an ADI for chlorantraniliprole of 0–2 mg/kg bw and concluded that an ARfD was unnecessary.

The 2008 JMPR also recommended the following residue definition for Chlorantraniliprole:

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

The residue is fat-soluble.

Chlorantraniliprole was last evaluated in 2016 for additional maximum residue levels. At the Fiftieth Session of the CCPR, chlorantraniliprole was listed for consideration of additional uses by the 2019 Extra JMPR. The Meeting received information on registered use patterns, supervised residue trials on beans, peas and oil palm with product labels from Malaysia and the USA.

RESIDUE ANALYSIS

Analytical methods

Chlorantraniliprole was first evaluated by the JMPR in 2008. Supervised field trials submitted to the current Meeting were analysed using a slightly modified method 13294 or method 13261 both already evaluated by the 2008 JMPR based on LC-MS/MS. The following additional recovery data were reported for these methods:

Table 1 Recovery data for method 13294 (modified) in plant matrices (Dorsey, S., 2018, CHLORANT19E_001)

Matrix	Fortification level (mg/kg)	n	Recovery in % (mean)	RSD (%)	Analyte
Peas, dry without shell	0.01	5	90-98 (92)	3	Chlorantraniliprole
	0.1	5	87-102 (96)	7	

Table 2 Recovery data for method 13261 in plant matrices (Petrova, D., 2017, CHLORANT19E_002)

Matrix	Fortification level (mg/kg)	n	Recovery in % (mean)	RSD (%)	Analyte, Mass transition
Palm oil fruit	0.01	7	74-89 (79)	6	Chlorantraniliprole
	0.1	7	87-107 (92)	8	m/z 484 → 453
	0.5	1	78	-	
	0.01	5	79-88 (84)	4	Chlorantraniliprole
	0.1	5	89-101 (93)	5	m/z 484 → 286
Kernel oil	0.01	7	89-108 (101)	7	Chlorantraniliprole
	0.1	7	69-81 (76)	5	m/z 484 → 453
	0.01	5	106-109 (108)	1	Chlorantraniliprole
	0.1	5	77-84 (81)	4	m/z 484 → 286

Matrix	Fortification level (mg/kg)	n	Recovery in % (mean)	RSD (%)	Analyte, Mass transition
Oil palm kernels	0.01	2	71-73 (72)	-	Chlorantraniliprole
	0.1	2	85-88 (86)	-	m/z 484 → 453
Oil palm Mesocarp oil	0.01	2	82-99 (95)	-	Chlorantraniliprole
	0.1	2	72-81 (76)	-	m/z 484 → 453
	1.0	1	93	-	
Oil palm Mesocarp cake	0.01	2	92-98 (95)	-	Chlorantraniliprole
	0.1	2	81-119 (100)	-	m/z 484 → 453
	0.5	1	82	-	
Oil palm Kernel cake	0.01	2	88-93 (90)	-	Chlorantraniliprole
	0.1	2	84-88 (86)	-	m/z 484 → 453

USE PATTERN

Chlorantraniliprole is intended for insecticidal use in beans, peas and oil palm by a foliar spray application in Malaysia and the USA.

Table 3 List of uses of Chlorantraniliprole

Crops or crop groups	Country	Application detail				
		kg ai/ha	Indoor/ Outdoor	No.	Interval in days	Pre harvest interval (PHI) in days
Legume Vegetables (succulent or dried) [a]	USA	0.11 (max. 0.225 kg ai/ha and season)	Outdoor	4	3	1
Oil Palm	Malaysia	0.03	Outdoor	2	14	1

[a] Including Bean (Lupinus) (includes grain lupin, sweet lupin, white lupin, and white sweet lupin); bean (Phaseolus) (includes field bean, kidney bean, lima bean, navy bean, pinto bean, runner bean, snap bean, tepary bean, wax bean); bean (Vigna) (includes adzuki bean, asparagus bean, blackeyed pea, catjang, Chinese longbean, cowpea, crowder pea, moth bean, mung bean, rice bean, southern pea, urd bean, yardlong bean); broad bean (fava); chickpea (garbanzo); guar; jackbean; lablab bean; lentil; pea (Pisum) (includes dwarf pea, edible-podded pea, English pea, field pea, garden pea, green pea, snowpea, sugar snap pea); pigeon pea; sword bean

RESULTS OF SUPERVISED RESIDUE TRIALS ON CROPS

Residue levels were reported as measured. Application rates were always reported as chlorantraniliprole 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. The values from the trials conducted according to maximum GAP selected for the estimation of maximum residue levels, STMR and HR are underlined.

Laboratory reports included method validation including batch recoveries with spiking at residue levels similar to those occurring in samples from the supervised trials. Dates of analyses or duration of residue sample storage were also provided. Field reports provided data on the sprayers used and their calibration, plot size, residue sample size and sampling date. Although trials included control plots, no control data are recorded in the tables except where residues in control samples exceeded the LOQ. Residue data are recorded unadjusted for pecent recovery.

Chlorantraniliprole - supervised residue trials

Commodity	Indoor/Outdoor	Treatment	Countries	Table
Beans	Outdoor	Foliar	USA	4
Peas	Outdoor	Foliar	USA	5
Oil palm	Outdoor	Foliar	Malaysia	6

Table 4 Residues of chlorantraniliprole following spray treatment on beans, dry

Location, Year (variety)	Application			Residues, mg/kg			Report/Trial No., Reference, analytical method, validation data, storage interval
	kg ai/ha	Inter-val days	kg ai/hL	Sample	DALA	Parent	
USA: 2×0.11 kg ai/ha (max. 0.225 kg ai/ha and season), 3 d RTI, 1 d PHI							
USA, Richland (IA) 2017 (Red Kidney Dark)	2 × 0.11	3	0.051	Dry seed	1	0.022, 0.028 (0.025)	48825-06, CHLORANT19E_001 Method: 13294 Storage interval:6 months
USA, York (NE) 2017 (Great Northern)	2 × 0.11	3	0.06	Dry seed	1	0.044, 0.058 (0.051)	48825-07, CHLORANT19E_001 Method: 13294 Storage interval:6 months
USA, Northwood (ND) 2017 (Medalist)	2 × 0.11	3	0.059	Dry seed	1	0.011, 0.011 (0.011)	48825-08, CHLORANT19E_001 Method: 13294 Storage interval:6 months
USA, Carlyle (IL) 2017 (Pinto)	2 × 0.11	3	0.062 0.085	Dry seed	1	0.01, 0.022 (0.016)	48825-09, CHLORANT19E_001 Method: 13294 Storage interval:6 months
USA, Vevla (ND) 2017 (T-9905)	2 × 0.11	3	0.096	Dry seed	1	0.013, 0.013 (0.013)	48825-10, CHLORANT19E_001 Method: 13294 Storage interval:6 months

DALA: days after last application

Table 5 Residues of chlorantraniliprole following spray treatment on peas, dry

Location, Year (variety)	Application			Residues, mg/kg			Report/Trial No., Reference, analytical method, validation data, storage interval
	kg ai/ha	Inter-val days	kg ai/hL	Sample	DALA	Parent	
USA: 2×0.11 kg ai/ha (max. 0.225 kg ai/ha and season), 3 day RTI, 1 day PHI							
USA, Jerome (ID) 2017 (Strike)	2 × 0.11	3	0.056	Dry seed	1	0.037, 0.035 (0.036)	48825-01, CHLORANT19E_001 Method: 13294 Storage interval:6 months
USA, Ephrata (WA) 2017 (Dundale)	2 × 0.11	3	0.059	Dry seed	1	0.059, 0.054 (0.056)	48825-02, CHLORANT19E_001 Method: 13294 Storage interval:6 months
USA, Oregon City (OR) 2017	2 × 0.11	3	0.048	Dry seed	1	0.19, 0.16 (0.18)	48825-03, CHLORANT19E_001 Method: 13294

Location, Year (variety)	Application			Residues, mg/kg			Report/Trial No., Reference, analytical method, validation data, storage interval
	kg ai/ha	Inter-val days	kg ai/hL	Sample	DALA	Parent	
(Columbia Green Peas)							Storage interval:6 months
USA, Payette (ID) 2017 (Wando)	2 × 0.11	3	0.04	Dry seed	1	0.021, 0.026 (0.024)	48825-04, CHLORANT19E_001 Method: 13294 Storage interval:6 months
USA, Parkdale (OR) 2017 (Progress #9)	2 × 0.11	3	0.058	Dry seed	1	0.047, 0.061 (0.054)	48825-05, CHLORANT19E_001 Method: 13294 Storage interval:6 months

DALA: days after last application

Table 6 Residues of chlorantraniliprole following spray treatment on oil palms

Location, Year (variety)	Application				Residues, mg/kg			Report/Trial No., Reference, analytical method, validation data, storage interval
	kg ai/ha	Inter-val days	kg ai/hL	Growth stage	Sample	DALA	Parent	
Malaysia: 2×0.03 kg ai/ha, 14 day RTI, 1 day PHI								
Malaysia, Selangor 2015 (Temera DxP)	2 × 0.03	14	2 × 0.015	BBCH 61-85	Palm fruit	0 1 3 7 14 21	0.091 0.25 0.27 0.22 0.29 0.38	MRID50234701-S15-04277-01, CHLORANT19E_002 Method: DuPont 13261 Storage interval: 4 months
	2 × 0.06	14	2 × 0.03	BBCH 61-85	Palm fruit	0 1 3 7 14 21	0.3 0.36 0.2 0.12 0.35 0.33	
Malaysia, Melaka 2015 (DxP)	2 × 0.03	14	2 × 0.015	BBCH 61-85	Palm fruit	1	0.18	MRID50234701-S15-04277-02, CHLORANT19E_002 Method: DuPont 13261 Storage interval: 4 months
	2 × 0.06	14	2 × 0.03	BBCH 61-85	Palm fruit	1	0.23	
Malaysia, Sungkai 2015 (Young Gambi)	2 × 0.03	14	2 × 0.015	BBCH 61-85	Palm fruit	1	0.2	MRID50234701-S15-04277-03, CHLORANT19E_002 Method: DuPont 13261 Storage interval: 4 months
	2 × 0.064	14	2 × 0.03	BBCH 61-85	Palm fruit	1	0.29	
Malaysia, Slim River 2015 (DxP)	2 × 0.03	14	2 × 0.015	BBCH 61-85	Palm fruit	1	0.19	MRID50234701-S15-04277-04 CHLORANT19E_002 Method: DuPont 13261 Storage interval: 4 months
	2 × 0.062	14	2 × 0.03	BBCH 61-85	Palm fruit	1	0.52	

DALA: days after last application

FATE OF RESIDUES DURING PROCESSING

Residues after processing

The fate of Chlorantraniliprole during processing of raw agricultural commodity (RAC) was investigated in supervised field trials on oil palms.

In the study conducted by Petrova, D. (2017, CHLORANT19E_002), oil palm fruits were processed into mesocarp oil and cake, kernels, kernel oil and kernel cake.

Processing to mesocarp cake and mesocarp oil: Spikelets were sterilised to gain sterilised fruits. To gain the mesocarp oil the sterilised fruits were pressed immediately after sterilization to receive raw mesocarp oil. After filtration/dehydration of the oil, fractions of samples were taken. The pressed fruits were separated into mesocarp cake and nuts.

Processing into kernels, kernel cake and kernel oil: After drying, an aliquot of the nuts were separated into kernels and shells. Sample fractions were taken from the dried kernels. The rest of the kernels were ground to raw kernel cake. An aliquot of the ground kernel was used for extraction to gain palm kernel oil and kernel cake after extraction.

In the following table the processing factors derived from the supervised field trial results (see section Residues from supervised field trials) are summarized:

Table 7 Processing factors for chlorantraniliprole in processed oil palm fruits based on supervised field trial data

Trial, Location	Application	Matrix	Chlorantraniliprole in mg/kg	PF
MRID50234701-S15-04277-01 Malaysia, Selangor	2 × 0.03 kg ai/ha, 14 d interval, 1 DALA	Oil palm fruits (RAC) Mesocarp oil Mesocarp cake Kernels Kernel oil Kernel cake	0.19 0.36 0.072 <0.01 0.02 <0.01	- 1.9 0.38 <0.05 0.11 <0.05
	2 × 0.06, 14 d interval, 1 DALA	Oil palm fruits (RAC) Mesocarp oil Mesocarp cake Kernels Kernel oil Kernel cake	0.26 1.0 0.37 <0.01 <0.01 <0.01	- 3.8 1.4 <0.04 <0.04 <0.04
MRID50234701-S15-04277-02 Malaysia, Melaka	2 × 0.03 kg ai/ha, 14 d interval, 1 DALA	Oil palm fruits (RAC) Mesocarp oil Mesocarp cake Kernels Kernel oil Kernel cake	0.25 0.47 0.27 <0.01 <0.01 <0.01	- 1.9 1.1 <0.04 <0.04 <0.04
	2 × 0.06 kg ai/ha, 14 d interval, 1 DALA	Oil palm fruits (RAC) Mesocarp oil Mesocarp cake Kernels Kernel oil Kernel cake	0.22 0.75 0.41 <0.01 <0.01 <0.01	- 3.4 1.9 <0.05 <0.05 <0.05
MRID50234701-S15-04277-03 Malaysia, Sungkai	2 × 0.03 kg ai/ha, 14 d interval, 1 DALA	Oil palm fruits (RAC) Mesocarp oil Mesocarp cake Kernels Kernel oil Kernel cake	0.21 0.34 0.18 <0.01 <0.01 <0.01	- 1.6 0.86 <0.05 <0.05 <0.05

Trial, Location	Application	Matrix	Chlorantraniliprole in mg/kg	PF
	2 × 0.064 kg ai/ha, 14 d interval, 1 DALA	Oil palm fruits (RAC) Mesocarp oil Mesocarp cake Kernels Kernel oil Kernel cake	0.32 1.0 0.38 <0.01 <0.01 <0.01	- 3.1 1.2 <0.03 <0.03 <0.03
MRID50234701-S15-04277-04 Malaysia, Slim River	2 × 0.03 kg ai/ha, 14 d interval, 1 DALA	Oil palm fruits (RAC) Kernels	0.19 <0.01	- <0.05
	2 × 0.062 kg ai/ha, 14 d interval, 1 DALA	Oil palm fruits (RAC) Kernels	0.52 <0.01	- <0.02

In a second study residues of chlorantraniliprole in palm oil (mesocarp oil) and palm kernel oil were measured in one supervised field trial in Malaysia (Loong *et al*., 2009, CHLORANT19E_003). Oil palm trees were treated with two spray application up to two weeks before harvest involving application rates of 0.01 or 0.02 kg ai/ha. Samples of palm fruits were collected immediately after the last application and after up to 14 days.

The palm fruits were processed into crude palm oil (=mesocarp oil) and palm kernel oil after the processing scheme:

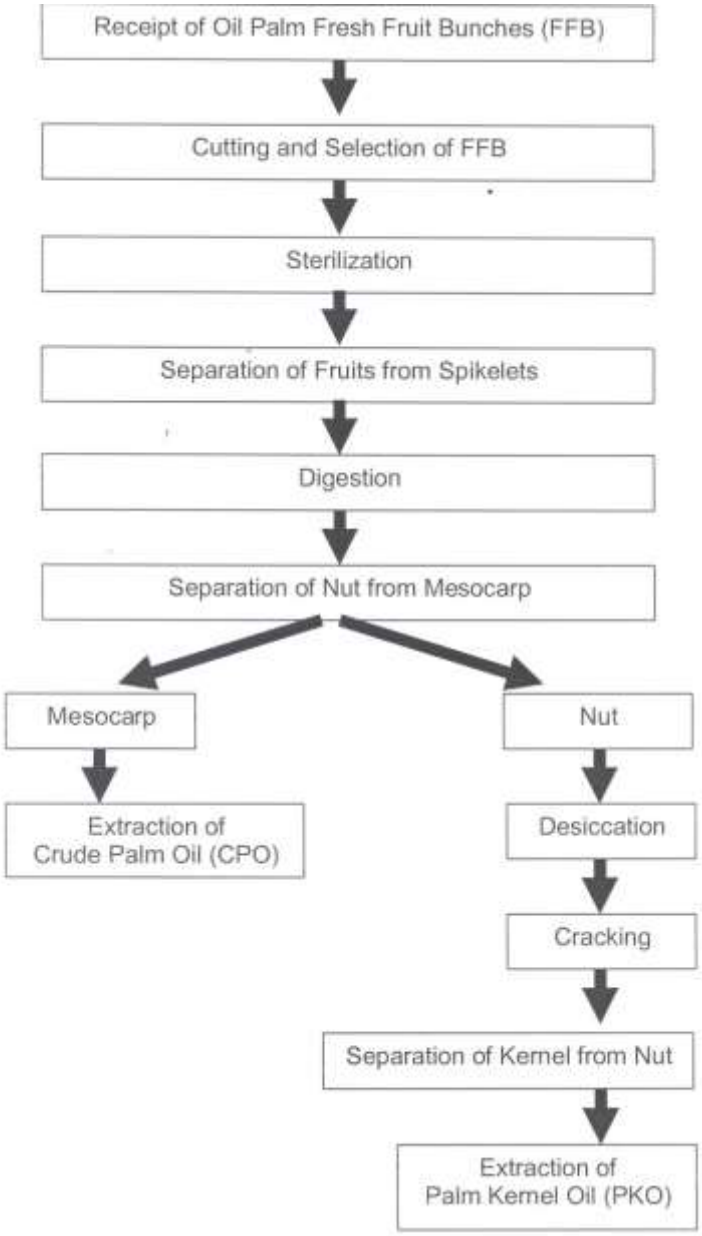


Figure 1 Process flow chart for palm oil

The digestion was conducted by a grinding machine at 15 rpm for 20 min, which mash the outer layers of the fruits but did not crack the nuts. The mashed mesocarp was pressed to gain the crude palm oil. The nuts were separated after the digestion, dried at 90±5 °C for 1 hour ("desiccation"), cracked and finally extracted after grinding by Soxhlet extraction.

All samples were analysed with a GC-ECD method supported with the concurrent recovery data (91–134%, 6% RSD).

In the following table the results from both plots are summarized. No raw palm fruits suitable as RAC were analysed, thus no processing factors can be derived from this study.

Table 8 Residues of chlorantraniliprole following spray treatment on oil palms

Location, Year (variety)	Application				Residues, mg/kg			Report/Trial No., Reference, analytical method, validation data, storage interval
	kg ai/ha	Inter-val days	kg ai/hL	Growth stage	Sample	DALA	Parent	
Malaysia, Sungkai 2009 (not stated)	2 × 0.01	14	2 × 0.005	Young mature up to fruiting stage	Crude Palm oil (=mesocarp oil)	0 1 3 7 14 21	0.01 0.02 0.02 0.01 <0.01 0.02	SIME DARBY-DP/2009/01, CHLORANT19E_003 Method: In-house GC-ECD method Storage interval: 2 months
					Palm kernel oil	0 1 3 7 14	<0.01 <0.01 <0.01 <0.01 <0.01	
	2 × 0.02	14	2 × 0.01	Young mature up to fruiting stage	Crude Palm oil (=mesocarp oil)	0 1 3 7 14 21	0.03 0.05 0.13 0.03 0.05 0.03	
					Palm kernel oil	0 1 3 7 14	<0.01 <0.01 <0.01 <0.01 <0.01	

DALA: days after last application

Table 9 Summary of processing factors

RAC	Processed commodity	Individual PF´s	Median or best estimate PF
Oil palm fruits	Mesocarp oil	1.6, 1.9, 1.9, 3.1, 3.4, 3.8	2.5
	Mesocarp cake	0.38, 0.86, 1.1, 1.2, 1.4, 1.9	1.2
	Kernels	<0.02, <0.03, <0.04, <0.04, <0.05(4)	<0.04
	Kernel oil	<0.03, <0.04, <0.04, <0.05, <0.05, 0.11	<0.04
	Kernel cake	<0.03, <0.04, <0.04, <0.05(3)	<0.04

APPRAISAL

Chlorantraniliprole is an insecticide that operates by a highly specific biochemical mode of action. It was first evaluated for residues and toxicological aspects by the 2008 JMPR. The 2008 JMPR established an ADI for chlorantraniliprole of 0–2 mg/kg bw and concluded that an ARfD was unnecessary.

The 2008 JMPR also recommended the following residue definition for Chlorantraniliprole:

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

The residue is fat-soluble.

Chlorantraniliprole was last evaluated in 2016 for additional maximum residue levels. At the Fiftieth Session of the CCPR (2018), chlorantraniliprole was listed for consideration of additional uses

by the 2019 Extra JMPR. The Meeting received information on registered use patterns, supervised residue trials on beans, peas and oil palm with product labels from Malaysia and the USA.

Methods of analysis

The current Meeting received additional concurrent recovery information for the analysis of chlorantraniliprole in plant matrices.

A minor modification of method 13261, which was previously evaluated by the 2008 JMPR, was additionally tested for dry peas, oil palm fruits, kernels and kernel oil as well as for the palm fruit mesocarb and mesocarb oil. The method involves analysis by LC-MS/MS techniques and was successfully validated at a LOQ of 0.01 mg/kg for all matrices investigated.

Results of supervised residue trials on crops

The Meeting received supervised trial data for applications of chlorantraniliprole on dry beans and peas as well as on oil palms conducted in the USA and Malaysia, respectively.

Dry beans (except dry soya beans) and dry peas

Chlorantraniliprole is registered for use on legume vegetables (succulent and dried) in the USA with a maximum GAP involving two foliar sprays of 0.11 kg ai/ha each (3 day interval), a maximum seasonal rate of 0.23 kg ai/ha and a PHI of 1 day.

Corresponding supervised field trials conducted in the USA on dry beans and dry peas matching this GAP were submitted.

Residues of chlorantraniliprole in beans, dry were (n=5): 0.011, 0.013, 0.016, 0.025 and 0.051 mg/kg.

Residues of chlorantraniliprole in peas, dry were (n=5): 0.024, 0.036, 0.054, 0.056 and 0.18 mg/kg.

The Meeting noted that residues in both commodities are not significantly different, which was confirmed by the Mann-Whitney-U Test. Since dry beans and peas are both representative commodities for the sub-groups dry beans (VD 2065) and dry peas (VD 2066), the Meeting decided to combine the datasets for mutual support.

Combined residues of chlorantraniliprole in beans, dry and peas, dry were (n=10): 0.011, 0.013, 0.016, 0.024, 0.025, 0.036, 0.051, 0.054, 0.056 and 0.18 mg/kg.

The US GAP does not include treatment of soya beans, which are also covered in the Codex sub-groups dry beans (VD 2065). Therefore the Meeting decided to exclude soya beans from its recommendations.

The Meeting estimated a maximum residue level of 0.3 mg/kg and a STMR value of 0.0305 mg/kg for chlorantraniliprole in dry beans (VD 2065), except dry soya beans and in dry peas (VD 2066).

Palm fruit

Chlorantraniliprole is registered for use on oil palms in Malaysia with two foliar sprays of 0.03 kg ai/ha each (14 day interval) and a PHI of 1 day. Four corresponding supervised field trial conducted in Malaysia were submitted.

Residues of chlorantraniliprole in palm fruits were (n=4): 0.18, 0.19, 0.2, 0.38 mg/kg.

The Meeting estimated a maximum residue level of 0.8 mg/kg and a STMR value of 0.195 mg/kg for chlorantraniliprole in palm fruits.

Fate of residues during processing

The fate of chlorantraniliprole residues has been examined under conditions simulating commercial processing of oil palm fruits.

Estimated processing factors for the commodities considered at this Meeting are summarized below.

Raw commodity	Processed commodity	Chlorantraniliprole			
		Individual processing factors	Mean or best estimate processing factor	STMR-P in mg/kg	Maximum residue level in mg/kg
Oil palm fruit (STMR: 0.195 mg/kg, maximum residue level: 0.8 mg/kg)	Mesocarp oil (= Palm oil)	1.6, 1.9, 1.9, 3.1, 3.4, 3.8	2.6	0.507	2
	Kernel oil (=Palm kernel oil, crude)	<0.03, <0.04, <0.04, <0.05, <0.05, 0.11	<0.05	0.0098	Not necessary
	Kernel cake (=Palm, kernel meal)	<0.03, <0.04, <0.04, <0.05(3)	<0.04	0.0078	Not necessary

For palm oil, crude (=mesocarp oil) the Meeting estimated a maximum residue level of 2 mg/kg and a STMR-P of 0.507 mg/kg, based on a mean processing factor of 2.6.

For palm kernel oil and palm kernel cake the Meeting estimated STMR-P values of 0.0098 mg/kg and 0.0078 mg/kg, respectively. No specific maximum residue levels are required since no accumulation of residues was observed.

Residues in animal commodities

The Meeting recalculated the livestock dietary burden based on the uses considered by the current and previous Meetings on the basis of diets listed in the 2016 edition of the FAO Manual Appendix IX (OECD Feedstuff Table). The maximum and mean dietary burdens for cattle of up to 36 ppm and 18 ppm, respectively, calculated by the 2016 Meeting are not changed by the addition of dry beans, except soya bean and dry peas (Median: 0.0305 mg/kg); and palm kernel cake (Median-P: 0.0078 mg/kg). The Meeting 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 in Annex 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 and animal commodities: *Chlorantraniliprole*

The residue is fat-soluble.

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg
		New	Previous	
VD 2065	Dry beans, Subgroup of (includes all commodities in this subgroup) (except soya beans)	0.3		0.0305
VD 2066	Dry peas, Subgroup of (includes all commodities in this subgroup)	0.3		0.0305
SO 3160	Palm fruit (African oil palm)	0.8		0.195
OC 0695	Palm oil, crude	2		0.507
OC1240	Palm kernel oil, crude			0.0098

Additional values used in estimating livestock dietary burdens.

Codex classification	Commodity	Median residue (-P) (mg/kg)	Highest residue (-P) (mg/kg)
	Palm, kernel meal	0.0078	

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for chlorantraniliprole is 0–2 mg/kg bw. The International Estimated Daily Intakes (IEDIs) for chlorantraniliprole 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 2019 Extra JMPR Report.

The IEDIs ranged from 0–1% of the maximum ADI. The Meeting concluded that long-term dietary exposure to residues of chlorantraniliprole 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 chlorantraniliprole was unnecessary. The Meeting therefore concluded that the acute dietary exposure to residues of chlorantraniliprole from the uses considered is unlikely to present a public health concern.

REFERENCES

Code	Author	Year	Title, Institute, Report reference
CHLORANT19E_001	Dorsey, S.	2018	Magnitude of chlorantraniliprole residues in dried, shelled peas and beans following foliar applications of Chlorantraniliprole (DPX-E2Y45) 20 SC [200 g/L (w/v); 18.4% (w/w)] - U.S., 2017; Analytical Bio-Chemistry Laboratories, Inc.; 48825; GLP: yes, unpublished
CHLORANT19E_002	Petrova, D.	2017	Determination of residues of chlorantraniliprole after two applications of Altacor 35 WG in oil palm trees at 4 sites in Malaysia in 2015; DuPont Malaysia; MRID 50234701; GLP: yes; unpublished
CHLORANT19E_003	Loong, L.K.; Siran, Y.M.; Keong, N.C.	2009	Foliar Applications of PREVATHON 5SC (Chlorantraniliprole) on Oil Palms and Sampling of Fresh Fruit Bunches for Residue Study on crude palm oil & palm kernel oil; DuPont Malaysia; SIME DARBY-DP/2009/01; GLP: yes; unpublished

CHLOROTHALONIL (081)

First draft prepared by Dr H Koayashi, Ministry of Agriculture, Forestry and Fisheries,Tokyo,Japan

EXPLANATION

Chlorothalonil, a benzene substituted with chlorines and dinitriles, is a non-systemic fungicide first evaluated by the JMPR in 1974 and a number of times subsequently. It was reviewed for toxicology by the 2009 and 2010 JMPR within the periodic review program of the CCPR. For the parent substance, chlorothalonil, an ADI of 0–0.02 mg/kg bw and an ARfD of 0.6 mg/kg bw were established. In addition, for the metabolite SDS-3701 (2,5,6-trichloro-4-hydroxyisophthalonitrile), an ADI of 0–0.008 mg/kg bw and an ARfD of 0.03 mg/kg bw were established.

The 2010 JMPR recommended the following residue definitions for chlorothalonil:

Definition of the residue for compliance with the MRL for plant commodities: chlorothalonil.

Definition of the residue for dietary risk assessment for plant commodities: chlorothalonil and SDS-3701, all considered separately.

Definition of the residue for compliance with the MRL and dietary risk assessment for animal commodities: SDS-3701.

At the Fiftieth Session of the CCPR (2018), chlorothalonil was scheduled for evaluation of an additional use on cranberry by the 2019 Extra JMPR. The current Meeting received new information on use patterns, additional analytical methods and supervised field trials on cranberry.

RESIDUE ANALYSIS

Analytical methods

For chlorothalonil and its metabolite SDS-3701, an additional method was provided for cranberry.

Method GRM005.03A (Huebner M.R., 2010)

Approximately 10 grams of homogenized cranberry sample are extracted with acetone- sulfuric acid (10 mol/L) solution (95:5, v/v, 100 mL). After the sample is centrifuged or allowed to settle, a 2-mL aliquot is diluted to 10 mL with water.

The extracts are cleaned up using solid phase extraction (SPE) on a C_{18} phase. Chlorothalonil and SDS-3701 are analysed by liquid chromatography-mass spectrometry/mass spectrometry (LC-MS/MS). Heated nebulizer APCI negative polarity multiple reaction monitoring (MRM) at m/z=245 → 245 for chlorothalonil and MRM at m/z=245 → 182 for SDS-3701. The procedural recoveries ranged from 65-108% for chlorothalonil and 75–100% for SDS-3701. The RSD values were ≦9.5% for chlorothalonil and ≦7.6% for SDS-3701. The LOQs for both analytes were 0.01 mg/kg. The recovery data are shown in Table 1.

Table 4 Recovery data for method GRM005.03A measuring chlorothalonil and SDS-3701

Matrix	analyte	Fortification level (mg/kg)	n	Recovery range (%)	Recovery, mean (%)	RSD (%)
Cranberry	Chlorothalonil	0.01	3	91-108	100	8.3
		0.5	3	87-94	89	4.4
		10	3	65-78	72	9.5
Cranberry	SDS-3701	0.01	3	85-95	90	5.9
		0.5	3	75-87	82	7.4
		10	3	86-100	92	7.6

USE PATTERN

The Meeting received GAP information for cranberry in the USA as shown in Table 2.

Table 2 Use pattern of chlorothalonil

Crop	Country	Conc. g ai/L or kg Formulation	Application					
			Type	kg ai/ha	Growth stage	No	PHI (days)	Note
Cranberry	USA	Bravo 720 719 g ai/L SC	Foliar	5.5	Not specified	3 (interval: 10 days or more)	50	

RESULTS OF SUPERVISED RESIDUE TRIALS ON CROPS

Residue levels were reported as measured. Application rates were always reported as chlorothalonil equivalents. When residues were not detected they are shown as below the LOQ, e.g., < 0.01 mg/kg. Application rates, concentrations and mean residue results have been rounded to two significant figures. Residue values from the trials conducted according to the maximum GAP were used for the estimation of maximum residue levels, STMR and HR. These results are underlined.

Laboratory reports included method validation including batch recoveries with spiking at residue levels similar to those occurring in samples from the supervised trials. Dates of analyses or duration of residue sample storage were also provided. Field reports provided data on the sprayers used and their calibration, plot size, residue sample size and sampling date.

Cranberries

Five residue trials were conducted on cranberries in the USA in 2016 (IR-4 PR No. 11846). Chlorothalonil was applied as a suspension concentrate (SC). Three foliar applications were made to the treated plot at each trial site at a target application rate of 5.5 kg ai/ha. The following adjuvants were mixed with chlorothalonil: Induce NIS in the trial conducted in Wareham; Attach NIS in Chatsworth, R-11 NIS in Warrenton, and Activator 90 NIS in Wisconsin Rapids and Warren. Fruit was sampled 48-51 days after the third (last) application. The residues of chlorothalonil were analysed using an LC/MS/MS method (Method GRM005.03A) with a LOQ of 0.01 mg/kg. Analyses were completed within 23 days of harvest. The residues of chlorothalonil (expressed as chlorothalonil) and SDS-3701 (expressed as SDS-3701) in the trials are shown in Table 3.

Table 3 Residues of chlorothalonil and SDS-3701 in cranberries (GRM005.03A, Storage interval: ≦22 days)

Cranberry Location, year (Variety)	Application						Residue (mg/kg)		Report no.
	Form	No	Intervals (days)	kg ai/ha	Sample	DALT (days)	Chloro-thalonil	SDS-3701 (as SDS-3701)	
Wareham, MA 2016 (Stevens)	SC	3	 10 10	5.5 5.6 5.6	berries	51	0.35, 0.47 (<u>0.41</u>)	<0.01, <0.01 (<u><0.01</u>)	IR-4 11846.16-MA185
Chatsworth, NJ 2016 (Stevens)	SC	3	 10 11	5.6 5.6 5.7	Berries	48	4.2, 1.8 (<u>3.0</u>)	<0.01, <0.01 (<u><0.01</u>)	IR-4 11846.16-NJ256
Warrenton, OR 2016 (Pilgrim)	SC	3	 10 11	5.7 5.7 5.8	Berries	50	7.7, 6.5 (<u>7.1</u>)	<0.01, <0.01 (<u><0.01</u>)	IR-4 11846.16-OR325
Wisconsin Rapids, WI 2016 (Stevens)	SC	3	 10 10	6.1 5.6 5.4	Berries	49	1.7, 2.0 (<u>1.8</u>)	0.019, 0.019 (<u>0.019</u>)	IR-4 11846.16-WI415
Warren, WI 2016 (Stevens)	SC	3	 10 10	6.1 6.1 6.1	Berries	49	4.3, 4.5 (<u>4.4</u>)	<0.01, <0.01 (<u><0.01</u>)	IR-4 11846.16-WI416

FATE OF RESIDUES DURING PROCESSING

No processing study was available for cranberry.

APPRAISAL

Chlorothalonil was reviewed for toxicology by the 2009 and 2010 JMPR within the periodic review program of the CCPR. For the parent substance an ADI of 0–0.02 mg/kg bw and an ARfD of 0.6 mg/kg bw were established. In addition to the parent substance, an ADI of 0–0.008 mg/kg bw and an ARfD of 0.03 mg/kg bw were established for the metabolite SDS-3701 (2,5,6-trichloro-4-hydroxyisophthalonitrile).

The 2010 JMPR recommended the following residue definitions for chlorothalonil:

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

Definition of the residue for dietary risk assessment for plant commodities: *chlorothalonil and SDS-3701, all considered separately*;

Definition of the residue for compliance with the MRL and dietary risk assessment for animal commodities: *SDS-3701*.

At the Fiftieth Session of the CCPR (2018), chlorothalonil was scheduled for evaluation of an additional use on cranberry by the 2019 Extra JMPR. The current Meeting received new information on use patterns for chlorothalonil on cranberry and additional analytical methods and supervised field trials.

Methods of analysis

The Meeting received an analytical method for chlorothalonil not previously evaluated by the Meeting. The method was used in the new supervised field trials.

Method GRM005.03A is applicable to cranberries and involved homogenisation with acetone and 10 mol/L sulfuric acid solution (95:5, v/v, 100 mL). Following solid phase extraction (SPE) clean-up, chlorothalonil and SDS-3701 were analysed by liquid chromatography-mass spectrometry/mass spectrometry (LC-MS/MS). The method was validated for both analytes in cranberries with a LOQ of 0.01 mg/kg.

Stability of pesticides in stored analytical samples

The 2015 JMPR concluded that chlorothalonil and SDS-3701 might degrade in cranberries and no acceptable storage interval above one month could be identified. In the supervised trials received by the current Meeting, the samples were analysed within 22 days of sampling and kept frozen during the storage interval. The Meeting concluded that the residue trial data could be used for evaluation.

Definition of the residue

Based on new information, the present Meeting reassessed the toxicity of the metabolites R611965 and R417888 and their relevance in dietary exposure.

The metabolite R611965 is covered by the ADI and ARfD of parent chlorothalonil, but it was noted that it is at least 10 times less potent. R611965 is the predominant residue in all rotational crops, representing 29–63% of the TRR in confined studies. Additionally, various field rotational crop studies were submitted to the 2010 JMPR, indicating potential residues of R611965 in succeeding crops:

Commodity group	Max. Residues of R611965 per trial [a] (mg/kg)	Field rotational crop median residue (mg/kg)	Field rotational crop highest residue (mg/kg)
Leafy and Brassica crops	<0.03, <0.05, <0.05, 0.18, 0.24, 1.1, 2.2	0.18	2.2
Legume vegetables	0.03, 0.14, 0.74, 1.0	0.44	1.0
Root tops	0.03, 0.04, 0.07, 0.1, 0.43, 0.49, 0.65, 0.91	0.265	0.91
Root and tuberous vegetables, bulb vegetables	0.02, 0.02, <0.03, 0.03, 0.08, 0.15, 0.2, 0.56, 0.59, 0.89	0.115	0.89
Cereal grains	<0.03, 0.04, 0.05, 0.06, 0.44, 0.56, 0.58	0.06	Not necessary
Oilseed and pulse crops	<0.03, <0.03, 0.04, 0.13	0.035	Not necessary
Fruiting vegetables	<0.03, <0.03, 0.14, 1.5	0.085	1.5

[a] up scaled (unless <LOQ) to the highest annual rate reported by the 2010 JMPR (20 kg ai/ha eq.)

Taking into account the 10 times lower potency of R611965, the Meeting concluded that its contribution to the overall dietary risk arising from its presence in commodities obtained from rotational crops is insignificant compared to parent chlorothalonil. The IESTI based on the median or highest residues found in field rotational crop studies represents a very small proportion of the ARfD (up to 5%).

Based on the chemical structure of R611965, conversion into SDS-3701 does not occur. SDS-3701 is the only chlorothalonil related residue found in commodities of animal origin. Therefore, the Meeting concluded that R611965 is irrelevant for the consideration of chlorothalonil residues in animal commodities.

The Meeting concluded that the inclusion of R611965 in the residue definition of chlorothalonil for dietary exposure purposes is unnecessary at this time. The relevance of this metabolite should be revisited in the next periodic review.

The metabolite R417888 is covered by the ADI and ARfD of chlorothalonil. Since the compound was not found in food or feed commodities, the Meeting concluded that its inclusion in the residue definition of chlorothalonil for dietary exposure purposes is unnecessary.

The Meeting confirmed its previous residue definition recommendations for chlorothalonil.

Results of supervised residue trials on crops

The Meeting received supervised trial data for applications of chlorothalonil on cranberries conducted in the USA.

Cranberry

The 2010 JMPR concluded that the overall information received was insufficient and recommended withdrawal of its previous recommendation for chlorothalonil in cranberries of 5 mg/kg.

The current Meeting received five new supervised field trials conducted in the USA and matching the GAP in the USA for cranberries at a rate of 3 × 5.5 kg ai/ha with a PHI of 50 days. In cranberries following treatment with chlorothalonil according to the US GAP, residues of chlorothalonil were (n=5): 0.41, 1.8, 3.0, 4.4 and 7.1 (highest individual value of 7.7) mg/kg.

In the same trials, the residues of SDS-3701 expressed as SDS-3701 were (n=5): < 0.01 (4), 0.019 mg/kg.

The Meeting estimated maximum residue level, STMR and HR values of 15, 3.0, and 7.7 mg/kg for chlorothalonil in cranberries, respectively. The Meeting also estimated STMR and HR values of 0.01 and 0.019 mg/kg, respectively, for SDS-3701.

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: *chlorothalonil*

Definition of the residue for dietary risk assessment for plant commodities: *chlorothalonil and SDS-3701 (2,5,6-trichloro-4-hydroxyisophthalonitrile), all considered separately.*

Definition of the residue for compliance with the MRL and dietary risk assessment for animal commodities: *SDS-3701 (2,5,6-trichloro-4-hydroxyisophthalonitrile).*

The residue is not fat-soluble.

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
FB0265	Cranberry	15	W	Chlorothalonil: 3.0 SDS-3701: 0.01	Chlorothalonil: 7.7 SDS-3701: 0.019

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for chlorothalonil and its metabolite SDS-3701 are 0–0.02 and 0–0.008 mg/kg bw, respectively. The International Estimated Daily Intakes (IEDIs) for chlorothalonil and SDS-3701 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 2019 Extra JMPR Report.

The IEDIs ranged from 10–50% and 4–10% of the maximum ADI for chlorothalonil and SDS-3701, respectively. The Meeting concluded that long-term dietary exposure to residues of chlorothalonil and its SDS-3701 metabolite from uses considered by the JMPR is unlikely to present a public health concern.

Acute dietary exposure

The ARfD for chlorothalonil and SDS-3701 are 0.6 and 0.03 mg/kg bw, respectively. The International Estimate of Short Term Intakes (IESTIs) for chlorothalonil and SDS-3701 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 2019 Extra JMPR Report.

For chlorothalonil, the IESTIs varied from 0–9% (children) and 0–3% (general population) of the ARfD. For SDS-3701, the IESTIs were 0% (children) and 0% (general population) of the ARfD. The Meeting concluded that acute dietary exposure to residues of chlorothalonil and SDS-3701 from uses considered by the present Meeting is unlikely to present a public health concern.

Threshold of toxicological concern (TTC) consideration for metabolites

The metabolite R613636 could be assessed using the TTC approach (Cramer Class III threshold of 1.5 µg/kg bw per day). Formation of R613636 was only observed following simulated sterilization (120 °C, 20 min, pH 6) but not after simulated pasteurization or cooking, and represented up to 23% (mean of individual samples) of the recovered residue. R613636 was not found in unprocessed plant or animal commodities.

The Meeting applied a factor of 0.23 to the maximum IEDI of 9.33 µg/kg bw for parent chlorothalonil estimated by the current Meeting to address the formation of R613636 during sterilization resulting in an estimated exposure of 2.37 µg/kg bw per day.

The Meeting noted that the 17 Cluster diets model contains no information allowing refinement for sterilized foods. However, considering the small difference (less than two-fold) between the estimated exposure and the threshold of toxicological concern for a Cramer Class III compound of 1.5 µg/kg bw per day, it seems very unlikely that the daily diet contains a high percentage (>50%) of foods subject to high temperature treatment. Therefore, noting that the current IEDI model does not include details of food processing, the Meeting concluded that exposure to R613636 is likely to be below the TTC for Cramer Class III compounds and that based on the uses evaluated by the JMPR, residues of R613636 are unlikely to present a public health concern.

The metabolites SYN548764 and R611968 could be assessed using the TTC approach (Cramer Class III threshold of 1.5 µg/kg bw per day). Since these metabolites were not identified in food or feed commodities, they are unlikely to present a public health concern.

REFERENCES

Code	Author(s)	Year	Title
GRM005.03A	Heubner M. R.	2010	Analytical Method for the Determination of Total Chlorothalonil Residues in Crop Commodities by LC-MS/MS Syngenta Method GRM005.03A Syngenta File No. R044686_50164 GLP unpublished
IR-4 PR No. 11846	Jolly C.	2017	Chlorothalonil: Magnitude of the Residue on Cranberry Report No. IR-4 PR No. 11846 Syngenta File No. R044686_52414 GLP unpublished

CYPRODINIL (207)

First draft prepared by Dr H Koayashi, Ministry of Agriculture, Forestry and Fisheries,Tokyo,Japan

EXPLANATION

Cyprodinil is a fungicide belonging to the anilinopyridine group. It is a systemic foliar and seed dressing fungicide that acts as an inhibitor of methionine biosynthesis. Cyprodinil has been registered in many countries to control a range of fungal diseases in cereals, grapes, pome fruit, stone fruit, strawberries, vegetables, field crops and ornamentals, and as a seed dressing for barley.

When Cyprodinil was first evaluated by the JMPR in 2003, an ADI of 0–0.03 mg/kg bw was established. The meeting decided that an ARfD was unnecessary. The residue definition for both plants and animal commodities, for both compliance with MRLs and dietary risk assessment, is cyprodinil. The residue is fat soluble. The JMPR evaluated new uses of cyprodinil for residues in 2013, 2015, 2017 and 2018.

At the Fiftieth Session of the CCPR (2018) cyprodinil was scheduled for consideration of an additional use for soya bean by the 2019 Extra JMPR. The Meeting received new information on a use pattern, analytical method and supervised trials in soya bean.

RESIDUE ANALYSIS

Analytical methods

An additional method was provided for determination of cyprodinil in soya bean.

Method POPIT MET.071.Rev11 (Dunchen M., 2010)

Cyprodinil is extracted by mechanical shaking with a methanol/water mixture (4:1; v/v). After centrifugation, and an aliquot of the extract is diluted with methanol and water. After filtration on a 0.22 µm syringe filter, the final determination of residues is performed by LC-MS/MS in multiple-reaction monitoring mode using the ion transition m/z = 226 → 93 for quantification. The procedural recoveries of cyprodinil ranged from 80–94% and the RSD values were ≦5.2%. The method LOQ is 0.01 mg/kg. The recovery data are shown in Table 1.

Table 1 Recovery data for method POPIT MET.071.Rev11 measuring cyprodinil

Matrix	Fortification level (mg/kg)	n	Recovery range (%)	Recovery, mean (%)	RSD (%)	Reference
Soya bean	0.01	7	77-83	80	2.9	M09160
	0.1	5	81-83	82	1.0	
Soya bean	0.01	7	84-100	94	5.2	M10115
	0.1	5	80-87	84	3.4	

USE PATTERN

The Meeting received GAP information for soya bean as shown in Table 2.

Table 2 Registered uses of cyprodinil on soya bean considered by the Meeting

Matrix	Country	Conc. Formulation	Application				
			Type	kg ai/ha	Growth stage	No	PHI Days
Soya bean	Brazil	0.750 kg ai/kg WG	Foliar	1.05	Beginning of crop flowering	2 (interval: 7 days)	30

RESULTS OF SUPERVISED RESIDUE TRIALS ON CROPS

Residue levels were reported as measured. Application rates were always reported as cyprodinil equivalents. When residues were not detected they were 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. The values from the trials conducted according to maximum GAP selected for the estimation of maximum residue levels, STMR and HR are underlined.

Laboratory reports included method validation including batch recoveries with spiking at residue levels similar to those occurring in samples from the supervised trials. Dates of analyses or duration of residue sample storage were also provided. Field reports provided data on the sprayers used and their calibration, plot size, residue sample size and sampling date.

Soya beans

The Meeting received 12 supervised residue trials conducted in Brazil with cyprodinil on soya beans, four each in 2009, 2010 and 2011.

Samples of dry seeds were collected at normal commercial harvest, 30 days after the last application and at 25 and 35 days after the last application.

Samples were frozen immediately after harvest and maintained in frozen storage for periods of up to 3 months (89 days) prior to extraction. The 2015 JMPR concluded that residues of cyprodinil were stable for at least 9 months in an oily matrix (rapeseed) during frozen storage at about -18 °C.

Residues of cyprodinil in soya bean were quantified using the analytical method POPIT MET.071 Rev11 with a LOQ of 0.01 mg/kg.

The results of the trials are summarized in Table 3.

Table 3 Residues of cyprodinil (WG, including fludioxonil at 0.275 kg ai/ha) in soya beans

	Application					Residue	
Soya bean Location, year (Variety)	No	Interval (days)	Application Rate (kg ai/ha)	Growth Stage at Application	DALT (days)	Residue Cyprodinil (mg/kg)	Reference
GAP		7	2 × 1		30		
Bandeirantes, Brazil 2009 (CD 202)	2	14	0.375 0.375	BBCH 77-85 BBCH 81	25 30 35	0.04 0.04 0.05	M09160-LZF Storage period=89 days
Carambei, Brazil 2009 (BRS 230)	2	14	0.375 0.375	BBCH 55-59 BBCH 61-65	25 30 35	<0.01 <0.01 <0.01	M09160-DMO1 Storage period=89 days
Goiânia, Brazil 2009 (NK 9074)	2	14	0.375 0.375	BBCH 79 BBCH 82	25 30 35	0.05 0.04 0.03	M09160-MFG Storage period=89 days
Uberlândia, Brazil 2009 (NK9074RR)	2	14	0.375 0.375	BBCH 77 BBCH 82	25 30 35	0.03 0.03 0.02	M09160- JJB Storage period=89 days
Engenheiro Coelho, Brazil 2010 (Valiosa)	2	7	0.375 0.375	BBCH 75-77 BBCH 77	25 30 35	0.04 0.03 0.02	M10115-LZF Storage period=73 days
Planaltina, Brazil 2010 (NK7074RR)	2	7	0.375 0.375	BBCH 79 BBCH 86	25 30 35	0.01 0.01 0.01	M10115-MFG Storage period=73 days

	Application					Residue	
Soya bean Location, year (Variety)	No	Interval (days)	Application Rate (kg ai/ha)	Growth Stage at Application	DALT (days)	Residue Cyprodinil (mg/kg)	Reference
GAP		7	2 × 1		30		
Carambei, Brazil 2010 (SYN1049RR)	2	7	0.375 0.375	BBCH 84-85 BBCH 85-86	25 30 35	0.01 0.01 < 0.01	M10115-DMO Storage period=73 days
Uberlândia, Brazil 2010 (NK7074)	2	7	0.375 0.375	BBCH 79 BBCH 81	25 30 35	< 0.01 < 0.01 < 0.01	M10115-JJB Storage period=73 days
Uberlândia, Brazil 2011 (SYN9070RR)	2	7	1.05 1.05	BBCH 75 BBCH 78	25 30 35	0.10 0.09 0.08	M11106-JJB Storage period=67 days
Cabeceiras, Brazil 2011 (9070RR)	2	7	1.05 1.05	BBCH 81 BBCH 86	25 30 35	0.10 0.08 0.10	M11106-MFG Storage period=67 days
Palmeira, Brazil 2011 (SYN1049RR)	2	7	1.05 1.05	BBCH 75-77 BBCH 77-79	25 30 35	0.04 0.04 0.04	M11106- DMO1 Storage period=67 days
Carambei, Brazil 2011 (Potência RR)	2	7	1.05 1.05	BBCH 75-77 BBCH 79-80	25 30 35	0.08 0.08 0.09	M11106- DMO2 Storage period=67 days

FATE OF RESIDUES DURING PROCESSING

No processing study was available for soya bean.

APPRAISAL

Cyprodinil is a fungicide belonging to the anilinopyridine group. When Cyprodinil was first evaluated by the JMPR in 2003, an ADI of 0–0.03 mg/kg bw was established. The 2003 JMPR decided that an ARfD was unnecessary. The residue definition for both plants and animal commodities, for both compliance with MRLs and dietary risk assessment, is cyprodinil. The residue is fat soluble.

At the Fiftieth Session of the CCPR (2018), cyprodinil was scheduled for evaluation of an additional use on soya bean by the 2019 Extra JMPR. The Meeting received new information on a use pattern, analytical method and supervised trials.

Methods of analysis

The Meeting received an analytical method for cyprodinil not previously evaluated by the Meeting. The method was used in the newly submitted supervised field trials.

Method POPIT MET.071.Rev11 is applicable to soya beans. Cyprodinil is extracted with methanol/water (4:1, v/v) and analysed by LC-MS/MS. The method was validated for cyprodinil in soya beans with a LOQ of 0.01 mg/kg.

Stability of pesticides in stored analytical samples

The stability of cyprodinil in rapeseed (commodity with high oil content) was evaluated by the 2015 JMPR, which confirmed that cyprodinil was stable for at least 9 months. In the supervised field trials, samples were stored at -18 °C for a maximum of 89 days. The Meeting concluded that the trials were suitable for evaluation.

Results of supervised residue trials on crops

The Meeting received supervised trial data for applications of cyprodinil on soya beans conducted in Brazil.

Soya beans

Cyprodinil is registered in Brazil for use on soya beans at a rate of 2 × 1.05 kg ai/ha with an interval of 7 days and a PHI of 30 days. Four supervised field trials from Brazil matched this GAP.

In soya beans following treatment with cyprodinil according to the Brazilian GAP, residues in ranked order were (n=4): 0.04, 0.09 (2), and 0.10 mg/kg.

Eight additional trials conducted in Brazil were at a lower application rate of 2 × 0.375 kg ai/ha, four of which were with longer intervals (14 days). The Meeting noted that the retreatment interval used in the submitted studies (7 to 14 days) did not appear to have a significant impact on residues of cyprodinil. The residues from these trials in ranked order were (n=8): < 0.01 (2), 0.01 (2), 0.03 (2), 0.04, and 0.05 mg/kg.

The Meeting decided to scale the residues, except those from two trials with the analytical value of <LOQ, to the Brazilian GAP rate in accordance with the proportionality principle (scaling factor=2.8). Scaled residues in ranked order (n=6) were: 0.03, 0.03, 0.08, 0.08, 0.11 and 0.14 mg/kg.

Combined residues matching the Brazilian GAP are (n=10): 0.03, 0.03, 0.04, 0.08, 0.08, 0.09, 0.09, 0.10, 0.11, 0.14 mg/kg.

The Meeting estimated a maximum residue level and STMR of 0.3 and 0.085 mg/kg, respectively, for cyprodinil in soya beans.

Animal feedstuffs

Farm animal dietary burden

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 OECD diets listed in Appendix IX of the 2016 edition of the FAO manual, are presented in Annex 6 and summarized below.

Livestock Dietary Burdens (ppm of dry matter diet) for cyprodinil

	USA/Canada		EU		Australia		Japan	
	Max	Mean	Max	Mean	Max	Mean	Max	Mean
Beef cattle	0.91	0.37	13.9	1.8	5.8	1.4	0.48	0.48
Dairy cattle	1.7	0.88	13.5	1.4	23.3[a]	1.8[b]	0.27	0.27
Poultry, broiler	0.51	0.51	0.81	0.55	0.13	0.13	0.66	0.07
Poultry, layer	0.51	0.51	1.4[c]	0.67[d]	0.13	0.13	-	-

[a] Highest maximum dairy cattle dietary burden suitable for MRL estimates for mammalian meat and milk

[b] Highest mean dairy cattle dietary burden suitable for STMR estimates for mammalian meat and milk

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

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

Animal commodity maximum residue levels

The animal dietary burdens for beef cattle and dairy cattle were the same as those calculated by the 2015 JMPR.

For poultry, based on the use patterns considered by the present and previous Meetings, the Meeting noted that the dietary burdens were lower than those calculated by the 2013 JMPR because the OECD Animal Feeding Table has been revised, removing kale leaves from the poultry diet.

Therefore, the Meeting confirmed the previous recommendations for animal commodities.

RECOMMENDATIONS

On the basis of the data from supervised trials the Meeting concluded that the residue levels listed 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: cyprodinil

Definition of the residue for compliance with the MRL and dietary risk assessment for animal commodities: cyprodinil

The residue is fat-soluble.

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
VD0541	Soya bean (dry)	0.3	-	0.085	-

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for cyprodinil is 0–0.03 mg/kg bw. 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 2019 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 for cyprodinil was unnecessary. The Meeting therefore concluded that the acute dietary exposure to residues of cyprodinil from the uses considered is unlikely to present a public health concern.

Consideration of threshold of toxicological concern (TTC) approach for metabolites

The metabolites CGA249287 and CGA304075 could be assessed using the TTC approach (Cramer Class III threshold of 1.5 µg/kg bw per day). These metabolites were found in the animal, plant and soil metabolism. The Meeting estimated maximum IEDIs of 0.6 and 0.4 µg/kg bw for CGA249287 and CGA304075, respectively, and concluded that they are unlikely to present a public health concern.

The metabolites CGA263208, CGA321915 and NOA422054 could be assessed using the TTC approach (Cramer Class III threshold of 1.5 µg/kg bw per day). These metabolites were observed mainly in rotational crops. The Meeting estimated maximum IEDIs of 0.6, 0.4 and 1.3 µg/kg bw for CGA263208,

CGA321915 and NOA422054, respectively, and concluded that they are unlikely to present a public health concern.

REFERENCES

Code	Author(s)	Year	Title
M09160	Casallanovo, F., Chicón, A.	2009	Switch - Magnitude of Residues of Cyprodinil and Fludioxonil in Soybeans - Brazil, 2008-09; Syngenta File No. A9219B_11564; Syngenta Report No. M09160
M10115	Casallanovo, F., Chicón, A., Duchen Silva, M.-A.	2010	Switch - Magnitude of Residues of Cyprodinil and Fludioxonil in Soybeans - Brazil, 2010; Syngenta File No. A9219B_11572; Syngenta Report No. M10115
M11106	Dos Santos Draetta, M.	2011	Unix 750 WG - Magnitude of Residues of Cyprodinil in Soybeans - Brazil, 2010-11; Syngenta File No. A8779A _10064; Syngenta Report No. M11106

DICAMBA (240)

First draft prepared by Dr M Doherty, Environmental Protection Agency, Washington, USA

EXPLANATION

Dicamba is a systemic broad-spectrum herbicide. It was first evaluated by the JMPR in 2010 (T, R). The most recent residue evaluation was completed in 2013 (R).

The 2010 JMPR established an ADI for dicamba of 0–0.3 mg/kg bw and an ARfD of 0.5 mg/kg bw. The residue definition for compliance with the MRL for plant commodities is parent dicamba. The residue definition for dietary risk assessment for plant commodities is the sum of dicamba and 5-OH dicamba, expressed as dicamba. The residue definition for compliance with the MRL and dietary risk assessment for animal commodities is the sum of dicamba and DCSA, expressed as dicamba. The residue is not fat soluble.

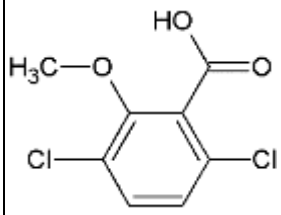
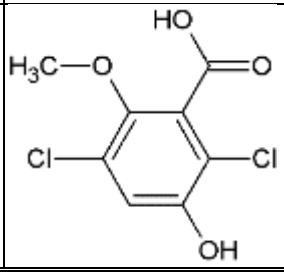
Dicamba was scheduled at the Fiftieth Session of the CCPR for the evaluation of additional uses by the 2019 Extra JMPR. Studies submitted by the sponsor include nature of the residue, field trials, and processing studies in soya bean, maize, and cotton, and storage stability in soya bean and cotton.

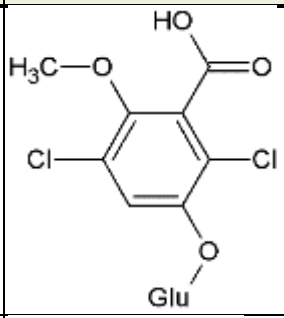
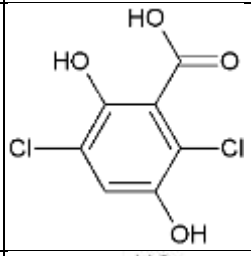
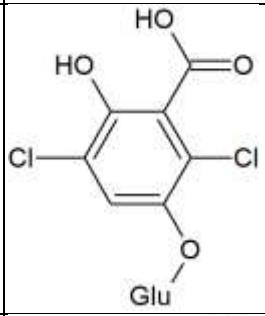
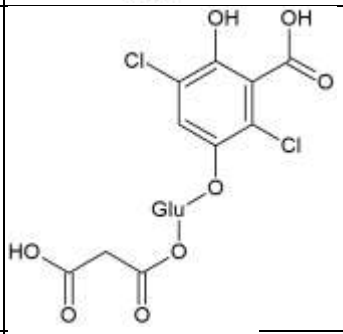
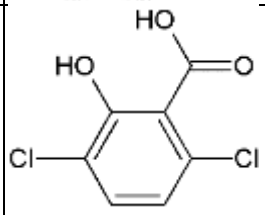
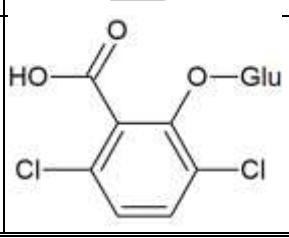
In this document, values in text are rounded to two significant figures; values in tables are presented as provided by the sponsor.

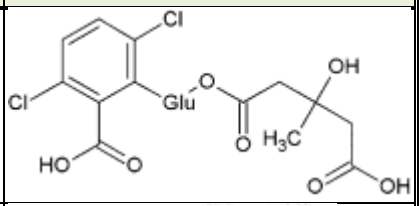
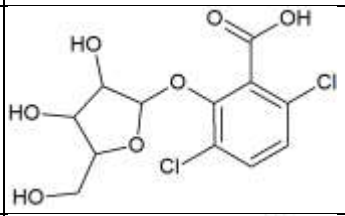
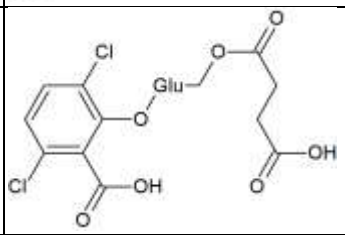
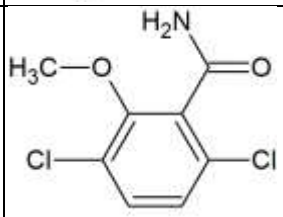
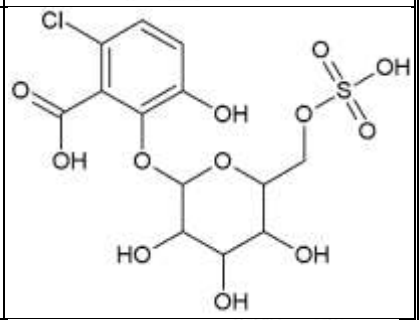
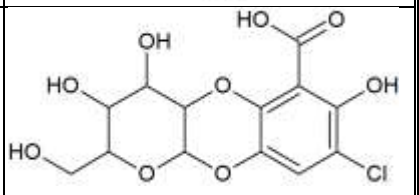
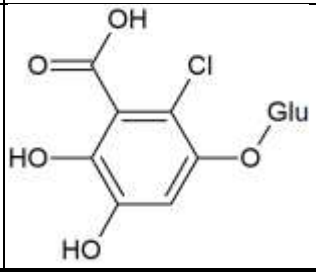
METABOLISM AND ENVIRONMENTAL FATE

The 2019 Extra Meeting received studies depicting the metabolism of dicamba by dicamba-tolerant soya bean, maize, and cotton. Tolerance is conveyed by expression of a dicamba mono-oxygenase protein system that oxidizes dicamba to DCSA. For all studies, dicamba was universally labelled in the phenyl ring, and applications were made either pre-emergence of the crop directly to the soil or post-emergence to the foliage. Control plants were interspersed amongst the treated plants and contained quantifiable levels of radioactivity, indicating potential volatilization and uptake of dicamba or volatile metabolites (e.g., $^{14}CO_2$).

Table 1 Metabolites and degradation products of dicamba observed in dicamba-tolerant crops

Common or code name	Chemical name molecular formula molar mass, g/mol	Structure
Dicamba	3,6-dichloro-2-methoxybenzoic acid $C_8H_6Cl_2O_3$ 221	
5-hydroxydicamba (5-OH Dicamba)	2,5-dichloro-3-hydroxy-6-methoxybenzoic acid $C_8H_6Cl_2O_4$ 237	

Common or code name	Chemical name molecular formula molar mass, g/mol	Structure
5-OH Dicamba Glucoside	2,5-dichloro-3-(β-D-glucopyranosyloxy)-6-methoxybenzoic acid $C_{14}H_{16}Cl_2O_9$ 399	
DCGA	2,5-dichloro-3,6-dihydroxybenzoic acid $C_7H_4Cl_2O_4$ 223	
DCGA Glucoside	2,5-dichloro-3-(β-D-glucopyranosyloxy)-6-hydroxybenzoic acid $C_{13}H_{14}Cl_2O_9$ 385	
DCGA Malonyl glucoside	3-[[6-O-(2-carboxyacetyl)-β-D-glucopyranosyloxy]-2,5-dichloro-6-hydroxybenzoic acid $C_{16}H_{16}Cl_2O_{12}$ 471	
DCSA	3,6-dichloro-2-hydroxybenzoic acid $C_7H_4Cl_2O_3$ 207	
DCSA Glucoside	3,6-dichloro-2-(β-D-glucopyranosyloxy) benzoic acid $C_{13}H_{14}Cl_2O_8$ 369	

Common or code name	Chemical name molecular formula molar mass, g/mol	Structure
DCSA HMG glucoside	2-[[6-O-(4-carboxy-3-hydroxy-3-methylbutyryl)-β-D-glucopyranosyl]oxy]-3,6-dichlorobenzoic acid $C_{19}H_{22}Cl_2O_{12}$ 513	
DCSA Pentoside	3,6-dichloro-2-(pentosyloxy)benzoic acid $C_{12}H_{12}Cl_2O_7$ 339	
DCSA Succinylglucoside	2-[[6-O-(3-carboxypropanoyl)-β-D-glucopyranosyl]oxy]-3,6-dichlorobenzoic acid $C_{17}H_{18}Cl_2O_{12}$ 485	
Dicamba Amide	3,6-dichloro-2-methoxybenzamide $C_8H_7Cl_2NO_2$ 220	
MCDHBA Glucoside Sulfate	6-Chloro-3-hydroxy-2-(3,4,5-trihydroxy-6-sulfooxymethyltetrahydro-pyran-2-yloxy)-benzoic acid $C_{13}H_{15}ClO_{12}S$ 431	
MCTHBA Cyclic Glucoside	7-Chloro-3,4,6-trihydroxy-2-hydroxymethyl-3,4,4a,9atetrahydro-2H-1,9.10-trioxaanthracene-5-carboxylic acid $C_{13}H_{13}ClO_9$ 349	
MCTHBA Glucoside	2-Chloro-5,6-dihydroxy-3-(β-D-glucopyranosyloxy)-benzoic acid $C_{13}H_{15}ClO_{10}$ 367	

Soya bean

The nature of the residue of dicamba in dicamba-tolerant soya bean was investigated by Miller and Mierkowski (2010; Report MSL0022659). Soya bean plants were grown in 12-inch pots in two greenhouses. Spray application of [phenyl-U-^{14}C]dicamba was made at a target rate of 2.8 kg ai/ha either on the day of planting (pre-emergence; PRE; n=29) or 29 days after planting at BBCH 60 (post-emergence; POE; n=32). Following treatment, the PRE and POE samples were placed in the same greenhouse but were physically separated. Pots with untreated control plants were interspersed amongst the treated plants for each group. Samples were collected as follows: Immature foliage 14 days after planting (PRE only), forage 36 (PRE) or 7 (POE) days after last application (DALA), hay 56 (PRE) or 27 (POE) DALA, and seed 112 (PRE) or 83 (POE) DALA. Samples were processed by grinding and were stored frozen.

Total radioactive residues (TRR) were determined by combustion/liquid scintillation counting (LSC). Residues were extracted with acetonitrile:water (2:3, v/v) from all matrices except seed, for which hexane was used first to extract oils. Seed was then extracted once with acetonitrile and then four times with acetonitrile:water (2:3, v/v). Unextracted radioactivity from the post-extraction solids (PES) was determined by combustion/LSC. Extracts were concentrated by rotary evaporation (analysis of the distillate indicated no significant amount of radioactivity was lost due to volatility). An aliquot of the concentrated aqueous extracts underwent hydrolysis (2 N HCl, ca. 100°C, 2 h). Aqueous extracts and hydrolysates were partitioned against ethyl acetate to assess partitioning behaviour. Specific residues were isolated using preparative HPLC analysis. Identification and characterization of metabolites was accomplished using HPLC-MS/MS as well as HPLC-UV and HPLC-RAD. Samples also underwent derivatization with trimethylsilyldiazomethane to discern the presence of carboxylic acid or phenolic groups and with acetic anhydride/pyridine to discern the presence of hydroxyl or phenolic groups. Finally, specific isolates underwent acid hydrolysis (1 N HCl, ca 100 °C, 1-2 hours), base hydrolysis (1 N NaOH, 58-65 °C, 5 hours) and ß-glucosidase digestion (37 °C, 69 hours) to identify/characterize conjugated metabolites. PES underwent further workup with dilute acid and base extraction, phosphate rinse, α-amylase digestion, protease digestion, EDTA extraction, oxidation with chlorite, hydrolysis with cellulase, and hydrolysis with KOH.

Total radioactive residues (extracted + unextracted) are presented in Table 1 (PRE) and Table 2 (POE). Control plants bore quantifiable levels of radioactivity, indicating potential volatilisation and uptake of dicamba or volatile metabolites. Levels were 0.08/0.28 mg eq/kg in PRE/POE forage and 0.17/0.14 mg eq/kg in PRE/POE seed.

Extractions with acetonitrile:water resulted in 91% TRR extracted from immature foliage, 91/94% from forage (PRE/POE), and 91/95% from hay (PRE/POE). Extraction efficiency was much lower for seed: 59% PRE and 64% POE (combined hexane, acetonitrile, and acetonitrile:water extractions). Further work with the PES showed that the vast majority of the unextracted radioactivity was associated with natural products (starch, lignin, cellulose, etc.).

A summary of extracted residues, PES, and hydrolysis and digestion products is shown in Tables 2 and 3.

Table 2 Summary of the nature of the residues in dicamba-tolerant soya bean following PRE application of dicamba (2.8 kg ai/ha)

	Immature foliage		Forage		Hay		Seed	
TRR, mg eq/kg	3.248		1.433		1.056		0.291	
Identification	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Triglycerides (from the hexane-extracted oil fraction)	Not Analysed						13.87	0.04
Acetonitrile/Water extracts	91.09	2.959	91.21	1.307	90.88	0.96	59.35	0.173
Dicamba	Not Analysed		1.61	0.023	0.85	0.009	0.2	0.001
DCSA			3.19	0.046	1.54	0.016	0.37	0.001
DCSA Glucoside			74.48	1.067	70.81	0.748	11.55	0.034
DCSA HMGglucoside			5.21	0.075	6.67	0.07	8.73	0.025

TRR, mg eq/kg	Immature foliage		Forage		Hay		Seed	
	3.248		1.433		1.056		0.291	
Identification	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Unk DCSA/DCGA Glucoside			0.55	0.008	0.51	0.005	ND	ND
Unk DCSA/DCGA Conj.			1.26	0.018	1.64	0.017	0.75	0.002
DCGA Glucoside			1.14	0.016	3.45	0.036	1.6	0.005
DCGA Malonyl glucoside			1.4	0.02	0.73	0.008	4.73	0.014
Sugars			0.96	0.014	1.08	0.011	8.42	0.025
Total Unknowns (n=11)[a]			1.39 [0.58]	0.02 [0.008]	3.59 [1.07]	0.038 [0.011]	4.09 [1.26]	0.013 [0.004]
Total Identified or Characterized Metabolites			89.81	1.287	87.29	0.922	50.21	0.146
PES	8.91	0.289	8.79	0.126	9.12	0.096	40.65	0.118
Phosphate	Not Analysed						0.25	0.001
Starch							1.29	0.004
Protein							10.06	0.029
Pectin							3.26	0.009
Cellulose							3.75	0.011
Hemicellulose							13.92	0.04
Unextracted							7.09	0.021

ND = Not detected

[a] Maximum individual values listed in brackets

Table 3 Summary of the nature of the residues in dicamba-tolerant soya bean following POE application of dicamba (2.8 kg ai/ha)

TRR, mg eq/kg	Forage		Hay		Seed	
	134.147		39.149		0.389	
Identification	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Triglycerides (from the hexane-extracted oil fraction)	Not Analysed				10.76	0.042
Acetonitrile/Water extracts	93.79	125.811	95.3	37.308	63.67	0.248
Dicamba	24.21	32.473	12.33	4.828	0.64	0.003
DCSA	4.08	5.473	1.93	0.757	0.46	0.002
DCSA Glucoside	60.32	80.913	67.26	26.333	15.27	0.059
DCSA HMGglucoside	1.14	1.535	2.48	0.97	9.61	0.037
Unk DCSA/DCGA Glucoside	0.12	0.164	ND	ND	ND	ND
Unk DCSA/DCGA Conj.	0.38	0.503	1.75	0.686	0.62	0.002
DCGA Glucoside	0.75	1.007	4.32	1.69	2.07	0.008
DCGA Malonyl glucoside	1.11	1.485	1.61	0.631	4.64	0.018
Sugars	ND	ND	0.49	0.19	9.15	0.036
Total Unknowns (n=11)[a]	1.52 [0.4]	2.038 [0.541]	3.13 [0.95]	1.227 [0.373]	4.27 [0.84]	0.0144 [0.003]
Total Identified or Characterized Metabolites	92.1	123.552	92.17	36.085	53.21	0.207
PES	6.21	8.336	4.7	1.841	36.33	0.141
Phosphate	Not Analysed				1.42	0.006
Starch					1.27	0.005
Protein					8.18	0.032
Pectin					2.78	0.011
Cellulose					3.46	0.013
Hemicellulose					12.9	0.05
Unextracted					7.6	0.03

ND = Not detected

[a] Maximum individual values listed in brackets

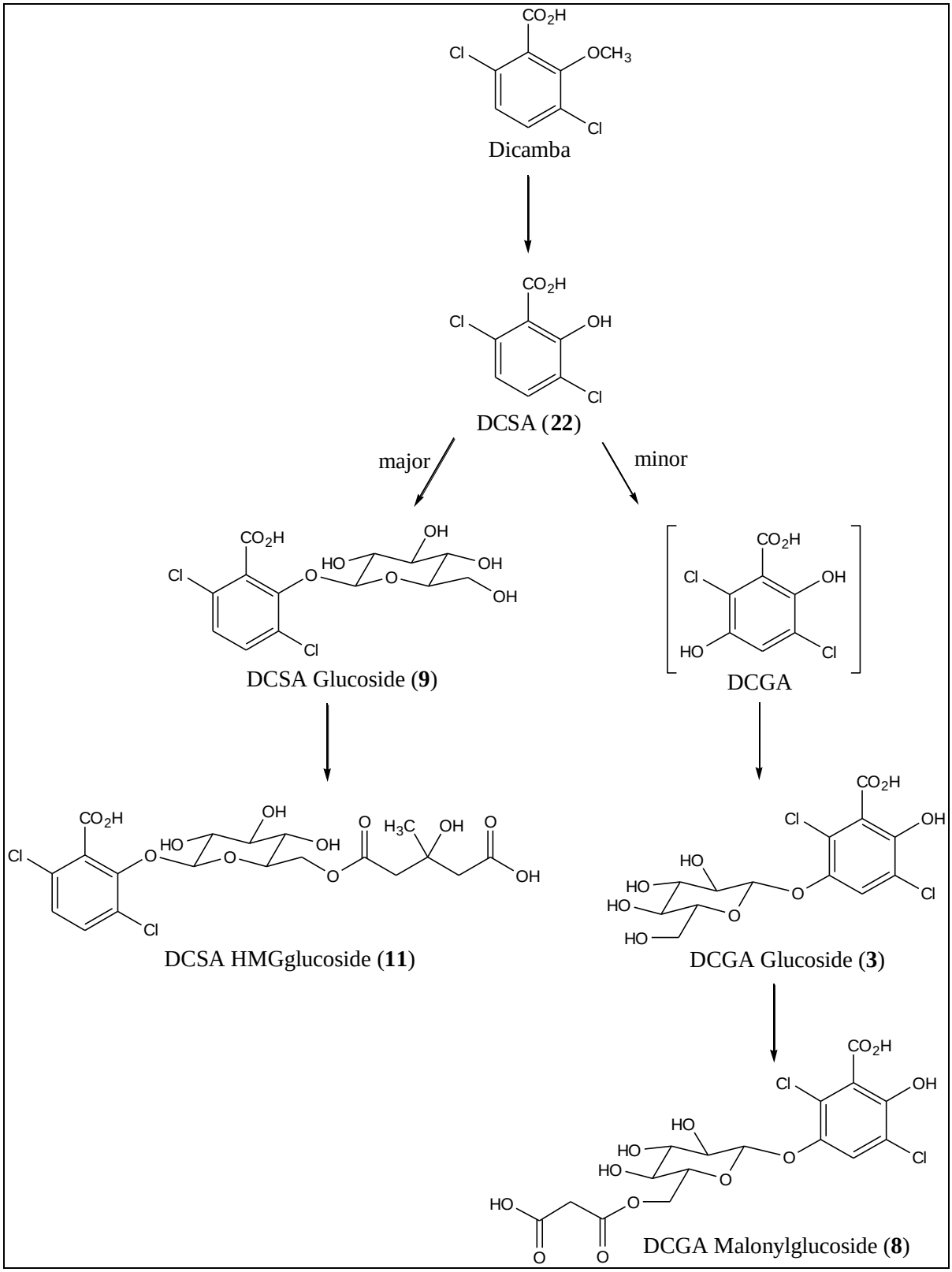


Figure 1 Proposed metabolic pathway of dicamba in dicamba-tolerant soya bean. (Provided by the sponsor)

Maize

The nature of the residue of dicamba in dicamba-tolerant maize was investigated by Adio and Feng (2015; Report MSL0025703). Maize plants were grown in small, outdoor box plots. Application of [phenyl-U-^{14}C]dicamba was made at a target rate of 2.24 kg ai/ha either on the day of planting (pre-emergence; PRE; n=42) or 30 days after planting (post-emergence; POE; n=42). Samples were collected as follows: Immature foliage 19 days after planting (PRE only), forage 80 (PRE) or 50 (POE) days after application, and stover and grain 114 (PRE) or 84 (POE) days after application. Samples were ground and then further homogenized by cryomilling.

Total radioactive residues (TRR) were determined by combustion/LSC. Residues were extracted with acetonitrile:water (2:3, v/v) from all matrices except grain, for which hexane was used first to extract oils (POE sample only). The remaining material was then rinsed with methanol and then extracted with acetonitrile:water (2:3, v/v). Unextracted radioactivity from the PES was determined by combustion/LSC. Extracts were concentrated by rotary evaporation (analysis of the distillate indicated no significant amount of radioactivity was lost due to volatility). An aliquot of the concentrated aqueous extracts underwent hydrolysis (2 N HCl, ca. 100 °C, 2 hours). Aqueous extracts and hydrolysates were partitioned against ethyl acetate to assess partitioning behaviour. Specific residues were isolated using preparative HPLC analysis. Identification and characterization of metabolites was accomplished using HPLC-MS/MS as well as HPLC-UV and HPLC-RAD. Samples also underwent derivatization with trimethylsilyldiazomethane to discern the presence of carboxylic acid or phenolic groups and with acetic anhydride/pyridine to discern the presence of hydroxyl or phenolic groups. Finally, specific isolates underwent acid hydrolysis (1 N HCl, ca 100 °C, 1-2 hours), base hydrolysis (1 N NaOH, ambient temperature, 1 h) and ß-glucosidase digestion (37 °C, 48 hours) to identify/characterize conjugated metabolites. PES underwent further workup with dilute acid and base extraction, phosphate rinse, α-amylase digestion, protease digestion, EDTA extraction, oxidation with chlorite, hydrolysis with cellulase, and hydrolysis with KOH.

Total radioactive residues (extracted + unextracted) from PRE application were 4.5 mg eq/kg in immature foliage, 0.075 mg eq/kg in forage, 0.24 mg eq.kg in stover, and 0.043 mg eq/kg in grain. Following POE application, TRR were 2.2 mg eq/kg in forage, 7.8 mg eq/kg in stover, and 0.062 mg eq/kg in grain. Radioactivity was not quantifiable in control plant matrices.

Extractions with acetonitrile:water resulted in 89% TRR extracted from immature foliage, 76/86% from forage (PRE/POE), and 74/83% from stover (PRE/POE). Extraction efficiency was much lower for grain: 7.4% PRE and 13% POE; inclusion of residues extracted into hexane gives 18% extracted (POE). Of the extracted radioactivity, most was associate with sugars and organic acids (9.4% TRR) and the majority of the remainder was in the form of dicamba-specific residues (e.g., dicamba and free and conjugated forms of 5-hydroxydicamba, DCSA, and DCGA). Further work with the PES showed that the vast majority of the unextracted radioactivity was associated with natural products (starch, lignin, cellulose, etc.).

A summary of extracted residues, PES, and hydrolysis and digestion products is shown in Tables 4 and 5.

Table 4 Summary of the nature of the residues in dicamba-tolerant maize following PRE application of dicamba

TRR, mg eq/kg	Immature foliage		Forage		Stover		Grain	
	4.513		0.075		0.243		0.043	
Identification	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Triglycerides (from the hexane-extracted oil fraction)	Not Analysed						4.64	0.002
Acetonitrile/water extracts	89.13	4.02	76.64	0.057	74.71	0.181	7.36	0.005
Dicamba	3.42	0.154	NQ	NA	0.15	0.0004	NQ	NA
5-OH Dicamba	1.09	0.049	2.01	0.001	1.82	0.004	0.02	<0.0001
5-OH Dicamba Glucoside	1.02	0.046	NQ	NA	0.08	0.0002	NQ	NA
DCSA	3.75	0.169	0.23	0.0002	4.35	0.011	NQ	NA
DCSA Glucoside	53.17	2.4	41.56	0.031	30.52	0.074	0.10	<0.0001
DCSA Pentoside/Unk DCGA Conj.	4.51	0.204	4.87	0.004	5.39	0.013	0.04	<0.0001

TRR, mg eq/kg	Immature foliage		Forage		Stover		Grain	
	4.513		0.075		0.243		0.043	
Identification	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
DCSA Succinylglucoside	NQ	NA	2.49	0.002	2.97	0.007	0.03	<0.0001
DCGA Glucoside/DCGA Pentosylglucoside	11.2	0.505	3.27	0.002	2.35	0.006	0.14	≤0.0001
Unk DCSA/DCGA Conj.	1.22	0.055	1.46	0.001	2.15	0.005	0.04	<0.0001
Unk DCSA/DCGA Conj.	0.16	0.007	1.17	0.0009	1.68	0.004	NQ	NA
Unk DCSA/DCGA Conj.	0.35	0.016	1.12	0.0008	1.47	0.004	NQ	NA
MCTHBA Glucoside	1.71	0.077	0.60	0.0004	1.43	0.003	0.03	<0.0001
MCTHBA Cyc Glucoside/DCSA HMGglucoside	3.07	0.139	2.41	0.002	2.94	0.007	0.04	<0.0001
Unk MCDHBA conj.	0.64	0.029	0.74	0.0006	0.57	0.001	0.04	<0.0001
Nat. prod. Organic Acids	1.74	0.079	2.43	0.002	0.95	0.002	0.22	0.0001
Sugars	1.12	0.051	6.23	0.005	6.37	0.015	2.50	0.0011
Total Unknowns (n≤12)[a]	0.96 [0.96]	0.043 [0.043]	5.99 [1.76]	0.0041 [0.001]	8.31 [1.96]	0.021 [0.005]	0.09 [0.05]	<0.0003 [<0.0001]
Total Identified or Characterized Metabolites	88.17	3.98	70.59	0.0529	65.19	0.1566	7.84	0.0032
PES	10.88	0.491	22.67	0.017	25.10	0.061	88.37	0.038
Phosphate	Not Analysed		1.80	0.001	3.19	0.008	1.21	0.001
Starch			6.36	0.005	6.76	0.016	21.48	0.009
Protein			1.31	0.001	1.62	0.004	11.51	0.005
Pectin			0.78	0.001	1.09	0.003	3.32	0.001
Lignin			2.31	0.002	3.85	0.009	6.61	0.003
Cellulose			6.27	0.005	5.55	0.013	26.72	0.011
Hemicellulose			4.27	0.003	2.22	0.005	16.67	0.007
Unextracted			0.27	0.0002	1.00	0.0024	0.49	0.0002

NQ = Not Quantified; there was insufficient material to quantify the amount of residue present in the sample.

NA = Not Applicable

[a] Maximum individual values listed in brackets

Table 5 Summary of the nature of the residues in dicamba-tolerant maize following POE application of dicamba

TRR, mg eq/kg	Forage		Stover		Grain	
	2.228		7.826		0.062	
Identification	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Triglycerides (from the hexane-extracted oil fraction)	Not Analysed				5.31	0.003
Acetonitrile/water extracts	85.82	1.912	83.28	6.518	12.78	0.008
Dicamba	8.64	0.193	6.27	0.491	0.01	<0.0001
5-OH Dicamba	3.01	0.067	3.85	0.301	0.11	≤0.0001
5-OH Dicamba Glucoside	0.36	0.008	0.56	0.044	0.04	<0.0001
DCSA	1.6	0.036	5.09	0.398	0.03	<0.0001
DCSA Glucoside	33.46	0.745	26.94	2.108	0.44	0.0003
DCSA Pentoside/Unk DCGA Conj.	3.65	0.081	3.43	0.269	0.20	0.0001
DCSA Succinylglucoside	3.62	0.081	4.26	0.333	0.12	≤0.0001
DCGA Glucoside/DCGA Pentosylglucoside	3.75	0.084	2.47	0.193	0.39	0.0002
Unk DCSA/DCGA Conj.	2.14	0.048	2.56	0.20	0.16	≤0.0001
Unk DCSA/DCGA Conj.	1.89	0.042	2.36	0.184	0.03	<0.0001
Unk DCSA/DCGA Conj.	1.58	0.035	1.88	0.147	0.04	<0.0001
MCTHBA Glucoside	2.32	0.052	1.87	0.146	0.09	≤0.0001
MCTHBA Cyc Glucoside/DCSA HMGglucoside	3.73	0.083	4.09	0.32	0.14	≤0.0001
Unk MCDHBA conj.	1.23	0.027	1.33	0.104	0.06	<0.0001
Nat. prod. Organic Acids	2.39	0.053	2.61	0.204	1.05	0.0006
Sugars	1.11	0.025	1.49	0.117	3.06	0.0019
Total Unknowns (n≤12)[a]	10.3 [2.27]	0.229 [0.05]	11.5 [2.66]	0.901 [0.208]	0.37 [0.1]	<0.0007 [≤0.0001]
Total Identified or Characterized Metabolites	74.48	1.66	71.06	5.559	11.28	0.0061
PES	14.18	0.316	16.71	1.308	80.65	0.05
Phosphate	2.04	0.045	2.59	0.20	1.67	0.001
Starch	6.43	0.143	7.20	0.56	18.62	0.011

TRR, mg eq/kg	Forage		Stover		Grain	
	2.228		7.826		0.062	
Identification	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Protein	1.04	0.023	1.61	0.13	8.32	0.005
Pectin	1.05	0.023	1.15	0.09	3.09	0.002
Lignin	2.29	0.051	2.57	0.20	6.70	0.004
Cellulose	0.74	0.017	0.75	0.06	26.59	0.016
Hemicellulose	0.57	0.013	0.76	0.06	15.91	0.01
Unextracted	0.02	0.0005	0.09	0.007	0.69	0.0004

[a] Maximum values listed in brackets

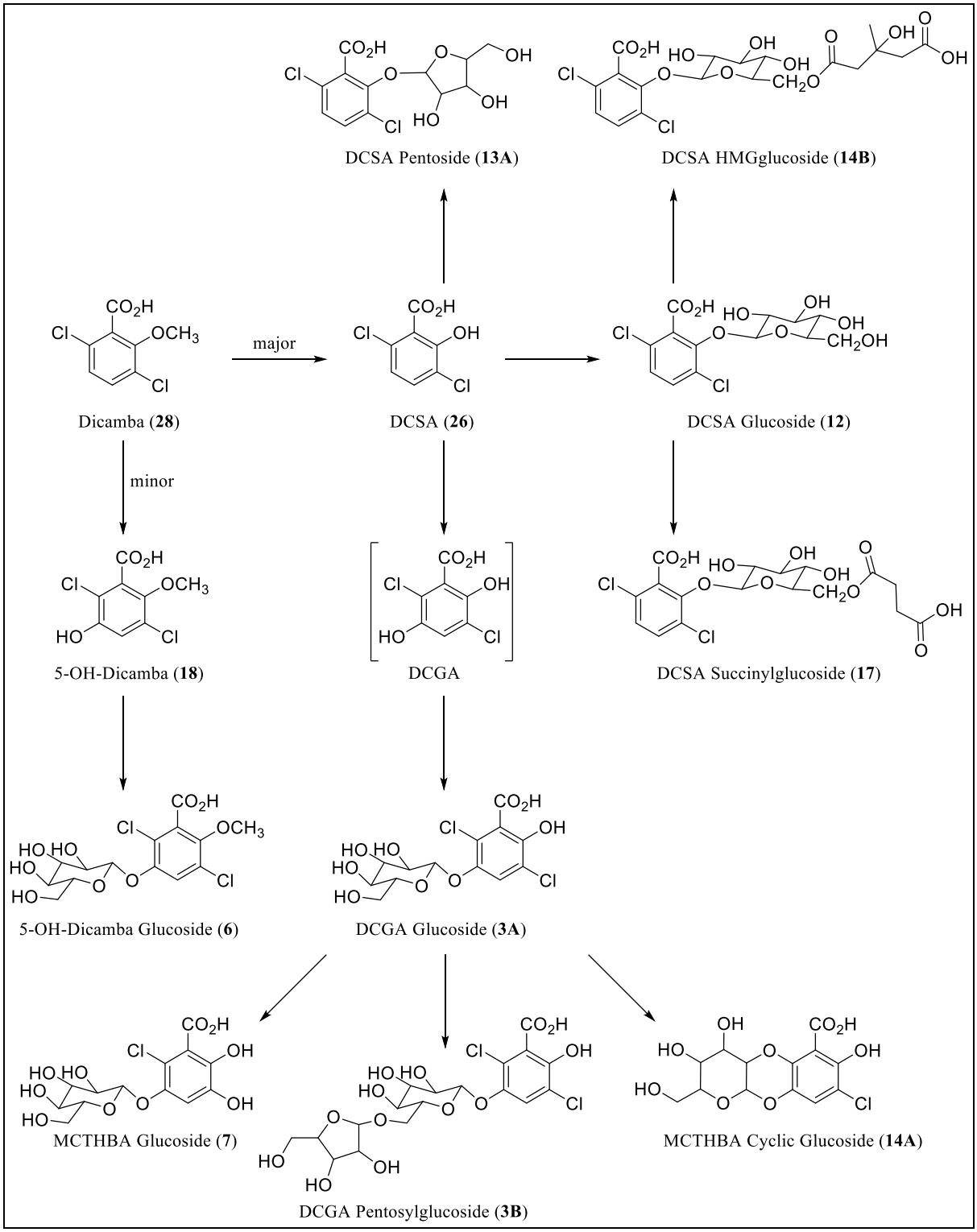


Figure 2 Proposed metabolic pathway of dicamba in dicamba-tolerant maize. (Provided by the sponsor)

Cotton

The nature of the residue of dicamba in dicamba-tolerant cotton was investigated by Whitehead *et al*. (2011; Report MSL0023760). Cotton plants were grown in small, outdoor box plots (~0.84 m^2). Application of [phenyl-U-^{14}C]dicamba was made at a target rate of 2.24 kg ai/ha either on the day of planting (pre-emergence; PRE) or 76 days after planting (post-emergence; POE). Samples of seed cotton (seeds + lint), leaves, and stems were collected 180/104 days after the PRE/POE treatments.

Leaves and stems served as surrogate gin trash samples. The gin trash samples were ground and frozen. Seed cotton samples were ginned to produce undelinted seed, which was homogenized by cryomilling.

Total radioactive residues (TRR) were determined by combustion/LSC. Residues were extracted from gin trash with acetonitrile:water (2:3, v/v). Samples of undelinted seed were extracted sequentially with hexane (three times), acetonitrile (one time), and acetonitrile:water (2:3, v/v; four times). The remaining material was then rinsed with methanol and then extracted with acetonitrile:water (2:3, v/v). Unextracted radioactivity from the PES was determined by combustion/LSC. Extracts were concentrated by rotary evaporation (analysis of the distillate indicated no significant amount of radioactivity was lost due to volatility). An aliquot of the concentrated aqueous extracts underwent hydrolysis (2 N HCl, approximately 100 °C, 2 hours or 2 N NaOH, 60 °C, 4 hours). Aqueous extracts and hydrolysates were partitioned against ethyl acetate to assess partitioning behaviour. Specific residues were isolated using preparative HPLC analysis. Identification and characterization of metabolites was accomplished using HPLC-MS/MS as well as HPLC-UV and HPLC-RAD. Samples also underwent derivatization with trimethylsilyldiazomethane to discern the presence of carboxylic acid or phenolic groups and with acetic anhydride/pyridine to discern the presence of hydroxyl or phenolic groups. Finally, specific isolates underwent acid hydrolysis (1 N HCl, approximately 100 °C, 1–2 hours) and base hydrolysis (1 M NaOH, ambient temperature, 1 hour). PES underwent further workup with dilute acid and base extraction, phosphate rinse, α-amylase digestion, protease digestion, EDTA extraction, oxidation with chlorite, hydrolysis with cellulase, and hydrolysis with KOH.

Total radioactive residues (extracted + unextracted) from PRE application were 0.85 mg eq/kg in gin trash and 0.16 mg eq/kg in undelinted seed. Following POE application, TRRs were 60 mg eq/kg in gin trash and 0.98 mg eq/kg in undelinted seed. Low levels of radioactivity (0.003–0.005 mg eq/kg) were observed in the control boxes, which were approx. 1.8 and 30 m from the PRE and POE treated boxes, respectively. This may be evidence of volatilisation and uptake of dicamba or volatile metabolites from treated plants/boxes to the control plants.

The extraction schemes resulted in 76/71% TRR extracted from gin trash (PRE/POE) and 31/38% from undelinted seed (PRE/POE). Of the material extracted from undelinted seed, 20% and 12% was associated with the hexane fraction for PRE and POE samples, respectively. Further work with the PES showed that the vast majority of the unextracted radioactivity was associated with natural products (starch, lignin, cellulose, etc.).

A summary of extracted residues, PES, and hydrolysis and digestion products is shown in Table 6.

Table 6 Summary of the nature of the residues in dicamba-tolerant cotton following PRE and POE application of dicamba

	PRE				POE			
	Gin trash		Seed		Gin trash		Seed	
TRR, mg eq/kg	0.8493		0.1621		60.0235		0.9778	
Identification	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Triglycerides (from the hexane-extracted oil fraction)	Not analysed		14.41	0.023	Not analysed		8.8	0.086
Acetonitrile/Water extracts	76.24	0.648	30.84	0.05	71.28	42.785	38.26	0.374
Dicamba	0.5	0.004	0.09	0.0001	4.48	2.691	0.85	0.008
Dicamba Amide	3.22	0.027	0.23	0.0004	ND	ND	ND	ND
DCSA	5.42	0.046	0.06	0.0001	13.39	8.035	1.91	0.019
DCSA Glucoside	27.77	0.236	0.73	0.0012	16.83	10.101	3.42	0.033
DCGA Glucoside	0.68	0.006	0.09	0.0001	2.46	1.476	1.1	0.011
MCTHBA Glucoside [A]	ND	ND	ND	ND	3.33	1.998	0.53	0.005
MCTHBA Cyc Glc	2.94	0.025	ND	ND	2.76	1.656	ND	ND
MCDHBA Glucoside Sulfate	ND	ND	ND	ND	4.71	2.828	ND	ND
MCDHBA Glucoside [a]	ND	ND	ND	ND	0.84	0.505	0.72	0.002
Sugars	8.94	0.076	4.59	0.0074	2.69	1.613	5.61	0.008
Total Unknowns (n=27)[b]	15.97 [2.91]	0.135 [0.025]	4.42 [0.9]	0.0045 [0.0007]	0.86 [0.86]	0.518 [0.518]	4.81 [0.81]	0.049 [0.008]

TRR, mg eq/kg	PRE				POE			
	Gin trash		Seed		Gin trash		Seed	
	0.8493		0.1621		60.0235		0.9778	
Identification	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Total Identified or Characterized Metabolites	49.49	0.42	19.96	0.032	51.49	30.903	21.69	0.165
PES	23.76	0.202	69.16	0.112	28.72	17.239	61.74	0.604
Phosphate	3.25	0.028	3.87	0.006	6.2	3.72	1.96	0.019
Starch	2.73	0.023	2.79	0.005	7.05	4.234	2.19	0.021
Protein	1.71	0.015	17.71	0.029	2.25	1.35	7.43	0.073
Pectin	1.91	0.016	7.4	0.012	2.66	1.595	4.04	0.039
Lignin	5.28	0.045	6.18	0.01	6.6	3.962	8.43	0.082
Cellulose	0.9	0.008	4.37	0.007	0.62	0.374	3.13	0.031
Hemicellulose	5.35	0.045	14.43	0.023	2.8	1.679	13.49	0.132
Sulfuric acid	Not analysed		5.07	0.008	Not analysed		3.21	0.031
Protein (2nd)			NA	NA			2.67	0.026
Hot DMSO			NA	NA			5.57	0.054
Unextracted	2.62	0.022	7.35	0.012	0.61	0.365	9.66	0.094

ND = Not Found

[a] Identity not confirmed

[b] Maximum individual values listed in brackets

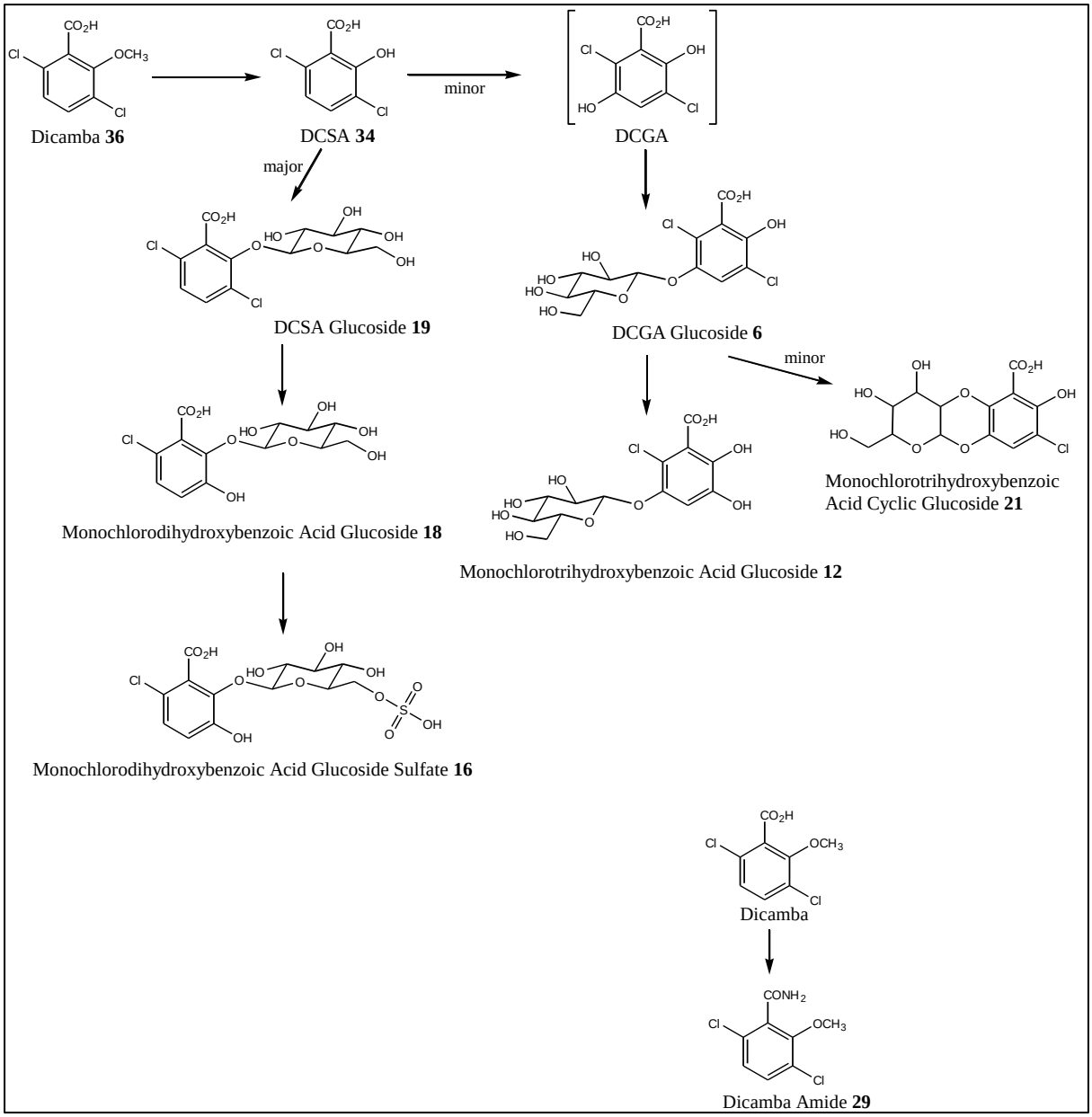


Figure 3 Proposed metabolic pathway of dicamba in dicamba-tolerant cotton. (Provided by the sponsor)

In summary, metabolism of dicamba by the dicamba-tolerant varieties reported above is similar. Demethylation of dicamba results in the formation of DCSA, which is subsequently either conjugated with glucose or hydroxylated to form DCGA, which also undergoes glucose conjugation. The predominant residue is free and glucose-conjugated DCSA, with most of those residues occurring in the conjugated form. In seeds/grain, a large percentage of the radioactive residue was shown to be incorporation of the radiolabelled carbon into natural plant constituents.

RESIDUE ANALYSIS

Analytical Methods

The Meeting received three methods for the analysis of dicamba, one each for dicamba-tolerant soya bean (AG-ME-1321-01), dicamba-tolerant maize (ME-1713), and dicamba-tolerant cotton (AG-ME-1381-01). All three methods are essentially the same and involve extraction of residues into acetonitrile:water, hydrolysis of conjugated metabolites to their free form by 1N HCl digestion at 95 °C

for 1 hour (AG-ME-1321-01, AG-ME-1381-01) or 1.5 hours (ME-1713), clean-up by liquid-liquid partitioning with ethyl acetate:isooctane (1:4, v/v ME-1713 and AG-ME-1381-01; 2:3, v/v AG-ME-1321-01), addition of ^{13}C-labelled internal standards of dicamba, 5-OH dicamba, DCSA, and DCGA, and analysis by LC-MS/MS. The hydrolysis step is similar to that used in the metabolism studies (2N HCl, 100 °C, 2 hours). For soya bean and cotton seed, the method LOQ was determined as the lowest fortification level giving acceptable accuracy (70-120% recovery) and precision (CV ≤ 20%) at that level and all greater levels; for other matrices, the LOQ was determined as the lowest limit of method validation (LLMV). When accuracy and precision were acceptable, but the number of replications was <5, the LOQ was considered to be the fortification level that brought the total number of replicates to at least 5 (see, for example, Table 7, defatted flour).

Soya bean

The analytical method for residues of dicamba in soya bean matrices was validated in seed, forage, hay, and numerous processed commodities (Table 7). Recoveries from samples fortified at 0.01 mg/kg were generally acceptable, with a few exceptions (e.g., 5-OH dicamba in soya bean seed). For some analytes and matrices, acceptable recovery was observed at the 0.005 mg/kg fortification level.

Table 7 Summary of recoveries of dicamba and metabolites from fortified soya bean matrices

Matrix	Fortification Level (mg/kg)	n	Mean recovery (%) (% RSD)			
			Dicamba	DCSA	5-OH Dicamba	DCGA
Seed	0 (Control) [a]	7	ND	ND	ND [b]	<0.001
	0.005	7	103 (7.35)	107 (5.18)	63.7 (11.1)	88.0 (5.54)
	0.01	7	99.8 (5.85)	106 (3.55)	66.9 (9.19)	95.9 (2.88)
	0.02	7	97.9 (4.81)	105 (2.39)	71.3 (6.53)	97.4 (3.52)
	0.1	7	97.1 (2.97)	104 (2.12)	102 (2.25)	101 (2.76)
	2	2	87.8	97.3	104	87.7
Forage	0 (Control) [a]	7	ND [b]	0.0014	ND	ND
	0.005	7	97.9 (7.49)	72.6 (5.57)	102 (5.22)	105 (9.30)
	0.01	7	99.3 (4.60)	80.9 (3.10)	110 (7.75)	101 (9.45)
	0.02	7	99.8 (3.52)	88.0 (2.77)	108 (2.42)	100 (5.83)
	0.1	7	105 (2.13)	90.0 (2.51)	106 (3.36)	105 (5.11)
	2	2	95.8	97.4	88.5	92.8
Hay	0 (Control) [a]	7	ND	ND	ND [b]	<0.001
	0.005	7	ND [c]	97.4 (4.55)	77.4 (8.73)	68.4 (2.69)
	0.01	7	86.3 (6.56)	105 (2.94)	80.0 (6.67)	82.9 (3.00)
	0.02	7	95.9 (10.5)	106 (2.01)	84.0 (9.13)	90.5 (1.82)
	0.1	7	105 (4.05)	107 (2.78)	102 (3.94)	100 (3.88)
	2	2	72.5	88.0	89.8	90.0
Hulls	0 (Control) [a]	3	ND [b]	<0.001	<0.001	<0.001
	0.01	2	92.5	115	74.4	78.4
	0.02	4	92.8 (6.80)	109 (1.42)	74.9 (15.3)	86.2 (8.39)
	0.05	3	96.1 (3.81)	104 (8.09)	74.8 (29.3)	88.3 (13.0)
	0.2	1	96.5	99.5	100	86.8
	0.4	2	101	98.3	91.6	90.9
	1.5	1	106	97.9	101	97.9
Defatted flour	0 (Control) [a]	4	<0.001	<0.001	0.007	<0.001
	0.01	2	79.3	102	31.0	83.0
	0.02	2	78.2	98.2	64.4	99.9
	0.05	1	96.3	103	84.3	84.8
	0.2	1	94.1	94.0	91.5	91.0
	0.4	3	103 (5.82)	104 (7.11)	99.4 (2.57)	96.0 (6.10)
	2	2	81.9	89.2	93.5	97.7
	3	1	97.6	92.3	98.5	96.3
Defatted meal	0 (Control) [a]	3	ND [b]	<0.002	<0.001	<0.001
	0.01	2	88.7	92.9	70.7	81.8
	0.02	4	97.5 (5.42)	105 (11.8)	86.6 (10.8)	92.8 (7.42)
	0.05	3	92.8 (10.8)	107 (7.70)	86.5 (9.74)	92.0 7.85)
	0.2	1	94.0	101	101	83.2
	0.4	2	96.7	102	85.7	92.9
	1.5	1	88.0	93.8	95.3	104

Matrix	Fortification Level (mg/kg)	n	Mean recovery (%) (% RSD)			
			Dicamba	DCSA	5-OH Dicamba	DCGA
Protein isolate	0 (Control) [a]	4	ND [b]	<0.001	ND	<0.001
	0.01	2	80.5	89.4	86.8	79.3
	0.02	2	87.1	94.7	94.1	82.9
	0.4	2	81.7	92.7	97.7	89.2
	0.8	2	87.1	100	98.0	93.6
	1.5	1	88.1	92.6	101	86.7
Protein concentrate	0 (Control) [a]	3	ND [b]	<0.001	ND	<0.001
	0.01	2	81.0	96.3	80.7	82.7
	0.02	1	90.1	103	84.8	89.2
	0.2	1	90.1	105	106	97.1
	0.4	2	114	97.3	100	95.9
	1.5	1	94.3	90.6	105	89.3
Crude lecithin	0 (Control) [a]	8	<0.001	<0.001	<0.001	ND [b]
	0.01	3	83.5 (18.1)	93.6 (4.90)	84.1 (7.93)	71.3 (7.54)
	0.2	3	86.1 (10.7)	96.7 (8.15)	98.9 (4.05)	78.8 (2.04)
	0.4	2	107	107	107	90.8
	2	2	107	108	103	101
Degummed oil	0 (Control) [a]	4	ND [b]	<0.001	<0.001	<0.001
	0.01	3	98.5 (3.61)	96.0 (3.19)	84.9 (2.65)	70.8 (14.5)
	0.02	3	93.5 (5.16)	99.1 (2.42)	87.7 (2.20)	77.9 (19.6)
	0.05	2	86.8	101	93.7	82.2
	0.4	3	96.5 (12.1)	104 (3.49)	99.3 (8.38)	92.6 (7.20)
	2	2	103	99.4	101	105
Refined oil	0 (Control) [a]	4	<0.001	<0.001	<0.003	<0.002
	0.01	3	88.9 (4.31)	95.1 (1.03)	80.4 (9.65)	63.6 (5.17)
	0.02	3	87.6 (18.3)	98.2 (5.38)	86.7 (7.48)	73.2 (7.96)
	0.05	2	91.1	96.2	100	79.3
	0.2	1	105	99.7	90.0	94.5
	0.4	3	104 (8.32)	103 (4.94)	102 (8.76)	99.4 (5.29)
	2	2	102	107	100	103
Soymilk	0 (Control) [a]	4	ND [b]	ND	<0.001	<0.001
	0.01	3	95.1 (6.26)	101 (5.10)	83.5 (1.52)	72.6 (4.58)
	0.02	3	96.2 (11.5)	103 (8.58)	88.9 (7.30)	83.3 (9.56)
	0.05	2	83.9	104	94.5	86.5
	0.2	1	64.0	105	87.5	107
	0.4	3	90.7 (11.2)	94.1 (5.58)	92.9 (2.38)	87.9 (7.92)
	2	2	77.7	97.8	92.4	95.2
Tofu	0 (Control)[a]	2	ND [b]	ND	<0.001	<0.001
	0.01	2	77.7	109	92.7	77.8
	0.02	1	80.5	101	96.4	78.2
	0.05	1	80.9	103	100	92.6
	0.2	1	97.6	91.6	94.7	85.6
	0.4	3	81.2 (16.3)	82.5 (13.4)	84.5 (11.7)	85.7 (11.8)
	1.5	1	91.1	80.5	83.3	93.3

[a] Values reported for unspiked control samples are mg/kg

[b] ND = not detected in any sample

[c] Mean residue in extracts stored 72 hrs was 0.0055 mg/kg (RSD = 0.1%)

Maize

The analytical method for residues of dicamba in maize matrices was validated in grain, forage, and stover (Table 8).

Table 8 Summary of recoveries of dicamba and metabolites from fortified maize matrices

Matrix	Fortification Level (mg/kg)	n	Mean recovery (%) (% RSD)			
			Dicamba	DCSA	5-OH Dicamba	DCGA
Grain	0.01	6	106 (8.8)	106 (3.3)	99 (15)	91 (12)

Matrix	Fortification Level (mg/kg)	n	Mean recovery (%) (% RSD)			
			Dicamba	DCSA	5-OH Dicamba	DCGA
	0.1	6	97 (5.9)	100 (6)	101 (9.5)	95 (3.8)
	0.5	5	101 (2.8)	105 (2.6)	102 (9)	110 (5)
Forage	0.01	6	98 (12)	106 (4.1)	107 (10)	95 (11)
	0.1	6	98 (5.7)	93 (8.3)	91 (10)	78 (7)
	0.5	6	98 (2.8	95 (9.9)	95 (12)	92 (4.8)
Stover	0.01	6	83 (16)	108 (4.2)	117 (1.6)	95 (14) [97 (17)] [a]
	0.1	6	99 (3.6)	98 (4.2)	97 (7.3)	77 (4.2) [101 (4.7)] [a]
	0.5	6	98 (4.7)	96 (5.7)	101 (6.3)	97 (7.3) [106 (4.3)] [a]

[a] Values in square brackets are from samples prepared by an alternate preparation which includes clean-up through a 96-well filter plate

Cotton

The analytical method for residues of dicamba in cotton matrices was validated in undelinted seed, gin trash, hulls, meal, and refined oil (Table 9). For Method AG-ME-1381-01, recoveries and relative standard deviations were acceptable at fortifications of 0.02 mg/kg and greater for all commodities and analytes. Recoveries and relative standard deviations were also acceptable at 0.005 mg/kg in undelinted seed for DCSA, 5-OH dicamba, and DCGA. For MethodME-1713, recoveries and standard deviations were acceptable at 0.01 mg/kg in undelinted seed for all analytes.

Table 9 Summary of recoveries of dicamba and metabolites from fortified cotton matrices

Matrix	Fortification Level (mg/kg)	n	Mean recovery (%) (% RSD)			
			Dicamba	DCSA	5-OH Dicamba	DCGA
Method AG-ME-1381-01						
Undelinted seed	0 (Control) [a]	2	ND [b]	<0.001	<0.001	<0.002
	0.005	5	128 (38.4)	101 (18.5)	82.8 (25.5)	114 (18.9)
	0.01	5	115 (25.1)	89.8 (14.7)	94.2 (16.2)	95.7 (12.0)
	0.02	5	113 (10.5)	93.9 (7.38)	108 (9.26)	87.1 (9.75)
	0.2	5	96.1 (2.25)	98.9 (4.08)	102 (2.90)	88.9 (4.81)
	10	5	89.6 (14.1)	91.6 (6.94)	87.1 (15.3)	88.6 (10.1)
Gin trash	0 (Control) [a]	2	ND [b]	ND [b]	0.010	0.012
	0.04	5	104 (14.6)	79.5 (6.14)	106 (10.1)	80.2 (14.0)
	0.4	5	99.5 (2.73)	89.0 (4.15)	111 (3.28)	97.0 (2.10)
	10	5	86.7 (3.55)	102 (7.40)	99.0 (10.1)	101 (4.30)
Hulls	0 (Control) [a]	2	<0.003	ND [b]	ND [b]	<0.003
	0.02	5	100 (3.38)	100 (6.17)	91.0 (11.5)	116 (2.94)
	0.2	5	100 (2.11)	97.4 (4.05)	108 (6.80)	85.2 (2.83)
Meal	0 (Control) [a]	2	<0.003	<0.003	ND [b]	ND [b]
	0.02	5	104(5.11)	96.5(4.52)	88.1(16.1)	102(3.21)
	0.2	5	96.9 (2.46)	86.2 (4.35)	93.4 (5.28)	86.0 (1.74)
Refined oil	0 (Control) [a]	2	<0.004	ND [b]	ND [b]	ND [b]
	0.02	5	97.6(1.22)	95.2(5.33)	90.8(8.82)	103(5.51)
	0.2	5	99.7 (2.85)	103 (2.01)	102 (8.78)	91.8 (3.07)
Method ME-1713						
Undelinted seed	0.01	6	102 (5.7)	97 (4)	98 (12)	94 (3.9)
	0.1	6	102 (3.3)	97 (1.2)	98 (5.8)	76 (8)
	0.5	6	103 (5.6)	100 (4.8)	101 (6.4)	83 (5.1)

[a] Values reported for unspiked control samples are mg/kg

[b] ND = not detected in any sample

In summary, the submitted analytical methods are suitable for the analysis of dicamba, 5-OH dicamba, and free and conjugated forms of both DCSA and DCGA in the commodities tested.

Stability of pesticide residues in stored analytical samples

The Meeting received data reporting on the stability of dicamba, 5-OH dicamba, DCSA, and DCGA in stored samples of soya bean (M. Mueth and J. Foster, 2012, Report MSL0027420) and cotton (D. Maher and J. Foster, 2012, Report MSL0023058). For both crops, stability was evaluated by using samples from field trials bearing incurred residues of dicamba and metabolites. A single sample for each matrix was kept frozen under conditions mimicking the storage conditions for residue samples and analysing the residues from those samples over time (0–24 months for soya bean seed and 0-9 months for cotton undelinted seed). Samples were analysed in duplicate and the analytical methods included a hydrolysis step; therefore, residues reported as DCSA and DCGA include both free and conjugated forms.

Soya bean

Residues in soya bean matrices were analysed using Method AG-ME-1321-01. Procedural recoveries for soya bean matrices were between 70 and 120% for all analytes-matrices except dicamba in forage (140% at 0.4 mg/kg, Day 0 sample set and 146% at 0.5 mg/kg, Day 61 sample set), dicamba in hay (127% at 0.4 mg/kg, Day 0 sample set), and DCGA (66% at 4 mg/kg, Days 54–61 sample set).

Residues of 5-OH dicamba were < 0.02 mg/kg in all samples, including Day 0; therefore, storage stability for that compound could not be evaluated. For dicamba, DCSA (free and conjugated) and DCGA (free and conjugated), residues from the storage stability samples are summarized in Table 10.

Table 10 Storage stability of dicamba, DCSA, and DCGA in soya bean matrices

Storage Period, months	Dicamba		DCSA		DCGA	
	mg/kg [mean]	% of Day 0	mg/kg [mean]	% of Day 0	mg/kg [mean]	% of Day 0
Forage						
0	0.650, 0.675 [0.6625]	100	33.1, 33.1 [33.1]	100	5.10, 4.82 [4.96]	100
2	1.17, 1.15 [1.16]	175	35.2, 36.3 [35.8]	108	5.12, 4.34 [4.73]	95
3	0.674, 0.614 [0.644]	97	38.8, 37.7 [38.2]	116	5.56, 5.58 [5.57]	112
6	0.676, 0.696 [0.686]	104	36.4, 35.4 [35.9]	108	2.96, 3.11 [3.04]	61
10	0.689, 0.739 [0.714]	108	36.2, 38.5 [37.4]	113	3.24, 3.13 [3.18]	64
12	0.683, 0.709 [0.696]	105	38.1, 35.7 [36.9]	111	3.23, 3.09 [3.16]	64
18	0.660, 0.711 [0.686]	103	40.2, 38.0 [39.1]	118	2.66, 2.28 [2.47]	50
24	0.760, 0.727 [0.744]	112	39.7, 33.1 [36.4]	110	1.80, 1.85 [1.82]	37
Hay						
0	0.129, 0.127 [0.128]	100	109, 117 [113]	100	7.92, 8.47 [8.20]	100
2	0.147, 0.140 [0.144]	112	92.3, 94.5 [93.4]	83	8.12, 8.24 [8.18]	100
3	0.126, 0.131 [0.128]	100	107, 108 [107.5]	95	9.59, 9.54 [9.56]	117
6	0.133, 0.122 [0.128]	100	112, 106 [109]	96	7.12, 7.32 [7.22]	88
10	0.121, 0.098 [0.110]	86	80.7, 92.1 [86.4]	76	6.40, 9.23 [7.82]	95
12	0.171, 0.193 [0.182]	142	88.8, 96.4 [92.6]	82	7.83, 8.76 [8.30]	101
18	0.145, 0.154 [0.150]	117	116, 123 [119.5]	106	7.95, 7.96 [7.96]	97
24	0.200, 0.227 [0.214]	167	109, 114 [111.5]	99	8.47, 8.56 [8.52]	104
Seed						
0	<0.02, <0.02 [<0.02]	--	0.512, 0.566 [0.539]	100	0.244, 0.261 [0.252]	100
2	<0.02, <0.02 [<0.02]	--	0.553, 0.564 [0.558]	103	0.289, 0.315 [0.302]	120
3	<0.02, <0.02 [<0.02]	--	0.590, 0.564 [0.577]	107	0.309, 0.303 [0.306]	121
6	<0.02, <0.02 [<0.02]	--	0.549, 0.540 [0.544]	101	0.243, 0.234 [0.238]	94
10	<0.02, <0.02 [<0.02]	--	0.552, 0.479 [0.516]	96	0.337, 0.339 [0.338]	134
12	<0.02, <0.02 [<0.02]	--	0.560, 0.568 [0.564]	105	0.375, 0.376 [0.376]	149
18	<0.02, <0.02 [<0.02]	--	0.604, 0.580 [0.592]	110	0.288, 0.303 [0.296]	117
24	<0.02, <0.02 [<0.02]	--	0.518, 0.536 [0.527]	98	0.306, 0.313 [0.310]	123

Cotton

Residues in undelinted cotton seed were analysed using Method AG-ME-1381-01, and procedural recoveries ranged from 74 to 133% for all analytes-matrices. Quantifiable residues were observed in 2 of 6 dicamba procedural recovery control samples, 6 of 6 DCSA samples, and 5 of 6 DCGA samples.

The values in the table do not include corrections for residues in the control samples. If corrections are made, the recoveries ranged from 71 to 122%.

Residues of 5-OH dicamba were <0.02 mg/kg in all samples, including Day 0; therefore, storage stability for that compound could not be evaluated. For dicamba, DCSA (free and conjugated) and DCGA (free and conjugated), residues from the storage stability samples are summarized in Table 11.

Table 11 Storage stability of dicamba, DCSA, and DCGA in cotton undelinted seeds

Storage Period, days	Dicamba		DCSA		DCGA	
	mg/kg [mean]	% of Day 0	mg/kg [mean]	% of Day 0	mg/kg [mean]	% of Day 0
0	1.01, 0.91, 0.78, 0.83, 0.78, 0.82 [0.85]	100	0.24, 0.24, 0.24, 0.22, 0.24, 0.23 [0.23]	100	0.13, 0.12, 0.12, 0.13, 0.11, 0.12 [0.12]	100
1	0.82, 0.76, 0.79, 0.75 [0.78]	92	0.22, 0.21, 0.23, 0.24 [0.22]	96	0.14, 0.14, 0.16, 0.15 [0.14]	117
2	0.7, 0.89, 0.9, 0.88 [0.84]	99	0.2, 0.24, 0.22, 0.25 [0.23]	100	0.12, 0.13, 0.13, 0.14 [0.13]	108
4	0.94, 0.82, 0.88, 1 [0.91]	107	0.21, 0.2, 0.19, 0.21 [0.2]	87	0.14, 0.13, 0.12, 0.12 [0.13]	108
6	0.54, 0.62, 0.7, 0.72, 0.64, 0.72 [0.66]	78	0.12, 0.18, 0.18, 0.17, 0.18, 0.18 [0.17]	74	0.13, 0.14, 0.15, 0.13, 0.12, 0.14 [0.13]	108
9	0.72, 0.7, 0.8, 0.75, 0.72, 0.71 [0.73]	86	0.2, 0.17, 0.18, 0.21, 0.18, 0.18 [0.19]	83	0.14, 0.13, 0.14, 0.15, 0.14, 0.13 [0.14]	117

In soya beans residues of dicamba and DCSA (incl. conjugates) are stable for at least 2 years in forage, hay, and seed. Residues of DCGA (incl. conjugates) showed a decline in forage during frozen storage, with stability demonstrated for only up to approximately 3 months; DCGA residues were stable in hay and seed for at least 2 years.

Residues of dicamba, DCSA, and DCGA (incl. conjugates) were stable for at least 9 months in undelinted cotton seed.

USE PATTERN

Registered labels describing the uses of dicamba on soya bean, maize, and cotton were provided to the Meeting (Table 12).

Table 12 Registered Uses of Dicamba on Dicamba-Tolerant Crops Submitted to the 2019 Extra JMPR

Crop	Country	Formulation		Application					PHI (days)
		g ai/L	Type	Timing	Method	kg ai/ha	Water L/ha	Number	
Pulses									
Soya bean	Canada	480 (formulated as diglycol-amine salt)	SL	Pre-plant or pre crop emergence	Broadcast	0.28-0.6	100-220	1 or more 0.6 kg ai/ha rate is to be used only once in a season and should be used replant, pre-emergence or in-crop early post-emergence. 7-day retreatment interval 1.18 kg ai/ha annual maximum	7 (forage) 13 (hay)
				Post crop emergence up to 8-leaf stage or 76 cm in height	Broadcast	0.28-0.6			

Crop	Country	Formulation		Application					PHI (days)
		g ai/L	Type	Timing	Method	kg ai/ha	Water L/ha	Number	
	USA	350 (formulated as diglycol-amine salt)	SL	Pre-plant, at-plant, or pre crop emergence	Broadcast	0.56 – 1.12	140	1 or more, not more than 1.12 kg ai/ha pre-emergence 7-day retreatment interval	None
				Post crop emergence up to and incl. R1 growth stage [BBCH 60]	Broadcast	0.56		Up to 2, not more than 1.12 kg ai/ha total post-emergence 7-day retreatment interval 2.24 kg ai/ha annual maximum	
Cereals									
Maize	Canada	480 (formulated as diglycol-amine salt)	SL	Pre-plant or pre crop emergence	Broadcast	0.28-0.6	100-220	1 or more The 0.6 kg ai/ha rate is to be used only once in a season and should be used pre-plant, pre-emergence or in-crop early post-emergence. 7-day retreatment interval	None
				Post crop emergence up to 8-leaf stage or 76 cm in height	Broadcast	0.28-0.6		1.18 kg ai/ha annual maximum	
Oilseeds									
Cotton	USA	350 (formulated as diglycol-amine salt)	SL	Pre-plant, at-plant, or pre crop emergence	Broadcast	0.56 – 1.12	140	1 or more, not more than 1.12 kg ai/ha pre-emergence 7-day retreatment interval	7
				Post crop emergence	Broadcast	0.56		1 or more, not more than 1.12 kg ai/ha total post-emergence 7-day retreatment interval 2.24 kg ai/ha annual maximum	

RESULTS OF SUPERVISED RESIDUE TRIALS ON CROPS

The Meeting received data from supervised residue trials conducted on dicamba-tolerant soya bean, maize, and cotton.

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. All values in the summary tables are reported in terms of dicamba equivalents. Factors for converting to dicamba equivalents are based on the molecular weights of dicamba (221.04 g/mol), 5-OH dicamba (237.04 g/mol), DCSA (207.01 g/mol), and DCGA (223.01 g/mol). The factors are 0.932 for 5-OH dicamba, 1.068 for DCSA, and 0.991 for DCGA.

When calculating average residues, values below the LOQ were assumed to be at the LOQ, and 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 double underlined, residues used for dietary risk estimation are underlined, and the highest individual values selected for estimating dietary intake are bolded.

When combining residues for estimation of maximum residue levels, values listed as <LOQ are assumed to be LOQ, and the combined residue is listed as '<' only when both residues were below their respective LOQs. When combining residues for risk assessment, residues of dicamba in soya bean seed and maize grain that were reported as <LOQ were assumed to be zero based on the results of metabolism and field trial studies. Similarly, residues of 5-OH dicamba reported as <LOQ were assumed to be 0 in all soya bean and cotton commodities.

Supervised trials for dicamba:

Category	Crop	Commodity	Table
Pulses	Soya bean	Seed (VD 0541)	13
Cereal	Maize	Grain (GC 0645)	14
Oilseeds	Cotton	Seed (SO 0691)	15
Feeds	Soya bean	Forage (AL 1265) and hay	16
	Maize	Forage (AF 0645) and stover	17
	Cotton	Gin trash	18

Pulses

Soya bean

Twenty-two residue trials were conducted on dicamba-tolerant soya beans (MON 87708 variety) in major soya bean-growing areas of the USA during the 2008 growing season (S. Moran and J. Foster, 2010, Report MSL0023061). Trials consisted of one control plot and one or more of the following treatment regimens:

Pre-emergence at 1.12 kg/ha + BBCH 14 at 0.56 kg/ha + BBCH 60 at 0.56 kg/ha,

BBCH 14 at 1.12 kg/ha + BBCH 60 at 1.12 kg/ha, or

Pre-emergence at 1.12 kg/ha + BBCH 60 at 2.24 kg/ha.

Treatments were made with dicamba formulated as either the diglycolamine salt or the monoethanolamine salt. Soya bean seed (without the pod) samples were harvested at maturity (73–98 days after the last application). Samples consisting of 1 kg of seed harvested from at least 12 separate areas of the plot were placed into frozen storage within four hours of collection and remained frozen during transportation to the analytical facility and prior to analysis.

Dicamba, 5-hydroxydicamba, DCSA, and DCGA were extracted and analysed using the method described above. Samples were stored for a maximum of 158 days prior to extraction and no more than three days passed between extraction and analysis for residues; residues were shown to be

stable for at least 72 hours. Concurrent recoveries ranged from 74 to 110% across all analytes and fortification levels (0.01 mg/kg to 5 mg/kg except DCGA at 2 mg/kg (121%) and 5 mg/kg (124%). Relative standard deviations across all fortification levels were 5 to 23% for dicamba, 4.7 to 39% for 5-hydroxydicamba, 1.8 to 13% for DCSA, and 6 to 19% for DCGA.

Table 13 Results of dicamba residue trials in dicamba-tolerant soya bean seed (variety MON 87708) in the USA (2008 growing season; Report MSL0023061)

Trial No. Location (Salt) [a]	Application			DALA	Dicamba-equivalent residues (mg/kg) [Mean]					
	Timing (interval, days)	kg ai/ha	L/ha		Dicamba	5-OH dicamba	DCSA	DCGA	For max. res.	For risk
Critical GAP USA	1 pre + up to 2 post, 7-day retreatment interval	1.12 + 0.56 + 0.56	140	Last application BBCH 60	--	--	--	--	--	--
AR Proctor, Arkansas (MEA)	Pre (--) BBCH 14 (34) BBCH 60 (8)	1.12 0.56 0.56	189 190 190	89	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.034, 0.047 [0.040]	0.011, 0.011 [0.011]	0.039, 0.052 [0.045]	0.044, 0.057 [0.051]
	Pre (--) BBCH 14 (8)	1.12 1.12	190 190	89	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.076, 0.062 [0.069]	0.015, 0.018 [0.016]	0.081, 0.067 [0.074]	0.091, 0.080 [0.086]
(DGA)	Pre (--) BBCH 14 (8)	1.12 1.12	190 190	89	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.076, 0.073 [0.075]	0.023, 0.021 [0.022]	0.081, 0.078 [0.080]	0.099, 0.094 [0.097]
GA Montezuma, Georgia (MEA)	Pre (--) BBCH 14 (33) BBCH 60 (21)	1.11 0.56 0.56	191 187 186	77	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.44, 0.44 [0.44]	0.13, 0.13 [0.13]	0.44, 0.44 [0.44]	0.56, **0.57** [0.56]
IA-1 Richland, Iowa (MEA)	Pre (--) BBCH 14 (23) BBCH 60 (22)	1.13 0.51 0.57	184 189 185	80	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.012, 0.014 [0.013]	0.012, 0.012 [0.012]	0.017, 0.019 [0.018]	0.024, 0.026 [0.025]
	Pre (--) BBCH 14 (22)	1.1 1.12	189 188	73	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.020, 0.019 [0.020]	0.024, 0.023 [0.023]	0.025, 0.024 [0.025]	0.044, 0.042 [0.043]
				80	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.019, 0.012 [0.016]	0.016, 0.016 [0.016]	0.024, 0.017 [0.021]	0.036, 0.028 [0.032]
				87	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.019, 0.020 [0.020]	0.021, 0.023 [0.022]	0.024, 0.025 [0.025]	0.041, 0.043 [0.042]
				94	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.017, 0.021 [0.019]	0.020, 0.026 [0.023]	0.022, 0.026 [0.024]	0.037, 0.046 [0.041]
IA-2 Hedrick, Iowa (MEA)	Pre (--) BBCH 14 (22) BBCH 60 (19)	1.12 0.58 0.56	187 189 183	95	<0.005, <0.005 [<0.005]	0.019, 0.019 [0.019]	0.011, 0.011 [0.011]	0.018, 0.014 [0.016]	0.016, 0.016 [0.016]	0.048, 0.044 [0.046]
IL-1 Wyoming, Illinois (MEA)	Pre (--) BBCH 14 (22) BBCH 60 (21)	1.13 0.56 0.56	190 188 184	95	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.017, 0.010 [0.013]	0.018, 0.011 [0.014]	0.022, 0.015 [0.018]	0.035, 0.021 [0.028]
IL-2 Carlyle, Illinois (MEA)	Pre (--) BBCH 14 (24) BBCH 60 (14)	1.15 0.57 0.56	190 195 185	74	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.048, 0.050 [0.049]	0.026, 0.023 [0.025]	0.053, 0.055 [0.054]	0.074, 0.073 [0.074]
	Pre (--) BBCH 14 (14)	1.11 1.11	191 182	74	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.11, 0.12 [0.11]	0.063, 0.066 [0.064]	0.12, 0.12 [0.12]	0.18, 0.18 [0.18]

Trial No. Location (Salt) [a]	Application			DALA	Dicamba-equivalent residues (mg/kg) [Mean]					
	Timing (interval, days)	kg ai/ha	L/ha		Dicamba	5-OH dicamba	DCSA	DCGA	For max. res.	For risk
(DGA)	Pre (--) BBCH 14 (14)	1.13 1.14	193 188	74	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.099, 0.11 [0.10]	0.058, 0.063 [0.060]	0.10, 0.11 [0.11]	0.16, 0.17 [0.16]
IN Rockville, Indiana (MEA)	Pre (--) BBCH 14 (21) BBCH 60 (21)	1.12 0.56 0.55	189 186 176	73	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.045, 0.041 [0.043]	0.038, 0.050 [0.044]	0.050, 0.046 [0.048]	0.082, 0.091 [0.087]
KS-1 Cunningham, Kansas (MEA)	Pre (--) BBCH 14 (28) BBCH 60 (8)	1.11 0.55 0.57	187 191 198	95	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.024, 0.020 [0.022]	0.011, 0.011 [0.011]	0.029, 0.025 [0.027]	0.034, 0.031 [0.032]
	Pre (--) BBCH 14 (8)	1.1 1.15	193 200	95	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.023, 0.045 [0.034]	0.017, 0.019 [0.018]	0.028, 0.050 [0.039]	0.040, 0.064 [0.052]
(DGA)	Pre (--) BBCH 14 (8)	1.11 1.18	193 205	95	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.049, 0.053 [0.051]	0.021, 0.020 [0.021]	0.054, 0.058 [0.056]	0.070, 0.073 [0.072]
KS-2 Hudson, Kansas (MEA)	Pre (--) BBCH 14 (32) BBCH 60 (13)	1.14 0.55 0.58	191 184 193	77	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.013, 0.016 [0.014]	0.016, 0.015 [0.016]	0.018, 0.021 [0.019]	0.028, 0.031 [0.030]
LA Washington, Louisiana (MEA)	Pre (--) BBCH 14 (15) BBCH 60 (15)	1.11 0.55 0.57	188 199 187	85	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.021, 0.017 [0.019]	0.011, 0.011 [0.011]	0.026, 0.022 [0.024]	0.031, 0.028 [0.029]
MI Conklin, Michigan (MEA)	Pre (--) BBCH 14 (35) BBCH 60 (26)	1.12 0.56 0.56	184 186 186	88	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.095, 0.090 [0.092]	0.061, 0.055 [0.058]	0.100, 0.095 [0.097]	0.16, 0.15 [0.15]
MN-1 Campbell, Minnesota (MEA)	Pre (--) BBCH 14 (25) BBCH 60 (17)	1.12 0.56 0.56	187 187 187	78	<0.005, 0.012 [0.0088]	<0.019, 0.019 [0.019]	0.054, 0.056 [0.055]	0.043, 0.045 [0.044]	0.059, 0.068 [0.063]	0.096, 0.13 [0.11]
	Pre (--) BBCH 14 (17)	1.12 1.13	188 188	78	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.082, 0.079 [0.081]	0.081, 0.079 [0.080]	0.087, 0.084 [0.086]	0.16, 0.16 [0.16]
				88	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.076, 0.074 [0.075]	0.061, 0.055 [0.058]	0.081, 0.079 [0.080]	0.14, 0.13 [0.13]
				92	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.082, 0.075 [0.079]	0.081, 0.080 [0.081]	0.087, 0.080 [0.084]	0.16, 0.16 [0.16]
				100	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.073, 0.071 [0.072]	0.072, 0.071 [0.072]	0.078, 0.076 [0.077]	0.14, 0.14 [0.14]
MN-2 Fergus Falls, Minnesota (MEA)	Pre (--) BBCH 14 (27) BBCH 60 (18)	1.12 0.56 0.56	188 188 187	78	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.071, 0.073 [0.072]	0.048, 0.053 [0.050]	0.076, 0.078 [0.077]	0.12, 0.13 [0.12]
	Pre (--) BBCH 14 (18)	1.12 1.13	187 188	78	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.16, 0.16 [0.16]	0.12, 0.12 [0.12]	0.17, 0.17 [0.17]	0.28, 0.29 [0.28]
(DGA)	Pre (--) BBCH 14 (18)	1.12 1.12	188 187	78	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.20, 0.18 [0.19]	0.14, 0.12 [0.13]	0.21, 0.18 [0.20]	0.34, 0.30 [0.32]

Trial No. Location (Salt) [a]	Application			DALA	Dicamba-equivalent residues (mg/kg) [Mean]					
	Timing (interval, days)	kg ai/ha	L/ha		Dicamba	5-OH dicamba	DCSA	DCGA	For max. res.	For risk
MO Fisk, Missouri (MEA)	Pre (--) BBCH 14 (24) BBCH 60 (17)	1.12 0.56 0.57	186 189 189	81	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.020, 0.025 [0.023]	0.014, 0.016 [0.015]	0.025, 0.030 [0.028]	0.033, 0.041 [0.037]
ND-1 Carrington, North Dakota (MEA)	Pre (--) BBCH 14 (31) BBCH 60 (29)	1.12 0.56 0.57	188 187 188	87	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.059, 0.051 [0.055]	0.048, 0.041 [0.044]	0.064, 0.056 [0.060]	0.11, 0.092 [0.099]
NE-1 York, Nebraska (MEA)	Pre (--) BBCH 14 (26) BBCH 60 (21)	1.13 0.56 0.56	188 187 187	87	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.028, 0.025 [0.027]	0.030, 0.029 [0.029]	0.033, 0.030 [0.032]	0.058, 0.054 [0.056]
NE-2 Osceola, Nebraska (MEA)	Pre (--) BBCH 14 (25) BBCH 60 (18)	1.1 0.56 0.57	182 187 188	86	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.029, 0.032 [0.031]	0.015, 0.016 [0.016]	0.034, 0.037 [0.036]	0.044, 0.049 [0.046]
SC Elko, South Carolina (MEA)	Pre (--) BBCH 14 (30) BBCH 60 (9)	1.12 0.56 0.56	189 193 186	88	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.020, 0.022 [0.021]	0.011, 0.011 [0.011]	0.025, 0.027 [0.026]	0.030, 0.032 [0.031]
SD-1 Centerville, South Dakota (MEA)	Pre (--) BBCH 14 (31) BBCH 60 (17)	1.09 0.55 0.56	180 184 185	76	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.12, 0.12 [0.12]	0.056, 0.051 [0.053]	0.13, 0.12 [0.12]	0.18, 0.17 [0.17]
SD-2 Britton, South Dakota (MEA)	Pre (--) BBCH 14 (32) BBCH 60 (10)	1.12 0.56 0.56	186 187 187	88	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.046, 0.050 [0.048]	0.011, 0.011 [0.011]	0.051, 0.055 [0.053]	0.057, 0.060 [0.058]
WI-1 Delavan, Wisconsin (MEA)	Pre (--) BBCH 14 (35) BBCH 60 (15)	1.12 0.52 0.56	183 173 188	85	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.079, 0.075 [0.077]	0.071, 0.064 [0.068]	0.084, 0.080 [0.082]	0.15, 0.14 [0.14]
WI-2 Fitchburg, Wisconsin (MEA)	Pre (--) BBCH 14 (29) BBCH 60 (6)	0.56+0 .57 0.57 0.56	195, 186 188 190	98	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.012, 0.0093 [0.011]	0.011, 0.011 [0.011]	0.017, 0.014 [0.016]	0.023, 0.020 [0.021]

[a] Formulation: MEA = Monoethanolamine salt, DGA = Diglycolamine salt

Cereal grains

Maize

Twenty-two residue trials were conducted on dicamba-tolerant maize (MON 87419 variety) in major maize-growing areas of the USA during the 2013 growing season (E. Urbanczyk-Wochniak, 2015, Report MSL0026526). Trials consisted of one control plot and one or more of the following treatment regimens:

Pre-emergence at 1.12 kg ai/ha + BBCH 14-16 at 0.56 kg ai/ha + BBCH 18 at 0.56 kg ai/ha,

BBCH 12-14 at 0.56 kg ai/ha + BBCH 14-16 at 0.56 kg ai/ha + BBCH 18 at 0.56 kg ai/ha + 122 cm [BBCH 19] at 0.56 kg ai/ha, or

Pre-emergence at 6 kg ai/ha + BBCH 14-16 at 3 kg ai/ha + BBCH 18 at 3 kg ai/ha.

For all regimens, application retreatment intervals were at a minimum of seven days. Treatments were made with dicamba formulated as the diglycolamine salt. Maize grain samples were harvested at maturity (64-132 days after the last application). Samples consisting of 1 kg of seed harvested from at least 12 separate areas of the plot were placed into frozen storage within four hours of collection and remained frozen during transportation to the analytical facility and prior to analysis.

Samples were stored for a maximum of 147 days prior to extraction and no more than two days passed between extraction and analysis for residues of dicamba, 5-hydroxydicamba, DCSA, and DCGA using the method described above. Concurrent recoveries ranged from 89 to 107% across all analytes and fortification levels (0.01 mg/kg to ca. 0.5 mg/kg). Relative standard deviations across all analytes and fortification levels were 5.1 to 18%.

Table 14 Results of dicamba residue trials in dicamba-tolerant maize grain (variety MON 87419) in the USA (2013 growing season; Report MSL0026526)

Trial No. Location (Salt) [a]	Application			DALA	Dicamba-equivalent residues (mg/kg) [Mean]					
	Timing (interval, days)	kg ai/ha	L/ha		Dicamba	5-OH dicamba	DCSA	DCGA	For max. res.	For risk
Critical GAP Canada	1 pre + 1 post up to BBCH 18	0.6+ 0.6	100	30	--	--	--	--	--	--
01PA Germansville, Pennsylvania	Pre (--) BBCH 14-15 (23) BBCH 17-18 (14)	1.14 0.57 0.57	192 190 191	113	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
	BBCH 13 BBCH 14-15 (7) BBCH 17-18 (14) BBCH 19 (8)	0.58 0.58 0.58 0.56	194 193 195 189	105	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
02GA Chula, Georgia	Pre (--) BBCH 15 (27) BBCH 18 (12)	1.11 0.54 0.57	182 185 183	86	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
	BBCH 13 BBCH 15 (14) BBCH 18 (12) BBCH 19 (10)	0.56 0.55 0.55 0.57	183 188 178 195	76	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
03NC Belvidere, North Carolina	Pre (--) BBCH 15-16 (28) BBCH 18 (14)	1.16 0.58 0.56	193 186 188	89	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
	BBCH 13 BBCH 15-16 (7) BBCH 18 (14) BBCH 19 (7)	0.56 0.56 0.56 0.57	183 181 187 188	82	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
04TX Uvalde, Texas	BBCH 0 BBCH 15-16 (47) BBCH 18 (7)	1.11 0.56 0.56	187 185 187	71	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.039, 0.031 [0.035]	0.021, 0.020 [0.020]	0.049, 0.041 [0.045]	0.070, 0.061 [0.066]
	BBCH 12 BBCH 15-16 (23) BBCH 18 (7) BBCH 51 (7)	0.57 0.56 0.56 0.57	188 188 187 189	64	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.033, 0.027 [0.030]	0.023, 0.017 [0.020]	0.043, 0.037 [0.040]	0.065, 0.054 [0.060]
05MI Wright, Michigan	Pre (--) BBCH 14 (34) BBCH 18 (13)	1.14 0.56 0.56	187 187 189	121	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
	BBCH 12 BBCH 14 (9) BBCH 18 (13) BBCH 19 (11)	0.56 0.56 0.56 0.56	186 186 190 189	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]	<0.03, <0.03 [<0.03]
06KS Troy, Kansas	BBCH 5 BBCH 13 (22) BBCH 18 (8)	1.13 0.57 0.65	190 195 221	132	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
	BBCH 12 BBCH 13 (9) BBCH 18 (8)	0.58 0.57 0.57	195 194 195	119	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]

Trial No. Location (Salt) [a]	Application			DALA	Dicamba-equivalent residues (mg/kg) [Mean]					
	Timing (interval, days)	kg ai/ha	L/ha		Dicamba	5-OH dicamba	DCSA	DCGA	For max. res.	For risk
	BBCH 39 (13)	0.57	196							
07KS St. John, Kansas	BBCH 1 BBCH 16 (32) BBCH 18 (7)	1.12 0.57 0.55	185 188 187	91	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
	BBCH 12 BBCH 16 (18) BBCH 18 (7) BBCH 19 (21)	0.57 0.56 0.55 0.58	188 185 185 191	70	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
08IA Black Hawk, Iowa	Pre (--) BBCH 15 (35) BBCH 18 (13)	1.11 0.55 0.56	186 183 186	106	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
	BBCH 13 BBCH 15 (12) BBCH 18 (13) BBCH 19 (7)	0.56 0.56 0.55 0.56	191 186 183 189	99	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
09IL Carlyle, Illinois	Pre (--) BBCH 15-16 (32) BBCH 18 (13)	1.13 0.57 0.56	196 194 193	91	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
	BBCH 14 BBCH 15-16 (9) BBCH 18 (13) BBCH 19 (7)	0.56 0.56 0.56 0.56	179 190 193 191	84	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
10IL Highland, Illinois	Pre (--) BBCH 15 (25) BBCH 18 (16)	1.16 0.57 0.56	196 182 194	100	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
	BBCH 13 BBCH 15 (7) BBCH 18 (16) BBCH 18 (9)	0.58 0.57 0.56 0.56	194 182 192 193	91	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
11IL Camp Grove, Illinois	BBCH 0 BBCH 14 (30) BBCH 18 (9)	1.13 0.56 0.56	188 185 183	100	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
	BBCH 13 BBCH 14 (7) BBCH 18 (9) BBCH 19 (15)	0.55 0.56 0.55 0.57	178 182 181 190	91	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
12IL Stewardson, Illinois	BBCH 0 BBCH 15 (28) BBCH 18 (12)	1.12 0.55 0.55	188 180 180	105	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
	BBCH 12 BBCH 15 (11) BBCH 18 (12) BBCH 19 (11)	0.56 0.56 0.56 0.56	192 184 184 187	94	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
13IL Duvall, Illinois	BBCH 3 BBCH 16 (25) BBCH 18 (16)	1.12 0.57 0.56	190 192 192	108	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
	BBCH 12 BBCH 16 (7) BBCH 18 (16) BBCH 24 (9)	0.56 0.58 0.57 0.57	190 196 196 189	96	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
14IL Highland, Illinois	BBCH 0 BBCH 15 (28) BBCH 18 (14)	1.12 0.57 0.57	195 195 195	97	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
	BBCH 12 BBCH 15 (14) BBCH 18 (14) BBCH 39 (9)	0.56 0.57 0.56 0.57	194 194 195 191	88	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
15IN Pickard, Indiana	BBCH 0 BBCH 15 (29) BBCH 18 (12)	1.16 0.56 0.56	194 188 193	119	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]

Trial No. Location (Salt) [a]	Application			DALA	Dicamba-equivalent residues (mg/kg) [Mean]					
	Timing (interval, days)	kg ai/ha	L/ha		Dicamba	5-OH dicamba	DCSA	DCGA	For max. res.	For risk
	BBCH 12 BBCH 15 (15) BBCH 18 (12) BBCH 19 (14)	0.57 0.56 0.57 0.56	193 188 195 187	105	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
16MO Kirksville, Missouri	Pre (--) BBCH 14 (26) BBCH 18 (14)	1.11 0.56 0.56	186 188 184	89	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
	BBCH 12 BBCH 14 (10) BBCH 18 (14) BBCH 19 (7)	0.56 0.56 0.56 0.57	189 187 185 192	82	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
17MO Broseley, Missouri	BBCH 0 BBCH 5 (21) BBCH 16 (9)	1.12 0.57 0.56	187 188 187	95	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
	BBCH 14 BBCH 16 (7) BBCH 18 (9) BBCH 19 (7)	0.57 0.56 0.56 0.57	188 187 187 188	88	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
18NE Tabor, Nebraska	BBCH 0 BBCH 15 (32) BBCH 18 (8)	1.11 0.56 0.57	178 179 184	118	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
	BBCH 12 BBCH 15 (18) BBCH 18 (8) BBCH 19 (7)	0.57 0.56 0.57 0.56	184 180 182 188	111	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
19NE Henderson, Nebraska	BBCH 0 BBCH 16 (34) BBCH 19 (7)	1.1 0.55 0.56	191 191 191	99	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
	BBCH 13 BBCH 16 (8) BBCH 19 (7) BBCH 19 (7)	0.55 0.56 0.57 0.56	190 194 191 194	92	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
20NE Brunswick, Nebraska	BBCH 0 BBCH 15 (30) BBCH 18 (7)	1.09 0.55 0.56	188 190 192	101	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
	BBCH 12 BBCH 15 (10) BBCH 18 (7) BBCH 19 (13)	0.56 0.56 0.56 0.56	190 191 191 194	88	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
21SD Bushnell, S. Dakota	BBCH 0 BBCH 16 (32) BBCH 18 (7)	1.12 0.58 0.56	188 193 182	117	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
	BBCH 13 BBCH 16 (12) BBCH 18 (7) BBCH 19 (7)	0.56 0.56 0.57 0.56	196 187 188 183	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]	<0.03, <0.03 [<0.03]
22WI Richmond, Wisconsin	Pre (--) BBCH 15 (33) BBCH 18 (14)	1.11 0.56 0.56	187 184 181	120	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
	BBCH 13 BBCH 15 (10) BBCH 18 (14) BBCH 19 (7)	0.56 0.56 0.56 0.56	189 185 181 187	113	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]

[a] Formulation: MEA = Monoethanolamine salt, DGA = Diglycolamine salt

Oilseeds

Cotton

Thirteen residue trials were conducted in major cotton growing areas of the USA during the 2010 growing season (D. Maher and J. Foster, 2010, Report MSL0024072). Trials consisted of one control plot and one or more of the following treatment regimens:

Pre-emergence at 1.12 kg/ha + BBCH 16 at 0.56 kg/ha + (BBCH 60 + 15 days) at 0.56 kg/ha,

Pre-emergence at 1.12 kg/ha + BBCH 80 at 0.56 kg/ha + BBCH 99 at 0.56 kg/ha (7 days preharvest), or

BBCH 16 at 0.56 kg/ha + (BBCH 60 + 15 days) at 0.56 kg/ha + BBCH 80 at 0.56 kg/ha + BBCH 99 at 0.56 kg/ha (7 days preharvest).

Treatments were made with dicamba formulated as either the diglycolamine salt (Treatments 1-3 above) or the monoethanolamine salt (Treatment 3 only). Cotton seed (undelinted) samples were harvested at maturity. Samples consisting of 1 kg of seed were placed into frozen storage within four hours of collection and remained frozen during transportation to the analytical facility and prior to analysis.

Samples were stored for a maximum of 169 days prior to extraction and no more than two days passed between extraction and analysis for residues of dicamba, 5-hydroxydicamba, DCSA, and DCGA using the method described above. Concurrent recoveries ranged from 85 to 108% across all analytes and fortification levels (0.02 mg/kg to 5 mg/kg (0.2 mg/kg 5-hydroxydicamba)). Relative standard deviations across all analytes and fortification levels were 4.3 to 17%.

Table 15 Results of dicamba residue trials in dicamba-tolerant cotton seed (variety MON 88701) in the USA (2010 growing season; Report MSL0024072)

Trial No. Location (Salt) [a]	Application			DALA	Residues (mg/kg) [Mean]					
	Timing (interval, days)	kg ai/ha	L/ha		Dicamba	5-OH dicamba	DCSA	DCGA	For max. res.	For risk
Critical GAP USA	1 pre + 2 post with a 7-day retreatment interval	1.12 + 0.56 + 0.56	140	7	--	--	--	--	--	--
AR1 Proctor, Arkansas (DGA)	ns (--) ns (29) Mid-bloom (38)	1.1 0.56 0.56	188 187 188	70	<0.02, <0.02 [<0.02]	<0.0093, <0.0093 [<0.0093]	0.053, 0.043 [0.048]	0.030, 0.030 [0.030]	0.073, 0.063 [0.068]	0.10, 0.092 [0.098]
	ns (--) ns (99) 95% Open bolls (31)	1.1 0.56 0.56	188 188 188	7	1.2, 0.59 [0.90]	<0.0093, <0.0093 [<0.0093]	0.11, 0.053 [0.080]	0.040, 0.030 [0.035]	1.3, 0.64 [0.98]	1.3, 0.67 [1.0]
	ns (--) ns (38) ns (32) 95% Open bolls (31)	0.56 0.56 0.56 0.56	188 189 188 188	7	0.33, 0.58 [0.45]	<0.0093, <0.0093 [<0.0093]	0.053, 0.075 [0.064]	0.040, 0.050 [0.045]	0.38, 0.65 [0.52]	0.42, 0.70 [0.56]
(MEA)	ns (--) ns (38) ns (32) 95% Open bolls (31)	0.56 0.56 0.56 0.56	187 189 189 188	7	0.72, 0.51 [0.62]	<0.0093, <0.0093 [<0.0093]	0.18, 0.064 [0.12]	0.079, 0.040 [0.059]	0.90, 0.57 [0.74]	0.98, 0.61 [0.80]
CA1 Porterville, California (DGA)	ns (--) ns (48) BBCH 65 (43)	1.1 0.56 0.57	192 190 188	81	<0.02, <0.02 [<0.02]	<0.0093, <0.0093 [<0.0093]	<0.0053, <0.0053 [<0.0053]	<0.0053, <0.0053 [<0.0053]	0.025, 0.025 [0.025]	0.030, 0.030 [0.030]
	ns (--) ns (136) BBCH 97 (28)	1.1 0.57 0.57	190 189 191	8	0.18, 0.19 [0.18]	<0.0093, <0.0093 [<0.0093]	<0.0053, 0.021 [0.013]	<0.0053, <0.0053 [<0.0053]	0.19, 0.21 [0.20]	0.19, 0.22 [0.20]

Trial No. Location (Salt) [a]	Application			DALA	Residues (mg/kg) [Mean]					
	Timing (interval, days)	kg ai/ha	L/ha		Dicamba	5-OH dicamba	DCSA	DCGA	For max. res.	For risk
	ns (--) ns (43) ns (45) BBCH 97 (28)	0.56 0.58 0.57 0.57	189 190 189 189	8	0.47, 0.39 [0.43]	<0.0093, <0.0093 [<0.0093]	0.096, 0.043 [0.069]	0.040, 0.020 [0.030]	0.57, 0.43 [0.50]	0.61, 0.45 [0.53]
(MEA)	ns (--) ns (43) ns (45) BBCH 97 (28)	0.56 0.58 0.56 0.56	189 189 188 187	8	0.23, 0.17 [0.20]	<0.0093, <0.0093 [<0.0093]	0.064, 0.021 [0.043]	0.020, 0.020 [0.020]	0.29, 0.19 [0.24]	0.31, 0.21 [0.26]
CA2 Visalia, California (DGA)	ns (--) ns (41) BBCH 65 (49)	1.1 0.56 0.57	192 188 194	103	<0.02, <0.02 [<0.02]	<0.0093, <0.0093 [<0.0093]	<0.0053, <0.0053 [<0.0053]	<0.0053, <0.0053 [<0.0053]	0.025, 0.025 [0.025]	0.030, 0.030 [0.030]
	ns (--) ns (178) BBCH 85 (7)	1.1 0.57 0.56	191 190 192	8	0.66, 0.64 [0.65]	<0.0093, <0.0093 [<0.0093]	0.021, 0.053 [0.037]	<0.0053, <0.0053 [<0.0053]	0.68, 0.69 [0.69]	0.69, 0.70 [0.69]
	ns (--) ns (49) ns (88) BBCH 85 (7)	0.57 0.56 0.57 0.57	193 190 191 195	8	1.0, 0.82 [0.94]	<0.0093, <0.0093 [<0.0093]	0.15, 0.17 [0.16]	0.030, 0.030 [0.030]	1.2, 0.99 [1.1]	1.2, 1.0 [1.1]
GA1 Chula, Georgia (DGA)	ns (--) ns (26) 15 Days after white flower (43)	1.1 0.56 0.57	192 192 189	84	<0.02, <0.02 [<0.02]	<0.0093, <0.0093 [<0.0093]	<0.0053, <0.0053 [<0.0053]	<0.0053, <0.0053 [<0.0053]	0.025, 0.025 [0.025]	0.030, 0.030 [0.030]
	ns (--) ns (104) 90% Open bolls (42)	1.1 0.58 0.56	193 191 191	7	0.76, 1.3 [1.0]	<0.0093, <0.0093 [<0.0093]	0.021, 0.021 [0.021]	<0.0053, <0.0053 [<0.0053]	0.78, 1.3 [1.1]	0.79, 1.3 [1.1]
	ns (--) ns (43) ns (35) 90% Open bolls (42)	0.57 0.56 0.57 0.56	195 187 185 191	7	0.80, 0.71 [0.76]	<0.0093, <0.0093 [<0.0093]	0.032, 0.021 [0.027]	0.030, 0.020 [0.025]	0.83, 0.73 [0.78]	0.86, 0.75 [0.81]
(MEA)	ns (--) ns (43) ns (35) 90% Open bolls (42)	0.57 0.56 0.56 0.56	195 187 182 189	7	0.68, 0.50 [0.59]	<0.0093, <0.0093 [<0.0093]	0.021, 0.021 [0.021]	0.020, 0.020 [0.020]	0.70, 0.52 [0.61]	0.72, 0.54 [0.63]
LA1 Cheneyville, Louisiana (DGA)	ns (--) ns (27) Mid-bloom (36)	1.1 0.57 0.57	192 178 180	73	<0.02, <0.02 [<0.02]	<0.0093, <0.0093 [<0.0093]	<0.0053, <0.0053 [<0.0053]	<0.0053, <0.0053 [<0.0053]	0.025, 0.025 [0.025]	0.030, 0.030 [0.030]
	ns (--) ns (97) BBCH 88 (33)	1.1 0.56 0.58	195 188 202	7	0.33, 0.45 [0.39]	<0.0093, <0.0093 [<0.0093]	<0.0053, <0.0053 [<0.0053]	0.020, 0.020 [0.020]	0.34, 0.46 [0.40]	0.36, 0.48 [0.42]
	ns (--) ns (36) ns (34) BBCH 88 (33)	0.58 0.57 0.57 0.57	180 181 192 198	7	0.82, 0.65 [0.74]	<0.0093, <0.0093 [<0.0093]	0.032, 0.064 [0.048]	0.030, 0.030 [0.030]	0.85, 0.71 [0.78]	0.88, 0.74 [0.81]
MO1 Fisk, Missouri (DGA)	ns (--) ns (29) BBCH 65 (42)	1.1 0.56 0.56	187 187 187	79	<0.02, <0.02 [<0.02]	<0.0093, <0.0093 [<0.0093]	0.021, 0.021 [0.021]	0.020, 0.020 [0.020]	0.041, 0.041 [0.041]	0.061, 0.061 [0.061]
	ns (--) ns (113) BBCH 89 (30)	1.1 0.56 0.56	187 187 187	7	0.97, 1.0 [0.98]	<0.0093, <0.0093 [<0.0093]	<0.0053, 0.064 [0.035]	0.020, <0.0053 [0.012]	0.98, 1.1 [1.0]	1.0, 1.1 [1.0]
	ns (--) ns (42) ns (42) BBCH 89 (30)	0.57 0.56 0.56 0.56	190 188 187 187	7	0.56, 1.2 [0.86]	<0.0093, <0.0093 [<0.0093]	0.021, 0.032 [0.027]	0.020, 0.020 [0.020]	0.58, 1.2 [0.89]	0.60, 1.2 [0.91]

Trial No. Location (Salt) [a]	Application			DALA	Residues (mg/kg) [Mean]					
	Timing (interval, days)	kg ai/ha	L/ha		Dicamba	5-OH dicamba	DCSA	DCGA	For max. res.	For risk
OK1 Hinton, Oklahoma (DGA)	ns (--) ns (24) BBCH 65 (49)	1.1 0.57 0.56	176 190 188	68	<0.02, <0.02 [<0.02]	<0.0093, <0.0093 [<0.0093]	0.032, 0.032 [0.032]	0.030, 0.030 [0.030]	0.052, 0.052 [0.052]	0.082, 0.082 [0.082]
	ns (--) ns (107) BBCH 89 (27)	1.1 0.56 0.57	178 185 193	8	0.33, 0.23 [0.28]	<0.0093, <0.0093 [<0.0093]	0.085, 0.032 [0.059]	0.050, 0.030 [0.040]	0.42, 0.26 [0.34]	0.46, 0.29 [0.38]
	ns (--) ns (49) ns (34) BBCH 89 (27)	0.57 0.55 0.57 0.57	190 186 189 193	8	0.35, 0.18 [0.26]	<0.0093, <0.0093 [<0.0093]	0.12, 0.075 [0.096]	0.089, 0.050 [0.069]	0.47, 0.25 [0.36]	0.56, 0.30 [0.43]
(MEA)	ns (--) ns (49) ns (34) BBCH 89 (27)	0.56 0.55 0.56 0.56	185 185 184 190	8	0.23, 0.23 [0.23]	<0.0093, <0.0093 [<0.0093]	0.021, 0.075 [0.048]	0.030, 0.040 [0.035]	0.25, 0.30 [0.28]	0.28, 0.34 [0.31]
OK2 Dill City, Oklahoma (DGA)	ns (--) ns (28) BBCH 65 (49)	1.1 0.56 0.55	178 188 184	82	<0.02, <0.02 [<0.02]	<0.0093, <0.0093 [<0.0093]	0.032, <0.0053 [0.019]	0.030, 0.020 [0.025]	0.052, 0.025 [0.039]	0.082, 0.045 [0.063]
	ns (--) ns (109) BBCH 97 (43)	1.1 0.56 0.56	180 190 188	7	0.070, 0.060 [0.065]	<0.0093, <0.0093 [<0.0093]	<0.0053, <0.0053 [<0.0053]	<0.0053, <0.0053 [<0.0053]	0.075, 0.065 [0.070]	0.080, 0.070 [0.075]
	ns (--) ns (49) BBCH 97 (32) ns (43)	0.57 0.56 0.56 0.55	189 188 190 184	7	0.12, 0.16 [0.14]	<0.0093, <0.0093 [<0.0093]	0.032, 0.032 [0.032]	0.020, 0.030 [0.025]	0.15, 0.19 [0.17]	0.17, 0.22 [0.20]
SC1 Elko, South Carolina (DGA)	ns (--) ns (29) BBCH 65 (38)	1.1 0.56 0.56	190 195 193	99	<0.02, <0.02 [<0.02]	<0.0093, <0.0093 [<0.0093]	<0.0053, <0.0053 [<0.0053]	<0.0053, <0.0053 [<0.0053]	0.025, 0.025 [0.025]	0.030, 0.030 [0.030]
				105	<0.02, <0.02 [<0.02]	<0.0093, <0.0093 [<0.0093]	<0.0053, <0.0053 [<0.0053]	<0.0053, <0.0053 [<0.0053]	0.025, 0.025 [0.025]	0.030, 0.030 [0.030]
				112	<0.02, <0.02 [<0.02]	<0.0093, <0.0093 [<0.0093]	<0.0053, <0.0053 [<0.0053]	<0.0053, <0.0053 [<0.0053]	0.025, 0.025 [0.025]	0.030, 0.030 [0.030]
				119	<0.02, <0.02 [<0.02]	<0.0093, <0.0093 [<0.0093]	<0.0053, <0.0053 [<0.0053]	<0.0053, <0.0053 [<0.0053]	0.025, 0.025 [0.025]	0.030, 0.030 [0.030]
	ns (--) ns (102) BBCH 95 (63)	1.1 0.56 0.56	189 195 187	7	0.18, 0.060 [0.12]	<0.0093, <0.0093 [<0.0093]	<0.0053, 0.021 [0.013]	<0.0053, 0.020 [0.012]	0.19, 0.081 [0.13]	0.19, 0.10 [0.15]
	ns (--) ns (38) ns (35) BBCH 95 (63)	0.56 0.56 0.56 0.56	194 193 193 186	1	0.21, 0.31 [0.26]	<0.0093, <0.0093 [<0.0093]	0.021, 0.032 [0.027]	0.020, 0.030 [0.025]	0.23, 0.34 [0.29]	0.25, 0.37 [0.31]
				7	0.14, 0.090 [0.12]	<0.0093, <0.0093 [<0.0093]	0.053, 0.021 [0.037]	0.059, 0.020 [0.040]	0.19, 0.11 [0.15]	0.25, 0.13 [0.19]
				14	0.090, 0.070 [0.080]	<0.0093, <0.0093 [<0.0093]	0.064, 0.032 [0.048]	0.099, 0.050 [0.074]	0.15, 0.10 [0.13]	0.25, 0.15 [0.20]

Trial No. Location (Salt) [a]	Application			DALA	Residues (mg/kg) [Mean]					
	Timing (interval, days)	kg ai/ha	L/ha		Dicamba	5-OH dicamba	DCSA	DCGA	For max. res.	For risk
				21	0.040, 0.080 [0.060]	<0.0093, <0.0093 [<0.0093]	0.096, 0.032 [0.064]	0.11, 0.040 [0.074]	0.14, 0.11 [0.12]	0.25, 0.15 [0.20]
TX1 Raymondville, Texas (DGA)	ns (--) ns (36) BBCH 65-67 (46)	1.1 0.58 0.58	190 194 195	43	<0.02, <0.02 [<0.02]	<0.0093, <0.0093 [<0.0093]	0.032, <0.0053 [0.019]	0.020, 0.020 [0.020]	0.052, 0.025 [0.039]	0.072, 0.045 [0.059]
				49	<0.02, <0.02 [<0.02]	<0.0093, <0.0093 [<0.0093]	0.021, <0.0053 [0.013]	<0.0053, 0.020 [0.012]	0.041, 0.025 [0.033]	0.046, 0.045 [0.046]
				56	<0.02, <0.02 [<0.02]	<0.0093, <0.0093 [<0.0093]	<0.0053, 0.032 [0.019]	<0.0053, 0.020 [0.012]	0.025, 0.052 [0.039]	0.030, 0.072 [0.051]
				63	<0.02, <0.02 [<0.02]	<0.0093, <0.0093 [<0.0093]	0.021, <0.0053 [0.013]	0.020, <0.0053 [0.012]	0.041, 0.025 [0.033]	0.061, 0.030 [0.046]
	ns (--) ns (119) BBCH 87 (5)	1.1 0.58 0.58	191 194 193	7	0.58, 0.19 [0.38]	<0.0093, <0.0093 [<0.0093]	0.075, <0.0053 [0.040]	<0.0053, <0.0053 [<0.0053]	0.65, 0.20 [0.43]	0.66, 0.20 [0.43]
	ns (--) ns (46) ns (37) BBCH 87 (5)	0.58 0.58 0.58 0.58	194 195 194 195	1	0.28, 0.23 [0.26]	<0.0093, <0.0093 [<0.0093]	0.032, 0.032 [0.032]	0.030, 0.030 [0.030]	0.31, 0.26 [0.29]	0.34, 0.29 [0.32]
				7	0.19, 0.17 [0.18]	<0.0093, <0.0093 [<0.0093]	0.096, 0.043 [0.069]	0.030, 0.020 [0.025]	0.29, 0.21 [0.25]	0.32, 0.23 [0.27]
				14	0.070, 0.040 [0.055]	<0.0093, <0.0093 [<0.0093]	0.043, 0.032 [0.037]	0.030, 0.020 [0.025]	0.11, 0.072 [0.092]	0.14, 0.092 [0.12]
				21	0.030, 0.040 [0.035]	<0.0093, <0.0093 [<0.0093]	0.032, 0.032 [0.032]	0.030, 0.030 [0.030]	0.062, 0.072 [0.067]	0.092, 0.10 [0.097]
TX2 Levelland, Texas (DGA)	ns (--) ns (36) BBCH 65 (41)	1.1 0.57 0.56	191 193 188	84	<0.02, <0.02 [<0.02]	<0.0093, <0.0093 [<0.0093]	0.25, 0.25 [0.25]	0.11, 0.11 [0.11]	0.27, 0.27 [0.27]	0.37, 0.37 [0.37]
	ns (--) ns (118) BBCH 89 (37)	1.1 0.55 0.55	190 184 185	6	0.84, 1.3 [1.1]	<0.0093, <0.0093 [<0.0093]	0.12, 0.21 [0.17]	0.069, 0.050 [0.059]	0.96, 1.5 [1.2]	1.0, 1.6 [1.3]
	ns (--) ns (41) ns (41) BBCH 89 (37)	0.55 0.55 0.56 0.55	187 187 191 187	6	1.3, 1.5 [1.4]	<0.0093, <0.0093 [<0.0093]	0.28, 0.29 [0.28]	0.11, 0.17 [0.14]	1.6, 1.8 [1.7]	1.7, 2.0 [1.8]
TX3 Wolfforth, Texas (DGA)	ns (--) ns (41) BBCH 65 (36)	1.1 0.57 0.55	188 194 185	84	<0.02, <0.02 [<0.02]	<0.0093, <0.0093 [<0.0093]	0.11, 0.043 [0.075]	0.069, 0.040 [0.055]	0.13, 0.063 [0.095]	0.20, 0.10 [0.15]
	ns (--) ns (125) BBCH 89 (30)	1.1 0.55 0.55	189 187 184	6	2.0, 0.80 [1.4]	<0.0093, <0.0093 [<0.0093]	0.17, 0.17 [0.17]	0.069, 0.099 [0.084]	2.1, 0.97 [1.6]	2.2, 1.1 [1.6]
	ns (--) ns (36) ns (48) BBCH 89 (30)	0.56 0.56 0.56 0.56	189 188 189 188	6	1.3, 1.1 [1.2]	<0.0093, <0.0093 [<0.0093]	0.12, 0.17 [0.14]	0.079, 0.12 [0.099]	1.4, 1.3 [1.3]	1.5, 1.4 [1.4]

Trial No. Location (Salt) [a]	Application			DALA	Residues (mg/kg) [Mean]					
	Timing (interval, days)	kg ai/ha	L/ha		Dicamba	5-OH dicamba	DCSA	DCGA	For max. res.	For risk
TX4 Uvalde, Texas (DGA)	ns (--) ns (49) BBCH 65 (47)	1.1 0.56 0.56	189 188 186	71	<0.02, <0.02 [<0.02]	<0.0093, <0.0093 [<0.0093]	<0.0053, <0.0053 [<0.0053]	<0.0053, <0.0053 [<0.0053]	0.025, 0.025 [0.025]	0.030, 0.030 [0.030]
	ns (--) ns (132) BBCH 89 (29)	1.1 0.58 0.55	186 201 184	6	0.86, 1.2 [1.0]	<0.0093, 0.019 [0.014]	0.032, 0.043 [0.037]	0.020, 0.030 [0.025]	0.89, 1.3 [1.1]	0.91, 1.3 [1.1]
	ns (--) ns (47) ns (36) BBCH 89 (29)	0.56 0.55 0.55 0.57	187 184 192 189	6	0.49, 0.44 [0.46]	<0.0093, <0.0093 [<0.0093]	0.032, 0.043 [0.037]	0.030, 0.030 [0.030]	0.52, 0.48 [0.50]	0.55, 0.51 [0.53]

[a] Formulation: MEA = Monoethanolamine salt, DGA = Diglycolamine salt

Legume animal feeds

Soya bean

Twenty-two residue trials were conducted on dicamba-tolerant soya beans (MON 87708 variety) as described above (Pulses, soya bean; Report MSL0023061). From those trials, samples of soya bean forage and hay were harvested 7–10 and 14–24 DALA, respectively. Samples of hay were dried in the field to a moisture content of 10–20%.

Soya bean forage samples were stored frozen for 119 to 292 days, and hay samples for up to 283 days. Residues of dicamba, 5-hydroxydicamba, DCSA, and DCGA were analysed using the method described above. Average concurrent recoveries from forage across all four analytes and across fortifications from 0.01 to 150 mg/kg ranged from 84 to 125%. Relative standard deviations ranged from 8.1 to 18%. Storage stability data indicate that residues of DCGA in forage are not stable for the storage period experienced by the samples in the study.

Results for soya bean forage and hay are shown in Table 16.

Table 16 Results of dicamba residue trials in dicamba-tolerant soya bean forage and hay (variety MON 87708) in the USA (2008 growing season; Report MSL0023061)

Trial No. Location Year (Salt) [a]	Application			Matrix	DALA	Dicamba-equivalent residues (mg/kg) [Mean]					
	Timing (interval, days)	kg ai/ha	L/ha			Dicamba	5-OH dicamba	DCSA	DCGA	For max. res.	For risk
Critical GAP USA	1 pre + 2 post with a 7-day retreatment interval	1.12 + 0.56 + 0.56	140	--	7 forage 14 hay	--	--	--	--	--	--
AR Proctor, Arkansas (MEA)	Pre (--) BBCH 14 (34) BBCH 60 (8)	1.12 0.56 0.56	189 190 190	Forage	7	0.051, 0.058 [0.054]	0.0067, 0.0086 [0.0076]	22, 19 [20]	2.0, 1.8 [1.9]	22, 19 [20]	24, 20 [22]
				Hay	15	0.049, 0.052 [0.050]	0.0093, 0.011 [0.0100]	30, 40 [35]	1.9, 2.5 [2.2]	30, 40 [35]	32, 42 [37]
	Pre (--) BBCH 14 (8)	1.12 1.12	190 190	Forage	7	0.17, 0.12 [0.15]	0.018, 0.016 [0.017]	43, 39 [41]	2.2, 2.9 [2.6]	44, 39 [41]	46, 42 [44]
				Hay	15	0.15, 0.11 [0.13]	0.027, 0.031 [0.029]	90, 76 [83]	5.3, 4.3 [4.8]	90, 76 [83]	95, 81 [88]

Trial No. Location Year (Salt) [a]	Application			Matrix	DALA	Dicamba-equivalent residues (mg/kg) [Mean]					
	Timing (interval, days)	kg ai/ha	L/ha			Dicamba	5-OH dicamba	DCSA	DCGA	For max. res.	For risk
(DGA)	Pre (--) BBCH 14 (8)	1.12 1.12	190 190	Forage	7	0.16, 0.13 [0.15]	0.021, 0.017 [0.019]	42, 39 [41]	3.0, 2.8 [2.9]	42, 40 [41]	45, 42 [44]
				Hay	15	0.14, 0.17 [0.15]	0.023, 0.032 [0.028]	76, 99 [88]	4.1, 5.4 [4.7]	76, 99 [88]	80, 100 [93]
GA Montezuma, Georgia (MEA)	Pre (--) BBCH 14 (33) BBCH 60 (21)	1.11 0.56 0.56	191 187 186	Forage	8	0.021, 0.021 [0.021]	0.0081, 0.0056 [0.0068]	51, 49 [50]	4.6, 5.9 [5.3]	51, 49 [50]	56, 55 [56]
				Hay	24	0.014, 0.014 [0.014]	<0.005, <0.005 [<0.0047]	12, 13 [13]	0.43, 0.37 [0.40]	12, 13 [13]	13, 14 [13]
IA-1 Richland, Iowa (MEA)	Pre (--) BBCH 14 (23) BBCH 60 (22)	1.13 0.51 0.57	184 189 185	Forage	7	0.021, 0.021 [0.021]	0.0047, 0.0047 [0.0047]	14, 14 [14]	1.5, 1.9 [1.7]	14, 14 [14]	16, 16 [16]
				Hay	17	0.014, 0.014 [0.014]	<0.005, <0.005 [<0.0047]	36, 32 [34]	3.7, 3.6 [3.7]	36, 32 [34]	40, 36 [38]
	Pre (--) BBCH 14 (22)	1.1 1.12	189 188	Forage	3	0.091, 0.073 [0.082]	0.0074, 0.0065 [0.0069]	49, 46 [47]	5.9, 4.9 [5.4]	49, 46 [47]	55, 51 [53]
					7	0.014, 0.015 [0.014]	0.0049, <0.005 [0.0048]	28, 28 [28]	2.8, 2.8 [2.8]	28, 28 [28]	31, 31 [31]
					10	0.020, 0.010 [0.015]	<0.005, <0.005 [<0.0047]	24, 28 [26]	2.6, 2.6 [2.6]	24, 28 [26]	26, 30 [28]
					14	<0.005, <0.005 [<0.005]	<0.005, <0.005 [<0.0047]	14, 15 [15]	1.9, 1.9 [1.9]	14, 15 [15]	16, 17 [16]
				Hay	17	0.017, 0.021 [0.019]	0.0055, 0.0056 [0.0055]	51, 53 [52]	4.4, 4.2 [4.3]	51, 54 [52]	55, 58 [56]
IA-2 Hedrick, Iowa (MEA)	Pre (--) BBCH 14 (22) BBCH 60 (19)	1.12 0.58 0.56	187 189 183	Forage	8	0.025, 0.030 [0.027]	<0.005, <0.005 [<0.0047]	10, 10 [10]	1.6, 1.7 [1.7]	10, 10 [10]	12, 12 [12]
				Hay	18	0.026, 0.021 [0.023]	0.011, 0.010 [0.011]	23, 24 [24]	6.2, 5.8 [6.0]	24, 24 [24]	30, 30 [30]
IL-1 Wyoming, Illinois (MEA)	Pre (--) BBCH 14 (22) BBCH 60 (21)	1.13 0.56 0.56	190 188 184	Forage	7	0.021, 0.029 [0.025]	<0.005, <0.005 [<0.0047]	14, 13 [14]	1.8, 1.8 [1.8]	14, 13 [14]	16, 15 [15]
				Hay	21	0.027, 0.032 [0.030]	<0.005, <0.005 [<0.0047]	21, 22 [21]	0.42, 0.76 [0.59]	21, 22 [21]	21, 23 [22]

Trial No. Location Year (Salt) [a]	Application			Matrix	DALA	Dicamba-equivalent residues (mg/kg) [Mean]					
	Timing (interval, days)	kg ai/ha	L/ha			Dicamba	5-OH dicamba	DCSA	DCGA	For max. res.	For risk
IL-2 Carlyle, Illinois (MEA)	Pre (--) BBCH 14 (24) BBCH 60 (14)	1.15 0.57 0.56	190 195 185	Forage	8	0.021, 0.021 [0.021]	<0.005, <0.005 [<0.0047]	13, 14 [14]	2.0, 2.2 [2.1]	13, 14 [14]	15, 16 [16]
				Hay	18	0.014, 0.014 [0.014]	<0.005, <0.005 [<0.0047]	14, 16 [15]	1.3, 1.4 [1.4]	14, 16 [15]	15, 17 [16]
	Pre (--) BBCH 14 (14)	1.11 1.11	191 182	Forage	8	0.030, 0.032 [0.031]	0.0063, 0.0061 [0.0062]	33, 33 [33]	1.3, 1.4 [1.3]	33, 33 [33]	34, 35 [34]
				Hay	18	0.018, 0.016 [0.017]	<0.005, <0.005 [<0.0047]	37, 36 [36]	2.0, 2.3 [2.1]	37, 36 [36]	38, 39 [39]
(DGA)	Pre (--) BBCH 14 (14)	1.13 1.14	193 188	Forage	8	0.027, 0.021 [0.024]	0.0047, 0.0053 [0.0050]	20, 26 [23]	0.89, 0.97 [0.93]	20, 26 [23]	21, 27 [24]
				Hay	18	<0.010, 0.014 [0.012]	<0.005, 0.0052 [0.0049]	25, 34 [29]	4.4, 2.2 [3.3]	25, 34 [29]	29, 36 [33]
IN Rockville, Indiana (MEA)	Pre (--) BBCH 14 (21) BBCH 60 (21)	1.12 0.56 0.55	189 186 176	Forage	7	1.1, 1.3 [1.2]	0.0082, 0.0095 [0.0089]	17, 15 [16]	2.2, 2.2 [2.2]	18, 16 [17]	20, 18 [19]
				Hay	15	0.28, 0.30 [0.29]	0.0070, 0.0076 [0.0073]	32, 27 [30]	6.4, 4.6 [5.5]	33, 27 [30]	39, 32 [35]
KS-1 Cunningham, Kansas (MEA)	Pre (--) BBCH 14 (28) BBCH 60 (8)	1.11 0.55 0.57	187 191 198	Forage	8	0.021, 0.021 [0.021]	<0.005, 0.0048 [0.0048]	19, 20 [19]	1.2, 1.1 [1.1]	19, 20 [19]	20, 21 [20]
				Hay	21	0.021, 0.017 [0.019]	<0.005, 0.0053 [0.0050]	48, 45 [47]	1.1, 1.5 [1.3]	48, 45 [47]	50, 46 [48]
	Pre (--) BBCH 14 (8)	1.1 1.15	193 200	Forage	8	0.024, 0.020 [0.022]	<0.005, 0.0062 [0.0055]	53, 58 [55]	1.1, 0.90 [1.0]	53, 58 [55]	54, 59 [56]
				Hay	21	0.040, 0.038 [0.039]	0.0093, 0.0070 [0.0082]	140, 120 [130]	2.8, 3.1 [2.9]	140, 120 [130]	140, 120 [130]
(DGA)	Pre (--) BBCH 14 (8)	1.11 1.18	193 205	Forage	8	0.019, 0.016 [0.018]	<0.005, <0.005 [<0.0047]	53, 46 [49]	0.96, 1.0 [0.98]	53, 46 [49]	54, 47 [50]
				Hay	21	0.042, 0.037 [0.040]	0.010, 0.0099 [0.0100]	140, 140 [140]	3.6, 3.7 [3.6]	140, 140 [140]	150, 140 [140]
KS-2 Hudson, Kansas (MEA)	Pre (--) BBCH 14 (32) BBCH 60 (13)	1.14 0.55 0.58	191 184 193	Forage	7	0.021, 0.021 [0.021]	<0.005, <0.005 [<0.0047]	21, 21 [21]	2.2, 2.2 [2.2]	21, 21 [21]	23, 23 [23]

Trial No. Location Year (Salt) [a]	Application			Matrix	DALA	Dicamba-equivalent residues (mg/kg) [Mean]					
	Timing (interval, days)	kg ai/ha	L/ha			Dicamba	5-OH dicamba	DCSA	DCGA	For max. res.	For risk
				Hay	18	0.021, 0.027 [0.024]	0.0082, 0.0081 [0.0082]	32, 37 [34]	1.4, 1.3 [1.4]	32, 37 [34]	33, 38 [36]
LA Washington, Louisiana (MEA)	Pre (--) BBCH 14 (15) BBCH 60 (15)	1.11 0.55 0.57	188 199 187	Forage	7	0.095, 0.097 [0.096]	<0.005, 0.0059 [0.0053]	19, 20 [19]	2.2, 2.1 [2.2]	19, 20 [19]	21, 22 [22]
				Hay	20	0.092, 0.11 [0.100]	<0.005, 0.0050 [0.0048]	46, 47 [47]	2.0, 1.9 [2.0]	46, 48 [47]	48, 49 [49]
MI Conklin, Michigan (MEA)	Pre (--) BBCH 14 (35) BBCH 60 (26)	1.12 0.56 0.56	184 186 186	Forage	7	0.92, 1.1 [1.0]	0.0059, 0.0062 [0.0061]	10, 8.9 [9.5]	1.3, 1.4 [1.3]	11, 10.0 [11]	12, 11 [12]
				Hay	14	1.2, 0.86 [1.0]	0.013, 0.0090 [0.011]	22, 18 [20]	3.5, 2.4 [2.9]	23, 19 [21]	26, 21 [24]
MN-1 Campbell, Minnesota (MEA)	Pre (--) BBCH 14 (25) BBCH 60 (17)	1.12 0.56 0.56	187 187 187	Forage	7	0.28, 0.16 [0.22]	<0.005, 0.0047 [0.0047]	14, 14 [14]	1.9, 2.0 [1.9]	14, 14 [14]	16, 16 [16]
				Hay	17	0.021, 0.014 [0.017]	<0.005, <0.005 [<0.0047]	34, 30 [32]	2.3, 2.0 [2.1]	34, 30 [32]	37, 32 [34]
	Pre (--) BBCH 14 (17)	1.12 1.13	188 188	Forage	3	1.1, 0.76 [0.92]	0.0062, 0.0082 [0.0072]	37, 32 [34]	2.9, 2.9 [2.9]	38, 33 [35]	41, 36 [38]
					7	0.45, 0.37 [0.41]	0.0062, <0.005 [0.0055]	26, 24 [25]	3.4, 2.8 [3.1]	27, 25 [26]	30, 27 [29]
					10	0.045, 0.046 [0.046]	<0.005, <0.005 [<0.0047]	22, 22 [22]	2.0, 1.6 [1.8]	22, 22 [22]	24, 24 [24]
					14	0.011, 0.016 [0.014]	<0.005, <0.005 [<0.0047]	16, 20 [18]	1.5, 1.8 [1.6]	16, 20 [18]	18, 21 [20]
				Hay	17	0.036, 0.049 [0.042]	<0.005, 0.0052 [0.0049]	64, 69 [67]	4.2, 4.1 [4.1]	64, 69 [67]	69, 73 [71]
MN-2 Fergus Falls, Minnesota (MEA)	Pre (--) BBCH 14 (27) BBCH 60 (18)	1.12 0.56 0.56	188 188 187	Forage	7	0.28, 0.37 [0.32]	<0.005, <0.005 [<0.0047]	15, 14 [15]	2.6, 2.4 [2.5]	15, 15 [15]	18, 17 [17]
				Hay	18	0.096, 0.13 [0.11]	<0.005, <0.005 [<0.0047]	41, 47 [44]	2.0, 4.1 [3.1]	41, 47 [44]	43, 51 [47]
	Pre (--) BBCH 14 (18)	1.12 1.13	187 188	Forage	7	0.54, 0.80 [0.67]	<0.005, 0.0049 [0.0048]	28, 31 [29]	4.0, 4.0 [4.0]	28, 32 [30]	32, 36 [34]

Trial No. Location Year (Salt) [a]	Application			Matrix	DALA	Dicamba-equivalent residues (mg/kg) [Mean]					
	Timing (interval, days)	kg ai/ha	L/ha			Dicamba	5-OH dicamba	DCSA	DCGA	For max. res.	For risk
(DGA)				Hay	18	0.25, 0.29 [0.27]	0.0094, 0.0085 [0.0090]	91, 97 [94]	5.9, 5.9 [5.9]	91, 97 [94]	97, 100 [100]
	Pre (--) BBCH 14 (18)	1.12 1.13	187 188	Forage	7	1.2, 1.1 [1.1]	<0.005, 0.0052 [0.0049]	36, 37 [36]	4.8, 5.2 [5.0]	37, 38 [37]	41, 43 [42]
				Hay	18	0.31, 0.32 [0.32]	0.013, 0.0077 [0.010]	100, 100 [100]	6.3, 6.7 [6.5]	100, 100 [100]	110, 110 [110]
MO Fisk, Missouri (MEA)	Pre (--) BBCH 14 (24) BBCH 60 (17)	1.12 0.56 0.57	186 189 189	Forage	7	0.063, 0.063 [0.063]	0.0056, 0.0047 [0.0051]	18, 16 [17]	0.36, 0.38 [0.37]	18, 16 [17]	19, 17 [18]
				Hay	19	0.014, 0.014 [0.014]	<0.005, <0.005 [<0.0047]	22, 22 [22]	0.77, 0.70 [0.73]	22, 22 [22]	22, 23 [23]
ND-1 Carrington, North Dakota (MEA)	Pre (--) BBCH 14 (31) BBCH 60 (29)	1.12 0.56 0.57	188 187 188	Forage	7	0.30, 0.29 [0.30]	<0.005, <0.005 [<0.0047]	12, 13 [12]	0.62, 0.84 [0.73]	12, 13 [13]	13, 14 [13]
				Hay	16	0.20, 0.12 [0.16]	0.0062, <0.005 [0.0054]	27, 26 [26]	4.7, 4.7 [4.7]	27, 26 [27]	32, 31 [31]
NE-1 York, Nebraska (MEA)	Pre (--) BBCH 14 (26) BBCH 60 (21)	1.13 0.56 0.56	188 187 187	Forage	7	0.35, 0.26 [0.31]	0.0059, <0.005 [0.0053]	13, 12 [12]	1.5, 1.4 [1.5]	13, 13 [13]	15, 14 [14]
				Hay	14	0.28, 0.29 [0.29]	0.0062, 0.0074 [0.0068]	33, 37 [35]	1.3, 1.5 [1.4]	34, 38 [36]	35, 39 [37]
NE-2 Osceola, Nebraska (MEA)	Pre (--) BBCH 14 (25) BBCH 60 (18)	1.1 0.56 0.57	182 187 188	Forage	8	0.043, 0.059 [0.051]	0.0074, 0.0055 [0.0064]	14, 14 [14]	1.5, 1.6 [1.6]	14, 14 [14]	15, 16 [16]
				Hay	14	0.014, 0.014 [0.014]	0.0066, 0.010 [0.0084]	38, 38 [38]	3.3, 3.6 [3.5]	38, 38 [38]	41, 42 [42]
SC Elko, South Carolina (MEA)	Pre (--) BBCH 14 (30) BBCH 60 (9)	1.12 0.56 0.56	189 193 186	Forage	7	0.068, 0.068 [0.068]	0.0053, <0.005 [0.0050]	20, 19 [19]	2.3, 2.1 [2.2]	20, 19 [20]	22, 21 [22]
				Hay	20	0.057, 0.065 [0.061]	<0.005, <0.005 [<0.0047]	29, 35 [32]	0.17, 0.18 [0.17]	29, 35 [32]	30, 35 [32]
SD-1 Centerville, South Dakota (MEA)	Pre (--) BBCH 14 (31) BBCH 60 (17)	1.09 0.55 0.56	180 184 185	Forage	7	2.3, 2.6 [2.5]	0.0052, 0.0056 [0.0054]	13, 15 [14]	1.6, 2.9 [2.3]	15, 18 [16]	17, 20 [19]
				Hay	18	0.18, 0.20 [0.19]	0.0047, <0.005 [0.0047]	37, 35 [36]	4.4, 3.7 [4.1]	37, 35 [36]	41, 39 [40]
SD-2 Britton, South Dakota	Pre (--) BBCH 14 (32) BBCH 60 (10)	1.12 0.56 0.56	186 187 187	Forage	7	0.42, 0.44 [0.43]	0.0051, 0.0053 [0.0052]	17, 16 [17]	2.7, 1.8 [2.3]	17, 17 [17]	20, 19 [19]

Trial No. Location Year (Salt) [a]	Application			Matrix	DALA	Dicamba-equivalent residues (mg/kg) [Mean]					
	Timing (interval, days)	kg ai/ha	L/ha			Dicamba	5-OH dicamba	DCSA	DCGA	For max. res.	For risk
(MEA)											
				Hay	14	0.063, 0.034 [0.048]	<0.005, <0.005 [<0.0047]	31, 30 [30]	2.0, 1.7 [1.8]	31, 30 [30]	33, 32 [32]
WI-1 Delavan, Wisconsin (MEA)	Pre (--) BBCH 14 (35) BBCH 60 (15)	1.12 0.52 0.56	183 173 188	Forage	7	0.56, 0.86 [0.71]	<0.005, <0.005 [<0.0047]	15, 16 [15]	4.0, 3.9 [4.0]	15, 17 [16]	19, 21 [20]
				Hay	18	0.16, 0.21 [0.18]	0.0054, <0.005 [0.0050]	31, 32 [32]	2.4, 2.4 [2.4]	32, 33 [32]	34, 35 [35]
WI-2 Fitchburg, Wisconsin (MEA)	Pre (--) BBCH 14 (29) BBCH 60 (6)	0.56, 0.57 0.57 0.56	195, 186 188 190	Forage	10	0.070, 0.057 [0.064]	<0.005, 0.0051 [0.0049]	16, 15 [15]	1.7, 1.9 [1.8]	16, 15 [15]	17, 17 [17]
				Hay	22	0.22, 0.15 [0.19]	0.0062, 0.0048 [0.0055]	60, 61 [61]	7.1, 7.3 [7.2]	60, 61 [61]	68, 68 [68]

[a] Formulation: MEA = Monoethanolamine salt, DGA = Diglycolamine salt

Straw, fodder, and forage of cereal grains and grasses

Maize

Twenty-two residue trials were conducted on dicamba-tolerant maize (MON 87419 variety) as described above (Cereal grains, maize; Report MSL0026526). From those trials, samples of maize forage and stover were harvested 29-71 and 64-132 DALA, respectively. The moisture content for the stover was not specified.

Maize forage samples were stored frozen for up to 225 days, and stover samples for up to 285 days. Residues of dicamba, 5-hydroxydicamba, DCSA, and DCGA were analysed using the method described above. Average concurrent recoveries from forage and stover across all four analytes and across fortifications from 0.01 to ca. 27 mg/kg depending on the matrix and analyte ranged from 94 to 109%. Relative standard deviations ranged from 3.2 to 16%.

Results for maize forage and hay are shown in Table 17.

Table 17 Results of dicamba residue trials in dicamba-tolerant maize forage and stover (variety MON 87419) in the USA (2013 growing season; Report MSL0026526)

Trial No. Location	Application			Matrix	DALA	Dicamba-equivalent residues (mg/kg) [Mean]					
	Timing (interval, days)	kg ai/ha	L/ha			Dicamba	5-OH dicamba	DCSA	DCGA	For max. res.	For risk
Critical GAP USA	1 pre + 2 post	1.12 + 0.56 + 0.56	140	--		--	--	--	--		
01PA Germansville, Pennsylvania	Pre (--) BBCH 14-15 (23) BBCH 17-18 (14)	1.14 0.57 0.57	192 190 191	Forage	68	<0.01, <0.01 [<0.01]	0.052, 0.038 [0.045]	0.86, 0.76 [0.81]	1.6, 1.6 [1.6]	0.87, 0.77 [0.82]	2.6, 2.4 [2.5]

Trial No. Location	Application			Matrix	DALA	Dicamba-equivalent residues (mg/kg) [Mean]					
	Timing (interval, days)	kg ai/ha	L/ha			Dicamba	5-OH dicamba	DCSA	DCGA	For max. res.	For risk
				Stover	107	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.42, 0.41 [0.42]	0.16, 0.22 [0.19]	0.43, 0.42 [0.43]	0.60, 0.65 [0.62]
	BBCH 13 BBCH 14-15 (7) BBCH 17-18 (14) BBCH 19 (8)	0.58 0.58 0.58 0.56	194 193 195 189	Forage	60	<0.01, <0.01 [<0.01]	0.049, 0.018 [0.033]	1.3, 0.82 [1.1]	1.6, 1.5 [1.6]	1.3, 0.83 [1.1]	3.0, 2.4 [2.7]
				Stover	99	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.67, 0.46 [0.57]	0.34, 0.24 [0.29]	0.68, 0.48 [0.58]	1.0, 0.73 [0.88]
02GA Chula, Georgia	Pre (--) BBCH 15 (27) BBCH 18 (12)	1.11 0.54 0.57	182 185 183	Forage	46	<0.01, <0.01 [<0.01]	0.045, 0.050 [0.048]	2.1, 2.0 [2.0]	1.4, 1.2 [1.3]	2.1, 2.0 [2.0]	3.5, 3.3 [3.4]
				Stover	86	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.29, 0.28 [0.28]	0.17, 0.11 [0.14]	0.30, 0.29 [0.29]	0.48, 0.41 [0.45]
	BBCH 13 BBCH 15 (14) BBCH 18 (12) BBCH 19 (10)	0.56 0.55 0.55 0.57	183 188 178 195	Forage	36	<0.01, <0.01 [<0.01]	0.050, 0.076 [0.063]	2.5, 3.4 [3.0]	1.4, 1.8 [1.6]	2.5, 3.4 [3.0]	3.9, 5.3 [4.6]
				Stover	76	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.35, 0.34 [0.34]	0.21, 0.26 [0.24]	0.36, 0.35 [0.35]	0.58, 0.62 [0.60]
03NC Belvidere, N Carolina	Pre (--) BBCH 15-16 (28) BBCH 18 (14)	1.16 0.58 0.56	193 186 188	Forage	54	<0.01, <0.01 [<0.01]	0.056, 0.050 [0.053]	1.2, 0.25 [0.73]	2.1, 0.43 [1.3]	1.2, 0.26 [0.74]	3.4, 0.73 [2.1]
				Stover	89	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.22, 0.20 [0.21]	0.12, 0.14 [0.13]	0.23, 0.22 [0.22]	0.36, 0.36 [0.36]
	BBCH 13 BBCH 15-16 (7) BBCH 18 (14) BBCH 19 (7)	0.56 0.56 0.56 0.57	183 181 187 188	Forage	47	<0.01, <0.01 [<0.01]	<0.01, 0.042 [0.026]	0.24, 0.87 [0.55]	1.8, 2.5 [2.1]	0.25, 0.88 [0.56]	2.0, 3.4 [2.7]
				Stover	82	0.013, <0.01 [0.011]	<0.01, <0.01 [<0.01]	0.16, 0.15 [0.16]	0.052, 0.050 [0.051]	0.17, 0.16 [0.17]	0.24, 0.22 [0.23]
04TX Uvalde, Texas	BBCH 0 BBCH 15-16 (47) BBCH 18 (7)	1.11 0.56 0.56	187 185 187	Forage	36	<0.01, 0.019 [0.014]	0.26, 0.50 [0.38]	4.0, 6.6 [5.3]	1.0, 1.4 [1.2]	4.0, 6.6 [5.3]	5.3, 8.5 [6.9]
				Stover	71	0.066, 0.048 [0.057]	1.6, 0.78 [1.2]	17, 14 [16]	3.0, 3.0 [3.0]	17, 14 [16]	22, 18 [20]
	BBCH 12 BBCH 15-16 (23) BBCH 18 (7) BBCH 51 (7)	0.57 0.56 0.56 0.57	188 188 187 189	Forage	29	0.043, 0.033 [0.038]	0.46, 0.40 [0.43]	7.6, 4.8 [6.2]	1.9, 1.6 [1.7]	7.7, 4.9 [6.3]	10, 6.9 [8.4]
				Stover	64	0.051, 0.10 [0.078]	1.0, 0.91 [0.96]	13, 13 [13]	3.8, 4.4 [4.1]	13, 13 [13]	18, 18 [18]
05MI Wright, Michigan	Pre (--) BBCH 14 (34) BBCH 18 (13)	1.14 0.56 0.56	187 187 189	Forage	71	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.23, 0.42 [0.33]	0.073, 0.19 [0.13]	0.24, 0.43 [0.34]	0.33, 0.63 [0.48]
				Stover	121	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.068, 0.052 [0.060]	0.038, 0.024 [0.031]	0.078, 0.062 [0.070]	0.13, 0.096 [0.11]

Trial No. Location	Application			Matrix	DALA	Dicamba-equivalent residues (mg/kg) [Mean]					
	Timing (interval, days)	kg ai/ha	L/ha			Dicamba	5-OH dicamba	DCSA	DCGA	For max. res.	For risk
	BBCH 12 BBCH 14 (9) BBCH 18 (13) BBCH 19 (11)	0.56 0.56 0.56 0.56	186 186 190 189	Forage	60	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.18, 0.37 [0.28]	0.066, 0.17 [0.12]	0.19, 0.38 [0.29]	0.27, 0.56 [0.41]
				Stover	110	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.077, 0.054 [0.065]	0.053, 0.030 [0.042]	0.087, 0.064 [0.075]	0.15, 0.10 [0.13]
06KS Troy, Kansas	BBCH 5 BBCH 13 (22) BBCH 18 (8)	1.13 0.57 0.65	190 195 221	Forage	55	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.81, 0.70 [0.76]	0.35, 0.33 [0.34]	0.82, 0.71 [0.77]	1.2, 1.1 [1.1]
				Stover	132	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.032, 0.050 [0.041]	<0.01, 0.022 [0.016]	0.042, 0.060 [0.051]	0.062, 0.093 [0.077]
	BBCH 12 BBCH 13 (9) BBCH 18 (8) BBCH 39 (13)	0.58 0.57 0.57 0.57	195 194 195 196	Forage	42	<0.01, <0.01 [<0.01]	0.034, 0.016 [0.025]	1.8, 1.8 [1.8]	1.8, 1.2 [1.5]	1.8, 1.8 [1.8]	3.7, 3.1 [3.4]
				Stover	119	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.18, 0.20 [0.19]	0.10, 0.11 [0.11]	0.19, 0.21 [0.20]	0.30, 0.32 [0.31]
07KS St. John, Kansas	BBCH 1 BBCH 16 (32) BBCH 18 (7)	1.12 0.57 0.55	185 188 187	Forage	58	<0.01, <0.01 [<0.01]	0.030, 0.010 [0.020]	0.63, 0.42 [0.53]	1.2, 0.15 [0.68]	0.64, 0.43 [0.54]	1.9, 0.60 [1.2]
				Stover	91	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.74, 0.53 [0.64]	0.12, 0.080 [0.10]	0.75, 0.54 [0.65]	0.88, 0.63 [0.76]
	BBCH 12 BBCH 16 (18) BBCH 18 (7) BBCH 19 (21)	0.57 0.56 0.55 0.58	188 185 185 191	Forage	37	<0.01, <0.01 [<0.01]	0.019, 0.033 [0.026]	0.81, 1.1 [0.94]	0.45, 0.76 [0.60]	0.82, 1.1 [0.95]	1.3, 1.9 [1.6]
				Stover	70	<0.01, <0.01 [<0.01]	0.016, 0.011 [0.014]	1.5, 1.0 [1.3]	0.29, 0.27 [0.28]	1.5, 1.0 [1.3]	1.8, 1.3 [1.6]
08IA Black Hawk, Iowa	Pre (--) BBCH 15 (35) BBCH 18 (13)	1.11 0.55 0.56	186 183 186	Forage	63	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.32, 0.30 [0.31]	0.39, 0.32 [0.35]	0.33, 0.31 [0.32]	0.72, 0.64 [0.68]
				Stover	106	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.29, 0.40 [0.34]	0.18, 0.21 [0.19]	0.30, 0.41 [0.35]	0.49, 0.62 [0.56]
	BBCH 13 BBCH 15 (12) BBCH 18 (13) BBCH 19 (7)	0.56 0.56 0.55 0.56	191 186 183 189	Forage	56	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.40, 0.47 [0.43]	0.44, 0.70 [0.57]	0.41, 0.48 [0.44]	0.86, 1.2 [1.0]
				Stover	99	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.35, 0.35 [0.35]	0.18, 0.20 [0.19]	0.36, 0.36 [0.36]	0.55, 0.56 [0.56]
09IL Carlyle, Illinois	Pre (--) BBCH 15-16 (32) BBCH 18 (13)	1.13 0.57 0.56	196 194 193	Forage	50	<0.01, <0.01 [<0.01]	0.040, 0.067 [0.053]	0.69, 0.72 [0.70]	0.51, 0.72 [0.61]	0.70, 0.73 [0.71]	1.2, 1.5 [1.4]
				Stover	91	<0.01, <0.01 [<0.01]	<0.01, 0.014 [0.012]	0.41, 0.48 [0.44]	0.18, 0.25 [0.22]	0.42, 0.49 [0.45]	0.61, 0.75 [0.68]

Trial No. Location	Application			Matrix	DALA	Dicamba-equivalent residues (mg/kg) [Mean]					
	Timing (interval, days)	kg ai/ha	L/ha			Dicamba	5-OH dicamba	DCSA	DCGA	For max. res.	For risk
	BBCH 14 BBCH 15-16 (9) BBCH 18 (13) BBCH 19 (7)	0.56 0.56 0.56 0.56	179 190 193 191	Forage	43	<0.01, <0.01 [<0.01]	0.083, 0.085 [0.084]	1.6, 1.4 [1.5]	1.4, 1.8 [1.6]	1.6, 1.5 [1.5]	3.1, 3.3 [3.2]
				Stover	84	<0.01, 0.015 [0.012]	<0.01, 0.024 [0.017]	0.62, 0.89 [0.76]	0.48, 0.84 [0.66]	0.63, 0.91 [0.77]	1.1, 1.8 [1.4]
10IL Highland, Illinois	Pre (--) BBCH 15 (25) BBCH 18 (16)	1.16 0.57 0.56	196 182 194	Forage	49	<0.01, <0.01 [<0.01]	0.033, 0.042 [0.038]	0.69, 1.1 [0.89]	0.77, 1.2 [0.98]	0.70, 1.1 [0.90]	1.5, 2.3 [1.9]
				Stover	100	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.31, 0.48 [0.40]	0.13, 0.18 [0.15]	0.32, 0.49 [0.40]	0.46, 0.68 [0.57]
	BBCH 13 BBCH 15 (7) BBCH 18 (16) BBCH 18 (9)	0.58 0.57 0.56 0.56	194 182 192 193	Forage	40	<0.01, 0.011 [0.011]	0.029, 0.092 [0.061]	0.93, 1.6 [1.3]	1.1, 1.6 [1.4]	0.94, 1.6 [1.3]	2.1, 3.3 [2.7]
				Stover	91	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.37, 0.66 [0.52]	0.29, 0.35 [0.32]	0.38, 0.67 [0.53]	0.69, 1.0 [0.86]
11IL Camp Grove, Illinois	BBCH 0 BBCH 14 (30) BBCH 18 (9)	1.13 0.56 0.56	188 185 183	Forage	64	<0.01, <0.01 [<0.01]	0.011, <0.01 [0.010]	0.20, 0.14 [0.17]	0.082, 0.070 [0.076]	0.21, 0.15 [0.18]	0.31, 0.23 [0.27]
				Stover	100	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.084, 0.10 [0.094]	0.040, 0.053 [0.046]	0.094, 0.11 [0.10]	0.14, 0.18 [0.16]
	BBCH 13 BBCH 14 (7) BBCH 18 (9) BBCH 19 (15)	0.55 0.56 0.55 0.57	178 182 181 190	Forage	49	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.32, 0.31 [0.31]	0.26, 0.32 [0.29]	0.33, 0.32 [0.32]	0.60, 0.65 [0.62]
				Stover	91	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.36, 0.26 [0.31]	0.21, 0.18 [0.19]	0.37, 0.27 [0.32]	0.59, 0.46 [0.52]
12IL Stewardson, Illinois	BBCH 0 BBCH 15 (28) BBCH 18 (12)	1.12 0.55 0.55	188 180 180	Forage	65	<0.01, <0.01 [<0.01]	0.038, 0.022 [0.030]	0.30, 0.31 [0.30]	0.12, 0.82 [0.47]	0.31, 0.32 [0.32]	0.47, 1.2 [0.82]
				Stover	105	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.025, 0.084 [0.055]	<0.01, 0.034 [0.022]	0.035, 0.094 [0.065]	0.055, 0.14 [0.097]
	BBCH 12 BBCH 15 (11) BBCH 18 (12) BBCH 19 (11)	0.56 0.56 0.56 0.56	192 184 184 187	Forage	54	<0.01, <0.01 [<0.01]	0.21, 0.099 [0.16]	0.62, 0.92 [0.77]	1.3, 1.8 [1.5]	0.63, 0.93 [0.78]	2.2, 2.8 [2.5]
				Stover	94	0.015, <0.01 [0.012]	<0.01, <0.01 [<0.01]	0.39, 0.24 [0.31]	0.13, 0.11 [0.12]	0.40, 0.25 [0.33]	0.54, 0.37 [0.46]
13IL Duvall, Illinois	BBCH 3 BBCH 16 (25) BBCH 18 (16)	1.12 0.57 0.56	190 192 192	Forage	67	<0.01, <0.01 [<0.01]	0.087, 0.024 [0.056]	0.57, 0.51 [0.54]	1.2, 0.49 [0.84]	0.58, 0.52 [0.55]	1.8, 1.0 [1.4]
				Stover	108	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.25, 0.33 [0.29]	0.087, 0.13 [0.11]	0.26, 0.34 [0.30]	0.36, 0.49 [0.42]
	BBCH 12 BBCH 16 (7) BBCH 18 (16)	0.56 0.58 0.57	190 196 196	Forage	55	<0.01, <0.01 [<0.01]	0.036, 0.012 [0.024]	0.49, 0.42 [0.46]	0.57, 0.28 [0.43]	0.50, 0.43 [0.47]	1.1, 0.72 [0.92]

Trial No. Location	Application			Matrix	DALA	Dicamba-equivalent residues (mg/kg) [Mean]					
	Timing (interval, days)	kg ai/ha	L/ha			Dicamba	5-OH dicamba	DCSA	DCGA	For max. res.	For risk
	BBCH 24 (9)	0.57	189	Stover	96	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.40, 0.56 [0.48]	0.33, 0.57 [0.45]	0.41, 0.57 [0.49]	0.75, 1.2 [0.95]
14IL Highland, Illinois	BBCH 0 BBCH 15 (28) BBCH 18 (14)	1.12 0.57 0.57	195 195 195	Forage	63	<0.01, <0.01 [<0.01]	0.030, 0.015 [0.023]	0.68, 0.47 [0.57]	0.60, 0.38 [0.49]	0.69, 0.48 [0.58]	1.3, 0.88 [1.1]
				Stover	97	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.25, 0.16 [0.20]	0.11, 0.089 [0.100]	0.26, 0.17 [0.22]	0.38, 0.27 [0.32]
	BBCH 12 BBCH 15 (14) BBCH 18 (14) BBCH 39 (9)	0.56 0.57 0.56 0.57	194 194 195 191	Forage	54	<0.01, <0.01 [<0.01]	0.058, 0.020 [0.039]	1.3, 1.0 [1.1]	1.1, 0.88 [0.99]	1.3, 1.0 [1.2]	2.4, 1.9 [2.2]
				Stover	88	<0.01, <0.01 [<0.01]	<0.01, 0.010 [0.010]	0.50, 0.83 [0.67]	0.72, 0.73 [0.73]	0.51, 0.84 [0.68]	1.2, 1.6 [1.4]
15IN Pickard, Indiana	BBCH 0 BBCH 15 (29) BBCH 18 (12)	1.16 0.56 0.56	194 188 193	Forage	58	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.14, 0.78 [0.46]	0.15, 0.94 [0.54]	0.15, 0.79 [0.47]	0.31, 1.7 [1.0]
				Stover	119	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.15, 0.23 [0.19]	0.15, 0.28 [0.21]	0.16, 0.24 [0.20]	0.32, 0.52 [0.42]
	BBCH 12 BBCH 15 (15) BBCH 18 (12) BBCH 19 (14)	0.57 0.56 0.57 0.56	193 188 195 187	Forage	44	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.30, 0.11 [0.21]	0.22, 0.050 [0.14]	0.31, 0.12 [0.22]	0.55, 0.18 [0.36]
				Stover	105	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.22, 0.24 [0.23]	0.19, 0.19 [0.19]	0.23, 0.25 [0.24]	0.43, 0.45 [0.44]
16MO Kirksville, Missouri	Pre (--) BBCH 14 (26) BBCH 18 (14)	1.11 0.56 0.56	186 188 184	Forage	57	<0.01, <0.01 [<0.01]	0.021, 0.015 [0.018]	0.45, 0.43 [0.44]	0.37, 0.27 [0.32]	0.46, 0.44 [0.45]	0.85, 0.73 [0.79]
				Stover	89	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.45, 0.58 [0.52]	0.14, 0.29 [0.21]	0.46, 0.59 [0.53]	0.61, 0.89 [0.75]
	BBCH 12 BBCH 14 (10) BBCH 18 (14) BBCH 19 (7)	0.56 0.56 0.56 0.57	189 187 185 192	Forage	50	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.68, 0.76 [0.72]	0.28, 0.29 [0.28]	0.69, 0.77 [0.73]	0.98, 1.1 [1.0]
				Stover	82	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.51, 0.45 [0.48]	0.19, 0.20 [0.20]	0.52, 0.46 [0.49]	0.72, 0.67 [0.70]
17MO Broseley, Missouri	BBCH 5 BBCH 16 (21) (9)	1.12 0.57 0.56	187 188 187	Forage	68	<0.01, <0.01 [<0.01]	0.011, 0.060 [0.036]	0.34, 1.1 [0.71]	0.063, 0.17 [0.12]	0.35, 1.1 [0.72]	0.42, 1.3 [0.87]
				Stover	95	<0.01, <0.01 [<0.01]	0.015, <0.01 [0.013]	0.58, 0.42 [0.50]	0.14, 0.13 [0.14]	0.59, 0.43 [0.51]	0.75, 0.57 [0.66]
	BBCH 14 BBCH 16 (7) BBCH 18 (9) BBCH 19 (7)	0.57 0.56 0.56 0.57	188 187 187 188	Forage	61	<0.01, <0.01 [<0.01]	<0.01, 0.016 [0.013]	0.42, 0.49 [0.45]	0.046, 0.044 [0.045]	0.43, 0.50 [0.46]	0.48, 0.56 [0.52]
				Stover	88	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.37, 0.28 [0.33]	0.12, 0.11 [0.12]	0.38, 0.29 [0.34]	0.52, 0.41 [0.47]

Trial No. Location	Application			Matrix	DALA	Dicamba-equivalent residues (mg/kg) [Mean]					
	Timing (interval, days)	kg ai/ha	L/ha			Dicamba	5-OH dicamba	DCSA	DCGA	For max. res.	For risk
18NE Tabor, Nebraska	BBCH 0 BBCH 15 (32) BBCH 18 (8)	1.11 0.56 0.57	178 179 184	Forage	56	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.50, 0.61 [0.55]	0.70, 0.70 [0.70]	0.51, 0.62 [0.56]	1.2, 1.3 [1.3]
				Stover	118	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.12, 0.13 [0.12]	0.10, 0.10 [0.10]	0.12, 0.14 [0.13]	0.24, 0.25 [0.24]
	BBCH 12 BBCH 15 (18) BBCH 18 (8) BBCH 19 (7)	0.57 0.56 0.57 0.56	184 180 182 188	Forage	49	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.53, 0.29 [0.41]	0.39, 0.23 [0.31]	0.54, 0.30 [0.42]	0.94, 0.54 [0.74]
				Stover	111	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.18, 0.15 [0.17]	0.13, 0.16 [0.14]	0.19, 0.16 [0.18]	0.33, 0.33 [0.33]
19NE Henderson, Nebraska	BBCH 0 BBCH 16 (34) BBCH 19 (7)	1.1 0.55 0.56	191 191 191	Forage	53	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	1.6, 1.8 [1.7]	0.96, 0.97 [0.97]	1.6, 1.8 [1.8]	2.6, 2.8 [2.7]
				Stover	99	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	1.1, 0.56 [0.85]	0.21, 0.22 [0.22]	1.2, 0.57 [0.86]	1.4, 0.79 [1.1]
	BBCH 13 BBCH 16 (8) BBCH 19 (7) BBCH 19 (7)	0.55 0.56 0.57 0.56	190 194 191 194	Forage	46	<0.01, <0.01 [<0.01]	0.049, 0.020 [0.034]	2.6, 2.8 [2.7]	1.1, 1.6 [1.4]	2.6, 2.8 [2.7]	3.8, 4.4 [4.1]
				Stover	92	<0.01, <0.01 [<0.01]	0.010, 0.013 [0.012]	1.1, 1.3 [1.2]	0.90, 0.81 [0.86]	1.1, 1.3 [1.2]	2.0, 2.1 [2.1]
20NE Brunswick, Nebraska	BBCH 0 BBCH 15 (30) BBCH 18 (7)	1.09 0.55 0.56	188 190 192	Forage	54	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.65, 0.18 [0.42]	0.50, 0.12 [0.31]	0.66, 0.20 [0.43]	1.2, 0.33 [0.74]
				Stover	101	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.19, 0.052 [0.12]	0.054, 0.020 [0.037]	0.20, 0.062 [0.13]	0.27, 0.093 [0.18]
	BBCH 12 BBCH 15 (10) BBCH 18 (7) BBCH 19 (13)	0.56 0.56 0.56 0.56	190 191 191 194	Forage	41	<0.01, <0.01 [<0.01]	0.026, 0.028 [0.027]	1.8, 0.78 [1.3]	2.9, 0.31 [1.6]	1.9, 0.79 [1.3]	4.8, 1.1 [3.0]
				Stover	88	0.017, 0.015 [0.016]	0.029, 0.032 [0.031]	1.9, 1.7 [1.8]	2.0, 1.4 [1.7]	2.0, 1.7 [1.8]	4.0, 3.1 [3.5]
21SD Bushnell, S. Dakota	BBCH 0 BBCH 16 (32) BBCH 18 (7)	1.12 0.58 0.56	188 193 182	Forage	64	<0.01, <0.01 [<0.01]	0.011, 0.016 [0.013]	1.8, 1.7 [1.7]	1.1, 1.0 [1.1]	1.8, 1.7 [1.7]	2.9, 2.7 [2.8]
				Stover	112	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.23, 0.25 [0.24]	0.091, 0.090 [0.090]	0.24, 0.26 [0.25]	0.34, 0.36 [0.35]
	BBCH 13 BBCH 16 (12) BBCH 18 (7) BBCH 19 (7)	0.56 0.56 0.57 0.56	196 187 188 183	Forage	57	<0.01, <0.01 [<0.01]	<0.01, 0.020 [0.015]	0.38, 1.5 [0.92]	0.19, 0.23 [0.21]	0.39, 1.5 [0.93]	0.58, 1.7 [1.1]
				Stover	105	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.11, 0.14 [0.12]	0.092, 0.081 [0.086]	0.12, 0.15 [0.13]	0.22, 0.24 [0.23]
22WI Richmond, Wisconsin	Pre (--) BBCH 15 (33) BBCH 18 (14)	1.11 0.56 0.56	187 184 181	Forage	67	<0.01, <0.01 [<0.01]	0.013, <0.01 [0.012]	0.49, 0.71 [0.60]	0.29, 0.16 [0.22]	0.50, 0.72 [0.61]	0.80, 0.88 [0.84]

Trial No. Location	Application			Matrix	DALA	Dicamba-equivalent residues (mg/kg) [Mean]					
	Timing (interval, days)	kg ai/ha	L/ha			Dicamba	5-OH dicamba	DCSA	DCGA	For max. res.	For risk
				Stover	120	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.29, 0.14 [0.21]	0.094, 0.081 [0.088]	0.30, 0.15 [0.22]	0.41, 0.24 [0.32]
	BBCH 13 BBCH 15 (10) BBCH 18 (14) BBCH 19 (7)	0.56 0.56 0.56 0.56	189 185 181 187	Forage	60	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.58, 1.3 [0.95]	0.11, 0.15 [0.13]	0.59, 1.3 [0.96]	0.71, 1.5 [1.1]
				Stover	113	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.42, 0.32 [0.36]	0.17, 0.15 [0.16]	0.42, 0.32 [0.38]	0.60, 0.49 [0.54]

Oilseeds

Cotton

Three residue trials were conducted on dicamba-tolerant cotton (MON 88701 variety) from which samples of gin trash were collected; a summary of the trials is described above (Oilseeds, cotton; Report MSL0024072).

Gin trash samples were stored frozen for up to 26 days. Residues of dicamba, 5-hydroxydicamba, DCSA, and DCGA were analysed using the method described above. Average concurrent recoveries across all four analytes and across fortifications from 0.04 to ca 100 mg/kg depending on the matrix and analyte ranged from 90 to 121%. Relative standard deviations ranged from 3.4 to 17%.

Results for maize forage and hay are shown in Table 18.

Table 18 Results of dicamba residue trials in dicamba-tolerant cotton gin trash (variety MON 88701) in the USA (2010 growing season; Report MSL0024072)

Trial No. Location Salt [a]	Application			DALA	Residues (mg/kg) [Mean]					
	Timing (interval, days)	kg ai/ha	L/ha		Dicamba	5-OH dicamba	DCSA	DCGA	For max. res.	For risk
Critical GAP USA	1 pre + 2 post with a 7-day retreatment interval	1.12 + 0.56 + 0.56	140	7	--	--	--	--	--	--
OK2 Dill City, Oklahoma (DGA)	ns (--) ns (28) BBCH 65 (49)	1.1 0.56 0.55	178 188 184	82	<0.04, <0.04 [<0.04]	<0.037, <0.037 [<0.037]	0.42, 0.49 [0.45]	0.17, 0.23 [0.20]	0.46, 0.53 [0.49]	0.62, 0.76 [0.69]
	ns (--) ns (49) BBCH 97 (32) ns (43)	0.57 0.56 0.56 0.55	189 188 190 184	7	3.2, 3.1 [3.1]	<0.037, <0.037 [<0.037]	1.8, 2.0 [1.9]	0.49, 0.40 [0.44]	5.0, 5.1 [5.0]	5.5, 5.5 [5.5]
TX2 Levelland, Texas (DGA)	ns (--) ns (36) BBCH 65 (41)	1.1 0.57 0.56	191 193 188	84	<0.04, <0.04 [<0.04]	<0.037, <0.037 [<0.037]	1.8, 1.5 [1.7]	0.42, 0.70 [0.56]	1.9, 1.6 [1.7]	2.3, 2.3 [2.3]
	ns (--) ns (41) ns (41) BBCH 89 (37)	0.55 0.55 0.56 0.55	187 187 191 187	6	16, 13 [15]	<0.037, <0.037 [<0.037]	6.4, 3.2 [4.8]	3.2, 1.5 [2.4]	23, 17 [20]	26, 18 [22]
TX3 Wolfforth, Texas (DGA)	ns (--) ns (41) BBCH 65 (36)	1.1 0.57 0.55	188 194 185	84	<0.04, <0.04 [<0.04]	<0.037, <0.037 [<0.037]	0.91, 0.52 [0.72]	0.32, 0.31 [0.31]	0.95, 0.56 [0.76]	1.3, 0.87 [1.1]

Trial No. Location Salt [a]	Application			DALA	Residues (mg/kg) [Mean]					
	Timing (interval, days)	kg ai/ha	L/ha		Dicamba	5-OH dicamba	DCSA	DCGA	For max. res.	For risk
	ns (--) ns (36) ns (48) BBCH 89 (30)	0.56 0.56 0.56 0.56	189 188 189 188	6	24, 22 [23]	<0.037, <0.037 [<0.037]	6.7, 6.5 [6.6]	4.3, 3.9 [4.1]	30, 29 [30]	35, 33 [34]

[a] Formulation: DGA = Diglycolamine salt

FATE OF RESIDUES DURING PROCESSING

Soya bean

Of the soya bean field trials described above (Report MSL0023061), two were designated for producing seed for processing into hulls, meal, oil, lecithin, flour, protein isolates, soya milk, and tofu. In those trials, soya bean plants were treated at an exaggerated rate consisting of 1.12 kg ai/ha preemergence and 2.24 kg ai/ha at BBCH 60. Soya bean seeds were harvested at maturity and transported frozen to the processing facility.

Processing was done under simulated commercial practices. Seeds were dried to a moisture content of <13.5% followed by cleaning via aspiration and screening. A portion of the cleaned seeds was soaked in water for 12 h, ground and filtered to remove solids, and the resulting soya milk was cooked (ca 93 °C, 10min). Calcium sulfate was slowly added to an aliquot of the cooked soya milk to produce tofu. Soya bean seeds not used for milk/tofu production were processed in a roller mill and aspirated to separate the kernel from the hull. Kernels were adjusted to a moisture content of 13.5%, allowed to equilibrate for 12 hours and then heated and flaked. Flakes were used to produce the remaining processed commodities. Flakes were extruded and turned into collets by steam injection and compression. Following drying, the collets were ground and extracted three times with hexane to extract oil. A portion of the extracted collets were dried, ground, and screened to produce defatted flour. A portion of the defatted flour was freeze dried to produce protein isolate and protein concentrate. A separate portion of the extracted collets were toasted and underwent steam injection to form toasted soya bean meal. Crude oil from the extraction of the collets underwent heated vacuum evaporation to remove the hexane. The crude oil was hydrated and filtered to produce degummed oil and crude lecithin. The degummed oil was processed with NaOH to produce refined oil and soapstock. Finally, the refined oil was bleached and deodorized.

The processed commodities were analysed for residues of dicamba, 5-hydroxydicamba, DCSA, and DCGA using the method described above. Residues of dicamba and 5-hydroxydicamba were below the LOQ (0.013 mg/kg and 0.021 mg/kg, respectively) in all samples of soya bean seed and processed commodities (Table 19).

Table 19 Residues of dicamba metabolites DCSA and DCGA in processed products derived from dicamba-tolerant soya beans (Report MSL0023061)

Matrix	Trial	Residues, mg/kg [mean]			
		Dicamba	5-OH Dicamba	DCSA	DCGA
Seed	NE-1	<0.005	<0.02	0.062, 0.064, 0.062, 0.071 [0.065]	0.055, 0.053, 0.056, 0.055 [0.055]
	WI-1	<0.005	<0.02	0.168, 0.172, 0.167, 0.183 [0.173]	0.139, 0.148, 0.138, 0.144 [0.142]
Hulls	NE-1	<0.02	<0.02	0.082, 0.082, 0.081, 0.082 [0.082]	0.052, 0.055, 0.056, 0.054 [0.054]
	WI-1	<0.02	<0.02	0.251, 0.254, 0.271, 0.276 [0.263]	0.136, 0.144, 0.146, 0.143 [0.142]
Toasted defatted meal	NE-1	<0.02	<0.02	0.076, 0.079, 0.074, 0.076 [0.076]	0.067, 0.068, 0.070, 0.072 [0.069]
	WI-1	<0.02	<0.02	0.254, 0.268, 0.253, 0.266 [0.260]	0.185, 0.198, 0.189, 0.193 [0.191]
Degummed oil	NE-1	<0.02	<0.02	<0.02, <0.02, <0.02, <0.02 [<0.02]	<0.02, <0.02, <0.02, <0.02 [<0.02]
	WI-1	<0.02	<0.02	<0.02, <0.02, <0.02, <0.02 [<0.02]	<0.02, <0.02, <0.02, <0.02 [<0.02]
RBD oil	NE-1	<0.02	<0.02	<0.02, <0.02, <0.02, <0.02 [<0.02]	<0.02, <0.02, <0.02, <0.02 [<0.02]

Matrix	Trial	Residues, mg/kg [mean]			
		Dicamba	5-OH Dicamba	DCSA	DCGA
	WI-1	<0.02	<0.02	<0.02, <0.02, <0.02, <0.02 [<0.02]	<0.02, <0.02, <0.02, <0.02 [<0.02]
Crude lecithin	NE-1	<0.2	<0.2	<0.2, <0.2, <0.2, <0.2 [<0.2]	<0.2, <0.2, <0.2, <0.2 [<0.2]
	WI-1	<0.2	<0.2	<0.2, <0.2, <0.2, <0.2 [<0.2]	<0.2, <0.2, <0.2, <0.2 [<0.2]
Defatted flour	NE-1	<0.05	<0.05	0.074, 0.070, 0.071, 0.069 [0.071]	0.068, 0.067, 0.067, 0.066 [0.067]
	WI-1	<0.05	<0.05	0.242, 0.243, 0.239, 0.251 [0.244]	0.187, 0.180, 0.181, 0.178 [0.182]
Protein isolate	NE-1	<0.02	<0.02	<0.02, <0.02, <0.02, <0.02 [<0.02]	<0.02, <0.02, <0.02, <0.02 [<0.02]
	WI-1	<0.02	<0.02	0.029, 0.028 [0.028]	<0.02, <0.02 [<0.02]
Protein concentrate	NE-1	<0.02	<0.02	<0.02, <0.02, <0.02, <0.02 [<0.02]	<0.02, <0.02, <0.02, <0.02 [<0.02]
	WI-1	<0.02	<0.02	<0.02, <0.02, <0.02, <0.02 [<0.02]	<0.02, <0.02, <0.02, <0.02 [<0.02]
Soya milk	NE-1	<0.02	<0.02	<0.02, <0.02, <0.02, <0.02 [<0.02]	<0.02, <0.02, <0.02, <0.02 [<0.02]
	WI-1	<0.02	<0.02	<0.02, <0.02, <0.02, <0.02 [<0.02]	<0.02, <0.02, <0.02, <0.02 [<0.02]
Tofu	NE-1	<0.2	<0.2	<0.2, <0.2, <0.2, <0.2 [<0.2]	<0.2, <0.2, <0.2, <0.2 [<0.2]
	WI-1	<0.2	<0.2	<0.2, <0.2, <0.2, <0.2 [<0.2]	<0.2, <0.2, <0.2, <0.2 [<0.2]

Table 20 Processing factor for residues of dicamba in soya bean matrices (Report MSL0023061)

Commodity	Mean residues, mg/kg		Processing factors [a]	
	Dicamba + total DCSA	Dicamba + 5-OH Dicamba + total DCSA + total DCGA	Dicamba + total DCSA	Dicamba + 5-OH Dicamba + total DCSA + total DCGA
Seed	0.065, 0.173	0.120, 0.315	--	--
Hulls	0.082, 0.263	0.136, 0.405	1.26, 1.52	1.13, 1.29
Toasted defatted meal	0.076, 0.260	0.145, 0.451	1.17, 1.50	1.21, 1.43
Degummed oil	<0.02, <0.02	<0.02, <0.02	<0.31, <0.12	<0.17, <0.06
RBD oil	<0.02, <0.02	<0.02, <0.02	<0.31, <0.12	<0.17, <0.06
Crude lecithin	<0.2, <0.2	<0.2, <0.2	<3.08, <1.16	<1.67, <0.63
Defatted flour	0.071, 0.244	0.138, 0.426	1.09, 1.41	1.15, 1.35
Protein isolate	<0.02, 0.028	<0.02, 0.048	<0.31, 0.16	<0.17, 0.15
Protein conc.	<0.02, <0.02	<0.02, <0.02	<0.31, <0.12	<0.17, <0.06
Soya milk	<0.02, <0.02	<0.02, <0.02	<0.31, <0.12	<0.17, <0.06
Tofu	<0.2, <0.2	<0.2, <0.2	<3.08, <1.16	<1.67, <0.63

[a] Residues of dicamba and 5-OH dicamba are assumed to be zero based on the results of metabolism and field trial data.

Maize

Of the maize field trials described above (Report MSL0026526), two were designated for producing grain for processing into grits, meal, flour starch, and oil. In those trials, maize plants were treated at an exaggerated rate consisting of 5.6 kg ai/ha pre-emergence and 2.8 kg ai/ha each at BBCH 15 and BBCH 18. Maize grain samples were harvested at maturity and transported frozen to the processing facility. Processing was done under simulated commercial practices. Samples were dried to a moisture content of 10–15%. Samples were then cleaned by aspiration and screening.

For dry milling, kernels were adjusted to a moisture content of 21% and then cracked in a disk mill. The resulting stock was dried and then put through a screening process to obtain bran, germ, large grits, grits, meal, and flour. A portion of the germ was heated, flaked, and then submerged three times in hexane to extract the oil. The crude oil was separated from hexane using heated vacuum evaporation. The crude oil was processed with NaOH to produce refined oil and soapstock. Finally, the refined oil was bleached and deodorized.

For wet milling, the grain was steeped in hot water for 22–48 hours, passed through a disc mill, and the resulting germ and hull fractions were separated by water centrifuge. The germ and hull were dried and then separated by aspiration and screening. Starch and gluten were isolated from the processing water by centrifugation. Germ samples were adjusted to a moisture content of 12% and

flaked. The flake was pressed to produce crude oil and presscake; the latter was extracted three times with hexane to liberate the remaining crude oil. Hexane was removed by heated vacuum evaporation and the pressed and extracted crude oil fractions were combined. The crude oil was alkali refined, bleached, and deodorized.

The processed commodities were analysed for residues of dicamba, 5-hydroxydicamba, DCSA, and DCGA using the method described above.

Residues of all four analytes were < 0.01 mg/kg in all samples of grain and processed maize commodities, and processing factors could not be calculated.

Cotton

Of the cotton field trials described above (Report MSL0024072), bulk seed samples were collected from the MO1 and TX4 sites for processing into oil. In those trials cotton plants were treated at rates consisting of either 1.12 kg ai/ha pre-emergence and 0.56 kg each at BBCH 16 and BBCH 60 + 15 days, or 0.56 kg ai/ha each at BBCH 16, BBCH 60 + 15 days, BBCH 80 and BBCH 99 (7 days pre-harvest). Processing was done under simulated commercial practices.

Samples of ginned cotton seed were delinted and cracked to separate the hull from the kernel. Kernels were stored frozen for a maximum of 28 days prior to processing. Kernels were heated and flaked, and the flaked material was processed through an extruder with steam injection. The resulting collets were dried and then extracted three times with hexane. The extract was separated from the meal, which was desolventized and placed into frozen storage. were dried to a moisture content of 10–15%. Samples were then cleaned by aspiration and screening. Crude oil from the extraction of the collets underwent vacuum evaporation to remove the hexane. The crude oil was processed with NaOH to produce refined oil and soapstock. Finally, the refined oil was bleached and deodorized.

The processed commodities were analysed for residues of dicamba, 5-hydroxydicamba, DCSA, and DCGA using the method described above.

Table 21 Residues of dicamba in processed products derived from dicamba-tolerant cotton (Report MSL0024072)

Matrix	Trial	Treatment	Residues, mg/kg			
			Dicamba	5-OH dicamba	DCSA	DCGA
Undelinted Seed	MO1	2[a]	<0.02	<0.01	0.02	<0.005
		4[b]	0.697	<0.01	0.027	0.035
	TX4	2	<0.02	<0.01	<0.005	<0.005
		4	0.602	<0.01	0.048	0.040
Hulls	MO1	2	<0.02	<0.02	0.021	0.022
		4	0.697	<0.02	0.026	0.045
	TX4	2	<0.02	<0.02	0.020	0.013
		4	0.158	<0.02	0.057	0.084
Meal	MO1	2	<0.02	<0.02	<0.02	<0.02
		4	0.217	<0.02	<0.02	0.02
	TX4	2	<0.02	<0.02	0.02	<0.02
		4	<0.02	<0.02	<0.02	<0.02
Alkali refined oil	MO1	2	<0.02	<0.02	<0.02	<0.02
		4	<0.02	<0.02	<0.02	<0.02
	TX4	2	<0.02	<0.02	<0.02	<0.02
		4	<0.02	<0.02	<0.02	<0.02
RBD oil	MO1	2	<0.02	<0.02	<0.02	<0.02
		4	<0.02	<0.02	<0.02	<0.02
	TX4	2	<0.02	<0.02	<0.02	<0.02
		4	<0.02	<0.02	<0.02	<0.02

[a] Treatment 2 (Diglycolamine salt): Pre-emergence at 1.12 kg/ha + BBCH 16 at 0.56 kg/ha + (BBCH 60 + 15 days) at 0.56 kg/ha

[b] Treatment 4 (Diglycolamine salt): BBCH 16 at 0.56 kg/ha + (BBCH 60 + 15 days) at 0.56 kg/ha, + BBCH 80 at 0.56 kg/ha + BBCH 99 at 0.56 kg/ha (7 days pre-harvest)

Since residues from Treatment 2 were <LOQ in all RAC samples of cotton seed, the Meeting calculated processing factors for cotton commodities (Table 22) based on Treatment 4 only.

Table 22 Processing factor for residues of dicamba in cotton seed matrices (Report MSL0024072)

Commodity	Mean residues, mg/kg		Processing factors [mean or best estimate] [a]	
	Dicamba + total DCSA	Dicamba + 5-OH Dicamba + total DCSA + total DCGA	Dicamba + total DCSA	Dicamba + 5-OH Dicamba + total DCSA + total DCGA
Seed	0.724, 0.650	0.759, 0.690	--	--
Hulls	0.723, 0.215	0.768, 0.299	0.999, 0.331 [0.665]	1.012, 0.433 [0.723]
Meal	0.237, <0.04	0.257, <0.06	0.365, <0.062 [0.365]	0.372, <0.087 [0.372]
Alkali refined oil	<0.04, <0.04	<0.06, <0.06	<0.055, <0.062	<0.079, <0.087
RBD oil	<0.04, <0.04	<0.06, <0.06	<0.055, <0.062	<0.079, <0.087

[a] Residues of 5-OH dicamba are assumed to be zero based on the results of metabolism and field trial data.

RESIDUE AND ANALYTICAL ASPECTS

Dicamba is a systemic broad-spectrum herbicide. It was first evaluated by the JMPR in 2010 (T, R). The latest residue evaluation was conducted in 2013 (R).

The 2010 JMPR established an ADI for dicamba of 0–0.3 mg/kg bw and an ARfD of 0.5 mg/kg bw. Also, the following residue definitions based on metabolism studies with conventional crops have been established:

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

Definition of the residue for dietary risk assessment for plant commodities: *sum of dicamba and 5-OH dicamba, expressed as dicamba;*

Definition of the residue for compliance with the MRL and dietary risk assessment for animal commodities: *sum of dicamba and DCSA, expressed as dicamba.*

The residue is not fat soluble.

Dicamba was scheduled at the Fiftieth Session of the CCPR for the evaluation of additional uses by the 2019 Extra JMPR. Studies submitted by the sponsor include nature of the residue studies, field trials, and processing studies in dicamba-tolerant varieties of soya bean, maize, and cotton, and storage stability in soya bean and cotton.

All application rates are expressed as dicamba acid-equivalents.

Plant metabolism

Plant metabolism studies were conducted with dicamba uniformly labelled in the phenyl ring. Treatments were made to dicamba-tolerant varieties of soya bean, maize, and cotton each, either as pre-emergence applications (PRE) on the day of planting or post-emergence of the crop (POE). Tolerance is conveyed by expression of a dicamba mono-oxygenase protein system that oxidizes dicamba to DCSA. The major residue profiles between the two treatment regimens were very similar across all matrices. Since residues were considerably higher following POE treatment than PRE treatment, the Meeting has focused on the POE treatments in its consideration of the plant metabolism studies. Quantifiable levels of radioactivity were observed in the control samples from the metabolism studies. The Meeting is not concerned about this given the interspersing of control plants with treated plants and the metabolism of dicamba to volatile radiolabelled compounds (e.g., $^{14}CO_2$) that could be taken up by the control plants.

In a study illustrating the metabolism of dicamba in dicamba-tolerant soya bean, [phenyl-U-^{14}C]dicamba was applied to greenhouse-grown soya bean either on the day of planting (PRE) or 29 days after planting (DAP) (POE; BBCH 60 (first flowers opened)) at a target rate of 2.8 kg ai/ha.

PRE samples consisted of immature foliage collected 14 DAP (3.2 mg eq/kg TRR), forage harvested 36 DAP (1.4 mg eq/kg), hay harvested 56 DAP (1.1 mg eq/kg) and seed harvested 112 DAP (0.29 mg eq/kg). POE samples were collected as forage 36 DALA (134 mg eq/kg), hay 56 days after last application (DALA) (39 mg eq/kg), and seed 112 DALA (0.39 mg eq/kg).

Extractability of radioactivity into acetonitrile/water solvent was high (>91% TRR) for leafy matrices and lower (ca. 60% TRR) for seeds regardless of the treatment timing. Hexane extracted an additional 11–14% TRR from seeds.

Dicamba was a major predominant residue following POE treatment in forage (24% TRR, 32 mg/kg) and hay (12% TRR, 4.8 mg/kg), but a minor component in seed (0.64% TRR, 0.003 mg/kg). The principal residue in all matrices was DCSA, mostly present as glucoside conjugate. The sum of free DCSA and its conjugates in foliage/hay ranged from 65 to 72% TRR (28-88 mg eq/kg) and represented 25% TRR (0.098 mg eq/kg) in the seed following POE treatment. Other compounds identified in soya bean matrices were conjugated forms of DCGA (1.9–6.7% TRR). In the seeds, a major part of the radioactivity was incorporated into natural products and no dicamba-related compounds occurred at levels exceeding 9.2% TRR or 0.036 mg eq/kg) in the post-extraction solids.

Dicamba-tolerant maize plants grown outdoors were spray treated with [phenyl-U-^{14}C]dicamba at 2.24 kg ai/ha either PRE or POE (30 DAP).

From PRE treatments, immature foliage was harvested 19 DAP (4.5 mg eq/kg), forage 80 DAP (0.075 mg eq/kg), stover 114 DAP (0.24 mg eq/kg), and grain 114 DAP (0.043 mg eq/kg). POE samples consisted of forage 50 DALA (2.2 mg eq/kg) and stover (7.8 mg eq/kg) and grain (0.062 mg eq/kg), each 84 DALA.

Extractability of POE residues into methanol/water was relatively high (>83% TRR) for residues in foliage and low (13% TRR) for residues in grain; hexane extracted an additional 5.3% TRR from grain.

In forage and stover dicamba was a minor residue in all POE matrices, comprising 8.6% TRR (0.19 mg/kg) and 6.3% TRR (0.49 mg/kg), respectively. The principal residue was DCSA, mostly present as glucoside conjugate. The sum of free DCSA and its conjugates represented 42% TRR in foliage (0.94 mg eq/kg) and 40% TRR (3.1 mg eq/kg) in stover. The sum of free and conjugated 5-OH dicamba accounted for <4.5% TRR (0.34 mg/kg). Residues identified as specific DCGA conjugates totalled 3.8% TRR (0.084 mg eq/kg) in forage, 2.5% TRR (0.19 mg eq/kg) in stover, and 0.39% TRR (0.0002 mg eq/kg) in grain; additional DCGA conjugates, totalling 5.6% TRR in forage, 6.8% TRR in stover, and 0.23% TRR in grain, were observed but could not be resolved from DCSA conjugates. Other residues occurred at minor individual levels of < 3.7% TRR (< 0.083 mg eq/kg) in forage and < 5.9% TRR (0.46 mg eq/kg) in stover.

In grain, no extracted residues occurred at >10% TRR or > 0.01 mg eq/kg. The most predominant radioactive residues in grain extracts were sugars (3.1% TRR, 0.0019 mg eq/kg) and natural organic acids (1.1% TRR, 0.0006 mg eq/kg); all other residues were ≤ 0.44% TRR.

Unextracted residues in the PES accounted for 14% TRR in forage, 16% TRR in stover, and 81% TRR in grain. In total, nearly 100% of these residues were comprised of starch, lignin, and phosphate compounds in the foliage and of cellulose, hemicellulose, and starch in the grain.

Following PRE or POE (76 DAP) spray applications of [phenyl-U-^{14}C]dicamba to outdoor grown dicamba-tolerant cotton at 2.24 kg ai/ha., TRR in seed were 0.16 mg eq/kg (PRE) and 0.98 mg eq/kg (POE). TRR in surrogate gin trash (consisting of leaves and stems) were 0.85 mg eq/kg (PRE) and 60 mg eq/kg (POE).

Extractability of POE residues into acetonitrile/water was high for gin trash (71%), but low for seed (38%); an additional 8.8% TRR was extracted from POE cotton seed using hexane.

Dicamba was a minor residue in both seed (0.85% TRR, 0.008 mg/kg) and gin trash (4.5% TRR, 2.7 mg/kg). DCSA glucoside was the predominant residue in both matrices (3.4% TRR, 0.033 mg eq/kg seed; 17% TRR, 10 mg eq/kg gin trash), with free DCSA making up an additional 1.9%

TRR (0.019 mg eq/kg) in seed and 13% TRR (8 mg eq/kg) in gin trash. Sugars accounted for 5.6% TRR in seed and 2.7% TRR in gin trash. All other residues, including DCGA (free and conjugated), were each < 5% TRR for both matrices. Of the 61% TRR in seed PES, approximately two-thirds of the radioactivity was associated with starch, protein, pectin, lignin, cellulose, and hemicellulose. In gin trash PES, approximately 98% of the radioactivity was associated with those natural plant constituents.

Methods of analysis

The analytical methods provided to the meeting were adequately validated for the analysis of dicamba, 5-hydroxydicamba (5-OH dicamba), DCSA, and DCGA in soya bean, maize and cotton matrices. The methods include a hydrolysis step (1 mol/L HCl, 95 °C, 1 or 1.5 hours) that is similar to that used in the metabolism studies (2 mol/L HCl, ca. 100 °C, 2 hours) and adequate to convert conjugated forms of DCSA and DCGA to their free equivalent, which are then determined as the free acid by LC-MS/MS. The LOQs are 0.01 mg/kg or lower in all tested matrices for all analytes except 5-OH dicamba in defatted soya flour (0.05 mg/kg), DCGA in refined soya oil (0.02 mg/kg), and all analytes in cotton hulls, meal, and refined oil (0.02 mg/kg), and gin trash (0.04 mg/kg).

Stability of residues in stored analytical samples

The Meeting received storage stability data for incurred dicamba, DCSA (incl. conjugates) and DCGA (incl. conjugates) in soya bean forage, hay, and seed, and cotton undelinted seed. Samples of soya bean matrices were stored frozen (-10 °C) for approximately 0, 2, 3, 6, 9, 12, 18, and 24 months. Residues of dicamba, DCSA (incl. conjugates) and DCGA (incl. conjugates) were shown to be stable for at least 24 months in all three soya bean matrices, except for DCGA (incl. conjugates) in forage, which was stable up to 3 months. Samples of cotton matrices were stored frozen (-10 °C) for approximately 0, 1, 2, 4, 6, and 9 months. Residues of dicamba, DCSA (incl. conjugates), and DCGA (incl. conjugates) were shown to be stable in cotton undelinted seed for at least 9 months.

Definition of the residue

The 2010 Meeting determined that the definition of the residue for enforcement of dicamba MRLs in conventional crops is dicamba only, noting that DCSA was found only at very low levels. For dietary risk assessment, the 2010 Meeting established a residue definition in plants of the sum of dicamba and 5-OH dicamba, expressed as dicamba.

In all three dicamba-tolerant crops evaluated by the current Meeting, dicamba was a major residue only in soya bean forage and hay and a minor residue in other matrices; in the seeds/grain, dicamba amounted to less than 1% TRR (≤ 0.008 mg/kg). 5-OH dicamba was observed only in maize matrices and only at low levels (free + conjugated < 3.4% in forage, <4.5% TRR in stover, and < 0.15% TRR in grain). The major residue in all matrices was DCSA glucoside, with lesser amounts of free DCSA and other sugar conjugates. In foliage, total DCSA (free + conjugated) accounted for 30 (gin trash) to 72% TRR (soya bean hay). In seeds/grain, total DCSA made up 25% TRR in soya bean, 0.79% TRR in maize grain, and 5.3% TRR in cotton. DCGA was observed only in a conjugated form. Although total DCGA occurred at low total levels (< 6.6% TRR in all matrices), it was a significant portion (13–21%) of the identified residues, especially in seeds and grain.

In supervised field trials on tolerant crops, parent dicamba was generally not present at levels above the LOQ in seeds/grain and only at low levels in forages and hays. Thus, dicamba is not a good marker residue for enforcement in the crops considered by the Meeting. Total DCSA and total DCGA were consistently found at levels >LOQ in field trials, and there was a tendency for total DCSA to be greater than total DCGA. Therefore, the Meeting decided to revise the current residue definition for enforcement of MRLs in soya bean, maize, and cotton commodities to be the sum of dicamba and 3,6-dichloro-2-hydroxybenzoic acid (DCSA; free and conjugated), expressed as dicamba. The Meeting noted that this decision may need to be revisited if other dicamba-tolerant crops are considered by future Meetings.

In considering the residue definition for dietary risk assessment, the Meeting confirmed the conclusion from the 2010 Meeting that DCSA and DCGA were considered to have toxicity similar to

or lower than the parent compound. Total residues of DCSA account for the majority of the residues observed in the dicamba-tolerant crops evaluated by the current Meeting. Consequently, the Meeting decided to include free and conjugated DCSA for dietary risk assessment of soya bean, maize, and cotton commodities. Residue data from field trials indicate that exposure to total DCGA may be similar to that of total DCSA and cannot be excluded from consideration of dietary risk assessment of soya bean, maize, and cotton commodities. Therefore, the Meeting decided that the residue definition for risk assessment in soya bean, maize, and cotton commodities should be revised to the sum of dicamba, 2,5-dichloro-3-hydroxy-6-methoxybenzoic acid (5-OH dicamba), 3,6-dichloro-2-hydroxybenzoic acid (DCSA; free and conjugated) and 2,5-dichloro-3,6-dihydroxybenzoic acid (DCGA; free and conjugated), expressed as dicamba.

Thus, the Meeting agreed to replace the previous definitions for dicamba in plant commodities as follows:

Definition of the residue for compliance with the MRL for soya bean, maize, and cotton commodities: *sum of dicamba and 3,6-dichloro-2-hydroxybenzoic acid (DCSA; free and conjugated), expressed as dicamba;* for other plant commodities: *dicamba*.

Definition of the residue for dietary risk assessment for soya bean, maize, and cotton commodities: *sum of dicamba, 2,5-dichloro-3-hydroxy-6-methoxybenzoic acid (5-OH dicamba), 3,6-dichloro-2-hydroxybenzoic acid (DCSA; free and conjugated) and 2,5-dichloro-3,6-dihydroxybenzoic acid (DCGA; free and conjugated), expressed as dicamba*; for other plant commodities: *sum of dicamba and 5-OH dicamba, expressed as dicamba*.

The residue is not fat-soluble.

The Meeting noted that because of the change to the residue definitions for soya bean, maize, and cotton commodities, all previous recommendations for these commodities needed to be withdrawn and replaced with new recommendations. The changes in definitions of the residue will not influence the numeric values of previous recommendations for conventional crops.

Since the animal dietary burdens are driven by residues arising from the use of dicamba on conventional crops, the definitions for dicamba in animal commodities do not need to be revised.

Results of supervised residue trials on crops

The Meeting received data from supervised residue trials conducted on dicamba-tolerant soya bean, maize, and cotton. All field trials were conducted in the USA, and the results are supported by adequate method and storage stability data, except for DCGA in soya bean forage, for which all samples were stored for approximately 4 to 9 months, which is longer than the demonstrated period of stability of 3 months.

For maximum residue estimation, residues of dicamba or DCSA (free + conjugated) that are <LOQ are assumed to be at the LOQ, and the combined residues are expressed as less than the combined LOQ only when both residues are <LOQ.

For dietary risk estimation, the Meeting noted that in the metabolism studies with dicamba-tolerant crops, dicamba made up 1.9% of the residue definition in soya bean seed and 0.6% in maize grain; furthermore, dicamba residues were reported as <LOQ in all field trial samples of these commodities. Therefore, the Meeting decided that the contribution of dicamba to dietary risk assessment of dicamba-tolerant soya bean seed and maize grain is negligible and could be assumed to be zero. Similarly, 5-OH dicamba was not observed in metabolism studies or field trials in dicamba-tolerant soya bean and cotton commodities and was assumed to be zero; 5-OH dicamba could not be excluded for maize commodities. For the remaining raw commodities considered by the current Meeting, the contribution of dicamba to dietary exposure could not be excluded. Therefore, residues reported as <LOQ in those commodities were assumed to be at the LOQ when deriving total residues for dietary risk assessment.

Estimation of residues for compliance with the MRL and for dietary risk assessment in commodities from dicamba-tolerant varieties for <LOQ residue results

	Residue, mg/kg (reported [assumed])				Combined estimate	
Commodity	Dicamba	5-OH Dicamba	Total DCSA	Total DCGA	Dicamba + DCSA	Dicamba + 5-OH dicamba + DCSA + DCGA
Soya bean seed	<0.005 [0]	<0.02 [0]	<0.005 [0.005]	<0.005 [0.005]	0.005	0.01
Soya bean forage and hay	<0.005 [0.005]	<0.005 [0]	<0.005 [0.005]	<0.005 [0.005]	0.01	0.015
Maize grain	<0.01 [0]	<0.01 [0.01]	<0.01 [0.01]	<0.01 [0.01]	0.01	0.03
Maize forage & stover	<0.01 [0.01]	<0.01 [0.01]	<0.01 [0.01]	<0.01 [0.01]	0.02	0.04
Cotton seed	<0.02 [0.02]	<0.01 [0]	<0.005 [0.005]	<0.005 [0.005]	0.025	0.03
Cotton gin trash	<0.04 [0.04]	<0.04 [0]	<0.04 [0.04]	<0.04 [0.04]	0.08	0.12

Soya bean

The critical GAP is from the registration in the USA (one pre-emergence application at 1.12 kg ai/ha and up to two post-emergence applications at least 7 days apart, each at 0.56 kg ai/ha; last application no later than BBCH 60).

Five field trials matching the critical GAP with respect to both application rate and retreatment interval are available. An additional 17 trials were provided that match the GAP for application rate but not for retreatment interval. The meeting noted that for soya bean seed, the retreatment interval used in the submitted studies (6 to 29 days) does not appear to have a significant impact on residues. Therefore, the Meeting decided to consider all trials approximating the critical GAP with respect to application rate. On that basis, there are 22 trials suitable for making residue estimates.

Residues of dicamba in soya bean seed from independent trials for estimation of maximum residues were (n=22): 0.016 (2), 0.018 (2), 0.019, 0.024, 0.026, 0.027, 0.028, 0.032, 0.036, 0.045, 0.048, 0.053, 0.054, 0.060, 0.063, 0.077, 0.082, 0.097, 0.12, and 0.44 mg/kg.

Residues of dicamba in soya bean seed from independent trials for estimation of dietary risk were (n=22): 0.021, 0.025, 0.028, 0.029, 0.030, 0.031, 0.032, 0.037, 0.046 (2), 0.051, 0.056, 0.058, 0.074, 0.087, 0.099, 0.11, 0.12, 0.14, 0.15, 0.17, and 0.56 mg/kg.

The previous recommendation for soya bean (dry) is 10 mg/kg and was derived from a pre-harvest desiccation GAP. As this value accommodates residues in dicamba-tolerant soya bean seeds, the Meeting withdrew the previous maximum residue level recommendation of 10 mg/kg and made a new recommendation of 10 mg/kg for soya bean seed (dry) according to the new residue definition. The Meeting estimated a STMR of 0.0535 mg/kg.

Maize

The critical GAP is from the registration in Canada (one pre-emergent application at 0.58 kg ai/ha and one post-emergent application at 0.6 kg ai/ha with a 30-day PHI). No trials available to the Meeting matched the Canadian GAP.

The Meeting withdrew the previous maximum residue level recommendation of 0.01(*) mg/kg and made a new recommendation of 0.01(*) mg/kg for maize according to the new residue definition. The Meeting confirmed the STMR of 0.02 mg/kg estimated by the 2010 JMPR.

Cotton

The critical GAP is from the registration in the USA (one pre-emergence application at 1.12 kg ai/ha and up to two post-emergence applications at least 7 days apart, each at 0.56 kg ai/ha; PHI of 7 days).

Two field trials matching the critical GAP with respect to application rate, retreatment interval, and PHI are available. An additional 11 trials are available that match the rate and PHI but not the retreatment interval. The meeting noted that for cotton seed, the retreatment interval used in the submitted studies (5 to 63 days) does not appear to have a significant impact on residues of dicamba for estimation of residues or for dietary risk assessment. Therefore, the Meeting decided to consider all trials approximating the critical GAP with respect to application rate and PHI. On that basis, there are 13 trials suitable for making residue estimates.

Residues of dicamba in cotton undelinted seed from independent trials for estimation of maximum residues were (n=13): 0.07, 0.13, 0.20, 0.34, 0.40, 0.43, 0.69, 0.98, 1.0, 1.1 (2), 1.2, and 1.6 mg/kg.

Residues of dicamba in cotton undelinted seed from independent trials for estimation of dietary risk were (n=13): 0.075, 0.15, 0.20. 0.38, 0.42, 0.43, 0.69, 1.0 (2), 1.1 (2), 1.3, and 1.6 mg/kg.

The Meeting withdrew the previous maximum residue level recommendation of 0.04 * mg/kg and made a new recommendation of 3 mg/kg for cotton seed according to the new residue definition. The Meeting estimated a STMR of 0.69 mg/kg.

Animal feedstuffs

Soya bean forage and hay

The critical GAP is from the registration in the USA (one pre-emergence application at 1.12 kg ai/ha and up to two post-emergence applications at least 7 days apart, each at 0.56 kg ai/ha; PHI = 7 days for forage and 14 days for hay).

The meeting noted that for soya bean forage, the samples were stored for 4 to 9.5 months, which is longer than the period of demonstrated stability (3 months). The Meeting decided that the soya bean forage data could not be used to estimate residues.

Four trials are available approximating the critical GAP for soya bean hay with respect to use pattern and harvest 14 DALA. Although specific residue decline data are not available for soya bean hay, the Meeting noted a tendency for higher residues from trials with harvest >14 DALA than those at 14 DALA. Therefore, the Meeting agreed to consider all trials that were harvested 14–24 DALA for estimating residues.

Residues of dicamba in soya bean hay (as received) from independent trials for estimation of maximum residues were (n=22): 13, 15, *21*, 21, 22, 24, 27, *30*, 30, 32 (3), 34 (2), 35, *36*, 36, *38*, 44, 47 (2), and 61 mg/kg (residues from at-GAP trials in italics).

Residues of dicamba in soya bean hay (as received) from independent trials for estimation of dietary burden were (n=22): 13, 16, 22, 23, *24*, 30, 31, *32*, 32, 34, 35 (2), 36, *37*, 37, 38, 40, *42*, 47, 48, 49, and 68 mg/kg.

The Meeting estimated a maximum residue level for soya bean fodder (dry) of 150 mg/kg (dw; based on a dry matter content of 85% from the OECD feed table), a median residue of 35 mg/kg in hay (as received), and a highest residue of 68 mg/kg (as received).

Maize forage and fodder

The critical GAP is from the registration in Canada (one pre-emergent application at 0.58 kg ai/ha and one post-emergent application at 0.6 kg ai/ha with a 30-day PHI). As noted above under maize grain, no trials matching the critical GAP were available to the Meeting.

Meeting withdrew the previous maximum residue level recommendation of 0.6 mg/kg and made a new recommendation of 0.6 mg/kg (dry weight) for maize fodder (dry) according to the new residue definition. The Meeting confirmed the median residue of 0.06 mg/kg (as received) and highest residue of 0.33 mg/kg (as received) for maize fodder as well as the median residue of 0.16 mg/kg (as received) and highest residue of 0.31 mg/kg (as received) for maize forage estimated by the 2010 JMPR.

Cotton gin trash

The critical GAP is from the registration in the USA (one pre-emergence application at 1.12 kg ai/ha and up to two post-emergence applications at least 7 days apart, each at 0.56 kg ai/ha; PHI of 7 days).

The meeting noted that for cotton gin trash, there appears to be a strong trend for lower residues at increased retreatment intervals used in the submitted studies (30 to 49 days. Therefore, the Meeting decided that the submitted trials for cotton gin trash were not suitable for estimating residues.

Fate of residues during processing

Residues after processing

The Meeting received data depicting the concentration/dilution of residues during processing of soya bean seed, maize grain, and undelinted cotton seed from dicamba-tolerant crops. For all crops, processed commodities were derived using simulated commercial practices. The resulting processing factors and STMR-P estimates for dicamba-tolerant varieties of soya bean and cotton are summarized below; residues were <LOQ in all maize RAC and processed commodity samples.

Raw agricultural commodity	Processed commodity	Processing factors [median/best estimate]		MRL, mg/kg	STMR-P, mg/kg
		Dicamba + DCSA	Dicamba + DCSA + DCGA [a)]		
Soya bean seed MRL = 10 mg/kg STMR = 0.054 mg/kg	Hulls	1.26, 1.52 [1.39]	1.13, 1.29 [1.21]	15	0.065
	Meal	1.17, 1.50 [1.34]	1.21, 1.43 [1.32]	15	0.071
	RBD oil [b)]	<0.31, <0.12	<0.17, <0.06 [0.06]	--	0.0032
	Soya milk	<0.31, <0.12	<0.17, <0.06 [0.06]	--	0.0032
	Tofu	<3.08, <1.16	<1.67, <0.63 [0.63]	--	0.0034
Cotton undelinted seed MRL = 3 mg/kg STMR = 0.69 mg/kg	Hulls	0.999, 0.331 [0.665]	1.01, 0.433 [0.723]	--	0.50
	Meal	0.365, <0.062 [0.365]	0.372, <0.087 [0.372]	--	0.26
	RBD oil [b]	<0.055, <0.062	<0.079, <0.087 [0.079]	--	0.055

a) Residues of 5-OH dicamba were assumed to be zero based on results from metabolism, field trials, and processing studies.

b) Refined, bleached and deodorized

Residues in animal commodities

Estimated maximum and mean dietary burdens of livestock

Dietary burden estimates from the 2010 Meeting have been recalculated to include contributions from commodities grown from dicamba-tolerant soya bean, maize, and cotton considered by the current Meeting. Estimated dietary burdens for Australia, the EU, Japan, and Canada/USA are summarized below. The livestock diets are listed in Annex 6.

Livestock Dietary Burdens (ppm of dry matter diet) for dicamba.

Livestock	Australia		EU		Japan		Canada/USA	
	Max	Mean	Max	Mean	Max	Mean	Max	Mean
Cattle (beef)	140 [A)]	45 [C)]	77	23	15	5.5	5.7	2.6
Cattle (dairy)	140 [B)]	45 [D)]	85	27	29	8.7	84	30
Poultry (broiler)	1.0	1.0	1.3	1.3	0.86	0.86	1.4	1.4
Poultry (layer)	1.0	1.0	13 [E)]	6.3 [F)]	0.74	0.74	1.4	1.4

A) Highest maximum dietary burden for beef or dairy cattle; suitable for estimating the maximum residue levels for mammalian meat, fat, and offal.

B) Highest maximum dietary burden for dairy cattle; suitable for estimating the maximum residue levels for milk.

C) Highest mean dietary burden for beef or dairy cattle; suitable for estimating STMRs for mammalian meat, fat, and

offal.

D) Highest mean dietary burden for dairy cattle; suitable for estimating the STMR for milk.

E) Highest maximum dietary burden for broiler chickens or laying hens; suitable for estimating the maximum residue levels for poultry meat, fat, offal, and eggs.

F) Highest mean dietary burden for laying hens; suitable for estimating the STMRs for poultry meat, fat, offal, and eggs.

Animal commodity maximum residue levels

The Meeting noted that the dietary burdens for cattle and poultry remain essentially unchanged compared to those derived in 2010 (maximum and mean burdens were 140 and 44 ppm for cattle and 15.6 and 6.0 ppm for poultry; poultry burdens are lower in this assessment due to the removal of some commodities from the current OECD poultry diets); therefore, 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 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 soya bean, maize, and cotton: *sum of dicamba and 3,6-dichloro-2-hydroxybenzoic acid (DCSA; free and conjugated), expressed as dicamba;* for other plant commodities: *dicamba*.

Definition of the residue for dietary risk assessment for soya bean, maize, and cotton: *sum of dicamba, 2,5-dichloro-3-hydroxy-6-methoxybenzoic acid (5-OH dicamba), 3,6-dichloro-2-hydroxybenzoic acid (DCSA; free and conjugated) and 2,5-dichloro-3,6-dihydroxybenzoic acid (DCGA; free and conjugated), expressed as dicamba*; for other plant commodities: *sum of dicamba and 5-OH dicamba, expressed as dicamba*.

Definition of the residue for compliance with the MRL and for estimation of dietary exposure for animal commodities: *sum of dicamba and DCSA, expressed as dicamba.*

The residue is not fat-soluble.

Commodity		Recommended MRL, mg/kg		STMR or STMR-P, mg/kg	Highest Residue mg/kg
CCN	Name	CCN	Previous		
VD 0541	Soya bean (dry)	W [a)]	10		
VD 0541	Soya bean (dry)	10 [a)]	--	0.0535	--
GC 0645	Maize	W [a)]	0.01*		
GC 0645	Maize	0.01* [a)]	--	0.02 [b)]	--
SO 0691	Cotton seed	W [A)]	0.04*		
SO 0691	Cotton seed	3	--	0.69	--
AL 0541	Soya bean fodder (dry)	150 (dw)	--	35 (as)	68 (as)
AB 0541	Soya bean hulls	15	--	0.065	--
AB 1265	Soya bean meal	15	--	0.071	--
AS 0645	Maize fodder (dry)	W [a)]	0.6 (dw)		
AS 0645	Maize fodder (dry)	0.6 (dw) [a)]	--	0.06 (dw) [b)]	0.33 (dw) [b)]
	Soya bean oil	--		0.0032	
	Soya milk	--		0.0032	
	Tofu	--		0.0034	
	Cotton seed oil	--		0.055	
	Maize oil, crude			0.00058 [b)]	
	Cotton seed meal	--		Median: 0.26	
	Maize forage			Median: 0.40 (dw) [b)]	Highest: 0.775 (dw) [b)]

[a)] To withdraw the previous recommendation and replace it with a new one at the same level based on a new residue definition for compliance with the MRL.

[b)] Recommended by 2010 JMPR based on conventional maize

(as) – as received; (dw) – dry weight

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for dicamba is 0–0.3 mg/kg bw. The International Estimated Daily Intakes (IEDIs) for dicamba 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 2019 Extra JMPR Report.

The IEDIs ranged from 0–1% of the maximum ADI. The Meeting concluded that long-term dietary exposure to residues of dicamba from uses considered by the JMPR is unlikely to present a public health concern.

Acute dietary exposure

The ARfD for dicamba is 0.5 mg/kg bw. The International Estimate of Short-Term Intakes (IESTIs) for dicamba 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 2019 Extra JMPR Report.

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

REFERENCES

Report	Author	Year	Title
MSL0022659	Miller, M.J. and Mierkowski, M.	2010	Amended Report for MSL20227: Metabolism of Dicamba in Dicamba-Tolerant Soybeans
MSL0022661	Foster, J.E., Miller, M.J., and Mierkowski, M.	2010	Amended Report for MSL0022390 and MSL0022582: Analytical Method for the Determination of Dicamba and Its Major Metabolites in Soy Matrices by LC/MS/MS
MSL0023058	Maher, D and Foster, J.E.	2012	Determination of the Stability of Dicamba Residues in Dicamba-Tolerant Cotton under Frozen Storage Conditions
MSL0023061	Moran, S.J. and Foster, J.E.	2010	Amended Report for MSL0022660: Magnitude of Residues of Dicamba in Soybean Raw Agricultural and Processed Commodities after Application to MON 87708
MSL0023759	Foster, J.E., Mierkowski, M., and Miller, M.J.	2011	Amended Report for MSL0023267: Analytical Method for the Determination of Dicamba and Its Major Metabolites in Cotton Matrices by LC/MS/MS
MSL0023760	Whitehead, T., Mierkowski, M., and Chott, R.	2011	Amended Report for MSL0021858: Metabolism of ^{14}C-Dicamba in Dicamba-Tolerant Cotton
MSL0024072	Maher, D and Foster, J.E.	2011	Amended Report for MSL0022663: Magnitude of Dicamba Residues in Cotton Raw Agricultural and Processed Commodities Following Applications of Dicamba-Based Formulations to MON 88701. 2010 U.S. Trials
MSL0025703	Adio, A.M. and Feng, X.	2015	Nature of ^{14}C-Dicamba Residues in Corn Raw Agricultural Commodities Following Pre-emergence or Post-emergence Application to Dicamba-Glufosinate Tolerant Corn
MSL0026344	Riter, L.S. Wujcik, C.E., and Jensen, P.K.	2015	Analytical Method for the Determination of Dicamba and Major Metabolites in Raw Agricultural Commodities by LC-MS/MS
MSL0026526	Urbanczyk-Wochniak, E.	2015	Amended from MSL0026331, Magnitude of Dicamba Residues in Corn Raw Agricultural and Processed Commodities Following Applications of a Dicamba-Based Formulation to Dicamba Glufosinate Tolerant Corn. 2013 U.S. Trials
MSL0027420	Mueth, M.G. and Foster, J.	2012	Amended from MSL0023813, Determination of the Stability of Dicamba and its Major Endogenous Metabolites in Dicamba-Tolerant Soybean MON 87708 × MON 89788 under Frozen Storage Conditions

FENAZAQUIN (297)

First draft prepared by Dr M Doherty, Environmental Protection Agency, Washington, USA

EXPLANATION

Fenazaquin is a quinazoline insecticide/acaricide that exhibits contact and ovicidal activity against a broad spectrum of mites in grapes, pome fruit, citrus, peaches, cucurbits, tomatoes, cotton and ornamentals. It was first evaluated by JMPR in 2017 for toxicology and residues.

The 2017 JMPR established an ADI for fenazaquin of 0–0.05 mg/kg bw and an ARfD of 0.1 mg/kg bw. The residue definition for compliance with the MRL and dietary risk assessment for plant commodities is parent fenazaquin. As listed in the 2017 Report, the residue definition for compliance with the MRL and dietary risk assessment for animal commodities is the sum of fenazaquin and the metabolites 2-(4-{2-[(2-hydroxyquinazolin-4-yl)oxy]ethyl}phenyl)-2-methylpropanoic acid (2-hydroxy-fenazaquin acid) and quinazolin-4-ol and 3,4-dihydroquinazolin-4-one (4-hydroxyquinazoline), expressed as fenazaquin equivalents. The residue is fat soluble.

The 2017 Meeting determined that the submitted storage stability data were inadequate to support the recommendation of a maximum residue level for almonds. At the Fiftieth Session of the CCPR (2018), fenazaquin was scheduled for a follow-up evaluation of additional uses by the 2019 Extra JMPR. The Meeting received additional storage stability and residue trial data for almond, an analytical method for fenazaquin residues in animal commodities, and a cattle-feeding study.

In this document, values in text are rounded to two significant figures; values in tables are generally presented to the level of precision provided by the sponsor.

RESIDUE ANALYSIS

Analytical Methods

No new methods of analysis of fenazaquin residues in plants were submitted to the Meeting. The analytical method used in the storage stability study and the crop field trials was ANADIAG R A4167, which is the same LC-MS/MS method that was reviewed and deemed acceptable by the 2017 Meeting for analysis of residues in several plant commodities. Validation data for almond nutmeat and almond shells (S. Carringer, 2015, Report TCI-12-349) submitted to the current Meeting are summarized in Table 1 (fenazaquin) and Table 2 (*p*-tert-butylphenylethanol (TBPE) and 4-hydroxyquinazoline (4-OHQ) metabolites). The Meeting noted that the shell is the fibrous material between the nutmeat and the hull, not the hull itself.

Table 1 Method validation for residues of fenazaquin in almond commodities

Matrix	Fortified Level (mg/kg)	Sample Size (n)	Recoveries (%)	Mean Recovery (%) ± std. dev.	RSD (%)
Almond Nutmeat	0.01	5	88, 85, 80, 83, 81	83 ± 3.2	3.8
	0.50	5	78, 72, 73, 70, 76	74 ± 3.2	4.3
Almond Shells	0.01	5	80, 97, 88, 87, 102	91 ± 8.7	9.6
	0.50	5	92, 86, 90, 84, 82	87 ± 4.1	4.8

Table 2 Method validation for residues of metabolites TBPE and 4-OHQ in almond commodities

Matrix	Analyte	Fortified Level (ppm)	Sample Size (n)	Recoveries (%)	Mean Recovery (%)	RSD (%)
Almond Nutmeat	TBPE	0.01	5	93, 84, 71, 83, 70	80	12
		0.10	5	84, 86, 91, 77, 90	86	6.5
	4-OHQ	0.01	5	103, 114, 105, 116, 102	108	6.0
		0.10	5	90, 94, 94, 92, 90	92	2.2

Matrix	Analyte	Fortified Level (ppm)	Sample Size (n)	Recoveries (%)	Mean Recovery (%)	RSD (%)
Almond Shells	TBPE	0.01	5	94, 90, 89, 100, 100	95	5.6
		0.50	5	75, 73, 78, 73, 81	76	4.6
	4-OHQ	0.01	5	88, 93, 86, 90, 85	88	3.6
		0.50	5	80, 84, 83, 82, 80	82	2.2

The Meeting received a cattle-feeding study with a description and validation of a method for the analysis of fenazaquin in milk, kidney, liver, and muscle, and 2-hydroxyfenazaquin acid in kidney, liver, and muscle (L. Ferguson, 2015, Report 029280). The method consists of extraction of residues sequentially into acetone and acetonitrile:water (4:1, v/v). Extracts are cleaned-up by solid-phase extraction on HLB sorbent and then residues were analysed by LC-MS/MS.

Table 3 Recovery of fenazaquin and 2-hydroxyfenazaquin acid from cattle matrices in the livestock feeding study

			Recovery, %			
			Primary (*m/z* 307.0→161.2)		Confirmatory (*m/z* 307.0→147.2)	
Analyte	Matrix	Fortification, mg/kg	Individual	Mean ± RSD	Individual	Mean ± RSD
Fenazaquin	Milk	0.01	73.5, 83.3, 77.5	78 ± 6.3	71.8, 81.8, 70.4	75 ± 8.3
		0.2	76.2, 78.5, 83.9	80 ± 5.0	77.2, 78.4, 82.4	79 ± 3.4
		2	78.6, 84.0, 81.3	81 ± 3.3	80.9, 85.4, 81.5	83 ± 2.9
	Liver	0.01	92.3, 95.3, 93.0	94 ± 1.7	94.5, 87.9, 92.3	92 ± 3.7
		0.2	87.9, 86.4, 87.7	87 ± 0.92	88.3, 87.9, 86.4	88 ± 1.1
		2	84.5, 87.0, 86.8	86 ± 1.6	86.6, 86.0, 87.5	87 ± 0.92
	Kidney	0.01	80.1, 81.7, 88.0	83 ± 5.0	79.1, 84.8, 81.8	82 ± 3.5
		0.2	84.8, 127.0, 85.4	99 ± 24	82.3, 128.0, 82.0	97 ± 27
		2	86.0, 83.2, 82.6	84 ± 2.1	86.3, 85.5, 82.3	85 ± 2.5
	Muscle	0.01	85.9, 88.3, 88.7	88 ± 1.7	81.0, 90.2, 85.4	86 ± 5.4
		0.2	83.1, 85.0, 83.1	84 ± 1.3	82.2, 83.8, 83.0	83 ± 0.96
		2	81.1, 83.8, 83.2	83 ± 1.7	80.6, 83.5, 81.9	82 ± 1.8
	Fat	0.01	85.2, 86.3, 89.2	87 ± 2.4	84.3, 97.6, 90.0	91 ± 7.4
		0.2	74.9, 68.6, 83.2	76 ± 9.7	74.1, 69.5, 82.9	76 ± 9.0
		2	79.3, 82.5, 82.6	82 ± 2.3	78.0, 82.2, 79.0	80 ± 2.8
2-OH-Fenazaquin acid	Liver	0.01	83.0, 84.8, 80.2	83 ± 2.8	90.5, 85.8, 76.4	84 ± 8.6
		0.2	82.8, 77.9, 93.2	85 ± 9.2	82.4, 76.8, 93.1	84 ± 9.9
		2	86.0, 91.8, 93.2	90 ± 4.2	85.8, 92.7, 91.9	90 ± 4.2
	Kidney	0.01	85.9, 77.5, 84.0	82 ± 5.3	83.7, 74.2, 84.8	81 ± 7.2
		0.2	70.4, 73.6, 81.9	75 ± 7.8	72.1, 75.1, 83.7	77 ± 7.8
		2	70.2, 82.2, 82.3	78 ± 9.0	71.0, 82.9, 82.3	79 ± 8.5
	Muscle	0.01	92.4, 93.2, 96.2	94 ± 2.1	97.9, 93.9, 99.4	97 ± 2.9
		0.2	97.2, 96.6, 92.9	96 ± 2.4	96.8, 95.0, 90.4	94 ± 3.5
		2	90.7, 103.0, 89.2	94 ± 8.1	92.2, 103.0, 88.2	94 ± 8.1

The methods described above are suitable for the analysis of fenazaquin, TBPE, and 4-OHQ in almond commodities and for fenazaquin and 2-hydroxyfenazaquin acid in animal matrices. Recovery of analytes consistently fell within the range of 70 to 120% and relative standard deviations were less than 20%.

Stability of pesticide residues in stored analytical samples

A new storage stability study on almond was submitted to the Meeting. In the study (Report TCI-12-349, Carringer, 2015), almond nutmeats and shells were fortified with fenazaquin, TBPE, or 4-OHQ at 0.5 mg/kg and placed into frozen storage (-25 to -10 °C) for approximately 1 and 8 (TBPE and 4-OHQ) or 17 (fenazaquin) months prior to analysis by the method cited above; 0-day samples were also analysed. Results from storage stability samples are summarized in Table 4.

Table 4 Summary of storage stability results for almond commodities submitted to the current Meeting. Fortification level = 0.5 mg/kg.

Analyte	Matrix	Storage time, months	Residues remaining, mg/kg		% residues remaining, mg/kg	Average % remaining (normalized to 0-day)
			Procedural sample	Stored sample		
Fenazaquin	Nutmeat	0	--	0.392, 0.362, 0.364, 0.350, 0.378	78, 72, 73, 70, 76	100
		1	0.374, 0.360	0.362, 0.366	72, 73	99
		17	0.427, 0.442	0.396, 0.398	79, 80	108
	Shells	0	--	0.458, 0.432, 0.448, 0.418, 0.408	92, 86, 90, 84, 82	100
		1	0.370, 0.348	0.320, 0.302	64, 60	72
		17	0.464, 0.455	0.502, 0.495	100, 99	115
TBPE	Nutmeat	0	--	0.422, 0.455	84, 91	100
		1	0.387, 0.424	0.425, 0.504	85, 101	106
		8	0.460, 0.418	0.399, 0.446	80, 89	96
	Shells	0	--	0.348, 0.364	70, 73	100
		1	0.402, 0.438	0.393, 0.462	79, 92	120
4-OHQ	Nutmeat	0	--	0.446, 0.453	89, 91	100
		1	0.476, 0.520	0.541, 0.520	108, 104	118
		8	0.488, 0.484	0.490, 0.491	98, 98	109
	Shells	0	--	0.425, 0.441	85, 88	100
		1	0.452, 0.464	0.454, 0.424	91, 85	101

The Meeting received new storage stability data as part of the cattle feeding study. Control matrices were fortified at 0.2 mg/kg fenazaquin and stored frozen (ca. -20 °C) for 0, 0.5, 1, 2, and 4 months; data on the stability of 2-OH fenazaquin acid were not provided. Residues of fenazaquin were determined using the method described above. Concurrent recoveries at 0.2 mg/kg ranged from 79 to 98%, with most recoveries being >80%. Residues of fenazaquin in the storage stability samples are summarized in Table 5.

Table 5 Stability of fenazaquin in animal matrices during frozen storage.

Matrix	Storage time, months	Mean concurrent recovery, %	Fenazaquin, mg/kg	Mean fenazaquin, mg/kg	Mean % remaining
Milk	0	81	0.166, 0.160, 0.162	0.163	81
	0.5	89	0.173, 0.177, 0.173	0.174	87
	1	88	0.184, 0.178, 0.172	0.178	89
	2	85	0.170, 0.172, 0.176	0.173	86
	4	87	0.166, 0.170, 0.171	0.169	84
Liver	0	88	0.189, 0.179, 0.185	0.184	92
	0.5	91	0.169, 0.174, 0.169	0.171	85
	1	82	0.168, 0.161, 0.161	0.163	82
	2	92	0.168, 0.156, 0.160	0.161	81
	4	98	0.160, 0.149, 0.156	0.155	77
Kidney	0	89	0.181, 0.189, 0.184	0.185	92
	0.5	80	0.147, 0.155, 0.139	0.147	74
	1	86	0.165, 0.160, 0.168	0.164	82
	2	84	0.170, 0.168, 0.160	0.166	83
	4	83	0.161, 0.168, 0.160	0.163	82
Muscle	0	85	0.170, 0.172, 0.176	0.173	86

Matrix	Storage time, months	Mean concurrent recovery, %	Fenazaquin, mg/kg	Mean fenazaquin, mg/kg	Mean % remaining
	0.5	88	0.184, 0.180, 0.181	0.182	91
	1	92	0.183, 0.172, 0.180	0.178	89
	2	92	0.174, 0.186, 0.181	0.180	90
	4	94	0.176, 0.181, 0.180	0.179	90
Fat	0	79	0.154, 0.161, 0.155	0.157	78
	0.5	87	0.180, 0.174, 0.174	0.176	88
	1	86	0.166, 0.172, 0.173	0.171	85
	2	84	0.166, 0.170, 0.169	0.168	84
	4	91	0.175, 0.176, 0.174	0.175	87

USE PATTERN

Information on the registered uses of fenazaquin was provided to the 2017 Meeting. Information relevant to almond is presented below (Table 6).

Table 5 Registered use of fenazaquin on almond

Country	End-use product	Application			Application rate per treatment			PHI (days)
		Method	No. per crop	Application interval (days)	kg ai/hL max	Water L/ha	kg ai/ha max	
TREE NUTS								
USA a	Soluble concentrate	Broadcast ground spray/ airblast	1	N/A	Not specified	935 (minimum recommended)	0.504	7

[a] In the USA, the registration is for US Crop Group 14-12, which includes African nut-tree, almond, beechnut, Brazil nut, Brazilian pine, bunya, bur oak, butternut, cajou nut, candlenut, cashew, chestnut, chinquapin, coconut, coquito nut, dika nut, ginkgo, Guiana chestnut, hazelnut (filbert), heartnut, hickory nut, Japanese horsechestnut, macadamia nut, mongongo nut, monkey-pot, monkey puzzle nut, okari nut, pachira nut, peach palm nut, pecan, pequi, pili nut, pine nut, pistachio, sapucaia nut, tropical almond, black walnut, English walnut, and yellowhorn.

RESULTS OF SUPERVISED RESIDUE TRIALS ON CROPS

The Meeting received data from supervised residue trials conducted on almond.

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 sample sizes, example chromatograms and example calculations. Samples were analysed by the method described above.

All harvested commodities were maintained whole in the field and not homogenized until they reached the analytical laboratory.

In the summary tables, values used for making maximum residue level recommendations are underlined. Where there is more than one sample per field trial, the highest individual values for estimating dietary intake are bolded. When calculating averages, residues reported as <0.01 mg/kg were assumed to be at 0.01 mg/kg. Analytical results described in the text are rounded to two significant figures according to ISO standards; values in tables appear as reported by the sponsor. In the summary tables, residue values leading to maximum residue estimations are double underlined, residues used for dietary risk estimation are underlined, and the highest individual values selected for estimating dietary exposure are bolded.

The field trial study designs included control plots. Data from field trials evaluated by the 2017 meeting are summarized separately from studies evaluated by the current Meeting.

Supervised trials for fenazaquin:

Category	Crop	Table
Tree nuts	Almond nutmeat (TN 0660)	7
	Almond and pecan nutmeat (TN 0660 and TN 0672) from 2017 JMPR	8

Tree nuts

Almond

Four trials were conducted in the USA during the 2012 growing season (S. Carringer, 2015, Study Report TCI-12-349). At each location, one control and one treated plot were established. The treated plot received a single application of fenazaquin by airblast sprayer as a soluble concentrate. The nominal application rate was 0.5 kg ai/ha; applications were made in 524 to 627 L/ha for concentrated sprays and in 1412–1543 L/ha for dilute sprays. All trials included a non-ionic surfactant in the tank mix. Samples consisted of at least 1 kg of tree nuts and 0.3 to 1.1 kg for shells. Samples were placed into freezers within 3.5 hours of harvest and were maintained frozen until analysis. Samples were homogenized in the presence of dry ice at the analytical facility. Average concurrent recoveries across all three analytes and across fortifications of 0.01 and 0.5 ranged from 87 to 121% with standard deviations ranging from 2.1 to 14%. Almond nutmeat samples were in frozen storage for a maximum of 438 days prior to analysis for fenazaquin and for a maximum of 605 days prior to analysis of TBPE and 4-OHQ. Since almond shells are neither a human food nor a livestock feed, residue levels have not been reported herein.

Table 7 Results of 2012 fenazaquin residue trials in almond provided to the current Meeting

Report Trial No. Location	Crop (Variety)	Application			Matrix	DALA	Residues [mean] (mg/kg)		
		No.	L/ha	kg ai/ha			Fenazaquin	TBPE	4-OHQ
Critical GAP [US]	Tree nuts	1	935 [a]	0.504	--	7	--	--	--
TCI-12-349 01 [b] Terra Bella, CA, US	Almond (Nonpareil)	1	627	0.511	Nutmeat	7	<0.01, <0.01 [<u>≤0.01</u>]	ND, ND [ND]	ND, ND [ND]
TCI-12-349 04 [b] Strathmore, CA, US	Almond (Fritz)	1	1543	0.506	Nutmeat	7	<0.01, <0.01 [<u>≤0.01</u>]	ND, ND [ND]	ND, ND [ND]
TCI-12-349 02 Wasco, CA, US	Almond (Non Pareil)	1	524	0.512	Nutmeat	7	<0.01, <0.01 [<u>≤0.01</u>]	ND, ND [ND]	ND, ND [ND]
TCI-12-349 03 Fresno, CA, US	Almond (Butte)	1	1412	0.507	Nutmeat	7	<0.01, 0.0113 [<u>0.011</u>]	ND, ND [ND]	ND, ND [ND]

[a] Minimum recommended on the label; not specified as part of GAP.

[b] Separated by approx. 20 km; application and harvest dates are offset by approximately 30 days

Almond and Pecan (Evaluated by 2017 Meeting)

Residues from field trials with almond and pecan evaluated by the 2017 Meeting are provided in Table 8.

Table 8 Summary of 2008 field trials residues in almond and pecan reviewed by the 2017 Meeting

Report Trial No. Location	Crop (Variety)	Application			Matrix	DALA	Residues [mean] (mg/kg) Fenazaquin
		No.	L/ha	kg ai/ha			
Critical GAP [USA]	Tree nuts	1	935	0.504	--	7	--
TCI-08-219 06 [a] Terra Bella, CA, US	Almond (Nonpareil)	1	571	0.500	Nutmeat	1	0.047, 0.023 [0.035]
						7	<0.01, 0.012 [0.011]
						14	<0.01, <0.01 [<0.01]
						21	**0.016**, <0.01 [0.013]
TCI-08-219 09 [a] Strathmore, CA, US	Almond (Nonpareil)	1	1104	0.520	Nutmeat	7	<0.01, <0.01 [<0.01]
TCI-08-219 07 Dinuba, CA, US	Almond (Carmel)	1	1151	0.490	Nutmeat	7	<0.01, <0.01 [<0.01]
TCI-08-219 08 [b] Wasco, CA, US	Almond (Price)	1	655	0.500	Nutmeat	7	<0.01, <0.01 [<0.01]
TCI-08-219 10 [b] Wasco, CA, US	Almond (Nonpareil)	1	589	0.530	Nutmeat	7	<0.01, <0.01 [<0.01]
TCI-08-219 01 Girard, GA, US	Pecan (Desirables)	1	963	0.520	Nutmeat	6	<0.01, <0.01 [<0.01]
TCI-08-219 02 Montezuma, GA, US	Pecan (Money Makers)	1	1600	0.500	Nutmeat	7	<0.01, <0.01 [<0.01]
TCI-08-219 03 Alexandria, LA, US	Pecan (Creek)	1	1001	0.490	Nutmeat	0	0.017, 0.019 [0.018]
						7	<0.01, <0.01 [<0.01]
						14	<0.01, <0.01 [<0.01]
						21	<0.01, <0.01 [<0.01]
TCI-08-219 04 Pearsall, TX, US	Pecan (Wichita)	1	561	0.490	Nutmeat	7	<0.01, <0.01 [<0.01]
TCI-08-219 05 Anton, TX, US	Pecan (Western Schley)	1	645	0.490	Nutmeat	7	0.013, 0.015 [0.014]

[a] Trials are separated by approximately 25 km.

[b] Application and harvest dates are offset by approximately 30 days

PRIMARY FEED COMMODITIES OF PLANT ORIGIN

Almond hulls (Evaluated by 2017 Meeting)

Residues from field trials with almond evaluated by the 2017 Meeting are provided in Table 9.

Table 9 Summary of 2008 field trials residues in almond hulls reviewed by the 2017 Meeting

Report Trial No. Location	Crop (Variety)	Application			Matrix	DALA	Fenazaquin Residues [average], mg/kg
		No.	L/ha	kg ai/ha			
Critical GAP [USA]	Almond	1	935	0.504	--	7	--
TCI-08-219 06 [a] Terra Bella, CA, US	Almond (Nonpareil)	1	571	0.500	Hulls	1	1.80, 1.91 [1.86]
						7	1.01, 1.17 [1.09]
						14	1.23, 1.52 [1.38]
						21	1.33, 1.22 [1.28]

Report Trial No. Location	Crop (Variety)	Application			Matrix	DALA	Fenazaquin Residues [average], mg/kg
		No.	L/ha	kg ai/ha			
TCI-08-219 09 [a] Strathmore, CA, US	Almond (Nonpareil)	1	1104	0.520	Hulls	7	1.28, 1.12 [1.20]
TCI-08-219 07 Dinuba, CA, US	Almond (Carmel)	1	1151	0.490	Hulls	7	1.67, 1.27 [1.47]
TCI-08-219 08 [b] Wasco, CA, US	Almond (Price)	1	655	0.500	Hulls	7	0.312, 0.461 [0.387]
TCI-08-219 10 [b] Wasco, CA, US	Almond (Nonpareil)	1	589	0.530	Hulls	7	0.217, 0.315 [0.268]

[a] Trials are separated by approximately 25 km.

[b] Application and harvest dates are offset by approximately 30 days

RESIDUES IN ANIMAL COMMODITIES

Farm animal feeding studies

In a feeding study conducted in lactating Holstein cows with fenazaquin (Ferguson, 2015, Report 029280), test animals were dosed with fenazaquin by capsule for 28 days. Holstein cows were dosed at 0 (2 cows), 12.5 ppm (3 cows), 37.5 ppm (3 cows), or 125 ppm (6 cows). Milk samples were collected twice daily and pooled. Skim milk and cream were obtained from the Day-25 milk sample. Other than the cows retained to study residue depuration, the test animals were sacrificed at the end of the dosing period and tissues were collected within approximately 8 hours of the final dose. Samples of liver, muscle, kidney, fat (perirenal, mesenterial, subcutaneous) were collected and immediately placed into frozen (-22 to -10 °C) storage. Samples were homogenized in the presence of dry ice and placed back into frozen storage prior to analysis. Analysis for fenazaquin and 2-OH fenazaquin acid was done using the method described above.

Residues of fenazaquin in milk plateaued in milk by Day 3 of dosing and declined to < 0.01 mg/kg at Depuration Day 3. Residues of fenazaquin in milk were <LOQ at all time points from the 37.5 ppm dose; residues of 2-OH fenazaquin acid were not measured and samples from the 12.5 ppm dose level were not analysed. Fenazaquin residues in milk from the 125 ppm dose group averaged 0.035 mg/kg, with a maximum of 0.046 mg/kg (Table 10). Residues of fenazaquin in skim milk were <0.01 mg/kg at all feeding levels. Quantifiable residues occurred in cream from the 37.5-ppm and 125-ppm dose groups at 0.037 mg/kg and 0.12 mg/kg, respectively.

Table 10 Residues of fenazaquin in milk from the 125 ppm dose group

Dosing Day	Fenazaquin, mg/kg						
Test animal	1	2	3	4	5	6	Mean
1	<0.01	0.017	<0.01	0.020	0.022	0.020	0.016
3	0.018	0.043	0.026	0.044	0.068	0.051	0.042
7	0.013	0.037	0.022	0.033	0.044	0.053	0.034
10	0.022	0.037	0.022	0.041	0.045	0.055	0.037
13	0.016	0.030	0.017	0.036	0.046	0.041	0.031
16	0.016	0.042	0.017	0.042	0.050	0.055	0.037
19	0.016	0.029	0.021	0.029	0.047	0.040	0.030
22	0.022	0.043	0.024	0.046	0.080	0.060	0.046
25	0.017	0.036	0.016	0.034	0.030	0.051	0.031
28	0.017	0.022	0.017	0.032	0.038	0.049	0.029
Depuration 1	0.018	0.036	0.030				0.028
Depuration 3	<0.01	<0.01	<0.01				<0.01
Depuration 7	<0.01	<0.01	<0.01				<0.01
Depuration 10	<0.01						<0.01
Depuration 14	<0.01						<0.01

Residues of fenazaquin in tissues were highest in fat and lower in liver and kidney. Residues in muscle were <LOQ at the highest dose. Similarly, residues of 2-OH fenazaquin acid were <LOQ in muscle; the highest levels of 2-OH fenazaquin acid were found in liver, with lesser amounts in kidney (Table 11).

Table 11 Residues of fenazaquin in cattle tissues

Tissue	Dose level, ppm	Residues, mg eq/kg [a]				
		Fenazaquin	2-OH fenazaquin acid	4-OH quinazoline [b]	Fenazaquin + 2-OH fenazaquin acid	Fenazaquin + 2-OH fenazaquin acid + 4-OH quinazoline
Liver	12.5	Not analysed	0.011, 0.017, 0.015 [0.014]	0.0083, 0.018, 0.018 [0.011]	0.021, 0.027, 0.025 [0.024]	0.029, 0.045, 0.043 [0.039]
	37.5	<0.01, <0.01, <0.01 [<0.01]	0.052, 0.046, 0.049 [0.049]	0.039, 0.034, 0.037 [0.037]	0.062, 0.056, 0.059 [0.059]	0.10, 0.090, 0.096 [0.096]
	125	0.059, 0.029, 0.012 [0.033]	0.15, 0.10, 0.12 [0.13]	0.12, 0.076, 0.091 [0.094]	0.21, 0.13, 0.13 [0.16]	0.33, 0.21, 0.22 [0.25]
Kidney	12.5	Not analysed	<0.009, 0.009, <0.009 [0.009]	<0.002, <0.002, <0.002 [<0.002]	<0.019, <0.019, <0.019 [<0.019]	<0.021, <0.021, <0.021 [<0.021]
	37.5	<0.01, <0.01, <0.01 [<0.01]	0.027, 0.026, 0.019 [0.024]	0.018, 0.018, 0.018 [0.01]	0.037, 0.036, 0.029 [0.034]	0.055, 0.054, 0.047 [0.052]
	125	<0.01, 0.022, 0.011 [0.013]	0.061, 0.052, 0.056 [0.056]	0.018, 0.018, 0.018 [0.014]	0.071, 0.074, 0.067 [0.071]	0.089, 0.092, 0.085 [0.089]
Muscle	12.5	Not analysed	<0.009, <0.009, <0.009 [<0.009]	Assumed 0	<0.019, <0.019, <0.019 [<0.019]	<0.019, <0.019, <0.019 [<0.019]
	37.5	Not analysed	<0.009, <0.009, <0.009 [<0.009]	Assumed 0	<0.019, <0.019, <0.019 [<0.019]	<0.019, <0.019, <0.019 [<0.019]
	125	<0.01, <0.01, <0.01 [<0.01]	<0.009, <0.009, <0.009 [<0.009]	Assumed 0	<0.019, <0.019, <0.019 [<0.019]	<0.019, <0.019, <0.019 [<0.019]
Perirenal Fat	12.5	0.028, 0.056, 0.052 [0.045]	Not analysed	Assumed 0	0.028, 0.056, 0.052 [0.045]	0.028, 0.056, 0.052 [0.045]
	37.5	0.12, 0.11, 0.091 [0.11]			0.12, 0.11, 0.091 [0.11]	0.12, 0.11, 0.091 [0.11]
	125	0.32, 0.42, 0.18 [0.31]			0.32, 0.42, 0.18 [0.31]	0.32, 0.42, 0.18 [0.31]
Mesenterial fat	12.5	0.020, 0.031, 0.044 [0.032]			0.020, 0.031, 0.044 [0.032]	0.020, 0.031, 0.044 [0.032]
	37.5	0.10, 0.11, 0.083 [0.098]			0.10, 0.11, 0.083 [0.098]	0.10, 0.11, 0.083 [0.098]
	125	0.26, 0.41, 0.11 [0.26]			0.26, 0.41, 0.11 [0.26]	0.26, 0.41, 0.11 [0.26]
Subcutaneous fat	12.5	<0.01, <0.01, 0.034 [0.014]			<0.01, <0.01, 0.034 [0.014]	<0.01, <0.01, 0.034 [0.014]
	37.5	0.042, 0.048, 0.046 [0.045]			0.042, 0.048, 0.046 [0.045]	0.042, 0.048, 0.046 [0.045]
	125	0.20, 0.20, <0.01 [0.13]			0.20, 0.20, <0.01 [0.13]	0.20, 0.20, <0.01 [0.13]

[a] For fenazaquin and 2-OH fenazaquin acid reported as not analysed or <LOQ mg/kg, residues were assumed to be 0.01 mg/kg and combined residues are listed as <combined fenazaquin-equivalent LOQs only when all residues were reported as <0.01 mg/kg.

[b] Calculated from 2-OH fenazaquin acid by a factor of 0.75 (liver) or 0.25 (milk) from the goat metabolism study evaluated by the 2017 JMPR. Residues in fat were assumed to be zero.

APPRAISAL

Fenazaquin is a quinazoline insecticide/acaricide that exhibits contact and ovicidal activity against a broad spectrum of mites in grapes, pome fruit, citrus, peaches, cucurbits, tomatoes, cotton and ornamentals. It was first evaluated by JMPR in 2017 for toxicology and residues.

The 2017 JMPR established an ADI for fenazaquin of 0–0.05 mg/kg bw and an ARfD of 0.1 mg/kg bw. The residue definition for compliance with the MRL and dietary risk assessment for plant commodities is parent fenazaquin. The residue definition for compliance with the MRL and dietary risk assessment for animal commodities is the sum of fenazaquin and the metabolites 2-(4-{2-[(2-hydroxyquinazolin-4-yl)oxy]ethyl}phenyl)-2-methylpropanoic acid (2-hydroxy-fenazaquin acid) and quinazolin-4-ol and 3,4-dihydroquinazolin-4-one (4-hydroxyquinazoline), expressed as fenazaquin equivalents. The residue is fat soluble.

The 2017 Meeting determined that the submitted storage stability data were inadequate to support the recommendation of a maximum residue level for almonds. At the Fiftieth Session of the CCPR (2018), fenazaquin was scheduled for a follow-up evaluation of additional uses by the 2019 Extra JMPR Meeting. The Meeting received additional storage stability and residue trial data for almond nutmeat and shells (the fibrous material between the nutmeat and the hull), an analytical method for the analysis of fenazaquin, 2-hydroxyfenazaquin acid, and 2-hydroxyfenazaquin-N-oxide in bovine matrices, and a cattle-feeding study.

Methods of analysis

Residue analysis for all almond sample results submitted to the 2019 Extra Meeting was done using Method ANADIAG R A4167. This LC-MS/MS method was found acceptable by the 2017 JMPR for analysis of residues in multiple plant commodities. The current meeting received validation data for residues of fenazaquin in almond nutmeat and almond shells.

For both matrices, validation data generated concurrently with the analysis of field trial samples demonstrated adequate method performance for residues of fenazaquin. The LOQ was 0.01 mg/kg for all analytes and matrices.

The Meeting received information on an analytical method that was validated in conjunction with a cattle-feeding study. Average recoveries of fenazaquin at fortification levels of 0.01, 0.2, or 2 mg/kg ranged from 76–99%. The RSDs were <10% with the exception of kidney fortified at 0.2 mg/kg, for which the RSD was 24%. The Meeting considered this to be a minor deviation. The LOQs were 0.01 mg/kg for both fenazaquin and 2-OH fenazaquin acid in all matrices.

Stability of residues in stored analytical samples

The 2017 Meeting could not conclude that stability of fenazaquin in stored almonds was adequately demonstrated based on the inconsistent percent remaining residues with increasing storage time in the previously submitted study.

Stability of fenazaquin was investigated in parallel with the field trial study submitted to the current meeting. Samples of almond nutmeat and shells were fortified with each analyte separately at 0.5 mg/kg each, stored frozen (-25 to -10 °C), and analysed using the method cited above. Residues were stable for all analytes in both matrices for at least 17 months.

Based on the results of the new study, the Meeting concluded that the new data are sufficient to support the field trials conducted in 2008 and 2012.

Definition of the residue

The current Meeting noted that there are discrepancies in the residue definitions for animal commodities provided in the 2017 report.

In examining the 2017 report for clarification, the Meeting specifically noted the following points taken from the residue definition section pertaining to residues in animals:

> "The Meeting concluded that fenazaquin and 2-hydroxy-fenazaquin acid are suitable markers for enforcement of MRLs for livestock commodities."
>
> "The metabolite 4-hydroxyquinazoline is predominantly found in milk accounting for 1.5-fold the fenazaquin residues. In tissues, 4-hydroxyquinazoline was either not detected or detected at lower levels than those of the metabolite 2-hydroxy-fenazaquin acid. The Meeting concluded

that the 2-hydroxy-fenazaquin acid and 4-hydroxyquinazoline metabolites are not likely to be more toxic than the parent fenazaquin."

The current Meeting recommended that the residue definitions for animal commodities be corrected as follows:

For compliance with the MRL: The sum of fenazaquin and the metabolite 2-(4-{2-[(2-hydroxyquinazolin-4-yl)oxy]ethyl}phenyl)-2-methylpropanoic acid (2-hydroxy-fenazaquin acid) expressed as fenazaquin equivalents.

For estimation of dietary risk: The sum of fenazaquin and the metabolites 2-(4-{2-[(2-hydroxyquinazolin-4-yl)oxy]ethyl}phenyl)-2-methylpropanoic acid (2-hydroxy-fenazaquin acid) and quinazolin-4-ol and 3,4-dihydroquinazolin-4-one (tautomeric forms of 4-hydroxyquinazoline), expressed as fenazaquin equivalents.

The residue is fat soluble.

As there were no recommendations made by the 2017 Meeting involving animal commodities, the previous recommendations are not affected by this correction.

Results of supervised residue trials on crops

The current Meeting received supervised trial data reflecting applications of fenazaquin to almond; these data are in addition to data reviewed by the 2017 Meeting for almond. The demonstrated period of stability of fenazaquin residues (17 months) covers the storage period for that analyte in nutmeats for the previously submitted trials (maximum storage period of 2 months) and the new trials (maximum storage period of 14 months).

A label for the end-use product containing fenazaquin was available from the USA describing the registered use of fenazaquin on the USA tree nuts crop group.

Tree nuts

The cGAP for tree nuts in the USA is a single application at up to 0.504 kg ai/ha with a 7-day PHI.

Nine trials in almonds, approximating the cGAP, were conducted in the USA. Residues of fenazaquin were (n=9): < 0.01(7), 0.011, and 0.013 mg/kg.

Five independent trials in pecan, approximating the cGAP, were conducted in the USA. Residues of fenazaquin were (n=5): < 0.01 (4), and 0.014 mg/kg.

Based on the observed similarity in residue levels for almond and pecan, the Meeting decided to use both data sets to mutually support a recommendation for tree nuts, except coconut. The combined dataset is (n=14): < 0.01(11), 0.011, 0.013 and 0.014 mg/kg.

The Meeting estimated a maximum residue level of 0.02 mg/kg, STMR of 0.01 mg/kg and HR of 0.016 mg/kg (based on highest individual value) for fenazaquin in Tree nuts (except coconut).

Animal feeds

Almond hulls

Residue data for fenazaquin in almond hulls were reviewed by the 2017 Meeting; however, that Meeting was unable to make residue estimates due to the lack of supporting storage stability data. The current Meeting decided to apply the new storage stability data for almond nutmeat and shells to the almond hull data reviewed previously.

Residues of fenazaquin in almond hulls from trials approximating cGAP were (n=5): 0.27, 0.39, 1.2, 1.4, and 1.5 mg/kg.

The Meeting estimated a maximum residue level of 4 mg/kg for almond hulls (dry; based on a dry matter content of 90% as per the OECD feed table), with a corresponding median residue of 1.2 mg/kg (as received).

Residues in animal commodities

Almond hulls are the only potential livestock feed item relevant to fenazaquin. Based on a median residue of 1.2 mg/kg, the maximum and mean dietary burdens are both 0.133 ppm. The burden is the same for beef and dairy cattle in both Australia and Canada/USA. In the feeding study, residues of fenazaquin were measured in milk and fenazaquin and 2-OH fenazaquin acids were measured in tissues. Measured residues of 2-OH fenazaquin acid were converted to fenazaquin-equivalents using the molecular weight factor of 0.869. The Meeting used results from the goat metabolism study to estimate unmeasured residues as follows:

Milk:2-OH fenazaquin acid = 0.25 × fenazaquin

4-OH quinazoline = 1.5 × fenazaquin

Liver: 4-OH quinazoline = 0.75 × 2-OH fenazaquin acid

Kidney:4-OH quinazoline = 0.25 × 2-OH fenazaquin acid

Muscle:4-OH quinazoline not detected, assumed to be zero

Fat:4-OH quinazoline not detected, assumed to be zero

Fenazaquin feeding study	Feed level (ppm) for milk residues	Residues (mg/kg) in milk	Feed level (ppm) for tissue residues	Residues (mg eq/kg)			
				Muscle	Liver	Kidney	Fat
MRL beef or dairy cattle							
Feeding study	12.5	<LOQ	12.5	<LOQ	0.027	<0.019	0.056
Dietary burden and high residue	0.133	<0.02	0.133	<0.02	<0.02	<0.02	<0.02
STMR beef or dairy cattle							
Feeding study	12.5	<0.01	12.5	<0.019	0.039	<0.019	0.045
Dietary burden and residue estimate	0.133	<0.0001	0.133	<0.0002	0.00041	<0.0002	0.00048
HR beef or dairy cattle							
Feeding study	12.5	<0.01	12.5	<0.019	0.045	<0.019	0.056
Dietary burden and residue estimate	0.133	<0.0001	0.133	<0.0002	0.00048	<0.0002	0.00060

Based on the anticipated residues, the Meeting estimated maximum residue levels of 0.02(*) mg/kg for edible offal (mammalian), mammalian fats (except milk fats), meat (from mammals other than marine mammals; as fat), milks, and milk fats. Corresponding STMRs and HRs are 0 mg/kg.

RECOMMENDATIONS

On the basis of the available data, the Meeting concluded that the residue levels listed below are suitable for establishing maximum residue levels and for IEDI and IESTI assessments.

The definition of the residue for compliance with the MRL and for dietary risk assessment for plant commodities is *fenazaquin*.

The definition of the residue for compliance with the MRL for animal commodities is *the sum of fenazaquin and the metabolite 2-(4-{2-[(2-hydroxyquinazolin-4-yl)oxy]ethyl}phenyl)-2-methylpropanoic acid (2-hydroxy-fenazaquin acid) expressed as fenazaquin equivalents*.

The definition of the residue for dietary risk assessment for animal commodities is *the sum of fenazaquin and the metabolites 2-(4-{2-[(2-hydroxyquinazolin-4-yl)oxy]ethyl}phenyl)-2-methylpropanoic acid (2-hydroxy-fenazaquin acid) and quinazolin-4-ol and 3,4-dihydroquinazolin-4-one (tautomeric forms of 4-hydroxyquinazoline), expressed as fenazaquin equivalents*.

The residue is fat soluble.

Commodity		Recommended MRL, mg/kg		STMR or STMR-P, mg/kg	HR or HR-P, mg/kg
CCN	Name	New	Previous		
TN 0085	Tree nuts, except coconut	0.02	--	0.01	0.016
AM 0660	Almond hulls	4 (dw)	--	Median: 1.2 (as)	--
MO 0105	Edible offal (mammalian)	0.02*	--	0	0
MF 0100	Mammalian fats (except milk fats)	0.02*	--	0	0
MM 0095	Meat (from mammals other than marine mammals)	0.02* (fat)	--	Muscle: 0 Fat: 0	Muscle: 0 Fat: 0
ML 0106	Milks	0.02*	--	0	0
FM 0183	Milk fats	0.02*	--	0	0

(as) – as received; (dw) – dry weight

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for fenazaquin is 0–0.05 mg/kg bw. The International Estimated Daily Intakes (IEDIs) for fenazaquin 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 2019 Extra JMPR Report.

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

Acute dietary exposure

The ARfD for fenazaquin is 0.1 mg/kg bw. The International Estimate of Short-Term Intakes (IESTIs) for fenazaquin 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 2019 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 fenazaquin from uses considered by the present Meeting is unlikely to present a public health concern.

REFERENCES

Report	Author	Year	Title
TCI-12-349	Carringer, S.J.	2015	Residues of Fenazaquin in or on Almonds Following One Application of GWN-1708 1.67 SC (2012)
029280	Ferguson, L-J.	2015	Magnitude of the Residue of Fenazaquin and Metabolite M29 N-Oxide in Bovine Tissues and Milk from a 28-Day Feeding Study

FLONICAMID (282)

First draft prepared by Dr N C Keong, Malaysian Agricultural Research and Development Institute, Kuala Lumpur, Malaysia

EXPLANATION

Flonicamid is the ISO approved common name for N-cyanomethyl-4-(trifluoromethyl)nicotinamide (IUPAC). Flonicamid (CAS No. 158062-67-0) is systemic pyridine carboxamide insecticide with selective activity against Hemipterous pests.

Flonicamid was first evaluated for residues and toxicological aspects by the 2015 JMPR. The 2015 JMPR established an ADI for flonicamid of 0–0.07 mg/kg bw and concluded that an ARfD was unnecessary.

The 2015 JMPR also recommended the following residue definition for flonicamid:

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

Definition of the residue for compliance with the MRL and dietary risk assessment in animal commodities: *Flonicamid and the metabolite TFNA-AM, expressed as parent.*

The residue is not fat-soluble.

Flonicamid was last evaluated in 2017 for additional maximum residue levels. At the Fiftieth Session of the CCPR (2017), flonicamid was listed for consideration of additional uses by the 2019 Extra JMPR. The Meeting received information on registered use patterns, analytical method information, storage stability data and supervised residue trials on citrus fruits with product labels from the USA.

RESIDUE ANALYSIS

Analytical methods

Flonicamid was first evaluated by the JMPR in 2015. Supervised field trials submitted to the current Meeting were analysed using method IB-2014-JLW-002-01-01 for citrus fruits. Additionally, methods H13-87 and 09604 were submitted used to investigated residues in stored analytical samples.

Table 1 Overview of analytical methods for flonicamid and its metabolites

Method	Matrix	Extraction	Clean-Up	Detection, LOQ
H13-87	High acid (citrus fruit)	methanol	Diatomaceous earth column Florisil SPE	GC-MS (peel/pulp) Flonicamid: m/z: 174, LOQ: 0.04/0.01 mg/kg TFNA: m/z: 174, LOQ: 0.04/0.01 mg/kg TFNG: m/z: 174, LOQ: 0.04/0.01 mg/kg
09604	High acid (strawberry)	acetonitrile:water (1:1, v/v)	none	LC-MS/MS Flonicamid: m/z: 230 → 203, LOQ: 0.0094 mg/kg TFNA: m/z: 192 → 148, LOQ: 0.0153 mg/kg TFNA-AM: m/z: 190 → 148, LOQ: 0.0134 mg/kg TFNG: m/z: 249 → 203, LOQ: 0.0104 mg/kg
IB-2014-JLW-002-01-01 PSM-16-06-02[a]	High acid (citrus fruit & pulp) Citrus oil	acetonitrile:water (1:1, v/v)	Partitioning with ethyl acetate	LC-MS/MS Flonicamid: m/z: 230 → 203, LOQ: 0.01 mg/kg TFNA: m/z: 192 → 148, LOQ: 0.01 mg/kg TFNA-AM: m/z: 191 → 148, LOQ: 0.01 mg/kg TFNG: m/z: 249 → 203, LOQ: 0.01 mg/kg

[a] Study PSM-16-06-02 used the same method IB-2014-JLW-002-01-01

Method H13-87 (Japan Food Research Laboratories, 2002, H13-87):

The method involves extraction with methanol, followed by clean-up on a diatomaceous earth column. Residues were eluted with ethyl acetate: acetic acid (99:1, v/v). The extract was cleaned-up with a florisil column. For the analysis of TFNA and TFNG, an additional derivatisation step was applied involving methylation of TFNA and TFNG using trimethylsilyldiazomethane after residue elution from the diatomaceous earth column and prior to clean-up on the florisil column. Final extracts were analysed by GC-MS.

LOQs for flonicamid, TFNA, TFNA-AM and TFNG were 0.04 and 0.01 mg/kg, in each of orange peel and pulp, respectively.

Table 2 Recovery data for method H13-87 in oranges

Matrix	Fortification level (mg/kg)	n	Recovery in % (mean)	RSD (%)	Analyte
Orange pulp	0.01	6	95-105 (100)	3.6	Flonicamid
	0.4	6	89-104 (99)	5.7	m/z 174
	0.01	6	94-112 (105)	8.0	TFNA
	0.4	6	79-86 (82)	2.9	m/z: 174
	0.01	6	84-96 (91)	4.4	TFNG
	0.4	6	85-101 (94)	6.0	m/z: 174
Orange peel	0.04	6	91-113 (102)	8.3	Flonicamid
	2.0	6	80-92 (87)	5.5	m/z 174
	0.04	6	80-112 (96)	14.2	TFNA
	2.0	6	90-103 (96)	5.2	m/z: 174
	0.04	6	82-108 (92)	10.7	TFNG
	2.0	6	84-97 (92)	5.0	m/z: 174

Method 09604 (Samoil, K. S., 2010, 09604):

Extraction with acetonitrile:water (1:1) followed by solvent removal and reconstitution in acetonitrile:water (7:3). Extracts were analysed in LC/MS/MS.

Table 3 Recovery data for method 09604 in strawberries

Matrix	Fortification level (mg/kg)	n	Recovery in % (mean)	RSD (%)	Analyte
Strawberry	0.02	7	90-100 (95)	5	Flonicamid m/z 230.023 → 203.125
	0.2	13	85-105 (99)	6	
	2.0	3	95-110 (100)	9	
	0.02	6	80-100 (88)	8	TFNA m/z : 192.008 → 148.060
	0.2	13	85-100 (93)	4	
	2.0	3	90-100 (93)	6	
	0.02	6	70-90 (79)	7	TFNA-AM m/z: 190.982 → 148.095
	0.2	13	75-95 (84)	5	
	2.0	3	80-90 (83)	6	
	0.02	6	70-80 (73)	5	TFNG m/z: 249.039 → 203.059
	0.2	13	70-90 (82)	6	
	2.0	3	80-90 (83)	6	

Method IB-2014-JLW-002-01-01 (Wiedmann, J. L. and McDonald, J. A., 2015, IB-2014-JLW-002-01-01):

For the analysis of residues in orange, orange pulp, orange juice, grapefruit and lemon, homogenised samples were extracted with acetonitrile:water (1:1, v/v) and the combined extracts were evaporated and reconstituted in HCl and water. The extract was partitioned three times with ethyl acetate. The ethyl acetate extract was dried and reconstituted in acetonitrile for LC/MS/MS analysis.

For analysis of orange pulp, homogenised samples were soaked in water, followed by extraction with acetonitrile. The extract aliquot was dried and reconstituted in water. For analysis of flonicamid and TFNA-AM, NaOH was added. For analysis of TFNA and TFNG, concentrated HCl was added. After addition of NaOH or HCl, the extract was partitioned 3 times with ethyl acetate. The ethyl acetate extract was dried and reconstituted in acetonitrile for LC/MS/MS analysis.

For analysis of residues in orange oil, samples were dissolved in hexane and partitioned with acetonitrile:water (50:50, v/v). The acetonitrile:water layer was removed and the oil was partitioned again. Collected acetonitrile:water extracts were diluted and further analysed by LC/MS/MS.

LOQs for flonicamid, TFNA, TFNA-AM and TFNG were 0.01 mg/kg, irrespective of orange, orange juice, orange pulp and orange oil.

Table 4 Recovery data for method IB-2014-JLW-002-01-01 in citrus fruits and processed products thereof

Matrix	Fortification level (mg/kg)	n	Recovery in % (mean)	RSD (%)	Analyte
Orange fruits	0.01	3	68-89 (82)	14.4	Flonicamid
	0.1	3	93-94 (94)	0.7	m/z: 230 → 203
	0.01	3	62-82 (75)	15.4	TFNA
	0.1	3	77-84 (80)	4.2	m/z: 192 → 148
	0.01	3	67-80 (75)	9.6	TFNA-AM
	0.1	3	77-83 (81)	4.0	m/z: 191 → 148
	0.01	3	81-90 (87)	5.6	TFNG
	0.1	3	89-91 (90)	1.3	249 → 203
Orange juice	0.01	3	86-95 (89)	6.0	Flonicamid
	0.1	3	91-96 (94)	2.9	m/z: 230 → 203
	0.01	3	97-115 (106)	8.5	TFNA
	0.1	3	91-102 (96)	5.9	m/z: 192 → 148
	0.01	3	71-80 (75)	6.2	TFNA-AM
	0.1	3	70-78 (75)	5.4	m/z: 191 → 148
	0.01	3	78-85 (83)	4.8	TFNG
	0.1	3	79-88 (83)	5.8	249 → 203
Orange pulp	0.01	3	83-96 (90)	7.3	Flonicamid
	0.1	3	83-87 (85)	2.1	m/z: 230 → 203
	2.0	3	80-83 (81)	1.8	
	0.01	3	77-113 (92)	20.7	TFNA
	0.1	3	78-85 (81)	4.8	m/z: 192 → 148
	0.01	3	79-82 (80)	2.0	TFNA-AM
	0.1	3	76-79 (78)	1.9	m/z: 191 → 148
	0.01	3	68-80 (73)	8.5	TFNG
	0.1	3	71-85 (76)	10.7	249 → 203
Orange oil	0.01	3	74-82 (79)	5.2	Flonicamid
	0.1	3	77-86 (82)	5.5	m/z: 230 → 203
	0.01	3	79-90 (84)	6.7	TFNA
	0.1	3	69-82 (76)	8.5	m/z: 192 → 148
	0.01	3	65-85 (75)	13.6	TFNA-AM
	0.1	3	71-80 (76)	6.1	m/z: 191 → 148
	0.01	3	68-91 (79)	15.0	TFNG
	0.1	3	78-93 (85)	8.6	249 → 203

Table 5 Recovery data of analytes in oranges for method used in PSM-16-06-02

Matrix	Fortification level (mg/kg)	n	Recovery in % (mean)	RSD (%)	Analyte
Orange fruits	0.01	3	80-103 (93)	12.4	Flonicamid
	0.1	3	82-85 (84)	2.0	m/z: 230 → 203
	0.01	3	81-87 (83)	4.1	TFNA
	0.1	3	86-91 (89)	2.6	m/z: 192 → 148
	0.01	3	81-93 (85)	8.0	TFNA-AM
	0.1	3	78-81 (79)	1.7	m/z: 191 → 148
	0.01	3	74-85 (79)	7.3	TFNG
	0.1	3	76-90 (82)	8.7	249 → 203

Stability of pesticide residues in stored analytical samples

The Meeting received two new studies investigating the storage stability of flonicamid and its metabolites in citrus fruits and strawberries.

In the first storage stability study (Japan Food Research Laboratories, 2002, H13-87), flonicamid, TFNA and TFNG in orange peel and orange pulp were investigated at -20 °C for at a period of 16 months (480-486 days).

In the second study (Samoil, K. S., 2010, 09604), flonicamid, TFNA-AM, TFNA and TFNG were investigated in strawberry for 460 days in storage at -20 °C.

In the following table, the recovered residues after storage are summarized.

Table 6 Storage stability of flonicamid in orange and strawberry matrices

Matrix	Storage in months (days)	Fortification level (mg/kg)	% remaining	Mean % remaining	Procedural recovery in % (n=1)
Flonicamid					
Orange pulp	14 (429) 16 (480)	1	89, 84 90, 84	86 87	Not reported
Orange peel	14 (435) 16 (486)	2	101, 93 96, 96	97 96	Not reported
Strawberry	15 (460)	0.2	95, 100, 100	98	100
TFNA					
Orange pulp	14 (429) 16 (480)	1	85,80 77, 77	82 77	Not reported
Orange peel	14 (435) 16 (486)	2	97, 92 94, 90	94 92	Not reported
Strawberry	15 (460)	0.2	85, 90, 90	88	90
TFNA-AM [a]					
Strawberry	15 (460)	0.2	80, 85, 85	83	80
TFNG					
Orange pulp	14 (429) 16 (480)	1	88, 86 92, 89	87 90	Not reported
Orange peel	14 (435) 16 (486)	2	96, 87 90, 89	92 90	Not reported
Strawberry	15 (460)	0.2	75, 80, 80	78	80

[a] No study of storage stability on TFNA-AM on orange pulp and peel

USE PATTERN

Flonicamid is intended for insecticidal use in citrus fruits by a foliar spray application in the USA.

Table 7 List of uses of flonicamid

Crops or crop groups	Country	Application detail					
		kg ai/ha	Growth stage at last treatment	Indoor/ Outdoor	No.	Interval in days	Pre harvest interval (PHI) in days
Citrus fruits	USA	0.1	At infestation	Outdoor	3	7	0

RESULTS OF SUPERVISED RESIDUE TRIALS ON CROPS

Residue levels were reported as measured. Application rates were always reported as flonicamid equivalents. When residues were not detected they are shown as below the LOQ, e.g., < 0.01 mg/kg. Application rates, spray concentrations and mean residue results have generally been rounded to the even with two significant figures. HR and STMR values from the trials conducted according to maximum GAP have been used for the estimation of maximum residue levels. These results are underlined.

Laboratory reports included method validation including batch recoveries with spiking at residue levels similar to those occurring in samples from the supervised trials. Dates of analyses or duration of residue sample storage were also provided. Field reports provided data on the sprayers used and their calibration, plot size, residue sample size and sampling date. Although trials included control plots, no control data are recorded in the tables except where residues in control samples exceeded the LOQ. Residue data are recorded unadjusted for % recovery.

Flonicamid - supervised residue trials

Commodity	Indoor/Outdoor	Treatment	Countries	Table
Lemons	Outdoor	Foliar	USA	8
Oranges	Outdoor	Foliar	USA	9
Grapefruits	Outdoor	Foliar	USA	10

Table 8 Residues of flonicamid following spray treatment on lemon trees

Location, year, trial reference, variety	Application					Matrix	DAT	Residues (mg/kg)	
	Spray Volume (L/ha)	Rate (kg ai/ha)	kg ai/hL	No.	RTI in days			Flonicamid	Mean
USA GAP: 3 × 0.1 kg ai/ha, PHI: 0 days									
Winter Garden, FL, USA 2014 IB-2014-JLW-002-19 Bearss	1100-1200	0.098-0.1	0.009	3	7-8	Lemon	0	0.22, 0.29	<u>0.25</u>
Newman, CA, USA 2014 IB-2014-JLW-002-20 Eureka	1200	0.095	0.008	3	7	Lemon	0	0.99, 0.43	<u>0.71</u>
Sanger, CA, USA 2015 IB-2014-JLW-002-21 Lisbon	1200	0.094-0.095	0.008	3	7	Lemon Lemon Lemon Lemon	0 3 7 10	0.17, 0.23 0.25, 0.20 0.13, 0.17 0.15, 0.16	0.20 <u>0.22</u> 0.15 0.15
Richgrove, CA, USA 2014 IB2014-JLW-002-22 Lisbon	1400	0.099-0.1	0.007	3	7	Lemon	0	0.12, 0.14	<u>0.13</u>
Yuma, AZ, USA 2014 IB-2014-JLW-002-23 Lisbon	1600	0.097-0.099	0.006	3	7	Lemon	0	0.10, 0.16	<u>0.13</u>

DAT: days after treatment

Table 9 Residues of flonicamid following spray treatment on orange trees

Location, year, trial reference, variety	Application					Matrix	DAT	Residues (mg/kg)	
	Spray Volume (L/ha)	Rate (kg ai/ha)	kg ai/hL	No.	RTI in days			Flonicamid	Mean
USA GAP: 3 × 0.1 kg ai/ha, PHI: 0 days									
Lake Wales, FL, USA 2014, IB-2014-JLW-002-01 Valencia	940	0.1	0.011	3	6-8	Orange	0	0.29, 0.18	0.24
Winter Garden, FL, USA I-2014-JLW-002-02 2014 Hamlin &	1200	0.1	0.009	3	7-8	Orange	0	0.13, 0.11	0.12
IB-2014-JLW-002-05 Easy Gold [1]	1200	0.1	0.008-0.009	3	7-8	Orange	0	0.23, 0.23	0.23
Umatilla, FL, USA 2014 IB-2014-JLW-002-03 Navel	1200	0.1	0.008-0.009	3	7	Orange Orange Orange Orange	0 3 7 10	0.049, 0.053 0.025, 0.028 0.018, 0.020 0.014, 0.017	0.051 0.027 0.019 0.016
De LeonSpring, FL, USA 2014 IB-2014-JLW-002-04 Navel	960-1000	0.1	0.01	3	7	Orange	0	0.11, 0.10	0.10
Winter Garden, FL, USA 2014 IB-2014-JLW-002-06 Hamlin	1700	0.1	0.006	3	7	Orange	0	0.15, 0.15	0.15
Oviedo, FL, USA 2014 IB-2014-JLW-002-07 Valencia	1400	0.1	0.007	3	7	Orange	0	0.11, 0.13	0.12
Bithlo, FL, USA 2014 IB-2014-JLW-002-08 Valencia	1400	0.099-0.1	0.007	3	7	Orange	0	0.12, 0.096	0.11
Raymondville, TX, USA 2014 IB-2014-JLW-002-09 Marrs	1000	0.1	0.01	3	7	Orange	0	0.066, 0.056	0.061
Porterville, CA, USA 2014 IB-2014-JLW-002-10 Valencia	1400	0.099	0.007	3	7-8	Orange	0	0.32, 0.12	0.22
Sanger, CA, USA 2015 IB-2014-JLW-002-11 Valencia	1400	0.094-0.095	0.007	3	6-8	Orange Orange Orange Orange	0 3 7 10	0.19, 0.25 0.19, 0.14 0.11, 0.092 0.066, 0.088	0.22 0.17 0.10 0.077
Orland, CA, USA 2014 IB-2014-JLW-002-12 Navel	940	0.1	0.011	3	7	Orange	0	0.059, 0.068	0.064
De Leon Spring, FL, USA 2016 Trial 01 PSM-16-06-02 Valencia	1000	0.099-0.1	0.01	3	6-8	Orange Orange Orange Orange	0 7 14 21	0.097, 0.078 0.075, 0.083 0.018, 0.019 <0.01, 0.015	0.088 0.079 0.018 0.012
Oak Hill, Florida, USA 2016 Trial 02 PSM-16-06-02 Valencia	1000	0.099-0.1	0.01	3	6-8	Orange Orange Orange Orange	0 7 14 21	0.081, 0.085 0.055, 0.086 0.017, 0.021 0.018, 0.017	0.083 0.070 0.019 0.018
Fresno, CA, USA 2016	940-950	0.1	0.011	3	7	Orange Orange	0 7	0.14, 0.21 0.075, 0.072	0.18 0.074

Location, year, trial reference, variety	Application					Matrix	DAT	Residues (mg/kg)	
	Spray Volume (L/ha)	Rate (kg ai/ha)	kg ai/hL	No.	RTI in days			Flonicamid	Mean
Trial 03 PSM-16-06-02 Navel						Orange Orange	14 21	0.072, 0.061 0.053, 0.040	0.066 0.046

DAT: days after treatment1: Same location, similar treatment dates, not considered independent

Table 10 Residues of flonicamid following spray treatment on grapefruit trees

Location, year, trial reference, variety	Application					Matrix	DAT	Residues (mg/kg)	
	Spray Volume (L/ha)	Rate (kg ai/ha)	kg ai/hL	No.	RTI in days			Flonicamid	Mean
USA GAP: 3 × 0.1 kg ai/ha, PHI: 0 d									
Lake Wales, FL, USA 2014 IB-2014-JLW-002-13 Flame	970	0.096-0.1	0.01	3	7-8	Grapefruit	0	0.13, 0.14	0.13
Umatilla, FL, USA 2014 IB-2014-JLW-002-14 Ray Red	1200	0.1	0.009	3	6-8	Grapefruit	0	0.031, 0.036	0.034
Oak Hill, Florida, USA 2014 IB-2014-JLW-002-15 Ray Red	1400	0.1	0.007	3	7	Grapefruit Grapefruit Grapefruit Grapefruit	0 3 7 10	0.061, 0.079 0.049, 0.046 0.016, 0.016 0.015, 0.011	0.070 0.048 0.016 0.013
Raymondville, TX, USA 2014 IB-2014-JLW-002-16 Rio Red	1000	0.1	0.01	3	7	Grapefruit	0	0.061, 0.053	0.057
Sanger, CA, USA 2014 IB-2014-JLW-002-17 Ruby Red	1400	0.093-0.094	0.007	3	7	Grapefruit	0	0.017, 0.021	0.019
Porterville, CA, USA 2014 IB-2014-JLW-002-18 Mellogold	1400	0.099-0.1	0.007	3	7	Grapefruit	0	0.077, 0.081	0.079

DAT: days after treatment

FATE OF RESIDUES DURING PROCESSING

Residues after processing

The fate of flonicamid during processing of raw agricultural commodity (RAC) was investigated in one supervised field trial on orange fruits. 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).

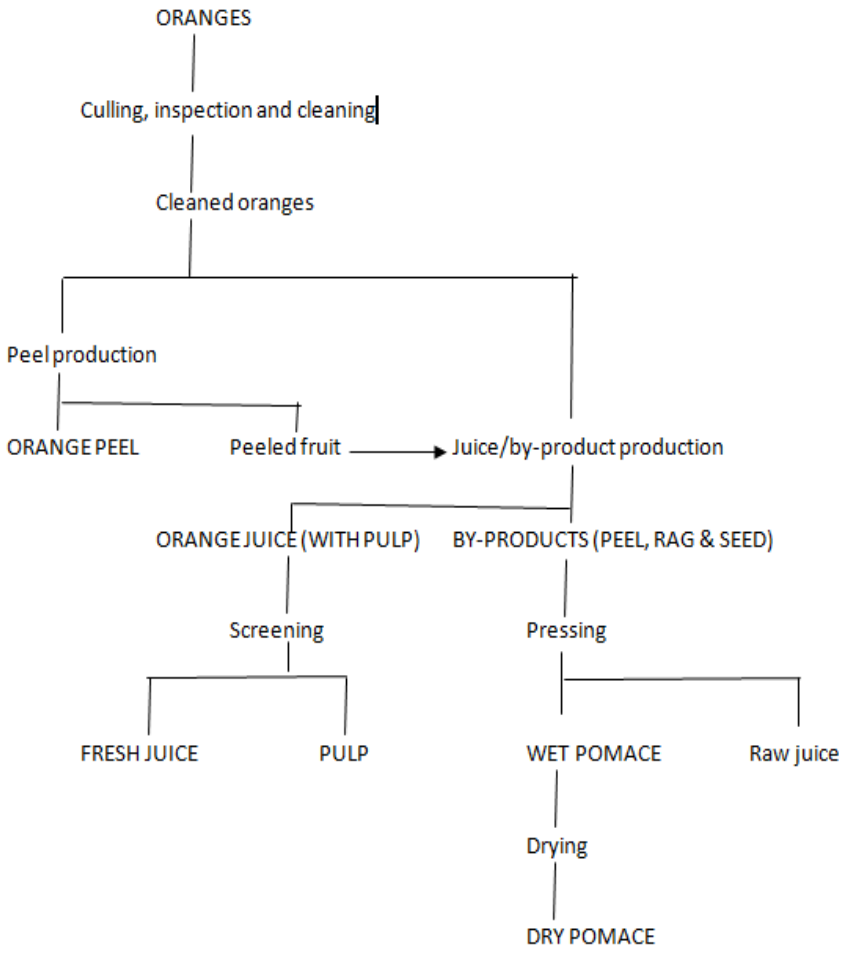
In the study IB-2014-JLW-002-01-01 (Wiedmann and McDonald 2015), only one supervised trial included a processing study. In the trial, orange trees were treated three times at minimum retreatment intervasl of 7 days, with each application of 0.5 kg ai/ha and harvested at 0 days. Orange fruits were processed into orange juice, dried orange pulp and orange oil.

To produce orange juice, whole cleaned orange were processed in a juicer machine to obtain juice and by-products (peel, rag and seed). Raw juice was sieved and pasteurised by heating.

To produce dried orange pulp (dry pomace), by-products from juice production were chopped and dewatered in hydraulic press. Solids were dried until moisture was <12%.

To produce peel oil, cleaned oranges were peeled with a modified abrasion peeler. During peeling, a spray of water was used to collect the peel oil being released. The liquid solution was sieved. The liquid was placed into a cooler and allow to separate into juice and an oil/water emulsion. Separation of peel oil from emulsion was achieved using centrifugation.

The flow chart of the processing study is shown in the following diagram.



In the following table the processing factors derived from the supervised field trial results are summarized:

Table 11 Processing factors for flonicamid in processed orange fruits based on one supervised field trial data

Trial, Location	Application	Matrix	Flonicamid in mg/kg	PF
IB-2014-JLW-002-10 Porterville, CA, USA	3 × 0.1 kg ai/ha, 7 d interval, 0 DALA	Orange (RAC) Orange, juice Orange, dried pulp Orange oil	0.544 <0.01 0.987 nd	- <0.02 1.81 <0.01

APPRAISAL

Flonicamid is the ISO approved common name for N-cyanomethyl-4-(trifluoromethyl)nicotinamide (IUPAC). Flonicamid (CAS No. 158062-67-0) is systemic pyridine carboxamide insecticide with selective activity against Hemipterous pests.

Flonicamid was first evaluated for residues and toxicological aspects by the 2015 JMPR. The 2015 JMPR established an ADI for flonicamid of 0–0.07 mg/kg bw and concluded that an ARfD was unnecessary.

The 2015 JMPR also recommended the following residue definition for flonicamid:

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

Definition of the residue for compliance with the MRL and dietary risk assessment in animal commodities: *Flonicamid and the metabolite TFNA-AM, expressed as parent*

The residue is not fat-soluble.

Flonicamid was last evaluated in 2017 for additional maximum residue levels. At the Fiftieth Session of the CCPR (2017), flonicamid was listed for consideration of additional uses by the 2019 Extra JMPR. The Meeting received information on registered use patterns, analytical method information, storage stability data and supervised residue trials on citrus fruits with product labels from the USA

Methods of analysis

The current Meeting received additional concurrent recovery information for the analysis of flonicamid in plant matrices.

Methods H13-87 and 09604 were used in the investigation of the storage stability in high acid matrices. In H13-87 method, methanol was used as extraction solvent. Residues were determined by GC-MS and individual LOQs of 0.01 mg/kg were validated for parent flonicamid and each of its metabolites TFNA, and TFNG in orange pulp. In orange peel, individual LOQs of 0.04 mg/kg were validated for parent flonicamid and each of its metabolites TFNA, and TFNG. The 09604 method involves extraction of the residue with acetonitrile:water (1:1). Determination was performed by LC-MS/MS. Based on concurrent recovery data, individual LOQs of 0.01 mg/kg were validated for parent flonicamid and each of its metabolites TFNA, TFNA-AM and TFNG.

Method IB-2014-JLW-002-01-01 was used for residue determination of field crop samples from the supervised trials. The method involves extraction of residues with acetonitrile:water (1:1; v/v). Determination was performed by LC-MS/MS and supported with concurrent recovery data suggesting individual LOQs of 0.01 mg/kg for parent flonicamid and each of its metabolites TFNA, TFNA-AM and TFNG.

Stability of residues in stored analytical samples

The Meeting received information on the storage stability of parent flonicamid and its metabolites TFNA, TFNA-AM and TFNG in high acidic matrices (citrus fruits and strawberries).

Flonicamid, TFNA and TFNG in orange peel and orange pulp were found to be stable in storage at -20 °C for at least 16 months (480–486 days). Flonicamid, TFNA, TFNA-AM and TFNG in strawberry were found to be stable in storage at -20 °C for at least 15 months (460 days).

Among all the samples from supervised trials in storage, the longest storage duration before analysis was 268 days. The Meeting concluded that all the residue results from supervised trials were analysed within acceptable storage intervals.

Results of supervised residue trials on crops

Flonicamid is registered for use on citrus fruits in the USA with a maximum GAP involving three foliar sprays of 0.1 kg ai/ha each (7 day interval), a maximum seasonal rate of 0.3 kg ai/ha and a PHI of 0 days. The Meeting received supervised trial data for applications of flonicamid on citrus fruits conducted in the USA.

Lemons and Limes

Corresponding supervised field trials conducted in the USA on lemons matching the GAP were submitted.

Residues of flonicamid in lemon fruits were (n=5): 0.13(2), 0.22 0.25 and 0.71 mg/kg.

The Meeting estimated a maximum residue level of 1.5 mg/kg and a STMR value of 0.22 mg/kg for flonicamid in the subgroup lemons and limes.

Oranges, Sweet, Sour

Corresponding supervised field trials conducted in the USA on oranges matching the GAP were submitted.

Residues of flonicamid in orange fruits were (n=14): 0.051, 0.061, 0.064, 0.083, 0.088, 0.10, 0.11, 0.12, 0.15, 0.18, 0.22(2), 0.23 and 0.24 mg/kg.

The Meeting noted that the US GAP involves treatment of all citrus fruit and decided to use oranges as representative commodity for the subgroup of oranges, sweet, sour.

The Meeting estimated a maximum residue level of 0.4 mg/kg and a STMR value of 0.115 mg/kg for flonicamid in the subgroup oranges, sweet, sour.

Pummelos and Grapefruits

Corresponding supervised field trials conducted in the USA on grapefruits matching the GAP were submitted.

Residues of flonicamid in grapefruits were (n=6): 0.019, 0.034, 0.057, 0.070, 0.079 and 0.13 mg/kg.

The Meeting noted that the US GAP involves treatment of all citrus fruit and decided to use graperuits as representative commodity for the subgroup of pummelos and grapefruit.

The Meeting estimated a maximum residue level of 0.3 mg/kg and a STMR value of 0.0635 mg/kg for flonicamid in the subgroup pummelos and grapefruit.

The Meeting noted that data from mandarins were not available therefore the Meeting did not consider a recommendation for the citrus group.

Fate of residues during processing

The fate of flonicamid residues has been examined simulating commercial processing of orange fruits.

Estimated processing factors for the commodities considered at this Meeting are summarized below.

Raw commodity	Processed commodity	Flonicamid			
		Individual processing factors	Mean or best estimate processing factor	STMR or STMR-P (mg/kg)	Maximum residue level (mg/kg)
Citrus fruits	Lemon (RAC)			0.22	1.5
	Juice	0.02 (from orange)	0.02	0.0044	-
	Dried pulp	1.8 (from orange)	1.8	0.396	3
	Oil	0.01 (from orange)	0.01	0.0022	-

The Meeting estimated a maximum residue level of 3 mg/kg for citrus pulp, dry on the basis of the processing factor of 1.8 for orange pulp, dry and the maximum residue level for lemon of 1.5 mg/kg.

Residues in animal commodities

The Meeting recalculated the livestock dietary burden based on the uses considered by the current and previous Meeting on the basis of diets listed in the 2016 edition of FAO Manual Appendix IX (OECD Feedstuff Table). The addition of citrus pulp, dry does not add significantly to the maximum and mean dietary burdens of up to 27.7 ppm and 15.3 ppm calculated by the 2016 JMPR. The Meeting confirmed its previous recommendations for flonicamid 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 assessment.

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

Definition of the residue for compliance with the MRL and dietary risk assessment for animal commodities: *Flonicamid and the metabolite TFNA-AM, expressed as parent*

The residue is not fat-soluble.

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
FC 002	Lemons and limes, Subgroup of	1.5	-	0.22	-
FC 0004	Oranges, Sweet, Sour, Subgroup of	0.4	-	0.115	-
FC 005	Pummelos and Grapefruit, Subgroup of	0.3	-	0.0635	-
AB0001	Citrus pulp, dry	3 (dw)	-	Median: 0.396	-
JF 0001	Citrus juice			0.0044	
OR 0001	Citrus oil, edible			0.0022	

(dw) – dry weight

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for flonicamid is 0–0.07 mg/kg bw. The International Estimated Daily Intakes (IEDIs) for flonicamid 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 2019 Extra JMPR Report.

The IEDIs ranged from 1–10% of the maximum ADI. The Meeting concluded that long-term dietary exposure to residues of flonicamid from uses considered by the JMPR is unlikely to present a public health concern.

Acute dietary exposure

The 2015 JMPR decided that an ARfD for flonicamid was unnecessary. The Meeting therefore concluded that the acute dietary exposure to residues of flonicamid from the uses considered is unlikely to present a public health concern.

REFERENCES

Code	Author	Year	Title, Institute, Report reference
H13-87	Anonymous	2002	Crop Residue Analysis Report. Japan Food Research Laboratories, Japan; report no. H13-87, GLP: no, Unpublished
09604	Samoil, K. S.	2010	Flonicamid: Magnitude of the residue on strawberry. IR-4, USA; report no. 09604, GLP: yes, Unpublished
IB-2014-JLW-002-01-01	Wiedmann, J. L. and McDonald, J. A.	2015	Magnitude of residues of Flonicamid on Citrus – USA in 2014. ISK Biosciences Corporation, USA; report no. IB-2014-JLW-002-01-01, GLP: yes, Unpublished
PSM-16-06-02	Schreier, T.	2017	Magnitude of The Residue of Beleaf 50 SG Insecticide in/on Orange, Precision Study Management LLC, USA; report no. PSM-16-06-02, GLP: yes, Unpublished

FLUPYRADIFURONE (285)

First draft prepared by Japan, and Dr Yukiko Yamada, Ministry of Agriculture, Forestry and Fisheries, Japan

EXPLANATION

Flupyradifurone, is an insecticide with the structure of butenolides. It acts as an agonist of nicotinic acetylcholine receptor.

Flupyradifurone was first evaluated by the Meeting for toxicology in 2015 as a new compound. It was evaluated for residues in 2016 and 2017.

The 2015 Meeting established an ADI of 0–0.08 mg/kg bw and an ARfD of 0.2 mg/kg bw.

The 2016 and 2017 Meetings reviewed information on identity, physical and chemical properties, 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: *Flupyradifurone.*

Definition of the residue (for dietary risk assessment) for plant commodities: *Sum of flupyradifurone, difluoroacetic acid (DFA) and 6-chloronicotinic acid (6-CNA), expressed as parent equivalents.*

Definition of the residue (for compliance with the MRL and dietary risk assessment) for animal commodities: *Sum of flupyradifurone and difluoroacetic acid, expressed as parent equivalents.*

The residue is not fat-soluble.

On a basis of the above residue definitions, the Meeting estimated maximum residue levels for a wide range of commodities.

Flupyradifurone was listed by the Forty-ninth CCPR for evaluation of additional uses by the current Meeting. The present Meeting received information on analytical methods, storage stability, use pattern, supervised residue trials and processing in support of estimation of maximum residue levels for blackberry, raspberry, avocado, pomegranate, cacao beans, coffee beans, and hops

RESIDUE ANALYSIS

Analytical methods

A number of analytical methods (for enforcement and data collection) for plant and animal matrices were submitted to and evaluated by the 2016 Meeting. The current Meeting received information on new analytical methods together with validation data for residues of flupyradifurone.

Method 01330/M002 (Rzepka, S., 2014, M-469883-02-1)

Analyte:	Flupyradifurone, DFA (for enforcement)
LOQ:	Flupyradifurone: 0.05 mg/kg for dried cacao beans; 0.10 mg/kg for green and roasted coffee beans DFA: 0.10 mg eq/kg dried cacao beans; 0.20 mg eq/kg for green and roasted coffee beans
Description:	Residues of flupyradifurone and DFA are extracted from dry or fermented cacao beans or green or roasted coffee beans twice with 25 ml mixture of acetonitrile and water (4:1, v/v) + 2.2 ml/L of formic acid. After centrifugation, an aliquot of the extract was mixed with C18-SPE and the resulting sample was analysed by reversed phase HPLC-MS/MS in positive ion mode for flupyradifurone and negative ion mode for DFA. Residues were quantified using solvent standards for the analysis of cacao beans and green coffee beans and matric matched standards for

roasted coffee beans. For quantitation and confirmation of flupyradifurone, transitions at *m/z* 289→126 and *m/z* 289→90 are monitored respectively, and for DFA, m/z 95 →19 and m/z 95 →51 respectively.

Method 01304 (RV-001-P10-02)(*Li,Y., 2010; M-401023-01-2* and *Li, Y.; Schoening, R.; 2012; M-415504-02-1*) and Method RV-001-P10-03 (slight modification of Method 01304 (Li, Y., 2012, M-433355-01-1)

Analyte: Flupyradifurone, DFS, 6-CNA and DFEAF (for data collection)
LOQ: Flupyradifurone, 6-CNA and DFEAF: 0.01 mg/kg (each in parent equivalents).
DFA: 0.02 mg/kg (in parent equivalents) in high water content and high acidic content matrices, and 0.05 mg/kg in high protein content, high starch content and high oil content matrices.
Description: Flupyradifurone residues are extracted twice from plant material with acetonitrile/water (4/1, v/v) with 2.2 mL/L formic acid. After rinsing and diluting with the same extraction solvent mixture, aliquots of the extracts are purified through a C-18 solid-phase extraction column, then amended with a mixture of stable, isotopically labelled internal standards. The final solution is analysed by HPLC-MS/MS. Two MRM transitions for quantitation and confirmation are monitored for flupyradifurone (*m/z* 289/126 or 90) and DFEAF (*m/z* 162/94 or 98). An HPLC-MS/MS method is highly specific, but the confirmatory ions were tested, and due to repeatability issues with flupyradifurone at the LOQ in some matrices, a second column system (Gemini C18, instead of HILIC as used in the primary method) was employed for confirmatory purposes with that compound. This column is also used as a confirmatory measurement of 6-CNA. For DFA, no second MRM transition is available. A Restek Allure Organic Acids HPLC column is therefore employed as a different separation system (as opposed to a HILIC column for the primary determination).

Method 01304/M001(Schoening, R.; Willmes, J.; 2014; M-476845-01-1)

Analyte: Flupyradifurone, DFA, 6-CNA and DFEAF (for data collection)
LOQ: Flupyradifurone and DFEAF: 0.01 mg/kg (each in parent equivalents) in cacao beans
DFA and 6-CNA: 0.02 mg/kg (in parent equivalents) in cacao beans
Description: Residues are extracted twice from cacao beans by blending with acetonitrile/water (4/1, v/v) + 2.2 mL/L formic acid. After centrifugation the clear supernatant was transferred into a volumetric flask and filled up to volume. For DFA and 6-CNA an aliquot of the crude extract was diluted with internal standard and acetonitrile/water (4/6, v/v) + 0.11 mL/L formic acid and the residues were quantified using reversed HPLC and MS/MS detection. For flupyradifurone and DFEAF an aliquot of the crude extract was evaporated to the aqueous reminder and cleaned up using a Chromabond XTR column. After elution of the residues with dichloromethane the eluate was evaporated to dryness and re-dissolved with acetonitrile/water (1/4, v/v). An aliquot of the solution was diluted with internal standard and acetonitrile/water (4/6, v/v) + 0.11 mL/L formic acid and the residues were quantified using reversed HPLC and MS/MS detection. One MRM transition was monitored for flupyradifurone, DFEAF, DFA and 6-CNA and each matrix tested: for flupyradifurone m/z 289 → 126, for DFEAF m/z 161 → 98, for DFA m/z 95 → 51 and for 6-CNA m/z 156 → 112.

Method Validation for Plant Commodities

Validation data for the methods used for determination of flupyradifurone residues in plant commodities for which supervised trial data were submitted to the current Meeting are summarized in Table 1 below. Concentrations are expressed in parent equivalents.

Table 1 Summary of method validation for plant commodities

Matrix	Fortification level (mg/kg)	n	Recoveries (%)	Mean recovery (%)	RSD (%)
Method 01330/M002 (Rzepta, S., 2014, M-469883-02-1)					
Flupyradifurone, m/z 289 → 126 for quantification					
Cacao, dried beans	0.05	5	92, 97, 98, 111, 101	100	7.1
	0.50	5	86, 82, 86, 73, 86	83	6.8

Matrix	Fortification level (mg/kg)	n	Recoveries (%)	Mean recovery (%)	RSD (%)
Coffee, green beans	0.10	5	72, 86, 79, 76, 70	77	8.2
	1.0	5	71, 81, 81, 80, 84	79	6.2
Coffee, roasted beans	0.10	5	78, 62, 70, 60, 92	72	18
	1.0	5	64, 95, 85, 79, 60	77	19
Flupyradifurone, m/z 289 → 90 for confirmation					
Cacao, dried beans	0.05	5	93, 99, 96, 110, 99	99	6.5
	0.50	5	86, 82, 87, 75, 86	83	6.0
Coffee, green beans	0.10	5	75, 86, 80, 76, 72	78	6.9
	1.0	5	73, 80, 81, 80, 84	80	5.1
Coffee, roasted beans	0.10	5	83, 63, 68, 64, 81	72	13
	1.0	5	66, 99, 88, 81, 61	79	20
DFA, m/z 95 → 19 for quantification					
Cacao, dried beans	0.10	5	85, 99, 87, 98, 85	91	7.8
	1.0	5	85, 91, 82, 89, 83	86	4.5
Coffee, green beans	0.20	5	92, 115, 106, 114, 111	108	8.7
	2.0	5	107, 110, 99, 97, 92	101	7.3
Coffee, roasted beans	0.20	5	101, 119, 87, 110, 79	99	17
	2.0	5	99, 101, 106, 105, 110	104	4.2
DFA, m/z 95 → 51 for confirmation					
Cacao, dried beans	0.10	5	82, 88, 89, 88, 82	86	4.1
	1.0	5	84, 92, 81, 88, 79	85	6.2
Coffee, green beans	0.20	5	86, 107, 106, 111, 111	104	10
	2.0	5	114, 116, 112, 104, 100	109	6.3
Coffee, roasted beans	0.20	5	97, 103, 80, 94, 74	90	14
	2.0	5	95, 102, 104, 108, 101	102	4.7
Method 01330/M002: Independent laboratory validation (Amic, S., 2014, M-493096-01-1)					
Flupyradifurone, m/z 289 → 126 for quantification					
Cacao, dried beans	0.05	5	78, 73, 77, 77, 80	77	3.3
	0.50	5	79, 82, 88, 87, 85	84	4.4
Coffee, green beans	0.10	4	90, 83, 86, 81	85	4.6
	1.0	5	85, 79, 82, 76, 83	81	4.4
Coffee, roasted beans	0.10	5	85, 87, 87, 91, 89	88	2.6
	1.0	5	101, 100, 99, 100, 96	99	1.9
Flupyradifurone, m/z 289 → 90 for confirmation					
Cacao, dried beans	0.05	5	77, 69, 75, 78, 81	76	5.9
	0.50	5	82, 83, 87, 85, 82	84	2.6
Coffee, green beans	0.10	4	88, 83, 89, 87	87	3.0
	1.0	5	82, 81, 84, 76, 82	82	4.3
Coffee, roasted beans	0.10	5	85, 86, 87, 87, 89	87	1.7
	1.0	5	106, 109, 95, 101, 94	101	6.5
DFA, m/z 95 → 19 for quantification					
Cacao, dried beans	0.10	5	80, 79, 85, 85, 82	82	3.4
	1.0	5	83, 82, 83, 84, 87	84	2.3
Coffee, green beans	0.20	5	95, 99, 97, 95, 95	96	1.9
	2.0	5	110, 102, 105, 100, 101	104	3.9
Coffee, roasted beans	0.20	5	78, 79, 77, 80, 81	79	2.0
	2.0	5	85, 88, 85, 83, 84	85	2.2
DFA, m/z 95 → 51 for confirmation					
Cacao, dried beans	0.10	5	79, 80, 86, 86, 82	83	4.0
	1.0	5	87, 84, 86, 86, 88	86	1.7
Coffee, green beans	0.20	5	94, 94, 98, 96, 96	96	1.8
	2.0	5	111, 106, 109, 105, 106	107	2.3
Coffee, roasted beans	0.20	5	79, 80, 79, 79, 81	80	1.1
	2.0	5	88, 89, 88, 86, 86	87	1.5
Method RV-001-P10-02 (Study IR-4 PR No. 10770)(including concurrent recoveries)					
Flupyradifurone					
Pomegranate, fruit	0.01	9	102, 99, 101, 93, 97, 89, 107, 86, 98	97	6.8
	1.0	3	106, 94, 95	98	6.8
DFA					
Pomegranate, fruit	0.02	9	90, 85, 93, 81, 101, 96, 101, 94, 90	92	7.3

Matrix	Fortification level (mg/kg)	n	Recoveries (%)	Mean recovery (%)	RSD (%)
	1.0	3	103, 107, 102	104	2.5
DFEAF					
Pomegranate, fruit	0.01	9	100, 101, 105, 92, 104, 96, 103, 94, 102	100	4.6
	1.0	3	107, 104, 105	105	1.5
6-CNA					
Pomegranate, fruit	0.01	9	108, 98, 107, 103, 103, 102, 105, 111, 99	104	4.1
	1.0	3	107, 102, 95	101	5.9
Method RV-001-P10-02 (Study RARVP074) (including concurrent recoveries)					
Flupyradifurone					
Coffee, green bean	0.01	20	85, 81, 84, 109, 90, 93, 99, 112, 92, 85, 105, 115, 105, 113, 104, 108, 101, 95, 89, 106	99	10.9
	0.5	2	92, 90	91	-
	1.0	6	86, 84, 86, 105, 104, 79	91	12.2
DFA					
Coffee, green bean	0.02	7	96, 91, 89, 101, 99, 103, 96	96	5.3
	0.05	13	98, 85, 85, 92, 90, 96, 95, 92, 86, 98, 100, 95, 102	93	6.1
	0.5	2	83, 82	83	-
	1.0	6	81, 82, 84, 92, 92, 84	86	5.7
DFEAF					
Coffee, green bean	0.01	20	103, 89, 74, 97, 76, 77, 91, 79, 81, 84, 87, 79, 92, 83, 86, 86, 86, 79, 89, 82	85	8.5
	0.5	2	89, 87	88	
	1.0	6	89, 90, 89, 85, 86, 77	86	5.6
6-CNA					
Coffee, green bean	0.01	20	78, 86, 77, 93, 81, 83, 88, 93, 80, 75, 98, 84, 97, 73, 86, 84, 89, 89, 89, 92	86	8.2
	0.5	2	94, 90	92	-
	1.0	6	89, 86, 88, 90, 91, 86	88	2.3
Method RV-001-P10-02 (Study I11-008)					
Flupyradifurone					
Coffee, green bean	0.01	5	87, 71, 96, 76, 74	81	12.9
	0.1	5	83, 84, 75, 92, 88	84	7.6
DFA					
Coffee, green bean	0.05	5	72, 70, 74, 74, 72	72	1.7
	0.5	5	76, 86, 82, 84, 86	83	4.1
DFEAF					
Coffee, green bean	0.05	5	81, 79, 84, 82, 75	80	4.3
	0.5	5	79, 79:75, 81, 79	79	2.8
6-CNA					
Coffee, green bean	0.01	5	97, 78, 90, 91, 105	92	10.8
	0.1	5	71, 76, 81, 88, 74	78	8.6
Method RV-001-P10-02 B (Study RARVY008)(including concurrent recoveries)					
Flupyradifurone					
Hops, kiln-dried cone	0.01	7	88, 91, 93, 97, 101, 96, 85	93	5.9
	2.4	3	99, 97, 97	98	1.2
	4.8	3	89, 89, 87	88	1.3
DFA					
Hops, kiln-dried cone	0.05	7	86, 88, 90, 89, 93, 95, 90	90	3.4
	2.4	3	87, 85, 82	85	3.0
	4.8	3	89, 91, 89	90	1.3
DFEAF					
Hops, kiln-dried cone	0.01	7	107, 94, 94, 97, 92, 95, 79	94	8.8
	2.4	3	99, 101, 103	101	2.0
	4.8	3	94, 92, 90	92	2.2

Matrix	Fortification level (mg/kg)	n	Recoveries (%)	Mean recovery (%)	RSD (%)
6-CNA					
Hops, kiln-dried cone	0.01	7	78, 81, 88, 91, 97, 85, 98	88	8.6
	2.4	3	100, 99, 99	99	0.6
	4.8	3	91, 95, 91	92	2.5
Method RV-001-P10-02 (Study RAGMN133-01) (including concurrent recoveries)					
Flupyradifurone					
Hops, kiln-dried cone	0.5	9	111, 84, 104, 90, 113, 98, 113, 92, 90	99	11.2
	20	6	109, 99, 118, 116, 102, 101	108	7.6
DFA					
Hops, kiln-dried cone	0.5	7	85, 62, 85, 72, 89, 88, 76	80	12.5
	20	6	96, 100, 98, 101, 95, 100	98	2.5
Method RV-001-P10-02 (Study 10-2225) (including concurrent recoveries)					
Flupyradifurone					
Hops, green cone	0.1	6	89, 89, 91, 94, 95, 107	94	7.2
	1.0	5	85, 86, 87, 92, 98	90	6.0
	5.0	1	87	-	-
Hops, kiln-dried cone	0.1	6	102, 103, 103,104, 105, 106	104	1.4
	1.0	5	107, 108, 111, 114, 115	111	3.2
	5.0	1	112	-	-
DFA					
Hops, green cone	0.2	6	91, 92, 95, 99, 100, 115	99	8.9
	1.0	5	76, 79, 83, 84, 94	83	8.2
	5.0	1	86	-	-
DFEAF					
Hops, green cone	0.1	6	68, 73, 79, 85, 95, 96	83	13.9
	1.0	5	76, 77, 78, 84, 91	81	7.8
	5.0	1	80	-	-
Hops, kiln-dried cone	0.1	6	89, 100, 106, 107, 107, 108	103	7.2
	1.0	5	108, 109, 110, 112, 114	111	2.2
	5.0	1	112	-	-
6-CNA					
Hops, green cone	0.1	6	85*, 92*, 93*, 95*, 96*, 111*	95	9.0
	1.0	5	83, 84, 89, 91, 95	88	5.6
	5.0	1	91	91	-
Hops, kiln-dried cone	0.1	6	90*, 95*, 99*, 101*, 103*, 106*	99	5.8
	1.0	5	108, 111, 112, 112, 117	112**	2.9
	5.0	1	111	-	-
Method RV-001-P10-02 (Study 10-3407) (including concurrent recoveries)					
Flupyradifurone					
Hops, beer	0.01	5	95, 100, 110, 114, 115	107	8.3
	0.10	3	105, 112, 116	111	5.0
Hops, brewer´s yeast	0.1	5	98, 109, 111, 111, 113	108	5.5
	1.0	3	77, 99, 102	93	14.7
Hops, draff	0.1	5	84, 94, 96, 105, 108	97	9.8
	1.0	3	101, 102, 105	103	2.0
DFA					
Hops, beer	0.02	5	93, 100, 110, 110, 113	105	8.0
	0.2	3	108, 108, 110	109	1.1
Hops, brewer´s yeast	0.2	5	99, 107, 109, 115, 116	109	6.3
	1.0	3	76, 90, 101	89	14.1
Hops, draff	0.2	5	98, 99, 108, 109, 111	105	5.8
	1.0	3	97, 101, 102	100	2.6
DFEAF					
Hops, beer	0.01	5	92, 94, 102, 111, 115	103	9.9
	0.1	3	102, 107, 112	107	4.7
Hops, brewer´s yeast	0.1	5	97, 107, 107, 110, 111	106	5.2
	1.0	3	85, 99, 109	98	12.3
Hops, draff	0.1	5	103, 105, 105, 105, 109	105	2.1
	1.0	3	100, 104, 107	104	3.4

Matrix	Fortification level (mg/kg)	n	Recoveries (%)	Mean recovery (%)	RSD (%)
6-CNA					
Hops, beer	0.01	5	83, 84, 98, 105, 109	96	12.4
	0.1	3	111, 111, 111	111	0.0
Hops, brewer´s yeast	0.1	5	107, 115, 116, 117, 118	115	3.8
	1.0	3	84, 98, 112	98	14.3
Hops, draff	0.1	5	92*, 98*, 105*, 116*, 133*	109	14.9
	1.0	3	111*, 111*, 111*	111**	0.0
Method RV-001-P10-03 (study AAFC12-054R)					
Flupyradifurone					
Blackberry, fruit	0.01	3	104, 106, 104	105	1.1
	0.02	3	107, 101, 98	102	4.5
	0.10	3	103, 95, 105	101	5.2
	2.4	3	107, 110, 109	109	1.4
Raspberry, fruit	0.01	3	99, 96, 99	98	1.8
	0.02	3	102, 97, 96	98	3.3
	0.10	3	102, 97, 104	101	3.6
	4.0	3	115, 108, 109	111**	3.4
Avocado	0.01	8	81, 73, 74, 85, 83, 80, 92, 80	81	7.5
	0.1	3	84, 100, 85	90	10.0
	0.5	3	93, 96, 83	91	7.5
DFA					
Blackberry, fruit	0.02	3	96, 102, 100	99	3.1
	0.04	3	94, 106, 102	101	6.1
	0.20	3	104, 101, 100	102	2.0
	2.4	3	111, 108, 104	108	3.3
Raspberry, fruit	0.02	3	90, 83, 89	87	4.3
	0.04	3	98, 101, 96	98	2.6
	0.20	3	97, 97, 100	98	1.8
	2.4	3	111, 110, 107	109	1.9
Avocado	0.05	5	116, 119, 98, 86, 116, 93, 86, 93	101	13.8
	0.50	5	95, 98, 97, 96, 80, 103	95	8.2
DFEAF					
Blackberry, fruit	0.01	3	87, 97, 102	95	8.0
	0.02	3	105, 99, 102	102	2.9
	0.10	3	108, 98, 104	103	4.9
	2.4	3	105, 102, 102	103	1.7
Raspberry, fruit	0.01	3	114, 105, 90	103	11.8
	0.02	3	102, 92, 97	97	5.7
	0.10	3	101, 97, 100	99	2.1
	4.0	3	109, 106, 105	107	2.0
6-CNA					
Blackberry, fruit	0.01	3	108, 114, 111	111	2.7
	0.02	3	108, 106, 111	108	2.3
	0.10	3	102, 103, 100	102	1.5
	2.4	3	87, 86, 82	85	3.1
Raspberry, fruit	0.01	3	98, 98, 88	95	6.1
	0.02	3	108, 105, 100	104	3.9
	0.10	3	101, 99, 101	100	1.2
	4.0	3	102, 103, 104	103	1.0
Method 01304/M001					
Flupyradifurone, m/z 289 → 126					
Cacao, green beans	0.01	5	90, 99, 101, 105, 106	100	6.4
	0.10	5	85, 98, 90, 91, 101	93	6.9
Cacao, fermented beans	0.01	5	108, 103, 104, 105, 104	105	1.8
	0.10	5	100, 103, 96, 102, 102	101	2.8
DFA, m/z 95 → 51					
Cacao, green beans	0.02	5	80, 92, 83, 93, 93	88	7.1
	0.20	5	89, 102, 101, 99, 100	98	5.4
Cacao, fermented beans	0.02	5	90, 81, 101, 99, 88	92	9.0
	0.20	5	100, 105, 100, 97, 100	100	2.9

Matrix	Fortification level (mg/kg)	n	Recoveries (%)	Mean recovery (%)	RSD (%)
DFEAF, m/z 161 → 98					
Cacao, green beans	0.01	5	85, 93, 88, 88, 86	88	3.5
	0.10	5	74, 80, 74, 77, 80	77	3.9
Cacao, fermented beans	0.01	5	88, 85, 80, 79, 79	82	5.0
	0.10	5	75, 78, 75, 77, 76	76	1.7
6-CNA, m/z 156 → 112					
Cacao, green beans	0.02	5	101, 101, 94, 98, 96	98	3.1
	0.20	5	84, 94, 86, 89, 90, 89	89	4.3
Cacao, fermented beans	0.02	5	106, 100, 102, 100, 100	102	2.6
	0.20	5	94, 94, 87, 89, 90	91	3.4

* recoveries were corrected for residue level detected in control sample

USE PATTERN

Flupyradifurone has been registered in many countries for use on crops including cane berries, avocado, pomegranate, hops, cacao and coffee for which supervised trial data were submitted to the current Meeting. The use pattern of flupyradifurone relevant to the supervised trials submitted to the current Meeting is summarized in Table 2. With the exception of coffee in Brazil, where a soil drench is possible, the application method for all other uses below are as foliar sprays in the field grown crops.

Table 2 Registered uses of flupyradifurone for the crops for which supervised trials were submitted.

Crop	Country	Conc. g ai/L or kg Form	Application					Minimum PHI, days (notes)
			Max No./crop/ season	Interval days	Water L/ha min-max	max g ai/ha (annual max)	g ai /hL	
Berries and other small fruits								
Cane berries (incl. blackberry & raspberry)	USA	200 SL	2	7	Min. 280 (ground) Min. 28 (aerial)	205 (410)		0
Assorted tropical and sub-tropical fruits – smooth inedible peel – large								
Avocado a/	USA	200 SL	2	14	Min. 234 (ground) Min. 94 (aerial)	205 (410)		1
Pomegranate [a]	USA	200 SL	2	7	Min. 234 (ground) Min. 94 (aerial)	205 (410)		0
Seeds for beverages								
Cacao beans	Ghana	75 EC [b]	4 (Aug, Sep, Oct, Dec)	-		15	37.5	7
Cacao beans	Côte d'Ivoire	75 EC [b]	2 (Dec/Jan, Jul/Aug)	-		18.75	47	-
Coffee beans	Brazil [b]	200 SL	1 (drench) 3 (foliar)	15 (for spray)[d]	50 ml/plant (drench) 400 (foliar)	600 (drench) (600) 200 (foliar) (600)		21
Dried herbs								
Hops, dry	USA	200 SL	1		Min. 234 (ground) Min. 94 (aerial)	154 (154)		21
Hops, dry	Canada	200 SL	1		Min. 100 (ground)	150 (150)		21

Crop	Country	Conc. g ai/L or kg Form	Application					Minimum PHI, days (notes)
			Max No./crop/ season	Interval days	Water L/ha min-max	max g ai/ha (annual max)	g ai /hL	
					Min. 20 (aerial)			
Hops, dry	Netherlands	200 SL	1 (BBCH 31-75)		2000-3300	150 (150)	Max 7.50	21

[a] They were included in a group of "tropical and subtropical, medium to large fruit, smooth, inedible peel", with different application intervals and PHIs.

[b] 75 g/L flupyradifurone with 10 g/L deltamethrin

[c] can be used also for drench treatment

[d] drench application: approximately 90 days before the first foliar application

RESULTS OF SUPERVISED RESIDUE TRIALS ON CROPS

The current Meeting received information on supervised trials using foliar spray of flupyradifurone conducted in support of estimating maximum residue levels for the following commodities: cane berries (blackberry and raspberry), avocado, pomegranate, cacao beans, coffee beans and hops, dry. The results of these supervised trials are summarized in the following tables:

Group/Sub-group	Commodity	Table No.
Berries and other small fruits (FB)		
Cane berries	Blackberry and Raspberry	3
Assorted tropical and sub-tropical fruits-inedible peel (FI)		
Assorted tropical and sub-tropical fruits – smooth inedible peel – large	Avocado	4
	Pomegranate	5
Seeds for beverages		
	Cacao beans	6
	Coffee beans	7
Dried herbs		
	Hops, dry	8

In addition to the description 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. Duration 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 trials used in the evaluation are summarized. 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 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 were used for the estimation of maximum residue levels, STMR and HR. Those results included in the tables are underlined.

For the calculation of sum of flupyradifurone, DFA and 6-CNA, expressed as parent equivalents (total residues), the Meeting used the approach agreed at the 2016 JMPR:

"Where parent or DFA residues were not detected or were less than the LOQ (*i.e.* < 0.01 mg/kg for parent or 0.05 mg/kg for DFA) the LOQ value was utilized for maximum residue estimation and dietary intake assessment. For 6-CNA, values less than the LOQ were not added for calculation of total residues of flupyradifurone."

The table below on how the total residues were calculated for each trial was copied from the Evaluation of the 2016 JMPR for easier reference.

Parent	DFA	6-CNA	Total
<0.01	0.05	0.01	0.07
0.01	<0.05	0.01	0.07
<0.01	<0.05	<0.01	<0.06
0.01	0.05	<0.01	0.06
0.01	0.05	0.01	0.07

All expressed in the parent equivalents.

The residue concentrations of DFEAF were also reported. While DFEAF is neither included in the residue definition for compliance with MRL nor the one for estimation of dietary exposure for plant commodities, DFEAF concentrations are shown in the following tables for consistency with the 2016 and 2017 JMPR Evaluations.

Berries and other small fruit

Cane berries

Eleven field trials were conducted on cane berries (four in blackberries and seven in raspberries) in Canada and the USA in the 2012-2014 growing seasons. Flupyradifurone SL 200 was applied as foliar broadcast sprays twice, with the interval of 6-8 days, on these crops at an application rate of 0.205 kg ai/ha each with the exception of two trials with lower rates (0.095-0.115 kg ai/ha). The second application was made on the day of harvest. An adjuvant was added to the spray mixtures, either non-ionic surfactant (0.05-0.2%), methylated seed oil (0.25%) or crop oil concentrate (1%).

For each trial, two independent samples of mature berries were harvested from the untreated and treated plots on the same day as the last application (0-day DALA). In addition to these samples, two independent treated raspberry (Trial AAFC12-054R-321) or blackberry (Trial AAFC12-054R-442) samples were harvested at 3, 7, 10 and 14-15 days after last application (DALA) to evaluate residue decline.

The residues of flupyradifurone, DFA and 6-CNA were determined with Method RV-001-P10-03 (HPLC-MS/MS). The LOQs were 0.01 mg/kg for flupyradifurone and 6-CNA and 0.02 mg/kg for DFA in these commodities, expressed in parent equivalents. Individual concurrent recoveries were all within the acceptable range of 70–120% and the RSD values were < 20%. In all the trials, fruits were analysed.

Table 3 Residues in blackberries and raspberries from supervised trials in the USA and Canada involving foliar application of flupyradifurone (200 SL formulation)

Trial No., Location Year (Blackberry Variety)	Application				DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage (GS)	Rate (g ai/ ha)	Vol. (L/ha)		Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
GAP USA Cane berries	2 (7)		205		0					
Blackberry										

Trial No., Location Year (Blackberry Variety)	Application				DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage (GS)	Rate (g ai/ ha)	Vol. (L/ha)		Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
GAP USA Cane berries	2 (7)		205		0					
AAFC12-054R AAFC12-054R-317 Holt, MI, USA 2012 (Illini)	2 (7)	fruiting plants fruiting	*110* *109*	151 149	0 0 mean	2.0 2.1 <u>2.1</u>	0.12 0.15 0.14	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	2.1 2.3 <u>2.2</u>
AAFC12-054R AAFC12-054R-322 Aurora, OR, USA 2012 (Marion)	2 (7)	mature & immature fruit mature & immature fruits	211 211	624 623	0 0 mean	1.5 1.6 <u>1.6</u>	0.13 0.11 0.12	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	1.6 1.7 <u>1.7</u>
AAFC12-054R AAFC12-054R-415 Holt, MI, USA 2013 (Illini)	2 (8)	fruiting mature, fruiting	*95* *115*	171 156	0 0 mean 3 3 mean 7 7 mean 10 10 mean 14 14 mean	0.56 0.29 0.43 0.54 0.44 <u>0.49</u> 0.29 0.28 0.29 0.13 0.30 0.22 0.16 0.14 0.15	0.022 <0.02 <0.02 0.063 0.052 0.058 0.085 0.072 0.079 0.061 0.11 0.086 0.14 0.10 0.12	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01	<0.01 <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	0.58 0.31 0.45 0.60 0.49 <u>0.55</u> 0.38 0.35 0.36 0.19 0.41 0.30 0.30 0.24 0.27
AAFC12-054R AAFC12-054R-442 Jordan, ON Canada 2014 (Chester)	2 (7)	mostly red berries mature berries	207 205	828 819	0 0 mean 3 3 mean 7 7 mean 10 10 mean 15 15 mean	0.81 0.81 <u>0.81</u> 0.52 0.55 0.54 0.51 0.46 0.49 0.39 0.34 0.37 0.27 0.28 0.28	0.023 0.032 0.028 0.040 0.041 0.041 0.077 0.072 0.075 0.10 0.092 0.096 0.13 0.13 0.13	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01	0.83 0.84 <u>0.84</u> 0.56 0.59 0.58 0.59 0.53 0.56 0.49 0.43 0.46 0.40 0.41 0.41
GAP USA Cane berries	2 (7)		205		0					
Raspberry										
AAFC12-054R AAFC12-054R-316 Cream Ridge, NJ USA 2012 (Heritage)	2 (7)	mature & immature fruit fruiting	206 208	286 293	0 0 mean	2.8 2.2 <u>2.5</u>	0.044 0.050 0.047	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	2.8 2.3 <u>2.5</u>
AAFC12-054R AAFC12-054R-318 Watsonville, CA USA 2012 (Z321.1)	2 (8)	mature canes with flowers and fruit mature canes with flowers and fruit	202 208	450 478	0 0 mean	0.66 0.39 0.53	<0.02 <0.02 <0.02	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	0.68 0.41 0.55

Trial No., Location Year (Blackberry Variety)	Application				DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage (GS)	Rate (g ai/ ha)	Vol. (L/ha)		Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
GAP USA Cane berries	2 (7)		205		0					
AAFC12-054R AAFC12-054R-319 Watsonville, CA USA 2012 (Z321.1)	2 (8)	mature canes with flowers and fruit mature canes with flowers and fruit	203 205	667 721	0 0 mean	1.6 0.45 <u>1.0</u>	<0.02 <0.02 <0.02	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	1.6 0.47 <u>1.0</u>
AAFC12-054R AAFC12-054R-320 Aurora, OR USA 2012 (Willamette)	2 (7)	mature & immature fruit mature fruit	206 206	377 378	0 0 mean	2.0 2.3 <u>2.2</u>	<0.02 <0.02 <0.02	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	2.0 2.3 <u>2.2</u>
AAFC12-054R AAFC12-054R-321 Jefferson, OR USA 2012 (Cascade Bounty)	2 (7)	mature & immature fruit Mature & immature fruit	211 213	478 482	0 0 mean 3 3 mean 7 7 mean 10 10 mean 14 14 mean	0.81 0.87 <u>0.84</u> 0.77 0.59 0.68 0.50 0.54 0.52 0.48 0.49 0.49 0.27 0.29 0.28	<0.02 <0.02 <0.02 <0.02 <0.02 <0.02 <0.02 <0.02 <0.02 0.029 0.034 0.032 0.028 0.039 0.034	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01	0.83 0.89 <u>0.86</u> 0.79 0.61 0.70 0.52 0.56 0.54 0.51 0.52 0.52 0.30 0.33 0.31
AAFC12-054R AAFC12-054R-323 Agassiz, BC Canada 2012 (Chemainus)	2 (8)	40% fruiting 85% fruiting	212 210	722 717	0 0 mean	1.2 0.98 <u>1.1</u>	<0.02 <0.02 <0.02	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	1.2 1.0 <u>1.1</u>
AAFC12-054R AAFC12-054R-335 Frelighsburg, QC Canada 2012 (Nova)	2 (6)	50% mature fruit 90% mature fruit	202 202	689 690	0 0 mean	2.4 2.6 <u>2.5</u>	0.029 0.04 0.035	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	2.4 2.6 <u>2.5</u>

No: number of applications; RTI: minimum retreatment intervalDALA: days after last application

Assorted tropical and sub-tropical fruits – smooth inedible peel – large

Avocado

Four supervised trials were conducted on avocado in the USA in 2013. In the supervised trials, avocado crops were sprayed twice with an SL formulation containing 200 g/L flupyradifurone at an application rate of approximately 0.205 kg ai/ha. In each plot, two different concentrations of flupyradifurone spray solutions were used in two parallel plots, but only the higher residue concentration of each trial was selected for estimation of maximum residue level. The first applications were made between BBCH 78 (development of fruit 80%) and 81 (beginning of ripening or fruit coloration). For all trials the interval between the two applications was 13 or 14 days. An adjuvant Dyne-Amic (0.25%, v/v) was added to the spray solutions. All applications were made using ground-based airblast equipment.

At each sampling, duplicate composite samples of avocado were harvested in the treated plots. Sampling took place when the plants were at BBCH 81. In the decline trials, avocado samples were collected at DALA of 0, 1, 7, 14, 21 and 28 days, between BBCH 79 (fruits have reached approximately 90% full size) and BBCH 81.

The residues of flupyradifurone and DFA were determined with Method RV-001-P10-03 (HPLC-MS/MS). The LOQs were 0.01 mg/kg for flupyradifurone and 0.05 mg eq/kg for DFA in avocado. Average concurrent recovery rates at the fortification levels of respective LOQs and higher concentrations were: 81–91% for flupyradifurone (fortification levels of 0.01–0.50 mg/kg) and 95–101% for DFA (fortification levels of 0.05–0.50 mg/kg). The RSD values were <20%. In all the trials, fruits were analysed.

Table 4 Residues in avocado from supervised trials in the USA involving foliar application of flupyradifurone (200 SL formulation)

Trial No., Location in the USA, Year (Avocado Variety)	Application				DALA	Residues as parent (mg/kg)*			
	No. (RTI, days)	GS	Rate (g ai/ha)	Vol. (L/ha)		Parent	DFA	6-CNA	Parent + DFA
GAP USA Avocado	2 (14)		205		1				
RARVN012 RV006-13HA RV006-13HA-TRTDC Homestead, FL, 2013 (Bonita)	2 (13)	81 85	197 202	487 519	1 1 *mean*	0.22 0.25 <u>0.24</u>	<0.05 <0.05 *<0.05*	n.a. n.a. *n.a.*	0.27 0.30 <u>0.29</u>
RARVN012 RV006-13HA RV006-13HA-TRTDD Homestead, FL, 2013 (Bonita)	2 (13)	81 85	201 208	4786 5080	1 1 mean	0.056 0.047 0.052	<0.05 <0.05 <0.05	n.a. n.a. n.a.	0.11 0.097 0.10
RARVN012 RV007-13DB RV007-13DB-TRTDC Arroyo Grande, CA, 2013 (Haas)	2 (14)	78 81	204 197	660 514	0 0 mean 1 1 mean 7 7 mean 14 14 mean 21 21 mean 28 28 mean	0.033 0.060 0.047 0.020 0.027 0.024 0.012 0.010 0.011 0.011 <0.01 0.011 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01	<0.05 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05	n.a. n.a. n.a. n.a. n.a. n.a. n.a. n.a. n.a. n.a. n.a. n.a. n.a. n.a. n.a. n.a. n.a. n.a.	0.083 0.11 0.097 0.070 0.077 0.074 0.062 0.060 0.061 0.061 <0.06 0.061 <0.06 <0.06 <0.06 <0.06 <0.06 <0.06
RARVN012 RV007-13DB RV007-13DB-TRTDD Arroyo Grande, CA, 2013 (Haas)	2 (14)	78 81	209 203	6742 6236	0 0 mean 1 1 *mean* 7 7 mean 14 14	0.048 0.030 0.039 0.028 0.023 <u>0.026</u> 0.019 0.016 0.017 0.021 0.017	<0.05 <0.05 <0.05 <0.05 <0.05 *<0.05* <0.05 <0.05 <0.05 <0.05 <0.05	n.a. n.a. n.a. n.a. n.a. *n.a.* n.a. n.a. n.a. n.a. n.a.	0.098 0.080 0.089 0.078 0.073 <u>0.076</u> 0.069 0.066 0.067 0.071 0.067

Trial No., Location in the USA, Year (Avocado Variety)	Application				DALA	Residues as parent (mg/kg)*			
	No. (RTI, days)	GS	Rate (g ai/ ha)	Vol. (L/ha)		Parent	DFA	6-CNA	Parent + DFA
GAP USA Avocado	2 (14)		205		1				
					mean	0.019	<0.05	n.a.	0.069
					21	<0.01	<0.05	n.a.	<0.06
					21	<0.01	<0.05	n.a.	<0.06
					mean	<0.01	<0.05	n.a.	<0.06
					28	<0.01	<0.05	n.a.	<0.06
					28	0.013	<0.05	n.a.	0.063
					mean	0.011	<0.05	n.a.	0.061
RARVN012 RV008-13DA RV008-13DA-TRTDC Riverside, CA, 2013 (Gwen)	2 (14)	79	205	686	0	0.32	0.077	n.a.	0.40
		79	206	662	0	0.26	0.093	n.a.	0.35
					mean	0.29	0.085	n.a.	0.37
					1	0.18	<0.05	n.a.	0.23
					1	0.20	0.058	n.a.	0.26
					mean	0.19	0.054	n.a.	0.24
					7	0.074	0.11	n.a.	0.19
					7	0.11	0.11	n.a.	0.22
					mean	0.091	0.11	n.a.	0.20
					14	0.084	0.18	n.a.	0.26
					14	0.11	0.17	n.a.	0.27
					mean	0.095	0.17	n.a.	0.27
					21	0.069	0.23	n.a.	0.30
					21	0.044	0.17	n.a.	0.22
					mean	0.056	0.20	n.a.	0.26
					28	0.12	0.25	n.a.	0.36
					28	0.064	0.20	n.a.	0.26
					mean	0.091	0.22	n.a.	0.31
RARVN012 RV008-13DA RV008-13DA-TRTDD Riverside, CA, 2013 (Gwen)	2 (14)	79	206	4922	0	0.11	<0.05	n.a.	0.16
		79	204	4949	0	0.11	<0.05	n.a.	0.16
					mean	0.11	<0.05	n.a.	0.16
					1	0.099	<0.05	n.a.	0.15
					1	0.12	<0.05	n.a.	0.17
					mean	0.11	<0.05	n.a.	0.16
					7	0.092	0.10	n.a.	0.19
					7	0.073	0.069	n.a.	0.14
					mean	0.082	0.086	n.a.	0.17
					14	0.051	0.085	n.a.	0.14
					14	0.059	0.083	n.a.	0.14
					mean	0.055	0.084	n.a.	0.14
					21	0.058	0.10	n.a.	0.16
					21	0.053	0.12	n.a.	0.17
					mean	0.055	0.11	n.a.	0.17
					28	0.052	0.14	n.a.	0.19
					28	0.035	0.14	n.a.	0.17
					mean	0.043	0.14	n.a.	0.18
RARVN012 RV009-13HA RV009-13HA-TRTDC Porterville, CA, 2013 (Zutano)	2 (14)	79	207	832	1	0.20	<0.05	n.a.	0.25
		81	206	819	1	0.25	<0.05	n.a.	0.30
					mean	0.22	*<0.05*	*n.a.*	0.27
RARVN012 RV009-13HA RV009-13HA-TRTDD Porterville, CA, 2013 (Zutano)	2 (14)	79	206	4927	1	0.081	<0.05	n.a.	0.13
		81	204	4967	1	0.053	<0.05	n.a.	0.10
					mean	0.067	<0.05	n.a.	0.12

Plot TRTDD: dilute spray application; Plot TRTDC: concentrated spray application;

No: number of applications;RTI: minimum retreatment interval;GS: growth stage;
DALA: days after last application; n.a.: not analysed
In these trials DFEAF was not analysed and therefore not included in the above table.

Pomegranate

Four supervised trials were conducted on pomegranate in the USA in 2012. In the supervised trials, pomegranate trees were sprayed twice with an SL formulation containing 200 g/L flupyradifurone at application rates of approximately 0.205 kg ai/ha. For all trials, the interval between the two applications was 6 or 7 days, except that in the trial 10770.12-CA10-T02, the interval was 11 days. An adjuvant was added to the spray solutions, either Silwet L-77 (silicone surfactant, 0.1% v/v) or Induce (non-ionic surfactant, 0.0125% v/v), or Dyne-Amic (vegetable oil, 0.33% v/v). In all the trails, pomegranate fruits were harvested 0, 7-8, 14, 27-29 and 33-36 days DALA.

The residues of flupyradifurone, DFA and 6-CNA were determined with Method RV-001-P10-02 (HPLC-MS/MS). The LOQs were 0.01 mg eq/kg for flupyradifurone and 6-CNA and 0.02 mg/kg for DFA in pomegranate. Average values of concurrent recovery rates at the fortification levels of respective LOQ and higher concentrations were: 97-98% for flupyradifurone; 92–104% for DFA; 100–105% for DFEAF; and 101–104% for 6-CNA. The RSD values were <20%. In all the trials, fruits were analysed.

Table 5 Residues in Pomegranate from supervised trials in the USA involving foliar application of flupyradifurone (200 SL formulation)

Trial No., Location in the USA, Year (Pomegranate Variety)	Application				DALA	Residues as parent (mg/kg)*				
	No. (RTI, days)	GS	Rate (g ai/ ha)	Vol. (L/ha)		Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
GAP USA Pomegranate	2 (7)		205		0					
IR-4 PR No. 10770	2	fruiting	205	355	0	0.23	<0.02	<0.01	<0.01	0.25
10770.12-CA08	(6)	fruiting	205	365	0	0.22	<0.02	<0.01	<0.01	0.24
10770.12-CA08-T02					mean	0.23	<0.02	<0.01	<0.01	0.25
Lost Hills, CA, 2012					8	0.20	<0.02	<0.01	<0.01	0.22
(Wonderful)					8	0.15	<0.02	<0.01	<0.01	0.17
					mean	0.18	<0.02	<0.01	<0.01	0.20
					14	0.14	<0.02	<0.01	<0.01	0.16
					14	0.15	<0.02	<0.01	<0.01	0.17
					mean	0.15	<0.02	<0.01	<0.01	0.17
					29	0.16	0.026	<0.01	<0.01	0.19
					29	0.076	0.023	<0.01	<0.01	0.099
					mean	0.12	0.025	<0.01	<0.01	0.14
					36	0.088	0.028	<0.01	<0.01	0.12
					36	0.14	0.042	<0.01	<0.01	0.18
					mean	0.11	0.035	<0.01	<0.01	0.15
IR-4 PR No. 10770	2	fruiting	207	748	0	0.14	<0.02	<0.01	<0.01	0.16
10770.12-CA09	(7)	fruiting	207	730	0	0.13	<0.02	<0.01	<0.01	0.15
10770.12-CA09-T02					mean	0.14	<0.02	<0.01	<0.01	0.16
USA					7	0.22	<0.02	<0.01	<0.01	0.24
Lost Hills, CA					7	0.075	<0.02	<0.01	<0.01	0.095
2012					mean	0.15	<0.02	<0.01	<0.01	0.17
(Wonderful)					14	0.10	<0.02	<0.01	<0.01	0.12
					14	0.10	<0.02	<0.01	<0.01	0.12
					mean	0.10	<0.02	<0.01	<0.01	0.12
					27	0.12	0.024	<0.01	<0.01	0.14
					27	0.038	0.022	<0.01	<0.01	0.060
					mean	0.079	0.023	<0.01	<0.01	0.10
					33	0.059	0.021	<0.01	<0.01	0.080
					33	0.039	0.034	<0.01	<0.01	0.073
					mean	0.049	0.028	<0.01	<0.01	0.077
IR-4 PR No. 10770	2	fruiting	196	851	0	0.20	<0.02	<0.01	<0.01	0.22

Trial No., Location in the USA, Year (Pomegranate Variety)	Application				DALA	Residues as parent (mg/kg)*				
	No. (RTI, days)	GS	Rate (g ai/ ha)	Vol. (L/ha)		Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
GAP USA Pomegranate	2 (7)		205		0					
10770.12-CA10	(11)	fruiting	196	851	0	0.16	<0.02	<0.01	<0.01	0.18
10770.12-CA10-T02					mean	0.18	<0.02	<0.01	<0.01	0.20
Davis, CA, 2012					7	0.073	0.025	<0.01	<0.01	0.098
(Wonderful)					7	0.12	0.024	<0.01	<0.01	0.14
					mean	0.097	0.025	<0.01	<0.01	0.12
					14	0.13	0.046	<0.01	<0.01	0.18
					14	0.06	0.031	<0.01	<0.01	0.091
					mean	0.095	0.039	<0.01	<0.01	0.13
					28	0.055	0.084	<0.01	<0.01	0.14
					28	0.063	0.022	<0.01	<0.01	0.085
					mean	0.059	0.053	<0.01	<0.01	0.11
					35	0.076	0.082	<0.01	<0.01	0.16
					35	0.077	0.093	<0.01	<0.01	0.17
					mean	0.077	0.088	<0.01	<0.01	0.16
IR-4 PR No. 10770	2	fruiting	216	692	0	0.20	<0.02	<0.01	<0.01	0.22
10770.12-CA11	(7)	fruiting	216	692	0	0.19	<0.02	<0.01	<0.01	0.21
10770.12-CA11-T02					mean	0.20	<0.02	<0.01	<0.01	0.22
Yuba City, CA					7	0.20	<0.02	<0.01	<0.01	0.22
2012					7	0.15	0.03	<0.01	<0.01	0.18
(Wonderful)					mean	0.18	0.025	<0.01	<0.01	0.20
					14	0.094	0.054	<0.01	<0.01	0.15
					14	0.12	0.058	<0.01	<0.01	0.18
					mean	0.11	0.056	<0.01	<0.01	0.16
					29	0.10	0.10	<0.01	<0.01	0.20
					29	0.088	0.14	<0.01	<0.01	0.23
					mean	0.094	0.12	<0.01	<0.01	0.21
					35	0.10	0.12	<0.01	<0.01	0.22
					35	0.063	0.12	<0.01	<0.01	0.18
					mean	0.082	0.12	<0.01	<0.01	0.20

No: number of applications;RTI: minimum retreatment interval;GS: growth stage;

DALA: days after last application

Trials CA08 and CA09 were conducted in the same location in different ranches with the application timing only a few days apart. Other differences in the trials were soil types (clay vs clay loam), age of trees (planted in 1999 vs 2006), different adjuvants used, and concentrations of spray solutions.

Seeds for beverages

Cacao beans

A total of nine supervised trials were conducted on cacao in Côte d'Ivoire and Ghana in 2014 and 2015. In the supervised trials, cacao trees were sprayed four times with an EC formulation containing 75g/L flupyradifurone and 10 g/L deltamethrin, at application rates in the range of 0.0155 to 0.021 kg ai/ha (flupyradifurone). The intervals between the applications were approximately one month.

Treated Samples of cacao pods were collected at BBCH 89 prior and directly after the last application, at DALA of 3, 7–10-11, 14–15, 20–21, 27-28 and 58–63, in accordance with the local practice. Pods were selected from all positions of the tree, high and low, exposed and covered by foliage. The quantity of pods picked was based on the density on the tree, i.e. more pods were taken from heavily laden parts.

Sampled pods were cut and dropped onto the ground, and afterwards they were picked from the ground and stored at ambient temperature for less than 24 hours, and then pulp with beans was removed from the peel. Peel was discarded. The pulp with the beans was wrapped into banana leaves and left into clean wooden boxes at ambient temperature during the fermentation process. After 6 to 7 days when the fermentation process ended, the wrapping material was removed and the beans were spread

over frames to dry. Frames were placed in open air but protected from rain. During drying, the beans were turned regularly to allow uniform drying. Each sample consisted of at least 1 kg of dry beans.

The residues of flupyradifurone, DFA and 6-CNA were determined with Method 01304/M001 (HPLC-MS/MS). The LOQs were 0.01 mg/kg for flupyradifurone and 0.02 mg eq/kg for DFA and 6-CNA. Average concurrent recovery rates at the fortification levels of respective LOQs and higher concentrations in dried cacao beans were: 95–98% for flupyradifurone; 95–104% for DFA; 98–99% for DFEAF; and 100–102% for 6-CNA. The RSD values were < 20%.

Table 6 Residues in cacao beans (dry) from supervised trials in Côte d'Ivoire and Ghana involving foliar application of flupyradifurone (85 EC formulation)

Trial No., Location, Year (Cacao Variety)	Application				DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	GS	Rate (g ai/ ha)	Vol. (L/ha)		Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
GAP GH Foliar Cacao	4 (-)[a]		15		7					
S14-00159	4	61-89	15.5	33	0*	<0.01	*0.033*	<0.01	<0.02	0.043
S14-00159-01	(30)	61-89	18.75	40	0	<0.01	0.041	<0.01	<0.02	0.051
S14-00159-01-2	(26)	61-89	18.75	40	0	<0.01	0.044	<0.01	<0.02	0.054
Plate Forme, Yamousoukro	(30)	61-89	18.75	40	mean	<0.01	0.043	<0.01	<0.02	0.053
Côte d'Ivoire					3	<0.01	0.066	<0.01	<0.02	0.076
2014					7	<0.01	0.043	<0.01	<0.02	0.053
(95% Forestiero, 5% Criollo)					7	<0.01	0.047	<0.01	<0.02	0.057
					mean	<u><0.01</u>	0.045	<0.01	<0.02	0.055
					11	<0.01	0.043	<0.01	<0.02	0.053
					15	<0.01	0.048	<0.01	<0.02	0.058
					20	<0.01	0.050	<0.01	<0.02	0.060
					28	<0.01	0.043	<0.01	<0.02	0.053
					58	<0.01	0.060	<0.01	<0.02	<u>0.070</u>
S14-00159	4	61-89	18.75	40	0*	<0.01	*0.049*	<0.01	<0.02	0.059
S14-00159-02	(30)	61-89	18.75	40	0	<0.01	0.033	<0.01	<0.02	0.043
S14-00159-02-2	(24)	61-89	21.0	45	3	<0.01	0.035	<0.01	<0.02	0.045
Bukaho, Agboville	(32)	61-89	18.75	40	7	<u><0.01</u>	0.055	<0.01	<0.02	0.065
Côte d'Ivoire					11	<0.01	0.070	<0.01	<0.02	0.080
2014					15	<0.01	0.065	<0.01	<0.02	0.075
(95% Forestero, 5% Criollo)					20	<0.01	0.057	<0.01	<0.02	0.067
					28	<0.01	0.075	<0.01	<0.02	0.085
					58	<0.01	0.089	<0.01	<0.02	<u>0.099</u>
S14-00159	4	61-89	19.9	58	0*	<0.01	<0.02	<0.01	<0.02	<0.03
S14-00159-03	(31)	61-89	18.75	55	0	<0.01	<0.02	<0.01	<0.02	<0.03
S14-00159-03-2	(28)	61-89	18.75	55	0	<0.01	<0.02	<0.01	<0.02	<0.03
Ntunkumso, Ashant	(28)	61-89	18.75	55	mean	<0.01	<0.02	<0.01	<0.02	<0.03
Ghana					3	<0.01	0.020	<0.01	<0.02	0.030
2014					7	<0.01	0.037	<0.01	<0.02	0.047
(Hybrid Bomso)					7	<0.01	0.037	<0.01	<0.02	0.047
					mean	<u><0.01</u>	0.037	<0.01	<0.02	0.047
					10	<0.01	0.034	<0.01	<0.02	0.044
					14	<0.01	0.041	<0.01	<0.02	0.051
					20	<0.01	0.030	<0.01	<0.02	0.040
					27	<0.01	0.040	<0.01	<0.02	0.050
					58	<0.01	0.049	<0.01	<0.02	<u>0.059</u>
S14-00159	4	61-89	18.75	55	0*	<0.01	<0.02	<0.01	<0.02	<0.03
S14-00159-04	(32)	61-89	18.75	55	0	<0.01	<0.02	<0.01	<0.02	<0.03
S14-00159-04-2	(28)	61-89	18.75	55	0	<0.01	<0.02	<0.01	<0.02	<0.03
Bosuso, Eastern Region	(25)	61-89	18.75	55	mean	<0.01	<0.02	<0.01	<0.02	<0.03
Ghana					3	<0.01	<0.02	<0.01	<0.02	<0.03
2014					7	<0.01	0.027	<0.01	<0.02	0.037

Trial No., Location, Year (Cacao Variety)	Application				DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	GS	Rate (g ai/ ha)	Vol. (L/ha)		Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
GAP GH Foliar Cacao	4 (-)[a]		15		7					
(99% Forestero 1% Criollo)					7	<0.01	<0.02	<0.01	<0.02	<0.03
					mean	<u><0.01</u>	0.024	<0.01	<0.02	0.034
					10	<0.01	0.029	<0.01	<0.02	0.039
					14	<0.01	0.039	<0.01	<0.02	0.049
					21	<0.01	0.026	<0.01	<0.02	0.036
					28	<0.01	0.041	<0.01	<0.02	<u>0.051</u>
					63	<0.01	0.029	<0.01	<0.02	0.039
S14-00159 S14-00159-05 S14-00159-05-2 Teawia, Easter Region NKwa Kwa, Ghana 2014 (95% Forestero 5% Criollo)	4 (31) (28) (25)	61-89 61-89 61-89 61-89	17.4 18.75 18.75 20.8	51 55 55 61	0* 0 3 7	<0.01 <0.01 <0.01 <0.01	<0.02 <0.02 <0.02 <0.02	<0.01 <0.01 <0.01 <0.01	<0.02 <0.02 <0.02 <0.02	<0.03 <0.03 <0.03 <0.03
					10	<0.01	0.034	<0.01	<0.02	0.044
					14	<0.01	0.030	<0.01	<0.02	0.040
					21	<0.01	0.023	<0.01	<0.02	0.033
					28	<0.01	0.028	<0.01	<0.02	0.038
					62	<0.01	<0.02	<0.01	<0.02	<0.03
S14-00159 S14-00159-06 S14-00159-06-2 Obugo, Ashant Ghana 2014 (95% Forestero 5% Criollo)	4 (31) (28) (28)	61-89 61-89 61-89 61-89	18.75 18.75 18.75 18.75	55 55 55 55	0* 0 3 7	<0.01 <0.01 <0.01 <u><0.01</u>	*0.027* 0.023 0.022 0.043	<0.01 <0.01 <0.01 <0.01	<0.02 <0.02 <0.02 <0.02	0.037 0.033 0.032 0.053
					10	<0.01	0.042	<0.01	<0.02	0.052
					14	<0.01	0.065	<0.01	<0.02	0.075
					20	<0.01	0.055	<0.01	<0.02	0.065
					27	<0.01	0.071	<0.01	<0.02	0.081
					58	<0.01	0.097	<0.01	<0.02	0.11
S15-04586 S15-04586-01 S15-04586-01-T2 Plate Forme, Yamoussoukro Côte d'Ivoire 2015 (95% Forestero, 5% Criollo)	4 (26) (28) (28)	61-89 61-89 61-87 61-89	18.75 18.75 18.75 20.4	40 40 40 44	0* 0 3 7	<0.01 <0.01 <0.01 <0.01	<0.02 <0.02 0.026 0.030	<0.01 <0.01 <0.01 <0.01	<0.02 <0.02 <0.02 <0.02	<0.03 <0.03 0.036 0.040
					11	<0.01	0.029	<0.01	<0.02	0.039
					15	<0.01	0.038	<0.01	<0.02	0.048
					22	<0.01	0.040	<0.01	<0.02	0.050
					27	<0.01	0.040	<0.01	<0.02	0.050
					62	<0.01	0.038	<0.01	<0.02	0.048
S15-04586 S15-04586-02 S15-04586-02-T2 Subiakro, Yamoussoukro Côte d'Ivoire 2015 (Forestero)	4 (26) (29) (27)	61-89 61-89 61-87 61-89	19.8 18.75 18.75 18.75	42 40 40 40	0* 0 3 7	<0.01 <0.01 <0.01 <u><0.01</u>	<0.02 0.034 0.027 0.030	<0.01 <0.01 <0.01 <0.01	<0.02 <0.02 <0.02 <0.02	<0.03 0.044 0.037 0.040
					10	<0.01	0.033	<0.01	<0.02	0.043
					14	<0.01	0.041	<0.01	<0.02	0.051
					21	<0.01	0.040	<0.01	<0.02	0.050
					26	<0.01	0.038	<0.01	<0.02	0.048
					61	<0.01	0.077	<0.01	<0.02	<u>0.087</u>
S15-04586 S15-04586-03 S15-04586-03-T2 Maoumou, Yamoussoukro Côte d'Ivoire 2015 (Forestero)	4 (27) (27) (29)	61-89 61-89 61-87 61-89	18.75 18.75 18.75 18.75	40 40 40 40	0* 0 3 7	<0.01 <0.01 <0.01 <u><0.01</u>	*0.025* 0.021 0.025 0.026	<0.01 <0.01 <0.01 <0.01	<0.02 <0.02 <0.02 <0.02	0.035 0.031 0.035 0.036
					9	<0.01	0.036	<0.01	<0.02	0.046
					13	<0.01	0.040	<0.01	<0.02	0.050
					20	<0.01	0.047	<0.01	<0.02	0.057
					25	<0.01	0.047	<0.01	<0.02	0.057
					60	<0.01	0.061	<0.01	<0.02	<u>0.071</u>

No: number of applications;RTI: minimum retreatment interval;GS: growth stage;

DALA: days after last application

[a] sprayed in a mixture with deltamethrin = EC formulation containing 75 g/L flupyradifurone and 10 g/L deltamethrin

* prior to last application

Coffee beans

A total of 16 supervised trials were conducted on coffee in Brazil, Colombia, Guatemala and Mexico in 2011 and 2012. In the supervised trials, a single drench application (114–118 days before harvest; at BBCH 72, 20% of fruit have reached final size, to BBCH 78, 89% of fruits have reached final size) was made followed by three broadcast foliar (airblast) spray treatment (BBCH 77, 70% of fruit have reached final size, to BBCH 88, nearly all fruits are fully ripe) to coffee trees with an SL formulation containing 200 g/L flupyradifurone. Rates of soil drench application ranged from 0.596 to 0.639 kg ai/ha. Individual foliar application rates ranged from 0.170 to 0.214 kg ai/ha per application. Total seasonal rates ranged from 1.118 to 1.240 kg ai/ha. The interval between the drench and the first foliar application was 86 to 91 days and interval between the foliar applications was 12–14 days. An adjuvant, methylated seed oil (MSO) or Dyne-Amic was used in all of the foliar applications at a rate of 0.25% (v/v).

Duplicate composite samples of coffee cherries were collected from the treated plots 0, 7–8, 13 to 15, 19 to 22 and 26 to 28 DALA. However, in Brazil in 2012, an additional sampling took place at 33–35 DALA.

Immediately after harvest, the coffee cherries were processed using the wet processing method typical of the region in which the trials were conducted. Using readily available hand operated equipment, the outer husk of the coffee cherries was removed and the remaining coffee beans were washed and allowed to ferment overnight in water to allow the mucilage (thin protective membrane surrounding the coffee beans) to loosen and be removed the next day by washing. For trial RV234-11DA, coffee cherries were not completely ripe and additional time was required to remove all of the husks, which made it impossible to remove all husks on the day of harvest. The coffee beans were spread out and allowed to air-dry in a protected area to avoid contamination. The coffee beans were turned regularly to promote drying. After the coffee beans, were allowed to dry to commercial dryness (8-11 days) the parchment (third layer of protective coating) was removed using hand operated equipment to yield the commodity, dried coffee bean, green.

The residues of flupyradifurone, DFA and 6-CNA were determined with Method RV-001-P10-02 (HPLC-MS/MS). The LOQs were 0.01 mg eq/kg for flupyradifurone and 6-CNA and 0.02 mg eq/kg for DFA for all sample materials. Average concurrent recovery rates at the fortification levels of respective LOQs and higher concentrations were: 91–99% for flupyradifurone, 83-96% for DFA, 85-88% for DFEAF and 86–92% for 6-CNA. The RSD values were <20%.

Table 6 Residues in coffee beans from supervised trials in Colombia, Brazil, Guatemala and Mexico involving drench application and foliar application of flupyradifurone (200 SL formulation)

Trial No., Location, Year (Coffee Variety)	Application				Sample	DALA	Residues as parent (mg/kg)*				
	No. (RTI, days)	GS	Rate (g ai/ ha)	Vol. (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
GAP Brazil Coffee	Drench 1 & foliar 3 (ca. 90 & 14)		Drench 600 & foliar 200			21					
RARVP074 RV232-11DA Cuilapa Guatemala 2011	4 (91) (13) (12)	78 79 80 88	600 199 201 201	227 394 412 367	bean, green	0	0.085	0.13	<0.01	<0.01	0.22
						0	0.079	0.23	<0.01	<0.01	0.31
						mean	0.082	0.18	<0.01	<0.01	0.26
						7	0.098	0.14	0.013	<0.01	0.24
						7	0.11	0.094	0.015	<0.01	0.20

Trial No., Location, Year (Coffee Variety)	Application				Sample	DALA	Residues as parent (mg/kg)*				
	No. (RTI, days)	GS	Rate (g ai/ ha)	Vol. (L/ha)			Parent	DFA	DFE AF	6-CNA	Parent + DFA + 6-CNA
GAP Brazil Coffee	Drench 1 & foliar 3 (ca. 90 & 14)		Drench 600 & foliar 200			21					
(Catuai)						mean	0.10	0.12	0.014	<0.01	0.22
						14	0.11	0.053	0.015	<0.01	0.17
						14	0.13	0.063	0.015	<0.01	0.19
						mean	0.12	0.058	0.015	<0.01	0.18
						21	0.12	0.1	0.014	<0.01	0.22
						21	0.11	0.097	0.018	<0.01	0.21
						mean	0.11	0.099	0.016	<0.01	0.21
						28	0.14	0.12	0.022	<0.01	0.26
						28	0.13	0.089	0.020	<0.01	0.22
						mean	0.14	0.11	0.021	<0.01	0.24
RARVP074	4	78	600	230	bean, green	0	0.047	0.10	<0.01	<0.01	0.15
RV233-11DA	(90)	81	199	401		0	0.055	0.12	<0.01	<0.01	0.18
Barberena	(14)	88	199	406		mean	0.051	0.11	<0.01	<0.01	0.16
Guatemala	(14)	88	199	370		7	0.045	0.11	<0.01	<0.01	0.15
2011						7	0.040	0.097	<0.01	<0.01	0.14
(Caturra)						mean	0.043	0.10	<0.01	<0.01	0.15
						14	0.061	0.12	<0.01	<0.01	0.18
						14	0.046	0.080	<0.01	<0.01	0.13
						mean	0.054	0.099	<0.01	<0.01	0.15
						21	0.063	0.14	<0.01	<0.01	0.20
						21	0.067	0.13	<0.01	<0.01	0.19
						mean	0.065	0.13	<0.01	<0.01	0.20
						28	0.052	0.12	<0.01	<0.01	0.17
						28	0.050	0.10	<0.01	<0.01	0.15
						mean	0.051	0.11	<0.01	<0.01	0.16
RARVP074	4	73	605	126	bean, green	0	0.21	0.35	0.012	0.011	0.57
RV234-11DA	(86)	77	199	394		0	0.19	0.67	0.017	<0.01	0.85
Zentla	(14)	79	197	395		mean	0.20	0.51	0.015	0.011	0.72
Mexico	(14)	81	199	402		7	0.16	0.65	0.023	0.013	0.82
2011						7	0.16	0.75	0.019	0.014	0.92
(Costa Rica)						mean	0.16	0.70	0.021	0.013	0.87
						14	0.10	0.22	0.015	<0.01	0.33
						14	0.13	0.40	<0.01	0.011	0.54
						mean	0.12	0.31	0.013	0.010	0.44
						21	0.14	0.50	<0.01	<0.01	0.65
						21	0.14	0.33	0.019	<0.01	0.47
						mean	0.14	0.42	0.015	<0.01	0.56
						28	0.12	0.33	0.015	<0.01	0.45
						28	0.11	0.51	0.019	<0.01	0.63
						mean	0.12	0.42	0.017	<0.01	0.54
RARVP074	4	72	609	195	bean, green	0	0.12	0.12	0.014	<0.01	0.24
RV246-11DA	(89)	81	197	397		0	0.12	0.11	0.014	<0.01	0.23
La Union,	(12)	85	195	393		mean	0.12	0.12	0.014	<0.01	0.24
Zihuateutla	(13)	85	203	414		7	0.25	0.13	0.028	<0.01	0.37
Mexico						7	0.24	0.13	0.030	<0.01	0.38
2011						mean	0.24	0.13	0.029	<0.01	0.37
(Caturra)						13	0.44	0.11	0.055	<0.01	0.55

Trial No., Location, Year (Coffee Variety)	Application				Sample	DALA	Residues as parent (mg/kg)*				
	No. (RTI, days)	GS	Rate (g ai/ ha)	Vol. (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
GAP Brazil Coffee	Drench 1 & foliar 3 (ca. 90 & 14)		Drench 600 & foliar 200			21					
						13	0.36	0.10	0.043	<0.01	0.46
						mean	0.40	0.11	0.049	<0.01	0.51
						20	0.46	0.12	0.064	0.010	0.59
						20	0.44	0.12	0.060	0.012	0.58
						mean	0.45	0.12	0.062	0.011	0.58
						26	0.59	0.31	0.090	0.020	0.91
						26	0.52	0.28	0.095	0.020	0.82
						mean	0.55	0.30	0.093	0.020	0.87
RARVP074 RV229-11DA Jardin Colombia 2012 (Castillo)	4 (90) (14) (14)	78 81 85 85	600 197 204 197	278 336 430 341	bean, green	0	0.13	0.29	<0.01	<0.01	0.41
						0	0.15	0.35	<0.01	<0.01	0.50
						mean	0.14	0.32	<0.01	<0.01	0.46
						7	0.17	0.36	0.010	<0.01	0.53
						7	0.18	0.31	0.011	<0.01	0.49
						mean	0.17	0.34	0.011	<0.01	0.51
						14	0.26	0.40	0.011	<0.01	0.65
						14	0.18	0.34	0.012	<0.01	0.52
						mean	0.22	0.37	0.012	<0.01	0.59
						21	0.24	0.40	0.013	<0.01	0.64
						21	0.19	0.39	0.012	<0.01	0.58
						mean	0.21	0.39	0.013	<0.01	0.61
						28	0.18	0.44	0.013	<0.01	0.62
						28	0.21	0.39	0.013	<0.01	0.60
						mean	0.20	0.41	0.013	<0.01	0.61
RARVP074 RV230-11DA Bolivar Colombia 2012 (2000)	4 (90) (14) (14)	78 81 85 89	600 198 201 200	278 329 484 339	bean, green	0	0.055	0.15	<0.01	<0.01	0.20
						0	0.036	0.12	<0.01	<0.01	0.15
						mean	0.045	0.13	<0.01	<0.01	0.18
						7	0.059	0.15	<0.01	<0.01	0.20
						7	0.062	0.15	<0.01	<0.01	0.21
						mean	0.061	0.15	<0.01	<0.01	0.21
						13	0.047	0.15	<0.01	<0.01	0.20
						13	0.082	0.15	<0.01	<0.01	0.23
						mean	0.065	0.15	<0.01	<0.01	0.22
						21	0.075	0.20	<0.01	<0.01	0.27
						21	0.099	0.23	<0.01	<0.01	0.33
						mean	0.087	0.21	<0.01	<0.01	0.30
						28	0.13	0.30	0.013	<0.01	0.43
						28	0.14	0.25	0.011	<0.01	0.40
						mean	0.14	0.27	0.012	<0.01	0.41
RARVP074 RV231-11DA Concordia Colombia 2012 (Caturra)	4 (90) (14) (14)	78 81 85 89	600 199 198 198	246 342 313 374	bean, green	0	0.061	0.066	<0.01	<0.01	0.13
						0	0.071	0.11	<0.01	<0.01	0.18
						mean	0.066	0.086	<0.01	<0.01	0.15
						7	0.09	0.081	<0.01	<0.01	0.17
						7	0.063	0.099	<0.01	<0.01	0.16
						mean	0.076	0.09	<0.01	<0.01	0.17
						13	0.094	0.079	<0.01	<0.01	0.17
						13	0.079	0.063	<0.01	<0.01	0.14
						mean	0.087	0.071	<0.01	<0.01	0.16

Trial No., Location, Year (Coffee Variety)	Application				Sample	DALA	Residues as parent (mg/kg)*				
	No. (RTI, days)	GS	Rate (g ai/ ha)	Vol. (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
GAP Brazil Coffee	Drench 1 & foliar 3 (ca. 90 & 14)		Drench 600 & foliar 200			21					
						20	0.081	0.12	<0.01	<0.01	0.2
						20	0.082	0.094	<0.01	<0.01	0.18
						mean	0.081	0.11	<0.01	<0.01	0.19
						27	0.15	0.12	0.012	<0.01	0.27
						27	0.17	0.13	0.014	<0.01	0.31
						mean	0.16	0.13	0.013	<0.01	0.29
I11-008 I11-008-01 Ribeirao Preto, Sao Paulo Brazil 2011 (Catuai)	4 (90) (15) (15)	81 85 88 89	600 202 208 186	## 400 400 400	bean	0	0.03	<0.05	<0.01	<0.01	0.08
						0	0.04	<0.05	<0.01	<0.01	0.09
						mean	0.04	<0.05	<0.01	<0.01	0.09
						7	0.03	<0.05	<0.01	<0.01	0.08
						7	<0.01	<0.05	<0.01	<0.01	<0.06
						mean	0.02	<0.05	<0.01	<0.01	0.07
						14	<0.01	<0.05	<0.01	<0.01	<0.06
						14	<0.01	<0.05	<0.01	<0.01	<0.06
						mean	<0.01	<0.05	<0.01	<0.01	<0.06
						21	<0.01	<0.05	<0.01	<0.01	<0.06
						21	<0.01	<0.05	<0.01	<0.01	<0.06
						mean	≤0.01	<0.05	<0.01	<0.01	≤0.06
						28	<0.01	<0.05	<0.01	<0.01	<0.06
						28	<0.01	<0.05	<0.01	<0.01	<0.06
						mean	<0.01	<0.05	<0.01	<0.01	<0.06
I11-008 I11-008-02 Paulinia, Sao Paulo Brazil 2011 (Catuai-Vermelho)	4 (90) (15) (14)	75 88 88 89	596 212 200 192	## 400 400 400	bean	0	0.04	<0.05	<0.01	0.01	0.10
						0	0.04	<0.05	<0.01	<0.01	0.09
						mean	0.04	<0.05	<0.01	0.01	0.10
						7	0.04	<0.05	<0.01	0.01	0.10
						7	0.03	<0.05	<0.01	<0.01	0.08
						mean	0.04	<0.05	<0.01	0.01	0.10
						14	0.03	<0.05	<0.01	<0.01	0.08
						14	0.04	<0.05	<0.01	<0.01	0.09
						mean	0.04	<0.05	<0.01	<0.01	0.09
						21	0.02	<0.05	<0.01	<0.01	0.07
						21	0.02	<0.05	<0.01	0.01	0.08
						mean	0.02	<0.05	<0.01	0.01	0.08
						28	0.07	0.09	0.01	0.01	0.17
						28	0.08	0.10	0.01	0.02	0.20
						mean	0.08	0.10	0.01	0.02	0.19
I11-008 I11-008-04 Londrina, Parana Brazil 2011 (Catuai)	4 (90) (15) (14)	73 85 88 89	598 206 170 214	## 400 400 400	bean	0	0.02	<0.05	<0.01	<0.01	0.07
						0	0.02	<0.05	<0.01	<0.01	0.07
						mean	0.02	<0.05	<0.01	<0.01	0.07
						7	n.d.	<0.05	<0.01	<0.01	<0.06
						7	n.d.	<0.05	<0.01	<0.01	<0.06
						mean	n.d.	<0.05	<0.01	<0.01	<0.06
						14	<0.01	<0.05	<0.01	<0.01	<0.06
						14	0.01	<0.05	<0.01	<0.01	0.06
						mean	0.01	<0.05	<0.01	<0.01	0.06
						21	0.05	<0.05	<0.01	<0.01	0.10
						21	0.05	<0.05	<0.01	<0.01	0.10
						mean	0.05	<0.05	<0.01	<0.01	0.10
						28	0.03	<0.05	<0.01	<0.01	0.08
						28	0.03	<0.05	<0.01	<0.01	0.08
						mean	0.03	<0.05	<0.01	<0.01	0.08

Trial No., Location, Year (Coffee Variety)	Application				Sample	DALA	Residues as parent (mg/kg)*				
	No. (RTI, days)	GS	Rate (g ai/ ha)	Vol. (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
GAP Brazil Coffee	Drench 1 & foliar 3 (ca. 90 & 14)		Drench 600 & foliar 200			21					
I11-008	4	85	606	##	bean	0	0.02	<0.05	<0.01	<0.01	0.07
I11-008-05	(90)	87	212	400		0	0.02	<0.05	<0.01	<0.01	0.07
Cristais Paulista, Sao Paulo	(15)	88	200	400		mean	0.02	<0.05	<0.01	<0.01	0.07
Brazil	(13)	89	202	400		7	0.02	<0.05	<0.01	<0.01	0.07
2011						7	0.01	<0.05	<0.01	<0.01	0.06
(Mundo Novo)						mean	0.02	<0.05	<0.01	<0.01	0.07
						14	n.d.	<0.05	<0.01	<0.01	<0.06
						14	<0.01	<0.05	<0.01	<0.01	<0.06
						mean	<0.01	<0.05	<0.01	<0.01	<0.06
						21	n.d.	<0.05	<0.01	<0.01	<0.06
						21	<0.01	<0.05	<0.01	<0.01	<0.06
						mean	<0.01	<0.05	<0.01	<0.01	<0.06
						28	<0.01	<0.05	<0.01	<0.01	<0.06
						28	<0.01	<0.05	<0.01	<0.01	<0.06
						mean	<0.01	<0.05	<0.01	<0.01	<0.06
I12-006	4	75	600	200	bean	0	0.20	<0.05	<0.01	<0.01	0.25
I12-006-01	(90)	81	200	400		7	0.23	<0.05	0.010	<0.01	0.28
Paulinia, Sao Paulo	(14)	83	200	400		14	0.20	0.10	<0.01	<0.01	0.30
Brazil	(15)	85	197.6	400		22	0.17	0.10	<0.01	<0.01	0.27
2012						28	0.20	0.09	<0.01	0.010	0.30
(Catuai Vermelho)						35	0.19	0.07	<0.01	<0.01	0.26
I12-006	4	73	600.6	200	bean	0	0.08	<0.05	0.01	<0.01	0.13
I12-006-02	(90)	85	208.4	400		8	0.08	<0.05	0.01	<0.01	0.13
Campinas, Sao Paulo	(15)	85	194.8	400		14	0.08	<0.05	<0.01	<0.01	0.13
Brazil	(15)	85	195.6	400		20	0.08	<0.05	<0.01	<0.01	0.13
2012						28	0.07	<0.05	<0.01	<0.01	0.12
(Catuai Vermelho)						35	0.22	0.08	0.02	<0.01	0.30
I12-006	4	73	602.4	200	bean	0	0.04	0.10	<0.01	<0.01	0.14
I12-006-03	(90)	79	190.6	400		7	0.02	0.10	<0.01	<0.01	0.12
Londrina, Parana	(15)	80	200.8	400		14	0.03	0.12	<0.01	<0.01	0.15
Brazil	(15)	81	200.2	400		19	0.02	<0.05	<0.01	<0.01	0.07
2012						28	0.02	0.07	<0.01	<0.01	0.09
(Catuai)						33	0.02	0.08	<0.01	<0.01	0.10
I12-006	4	81	639.2	200	bean	0	0.01	<0.05	<0.01	<0.01	0.06
I12-006-04	(90)	81	205.8	400		7	0.05	<0.05	<0.01	0.01	0.11
Ribeirao Preto, Sao Paulo	(15)	81	200.2	400		14	0.08	<0.05	<0.01	<0.01	0.13
Brazil	(16)	81	194.6	400		21	0.21	0.14	0.01	0.02	0.37
2012						28	0.27	0.12	0.01	0.01	0.40
(Catuai)						35	0.60	0.15	0.01	0.02	0.77
I12-006	4	75	617.4	200	bean	0	0.04	<0.05	<0.01	<0.01	0.09
I12-006-05	(90)	81	195.8	400		7	0.24	0.06	<0.01	0.02	0.32
Cristais Paulista, Sao Paulo	(15)	81	204.2	400		15	0.25	0.06	0.01	0.01	0.32
Brazil	(16)	85	192	400		20	0.35	0.12	0.02	0.02	0.49
2012						28	0.18	0.09	<0.01	<0.01	0.27
(Mundo novo)						35	0.26	0.10	<0.01	<0.01	0.36

No: number of applications;

RTI: minimum retreatment interval;

GS: growth stage;

DALA: days after last application

no information in report

Dried herbs

Hops, dry

A total of 12 field trials (four in the USA and eight in Germany) were conducted on hops in the 2010, 2011 and 2015 growing seasons. Flupyradifurone 200 SL was applied once as foliar spray at rates of 0.150-0.156 kg ai/ha except that in four trails in Germany the rate was 0.120 kg ai/ha

USA trials

Four field trials were conducted on hops following one broadcast foliar spray applications of flupyradifurone 200 SL in 2011 (3) and 2015 (1). Diluted and concentrated foliar airblast applications were tested in parallel plots. Individual application rates ranged from 0.154 to 0.156 kg ai/ha for the concentrated plot and from 0.152 to 0.155 kg ai/ha for the diluted plot. All applications were made at BBCH growth stage 85 (advanced ripening). All applications were made using ground-based equipment. Adjuvants were used in the trials, such as a non-ionic surfactant (NIS) at 0.2% (v/v), a crop oil concentrate (COC) at 1.0% (v/v), and a methylated seed oil (MSO) at 0.25% (v/v). The same adjuvant was used for the pair of plots for concentrated and diluted applications.

In the 2011 trials, single composite samples of fresh hop cones from both the concentrated and diluted spray plots, along with an untreated control sample, were collected 21 days after the application. The fresh hops were kiln-dried on the day of harvest to generate the dried hop cones. In the 2015 trial, duplicate composite samples of fresh hop cones were collected from the treated plot at the DALA of 0, 7, 14, 21, 28 and 35 (BBCH 85-89).

The residues of flupyradifurone, DFA and 6-CNA were determined with Method RV-001-P10-02 (HPLC-MS/MS). The LOQs in the 2011 trials were 0.01 mg eq/kg for flupyradifurone and 6-CNA: and 0.05 mg eq/kg for DFA in dried hop cone. Average recoveries at fortification levels of respective LOQ and higher were all within the acceptable range of 70–120%. The RSD values were < 20%.

The LOQs in the 2015 trial were 0.5 mg eq/kg for flupyradifurone and DFA in dried hop cone. Concurrent recoveries were within the acceptable range of 70–120%. The RSD values were < 20%. 6-CNA was not analysed in this trial, but is relevant to the residue definition for risk assessment. Assuming that the LOQ for 6-CNA was 0.5 mg eq/kg, this value was added to the sum of flupyradifurone and DFA-residues to estimate the total residue. In all the trials on hops according to the cGAP and where 6-CNA was determined, the concentrations of 6-CNA were < 0.5 mg eq/kg, except for one trial showing 0.73 mg/kg. Therefore, addition of 0.5 mg eq/kg as 6-CNA covers most of cases occurring in reality.

German trials

Eight residue trials were conducted on hops in the 2010 (4) and 2011 (4) seasons in Germany. Flupyradifurone 200 SL was applied once at BBCH 71–86 as foliar spray at application rates of 0.12 kg ai/ha (2010 trials) or 0.15 kg ai/ha (2011 trials). All applications were made using ground-based equipment, without adjuvant.

Composite samples of fresh hop cones were collected from the treated plot at the DALA of 0, 7–8, 13–14, 20-22 and 26-28 (BBCH 71-91). The fresh hops were kiln-dried on the day of harvest to generate the dried hop cones except for the samples harvested directly after the treatment and 7-8 days after application. The samples were then deep-frozen within 14.5- 25 hours after sampling.

The residues of flupyradifurone, DFA and 6-CNA were determined with Method RV-001-P10-02 (HPLC-MS/MS). The LOQs were 0.1 mg eq/kg for flupyradifurone and 6-CNA and 0.2 mg eq/kg for DFA in dried hop cone. Average recoveries at fortification levels of respective LOQs and higher concentrations were all within the acceptable range of 70–120%. The RSD values were < 20%.

Table 8 Residues in dried hops from supervised trials in Germany and the USA involving foliar application of flupyradifurone (200 SL formulation)

Trial No., Location Year (Type-Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage (BBCH)	Rate (g ai/ ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
GAP NL Foliar Hops	1		150			21					
10-2225	1	73-74	120	3000	cone, green	0	1.3	<0.2	<0.1	<0.1	1.5
10-2225-01						7	0.62	<0.2	<0.1	0.15	0.97
Ellingen						14	0.29	<0.2	<0.1	<0.1	0.49
Germany						21	0.52	<0.2	<0.1	<0.1	0.72
2010						28	0.16	<0.2	<0.1	<0.1	0.36
(Hallertauer Gold)					cone, kiln-dried	14	1.5	0.27	<0.1	0.16	1.9
						21	0.81	0.20	<0.1	<0.1	1.0
						28	1.1	0.40	<0.1	0.15	1.7
10-2225	1	75	120	2200	cone, green	0	0.49	<0.2	<0.1	<0.1	0.69
10-2225-02						8	0.27	<0.2	<0.1	<0.1	0.47
Luetzensoemmern						13	0.19	<0.2	<0.1	<0.1	0.39
Germany						20	<0.1	<0.2	<0.1	<0.1	<0.3
2010						27	<0.1	<0.2	<0.1	<0.1	<0.3
(Magnum)					cone, kiln-dried	13	0.54	<0.2	<0.1	0.73	1.5
						20	0.48	<0.2	<0.1	0.73/ 0.64 [a]	1.4/ 0.94 [a]
						27	<0.1	<0.2	<0.1	0.75	1.1
10-2225	1	71-75	120	2200	cone, green	0	1.4	<0.2	<0.1	<0.1	1.6
10-2225-03						7	0.54	<0.2	<0.1	<0.1	0.74
Muegeln						14	0.36	<0.2	<0.1	<0.1	0.56
Germany						21	0.20	<0.2	<0.1	<0.1	0.40
2010						28	<0.1	<0.2	<0.1	<0.1	<0.3
(Hallertauer Magnum)					cone, kiln-dried	14	1.4	0.25	<0.1	0.13	1.8
						21	0.77	0.28	<0.1	0.13/ 0.10 [a]	1.2/ 0.40 [a]
						28	0.32	<0.2	<0.1	0.15	0.67
10-2225	1	85	120	2200	cone, green	0	0.56	<0.2	<0.1	<0.1	0.76
10-2225-04						8	0.27	<0.2	<0.1	<0.1	0.47
Tettnang						14	0.17	<0.2	<0.1	<0.1	0.37
Germany						21	**0.14**	<0.2	<0.1	<0.1	**0.34**
2010						28	<0.1	<0.2	<0.1	<0.1	<0.3
(Hallertauer Tradition)					cone, kiln-dried	14	0.54	<0.2	<0.1	0.16	0.90
						21	0.90	0.21	<0.1	0.26/ 0.16 [a]	1.4/ 0.46 [a]
						28	0.49	<0.2	<0.1	0.15	0.84
11-2076	1	75	150	2500	cone, green	0	2.4	<0.2	<0.1	<0.1	2.6
11-2076-01						14	0.47	<0.2	<0.1	<0.1	0.67
Ellingen						21	0.51	<0.2	<0.1	<0.1	0.71
Germany						28	0.39	<0.2	<0.1	<0.1	0.59
2011					cone, kiln-dried	21	1.0	0.36	<0.1	<0.1	1.4
(Hallertauer mittelfrüh)						28	1.8	0.50	<0.1	0.12	2.4
11-2076	1	73	150	2500	cone, green	0	0.55	<0.2	<0.1	<0.1	0.75
11-2076-02						14	0.21	<0.2	<0.1	<0.1	0.41
Luetzensoemmern						21	<0.1	<0.2	<0.1	<0.1	<0.3
Germany						28	0.10	<0.2	<0.1	<0.1	0.30

Trial No., Location Year (Type-Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage (BBCH)	Rate (g ai/ ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
GAP NL Foliar Hops	1		150			21					
2011 (Magnum)					cone, kiln-dried	21	0.26	<0.2	<0.1	0.24/ 0.26 [a]	0.70/ 0.56 [a]
						28	0.31	<0.2	<0.1	0.22	0.73
11-2076 11-2076-03 Meinitz Germany 2011 (Hallertauer Tradition)	1	84-86	150	2500	cone, green	0	2.1	<0.2	<0.1	<0.1	2.3
						13	0.78	<0.2	<0.1	<0.1	0.98
						20	0.57	<0.2	<0.1	<0.1	0.77
						26	0.23	<0.2	<0.1	<0.1	0.43
					cone, kiln-dried	20	2.0	0.27	<0.1	<0.1	2.3
						26	0.49	<0.2	<0.1	<0.1	0.69
11-2076 11-2076-04 Tettnang Germany 2011 (Tettnanger)	1	75-78	150	2000	cone, green	0	0.61	<0.2	<0.1	<0.1	0.81
						13	0.11	<0.2	<0.1	<0.1	0.31
						22	<0.1	<0.2	<0.1	<0.1	<0.3
						28	0.11	<0.2	<0.1	<0.1	0.31
					cone, kiln-dried	22	0.43	<0.2	<0.1	<0.1	0.63
						28	0.29	<0.2	<0.1	<0.1	0.49
GAP US Foliar Hops	1		154			21					
RARVY008 RV047-11HA RV047-11HA-TRTDC Wilder, ID USA 2011 (Apollo)	1	85	156	422	cone, kiln-dried	21	2.4	0.90	0.011	0.092/ 0.064 [a]	3.4/ 0.12 [a]
RARVY008 RV047-11HA RV047-11HA-TRTDD Wilder, ID USA 2011 (Apollo)	1	85	155	1178	cone, kiln-dried	21	2.2	0.96	<0.01	0.089/ 0.064	3.2/ 0.12 [a]
RARVY008 RV048-11HA RV048-11HA-TRTDC Ephrata, WA USA 2011 (Cascade)	1	85	155	421	cone, kiln-dried	21	4.6	3.3	0.037	0.19/ 0.017 [a]	8.1/ 0.077 [a]
RARVY008 RV048-11HA RV048-11HA-TRTDD Ephrata, WA USA 2011 (Cascade)	1	85	154	974	cone, kiln-dried	21	4.7	3.0	0.07	0.24/ 0.017 [a]	7.9/ 0.077 [a]

Trial No., Location Year (Type-Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	Growth Stage (BBCH)	Rate (g ai/ ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
GAP NL Foliar Hops	1		150			21					
RARVY008 RV049-11HA RV049-11HA-TRTDC St. Paul, OR USA 2011 (Nugget)	1	85	154	315	cone, kiln-dried	21	2.3	0.80	<0.01	0.051/ 0.016 [a]	3.1/ 0.076 [a]
RARVY008 RV049-11HA RV049-11HA-TRTDD St. Paul, OR USA 2011 (Nugget)	1	85	152	595	cone, kiln-dried	21	2.7	0.64	<0.01	0.047/ 0.016 [a]	3.4/ 0.076 [a]
RAGMN133-01 GM007-15DA GM007-15DA-TRTD Ephrata, WA USA 2015 (Cascade)	1	85	153	466	cone, kiln-dried	0	12	<0.5	n.a.	n.a.	13
						0	16	<0.5	n.a.	n.a.	17
						mean	14	<0.5	n.a.	n.a.	15
						7	6.3	<0.5	n.a.	n.a.	6.8
						7	7.4	<0.5	n.a.	n.a.	7.9
						mean	6.9	<0.5	n.a.	n.a.	7.4
						14	5.9	<0.5	n.a.	n.a.	6.4
						14	4.2	<0.5	n.a.	n.a.	4.7
						mean	5.1	<0.5	n.a.	n.a.	5.6
						21	2.4	<0.5	n.a.	n.a.	2.9
						21	2.6	<0.5	n.a.	n.a.	3.1
						mean	2.5	<0.5	n.a.	n.a.	3.0
						28	2.4	<0.5	n.a.	n.a.	2.9
						28	3.0	<0.5	n.a.	n.a.	3.5
						mean	2.7	<0.5	n.a.	n.a.	3.2 [b]
						35	2.5	<0.5	n.a.	n.a.	3.0
						35	2.4	<0.5	n.a.	n.a.	2.9
						mean	2.5	<0.5	n.a.	n.a.	3.0

Plot TRTDD: dilute spray application; Plot TRTDC: concentrated spray application;

No: number of applications;RTI: minimum retreatment interval;GS: growth stage;

DALA: days after last application

[a] Residue detected in control sample

[b] Parent + DFA

FATE OF RESIDUES DURING PROCESSING

Information and data from residues in processed commodities

A study on the effects of heating at different pH and temperature on the flupyradifurone residues was evaluated by the 2016 JMPR which concluded that flupyradifurone was not degraded during the simulation of pasteurization (pH 4, 90 °C, 20 minutes), baking, boiling or brewing (pH 5, 100 °C, 60 minutes) or during sterilization (pH 6, 120 °C, 20 minutes).

The effects of processing on the concentrations of flupyradifurone residues were also evaluated by the 2016 JMPR for citrus fruit (orange), pome fruit (apples), grapes, strawberries, brassica vegetables (broccoli), fruiting vegetables (summer squash, tomato, cucumber), leafy vegetables (Indian mustard, spinach), pulses (soya bean), root and tuber vegetables (carrot, potato), cereals (barley, maize, wheat), oilseed crops (cotton, peanut), coffee, and hops. In these crops, residues may occur in the Raw Agricultural Commodity (RAC) and thus may be carried over into processed products. In addition processing studies on peaches, plums and cherries were evaluated by the 2017 JMPR.

The current Meeting received information on the processing of cacao beans, coffee beans and dried hops processed commodities, relevant to the current evaluation.

Cacao beans (Petrova, D.,2017, S15-04586)

The samples of cacao beans taken in the two supervised residue trials conducted in Cote d'Ivoire in 2015 on cacao (Table 6) were used in the processing study. After the collection of cacao pods, fermented and dried cacao beans were obtained as the "unprocessed" commodity and they were processed into roasted beans (nibs), cocoa powder and chocolate. The processing procedures simulated industrial practices at a laboratory scale.

Breaking of beans

Cacao beans were placed into a roller mill for breaking them into smaller pieces generating nibs and shells. After breaking, the shells and shell components were separated from the nibs by an air separation process.

Roasting of cocoa nibs

The shell-free nibs were placed into a pre-heated (up to 125 °C) air convection drying cabinet (oven) for 20 minutes. A fraction of the roasted nibs was sampled and stored deep-frozen.

Milling / Cocoa liquor production

For milling the roasted nibs a ball mill was used. The ball mill works on the principle of impact and attrition – size reduction done by impact and/or friction of steel balls with the substance for milling. The rest of roasted nibs were placed together with steel balls into a ball mill for production of cocoa liquor. The steel balls were previously warmed up into a heating cabinet. The ratio of steel balls and cocoa nibs for the milling process was approximately 10:1. During the milling process the "thermo jacket", a compartment of the mill where the roasted nibs and steel balls were placed, was constantly supplied with warm air coming from a water based heater programmed for 50 °C. The milling / cocoa liquor production lasted 30 min. The produced cocoa liquor was split into two parts – one for cocoa powder extraction and the other for chocolate production.

Cocoa powder extraction

The cocoa powder extraction was performed using petrol-based solvent at a ratio 2:1 (solvent: cocoa liquor). The flasks containing petrol based solvent and cocoa liquor (previously mixed with lab shaker in order to homogenize the mixture) were placed into a centrifuge for 5 minutes at relative centrifugal force of 500 xg and speed of 5330 rpm. After the centrifugation, the solvent containing the dissolved cocoa fat was transferred for filtering into cellulose thimble filters. The remaining solid in the flasks was again mixed with solvent at a ratio of 1 cocoa solid to 1.5 solvent. The whole process of mixing and centrifuge was repeated. The liquid (cocoa fat and solvent) was separated by filtration and the solid was moved out in a recipient and left for couple of hours in order to evaporate any rests of the solvent. The resulted dry substance was the cocoa powder. An aliquot of the solvent was taken and stored at ambient temperature. Fractions of cocoa powder were sampled and stored deep frozen.

Chocolate production

1. Refining

The remaining part of cocoa liquor was mixed manually with sugar and lecithin. The ratio was as follows: 800 g cocoa liquor, 10 g of lecithin, and 190 g of commercial sugar. Exact amount was adjusted according real weight of cocoa liquor. The mixture was named chocolate mass. The chocolate mass was placed into the ball mill together with steel balls (ratio= 1:10). The milling/ refining of the chocolate mass lasted for 40 min. Temperature of the "thermo jacket" where the chocolate mass and the steel balls were placed, was constantly supplied with warm air at the temperature of 42 °C. Aliquots of the commercial sugar and lecithin were taken and stored at ambient temperature.

2. Conching

Conching of chocolate targets the improvement of the flow properties and viscosity of the chocolate. The refined chocolate mass was placed into a labour kneader for conching. The kneader was adjusted to air flow at 1500 L/h and speed of kneading was 130 rpm. The "thermo jacket" where the refined chocolate was introduced was constantly supplied with warm air at the temperature of 75°C. The process of conching lasted for 4 hours. Fractions of chocolate were sampled and stored deep frozen.

Analysis

The residues of flupyradifurone and its metabolites DFA and 6-CNA were quantitated with Method 01304/M001 (HPLC/MS/MS). In all sample matrices the LOQ for flupyradifurone was 0.01 mg/kg and 0.02 mg eq/kg for DFA and 6-CNA. Average recoveries at fortification levels were within a range of 70–110%, and average RSDs were < 20% for all the related analytes.

Table 9 Processing of cacao beans from Study SI5-04586 to roasted bean, cocoa powder and chocolate

Trial No., Location, Year (Type-Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	GS	Rate (g ai/ ha)	Vol. (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
S15-04586	4	61-89	93.8	40	bean, dry	0*	<0.01	0.070	<0.01	<0.02	0.080
S15-04586-01	(26)	61-89	93.8	40		0	0.016	0.052	<0.01	<0.02	0.068
S15-04586-01-T3	(28)	61-87	93.8	40		7	<0.01	0.057	<0.01	<0.02	0.067
Côte d'Ivoire	(28)	61-89	93.8	40		7	<0.01	0.089	<0.01	<0.02	0.099
Plate Forme, Yamoussoukro						mean	<0.01	0.073	<0.01	<0.02	0.083
Africa, West					bean, roasted	7	<0.01	0.038	<0.01	<0.02	0.048
2015					cocoa powder	7	0.014	0.073	<0.01	<0.02	0.087
(95% Forastero, 5% Criollo)					chocolate	7	<0.01	0.034	<0.01	<0.02	0.044
S15-04586	4	61-89	93.8	40	bean, dry	0*	<0.01	0.084	<0.01	<0.02	0.094
S15-04586-02	(26)	61-89	93.8	40		0	<0.01	0.046	<0.01	<0.02	0.056
S15-04586-02-T3	(29)	61-87	93.8	40		7	0.01	0.075	<0.01	<0.02	0.085
Côte d'Ivoire	(27)	61-89	93.8	40		7	<0.01	0.085	<0.01	<0.02	0.095
Subiakro, Yamoussoukro						mean	0.01	0.080	<0.01	<0.02	0.090
Africa, West					bean, roasted	7	<0.01	0.076	<0.01	<0.02	0.086
2015					cocoa powder	7	0.013	0.15	<0.01	0.040	0.20
(Forastero)					chocolate	7	0.013	0.065	<0.01	<0.02	0.078

No: number of applications;RTI: minimum retreatment interval;GS: growth stage at last application;

DALA: days after last application

sprayed in a mixture with deltamethrin = Sivanto Energy containing 75 g/L flupyradifurone and 10 g/L deltamethrin

* prior to last application

The following table indicates estimated processing factors (either median or best estimate) for flupyradifurone and the total flupyradifurone residues.

Table 10 Summary of processing factors of flupyradifurone, or total flupyradifurone residues (cocoa dry bean to processed products)

Portion analysed	Individual trial residues (mg/kg)		Processing factors		
	S15-04586-01	S15-04586-02	S15-04586-01	S15-04586-02	Median/ best estimate
Flupyradifurone					
Cocoa dry bean (RAC)	<0.01 [a]	0.01 [a]	--		--
Roasted cocoa bean	<0.01	<0.01	-	<1	<1
Cocoa powder	0.014	0.013	>1.4	1.3	>1.4
Chocolate	<0.01	0.013	-	1.3	1.3
Total flupyradifurone residue					
Cocoa dry bean (RAC)	0.083 [a]	0.090 [a]	--		--
Roasted cocoa bean	0.048	0.086	0.58	0.96	0.77
Cocoa powder	0.087	0.20	1.05	2.22	1.64
Chocolate	0.044	0.078	0.53	0.87	0.70

[a] Mean value of two samples used for calculation

n.c. = not calculated

Coffee beans (Hoag, R.R.,2012, RARVP075)

The samples of coffee beans taken in the two field trials conducted in Brazil and Mexico with a single soil drench application followed by three broadcast foliar spray applications of flupyradifurone 200 SL at 2× exaggerated application rates were used for the processing study.

Single composite samples of coffee cherries were collected from the treated plots at a pre-harvest interval (PHIs) of 14 days. According to normal commercial practice in Brazil (trial RV235-11PA) and in various regions in Mexico (trial RV247-11PA) coffee cherries were allowed to air-dry before removing the outer hull and parchment using a machine that simulates large-scale commercial production of coffee beans, green. For trial RV235-11PA (Brazil) the cherries were allowed to air-dry for 10 days before removing the outer hull and parchment. For trial RV247-11PA (Mexico), coffee cherries were placed into forced-air drying ovens at a temperature of 122ºF (50 °C) for four days, followed by air-drying for eight days to yield the required sample size of coffee bean, green after removing the outer hull and parchment. At each processing laboratory, triplicate subsamples of coffee RAC (coffee bean, green) were removed from the bulk samples for analysis of flupyradifurone residues. The remainder of each bulk sample was used to generate the processed commodities coffee bean, roasted, and coffee, instant.

Roasting

The moisture content of green beans was determined and in case the moisture was greater than 13% the beans were dried at 30–40 °C in an oven until the moisture was 10-13%. Whole green coffee beans were aspirated with a Kice aspiration unit to remove light impurities such as light plant particles, dust and soil. After aspiration, a Hance seed cleaner was used to separate whole beans from extraneous material, e.g., small and large plant material. Samples from trial RV235-11 PA required aspiration. Samples from both trials were screened. A modified table top roaster, was utilized for roasting the clean green beans. Due to the variety of roast levels in commerce, a level similar to a “mild roast” was applied to provide a "worst case scenario" for residue purposes.

Clean green beans were roasted at a temperature of 199–216 °C and maintained for 10 to 30 minutes. After roasting the beans were allowed to cool. Resulting fraction was roasted coffee beans. Samples of dry roasted coffee beans were collected and placed into frozen storage.

Instant coffee processing

Roasted coffee beans were ground with Glen Mills disc mill to produce material to extract soluble substances for instant coffee production. After grinding, the material was sifted with a Great Western sample sifter equipped with 18 and 36 mesh sieves. Material below the 18-mesh sieve and remaining on the top of the 36-mesh sieve was used for extraction. Ground material was extracted to remove soluble substances in a fabricated extraction system. The system consisted of two steam stainless steel jacketed vessels, in-line pressure regulator to raise internal pressure above atmospheric pressure, 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 was pumped through the system until the exit solution became amber in colour. Exit temperature of liquid extract from top vessel was 129–163 °C. Liquid extract entered the chilled-water heat exchanger, and was decreased to 13–24 °C under atmospheric conditions. Extracts were filtered with a 10-mesh screen upon exiting chilled-water heat exchanger. After filtering, the solution was centrifuged and screened again utilizing a 120-mesh screen. Resulting fraction was coffee extract. "Spent grounds" from both vessels were dried at 54–71 °C in an oven until the moisture level was less than 12%. Resulting fraction was dried spent grounds. Spent grinds were not dried as they were not subjected to analysis.

Coffee extract was concentrated in a laboratory vacuum evaporator until the solids content was 15-30%. Temperature was maintained below 79 °C during the concentration. Extract was filtered with a 125-mesh screen. Resulting fractions were "liquor extract" and "processing water". Liquor extract were placed in freezer dryer containers and frozen. Frozen extract was freeze dried on a freeze dryer. After freeze drying, the product was reduced to granules and collected. Resulting fractions of freeze-dried coffee were collected and placed into frozen storage.

Analysis

The residues of flupyradifurone and its metabolite DFA and 6-CNA were analysed using Method RV-001-P10-02 (HPLC-MS/MS). The LOQ were 0.01 mg eq/kg for flupyradifurone and 6-CNA and 0.05 mg eq/kg for DFA in green and roasted beans. In instant coffee, the LOQ were 0.05 mg eq/kg for each analyte.

Prior and parallel to the residue analysis, the method was validated by recovery experiments. Average concurrent recoveries at fortification levels of respective LOQ and higher were within the acceptable range of 70–110%. Average RSD values were < 20%.

Table 11 Processing of coffee beans from RARVP075 study to roasted coffee beans and instant coffee

Trial No., Location, Year (Type-Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	GS	Rate (g ai /ha)#	Vol (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
RARVP075 RV235-11PA	4	77	1225	149	bean, green*	14	0.37	0.12	0.022	<0.01	0.49
Brazil	(91)	79	409	411		14	0.34	0.11	0.019	<0.01	0.44
Paulinia	(14)	80	396	374		mean	0.35	0.11	0.021	<0.01	0.47
2011	(14)	85	401	398	bean, roasted	14	0.20	0.092	0.014	0.014	0.31
(Catuai Vermelho)						14	0.19	0.080	<0.01	0.023	0.30
						mean	0.20	0.086	0.012	0.019	0.30
					coffee, instant	14	0.36	0.38	<0.05	0.049	0.79
						14	0.87	0.57	<0.05	0.053	1.5
						mean	0.62	0.48	<0.05	0.051	1.1
RARVP075 RV247-11PA	4	72	1206	201	bean, green	14	0.98	0.50	0.11	0.15	1.6
Mexico	(85)	81	401	402		14	1.1	0.52	0.11	0.024	1.6
	(12)	81	399	403		mean	1.0	0.51	0.11	0.088	1.6

Trial No., Location, Year (Type-Variety)	Application				Sample	DALA	Residues as parent (mg/kg)				
	No. (RTI, days)	GS	Rate (g ai /ha)#	Vol (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
La Union Zihuateutla 2011 (Caturra)	(13)	85	398	402	bean, roasted	14	0.73	0.55	0.064	0.045	1.3
						14	0.57	0.60	0.049	0.021	1.2
						mean	0.65	0.57	0.057	0.033	1.3
					coffee, instant	14	2.4	2.3	0.27	0.19	4.9
						14	1.8	3.2	0.20	0.095	5.1
						mean	2.1	2.8	0.23	0.14	5.0

No: number of applications;RTI: minimum retreatment interval;GS: growth stage at last application;

DALA: days after last application

combined drench (1 x 1200 g ai/ha) and spray application (3 x 400 g ai /ha)

* Calculated as average residue determined in triplicate subsamples

The effect of processing on coffee green bean was determined and the processing factors of flupyradifurone and of the total flupyradifurone residue for each processed commodity were calculated.

Table 12 Summary of processing factors of flupyradifurone or total flupyradifurone residues (coffee beans to their processed products)

Portion analysed	Individual trial residues (mg/kg)				Processing factors				
	RV235-11PA (A)	RV235-11PA (B)	RV247-11PA (B)	RV247-11PA (A)	RV235-11PA (A)	RV235-11PA (B)	RV247-11PA (B)	RV247-11PA (A)	Median or best estimate
Flupyradifurone									
Coffee green bean, RAC	0.37	0.34	0.98	1.1	--				--
Roasted bean	0.20	0.19	0.73	0.57	0.54	0.57	0.74	0.52	0.56
Instant coffee	0.36	0.87	2.4	1.8	0.98	2.58	2.42	1.64	2.0
Total flupyradifurone residue									
Coffee green bean, RAC	0.49	0.44	1.6	1.6	--				--
Roasted bean	0.31	0.30	1.3	1.2	0.63	0.68	0.81	0.75	0.72
Instant coffee	0.79	1.5	4.9	5.1	1.6	3.4	3.1	3.2	3.2

Hops, dry (Schulte, G., Bauer, J., 10-3407)

The field samples of green cone to be processed were sampled 21 days after treatment, at BBCH 89, in two trials conducted in Germany in 2015. The effects of processing on flupyradifurone residues in dried hops cone to hops draff, brewer's yeast and beer were studied.

Samples of harvested green cones were first dried to create the kiln-dried cone, which were stored deep-frozen within 24 hours after sampling at -18 °C or below until further processing. The processing of the defrosted kiln-dried cones into processed fractions (hops draff, brewer's yeast and beer) was performed in the processing facility in Germany. The processing procedures simulated industrial practices at a laboratory scale. Following defrosting the hop field specimens were transported to the processing facility at ambient temperature. Processing started with milling the dried hop specimens to hop powder within 48 h (after freezer output).

Brewing

For the brewing process the ingredients hops (dry cone), commercially bought malt and yeast and drinking water were used.

Mashing

Mashing is the homogeneous mixing of ground malt and water according to a definite temperature time regime (mash program). The main purpose of mashing was the dissolution and enzymatic conversion of ingredients. Before mashing, the brewer's malt was dry-milled in a special malt mill. The crushed malt was mixed with brew water. Mashing was started in a heatable tun. To produce Pilsner-style beer, the mash program lasted approximately 1 hour and 40 minutes at temperatures of 46 to 76 °C.

Lautering: Wort extraction and separation

After mash boiling, the wort was separated from the insoluble malt components (brewer's grain). The extract remaining in the brewer's grain was extracted by washing with hot water (first filter runnings). The wort separation was done using a refining vat and took 2–3 hours.

Wort boiling and conditioning

After addition of hop pellets, the separated wort was boiled for about 90 minutes at normal pressure. This deactivates the enzymes of the malt, sterilizes the wort, extracts and isomerizes the essential components of the hops, precipitates high molecular proteins (called "Bruch") and expels 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. For cooling and ventilating the wort, an intra-plant circulation was used. By adding oxygen (intra-plant circulation) the conditions for the start of the fermentation were prepared. Samples of hops draff were sampled.

Fermentation and maturation

In the pilot plant, the classical primary fermentation (low fermentation) was carried out in bottom fermentation containers. The fermentation temperature was approximately 9 °C. Fermentation heat was dissipated by means of room ventilation.

The duration of main fermentation depends on temperature, on starting extract concentration of the finished wort, on the ratio of non-fermentable sugars to the extract, on the final attenuation and on the yeast cell number (exact duration was recorded). As soon as the extract content of the fermented young beer was 2% higher than the final attenuation, the storing time began. Before maturation the young beer was cooled down. During the main fermentation the yeast deposits on the tank bottom and was sampled as brewer's yeast. At the beginning of maturation, the young beer was stored at room temperature (warm maturation to break down the diacetyl) in casks. Then the young beer was stored under pressure (approx. 1–0.7 bar) at approximately 2 °C (cold maturation) for about 3–4 weeks. In this time the remaining extract was fermented. Unwanted flavour and odorous substances were decomposed or expelled. Sludge particles and yeast settle at the bottom. 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 sampled.

Analysis

The residues of flupyradifurone and its metabolite DFA and 6-CNA were quantitated with Method 01304 (HPLC/MS/MS). In the sample matrices cone, green, cone, kiln-dried, hops draff and brewer's yeast, the LOQ was 0.1 mg eq/kg for flupyradifurone and 6-CNA and 0.2 mg eq/kg for DFA. In the beer the LOQ was 0.01 mg eq/kg for flupyradifurone and 6-CNA and 0.02 mg eq/kg for DFA.

The apparent residues in the control sample used for fortification experiments were below 30% of the LOQ, but for flupyradifurone in kiln-dried cone and for 6-CNA in green cone and kiln-dried cone concentration was at the level of 0.1 mg eq/kg, and also in hops draff at the level of 0.1 and 1.0 mg eq/kg. Therefore, recoveries were corrected for apparent residues in the corresponding control samples. Average recoveries at the fortification levels of respective LOQ and higher were within a range of 70 – 120%. Average RSD were <20%.

Table 13 Processing of hops from 10-3407 study beer

Trial No., Location, Year (Type-Variety)	Application				Sample	DALA	Residues as parent (mg/kg)*				
	No. (RTI, days)	Growth Stage	Rate (g ai /ha)	Volume (L/ha)			Parent	DFA	DFEAF	6-CNA	Parent + DFA + 6-CNA
GAP (EU); Foliar Hops	1		150			21					
10-3407 10-3407-01 Germany 04685 Golzern Europe, North 2010 (Nugget)	1	71	360	3000	cone, green	21	0.43	<0.2	<0.1	0.29/ 0.27**	0.92/ 0.57**
					cone, kiln-dried	21	2.2	0.72	<0.1	1.6/ 1.7**	4.5/ 2.0**
					hops draff	21	<0.1	<0.2	<0.1	0.16/ 0.15**	0.46/ 0.45**
					brewer's yeast	21	<0.1	<0.2	<0.1	<0.1	<0.3
					beer	21	0.01	<0.02	<0.01	<0.01	0.03
10-3407 10-3407-02 Germany 99706 Hohenebra Europe, North 2010 (Nordischer Brauer)	1	75	360	3000	cone, green	21	1.1	0.37	<0.1	0.24/ 0.16**	1.7/ 0.46**
					cone, kiln-dried	21	4.2	0.76	<0.1	0.77/ 1.3**	5.7/ 1.6**
					hops draff	21	<0.1	<0.2	<0.1	0.15	0.45
					brewer's yeast	21	<0.1	<0.2	<0.1	<0.1	<0.3
					beer	21	0.02	<0.02	<0.01	<0.01	0.04

No: number of applications;RTI: minimum retreatment interval;GS: growth stage at last application;

DALA: days after last application

** residue in control

The effect of processing on hops was determined and the processing factors of flupyradifurone and of the total flupyradifurone residue for each processed commodity were calculated.

Table 14 Summary of processing factors of flupyradifurone and total flupyradifurone residues (hops to beer)

Portion analysed	Individual trial residues (mg/kg)		Processing factors		
	10-3407-01	10-3407-02	10-3407-01	10-3407-02	Median or best estimate
Flupyradifurone					
Cone, kiln-dried	2.2	4.2	--		--
Hops draff	<0.1	<0.1	<0.05	<0.02	<0.03
Brewer's yeast	<0.1	<0.1	<0.05	<0.02	<0.03
Beer	0.01	0.02	0.005	0.005	0.005
Total flupyradifurone residue					
Cone, kiln-dried	4.5	5.7	--		--
Hops draff	0.46	0.45	0.10	0.08	0.09
Brewer's yeast	<0.3	<0.3	<0.07	<0.05	<0.06
Beer	0.03	0.04	0.01	0.01	0.01

APPRAISAL

Flupyradifurone, is an insecticide with the structure of butenolides. It acts as an agonist of the nicotinic acetylcholine receptor.

Flupyradifurone was first evaluated by the Meeting for toxicology in 2015 as a new compound. It was evaluated for residues in 2016 and 2017.

The 2015 Meeting established an ADI of 0–0.08 mg/kg bw and an ARfD of 0.2 mg/kg bw.

The 2016 and 2017 Meeting recommended the following residue definitions:

Definition of the residue (for compliance with the MRL) for plant commodities: *Flupyradifurone*

Definition of the residue (for dietary risk assessment) for plant commodities: *Sum of flupyradifurone, difluoroacetic acid (DFA) and 6-chloronicotinic acid (6-CNA), expressed as parent equivalents*

Definition of the residue (for compliance with the MRL and dietary risk assessment) for animal commodities: *Sum of flupyradifurone and difluoroacetic acid, expressed as parent equivalents*

The residue is not fat-soluble.

On a basis of the above residue definitions, the Meeting estimated maximum residue levels for a wide range of commodities.

Flupyradifurone was listed by the Forty-ninth CCPR for evaluation of additional uses by the current Meeting. The present Meeting received information on analytical methods, storage stability, use pattern, supervised residue trials and processing in support of estimation of maximum residue levels for blackberry, raspberry, avocado, pomegranate, cacao beans, coffee beans, and hops.

Methods of analysis

A number of analytical methods for plant and animal matrices were submitted to and evaluated by the 2016 Meeting. The current Meeting received information on new analytical methods (modified methods of those already reviewed) using HPLC-MS/MS together with validation data for residues of flupyradifurone. They were validated with the LOQs ranging from 0.01–0.5 mg eq/kg for flupyradifurone, DFA and 6-CNA in the plant commodities for which supervised trial or processing study data were submitted to this Meeting.

The Meeting evaluated in 2016 and 2017 storage stability data on flupyradifurone residues in various plant matrices stored frozen. The 2017 Meeting concluded that flupyradifurone, DFA and 6-CNA are stable for at least 52 months (1556 to 1572 days) in high water, high acid, high oil, high protein, and high starch content matrices, when stored frozen at approximately -18 °C. The frozen storage periods of samples in the trial studies submitted to the current Meeting were, at the longest, 841 days.

Results of supervised residue trials on crops

The current Meeting received information on supervised trials using foliar sprays of flupyradifurone conducted in support of estimating maximum residue levels for the following commodities: cane berries (blackberry and raspberry), avocado, pomegranate, cacao beans, coffee beans (drench and foliar applications) and hops, dry.

For the calculation of the sum of flupyradifurone, DFA and 6-CNA, expressed as parent equivalents (total residues), the Meeting used the approach agreed at the 2016 JMPR:

"Where parent or DFA residues were not detected or were less than the LOQ (*i.e.* < 0.01 mg/kg for parent or 0.05 mg/kg for DFA) the LOQ value was utilized for maximum residue estimation and dietary exposure assessment. For 6-CNA, values less than the LOQ were not added for calculation of total residues of flupyradifurone."

The table below on how the total residues were calculated for each trial was copied from the Evaluation of the 2016 JMPR for easy reference.

Parent	DFA	6-CNA	Total
<0.01	0.05	0.01	0.07
0.01	<0.05	0.01	0.07
<0.01	<0.05	<0.01	<0.06

Parent	DFA	6-CNA	Total
0.01	0.05	<0.01	0.06
0.01	0.05	0.01	0.07

All expressed in parent equivalents (concentrations are described in mg eq/kg in this evaluation).

Cane berries (Blackberry and raspberry)

Critical GAP in the USA for the cane berry crop sub-group allows two foliar applications at a maximum rate of 205 g ai/ha with an interval of 7 days, and PHI of 0 days.

Four field trials were conducted on blackberries in Canada and the USA in the 2012–2014 growing seasons.

Flupyradifurone residues from independent trials on blackberry following the above GAP were in rank order (n=2): 0.81 and 1.6 mg/kg.

In other two trials, application rates were 95–115 g ai/ha, lower than the critical GAP rate, and residues from these trials were in rank order (n=2): 0.49 and 2.1 mg/kg.

The Meeting decided to apply the proportionality principle to the residues from trials conducted with rates about half of the critical GAP rate.

The residues from the trials following the GAP and with the lower application rates, after scaling to the critical GAP rate of 205 g ai/ha, were in rank order (n=4): 0.81, 0.96, 1.6 and 3.9 mg/kg.

Corresponding total residues from the trials following the US GAP were (n=2): 0.84 and 1.7 mg/kg. Total residues from the trials using the application rates (95-115 g ai/ha) lower than the critical GAP rate were (n=2): 0.55 and 2.2 mg/kg.

The total residues from the trials following the GAP and with the lower application rates, after scaling to the GAP rate of 205 g ai/ha were: 0.84, 1.1, 1.7 and 4.1 mg/kg (highest individual residue: 4.3 mg/kg).

Seven field trials were conducted on raspberries in Canada and the USA in the 2012 growing seasons.

Flupyradifurone residues from independent trials on raspberry following the US GAP were in rank order (n=6): 0.84, 1.0, 1.1, 2.2, 2.5 and 2.5 mg/kg.

Corresponding total residues were: 0.86, 1.0, 1.1, 2.2, 2.5 and 2.5 mg/kg (highest individual residue: 2.8 mg/kg).

The US GAP is for the cane berry crop sub-group including blackberry and raspberry, and blackberry or raspberry is a representative commodity for the cane berries sub-group in the Codex classification. As the Mann-Whitney U-test on the residue populations of blackberry and raspberry indicated that these populations were not significantly different, the Meeting decided to combine these two populations to estimate a maximum residue level, STMR and HR for the subgroup of cane berries.

Combined flupyradifurone residues in rank order were (n=10): 0.81, 0.84, 0.96, 1.0, 1.1, 1.6, 2.2, 2.5, 2.5 and 3.9 mg/kg.

The Meeting estimated a maximum residue level of 6 mg/kg for the cane berries sub-group.

Corresponding combined total residues were in rank order (n=10): 0.84, 0.86, 1.0, 1.1, 1.1, 1.7, 2.2, 2.5, 2.5, and 4.1 mg/kg (highest individual residue: 4.3 mg/kg).

The Meeting estimated a STMR and HR of 1.4 mg/kg and 4.3 mg/kg, expressed in parent equivalents, respectively for the cane berries sub-group.

Avocado

Critical GAP in the USA for avocado, in the group of "tropical and subtropical, medium to large fruit, smooth, inedible peel", allows two foliar applications at a maximum individual rate of 205 g ai/ha with an interval of 14 days, and a PHI of 1 day. Four supervised trials were conducted on avocado in the USA in 2013.

Flupyradifurone residues from independent trials on avocado following the above GAP were in rank order (n=4): 0.026, 0.19, 0.22 and 0.24 mg/kg.

The Meeting estimated a maximum residue level of 0.6 mg/kg for avocado.

The Corresponding total residues were: 0.076, 0.27, 0.29 and 0.31 mg/kg (highest individual residue: 0.36 mg/kg).

The Meeting estimated a STMR and HR of 0.28 mg/kg and 0.36 mg/kg, expressed in parent equivalents, respectively for avocado.

Pomegranate

Critical GAP in the USA for pomegranate, in the group of "tropical and subtropical, medium to large fruit, smooth, inedible peel", allows two foliar applications at a maximum individual rate of 205 g ai/ha with an interval of 7 days, and a PHI of 0 days. Four supervised trials were conducted on pomegranate in the USA in 2012. Two trials were conducted in close proximity to each other with the application timing only a few days apart. Since other differences in the trial parameters would not affect the residue concentrations significantly, the Meeting considered that these trials were not independent.

Flupyradifurone residues from independent trials on pomegranate following the above GAP were in rank order (n=3): 0.18, 0.20 and 0.23 mg/kg.

The corresponding total residues were: 0.20, 0.22 and 0.25 mg/kg

According to the Codex document on minor crops, pomegranate requires 4 trials for estimating maximum residue level. The Meeting concluded that the data from 3 trials were insufficient to estimate a maximum residue level for pomegranate.

Cacao beans

The critical GAP is from Ghana, which allows 4 foliar applications in August, September, October and December at a maximum rate of 15 g ai/ha each with a PHI of 7 days. A total of nine supervised trials were conducted on cacao in Côte d'Ivoire and Ghana in 2014 and 2015.

Flupyradifurone residues dried cacao bean from trials approximating the GAP in Ghana were (n=7) all < 0.01 mg/kg.

The Meeting estimated a maximum residue level of 0.01(*) mg/kg for cacao beans.

Among nine decline trials, the total residue concentrations increased in two trials up to the longest days after the last application (DALA) interval, while in the others the total residue concentrations seemed to reach a peak or plateau. The Meeting considered that the dataset of total residues, regardless of DALA, would adequately cover the expected residues.

The total residues from trials approximating the GAP were (n=7): 0.051, 0.059, 0.070, 0.071, 0.087, 0.099 and 0.11 mg/kg.

The Meeting estimated a STMR of 0.071 mg/kg, expressed in parent equivalents, for cacao beans.

Coffee beans

Critical GAP in Brazil for coffee allows one drench application at 600 g ai/ha and three foliar spray applications at an application rate of 200 g ai/ha each with an interval of 15 days between foliar applications, and a PHI of 21 days. The drench application should be approximately 90 days before the

spray applications. The total annual application rate for drench or foliar applications is 600 g ai/ha. A total of 16 supervised trials were conducted on coffee in Brazil, Colombia, Guatemala and Mexico in 2011 and 2012 following the GAP in Brazil.

Flupyradifurone residues in dried coffee bean, green, from independent trials on coffee following the above GAP were in rank order (n=16): < 0.01 (2), 0.02, 0.05, 0.065, 0.08, 0.14, 0.14, 0.14, 0.16, 0.20, 0.21, 0.22, 0.35, 0.55 and 0.60 mg/kg.

The Meeting estimated a maximum residue level of 0.9 mg/kg for coffee beans.

Among the 12 decline trials, the total residue concentrations steadily increased in four trials up to the longest DALA, while in the others the total residue concentrations seemed to reach a peak or plateau. The Meeting considered that the dataset of total residues, regardless of DALA, would adequately cover the expected residues.

The total residues in these trials were (n=16): < 0.06, < 0.06, 0.10, 0.10, 0.19, 0.20, 0.24, 0.29, 0.30, 0.30, 0.41, 0.49, 0.56, 0.61, 0.77 and 0.87 mg/kg.

The Meeting estimated a STMR of 0.295 mg/kg, expressed in parent equivalents, for coffee beans.

Hops, dry

A total of 12 trials were conducted on hops in Germany and the USA.

Critical GAP in the Netherlands allows one foliar application at a rate of 150 g ai/ha and a PHI of 21 days. Eight residue trials were conducted on hops in the 2010 (4) and 2011 (4) seasons in Germany.

In four trials, 6-CNA residues were detected above the LOQ in control samples of dried hop cone. Among them, in three trials, the levels were more than 25% of the total residues, and the Meeting did not use these trials in the evaluation.

Flupyradifurone residues from trials on hops in Germany approximating the GAP in the Netherlands were in rank order (n=5): 0.31, 0.43, 1.1, 1.8 and 2.0 mg/kg.

Corresponding total residues from the German trials were (n=5): 0.63, 0.73, 1.7, 2.3 and 2.4 mg/kg.

Critical GAP in the USA on hops allows one foliar application at an application rate of 154 g ai/ha and a PHI of 21 days. Four field trials were conducted on hops in the USA following the US GAP in 2011 (three trials) and 2015 (one trial).

Flupyradifurone residues in the dried hop cone from independent trials in the USA on hops following the above GAP were in rank order (n=4): 2.4, 2.7, 2.7 and 4.7 mg/kg.

In one trial in the USA, 6-CNA was not analysed in dried hop cone. Assuming that the LOQ for 6-CNA was the same as for flupyradifurone and DFA (0.5 mg eq/kg), the Meeting agreed to add 0.5 mg eq/kg as 6-CNA to the sum of flupyradifurone and DFA residues to make a conservative estimate of the total residue.

The Corresponding total residues from the USA trials were (n=4): 3.4, 3.4, 3.7, 8.1 mg/kg.

Since the data from the USA trials would lead to a higher maximum residue level, the Meeting used these trials for the estimation of the maximum residue level for hops, dry.

The Meeting estimated a maximum residue level of 10 mg/kg and a STMR of 3.55 mg/kg, expressed in parent equivalents, for hops, dry.

Fate of residues during processing

The effects of processing on the concentrations of flupyradifurone residues were evaluated by the 2016 and 2017 JMPR for a wide range of commodities for which maximum residue levels were recommended.

The current Meeting received information on the processing of cacao beans, coffee beans and dried hops to processed commodities, relevant to the current evaluation.

The calculated processing factors for these commodities together with calculated STMR-Ps are summarized below.

Total Residues			
Processed commodity	Individual processing factor	Median or best estimate	STMR/ STMR-P
Cacao dry bean (RAC)			0.071
Roasted cacao bean	0.58, 0.96	0.77	0.0547
Cocoa powder	1.05, 2.22	1.64	0.116
Chocolate	0.53, 0.87	0.70	0.0497
Coffee green bean (RAC)			0.295
Roasted coffee bean	0.63, 0.68, 0.75, 0.81	0.72	0.21
Instant coffee	1.6, 3.1, 3.2, 3.4	3.2	0.94
Hops, dry (RAC)			3.55
Beer (hops)	0.01, 0.01	0.01	0.0355

Animal commodity maximum residue levels

As none of the commodities evaluated, or their by-products, for which supervised trial data were submitted to the current Meeting are fed to animals, the Meeting concluded that there was no need to revisit the previous recommendations for flupyradifurone in animal commodities.

RECOMMENDATIONS

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

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

Definition of the residue for dietary risk assessment for plant commodities: *Sum of flupyradifurone, difluoroacetic acid (DFA) and 6-chloronicotinic acid (6-CNA), expressed as parent equivalents.*

Definition of the residue for compliance with the MRL and for dietary risk assessment for animal commodities: *Sum of flupyradifurone and difluoroacetic acid, expressed as parent equivalents*.

The residue is not fat-soluble.

CCN	Commodity	Recommended maximum residue level mg/kg		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
FB 2005	Cane berries	6	-	1.4	4.3
FI 0326	Avocado	0.6	-	0.28	0.36
SB 0715	Cacao beans	0.01 *	-	0.071	-
SB 0716	Coffee beans	0.9	-	0.295	-
DH 1100	Hops, dry	10	-	3.55	-
	Cacao beans, roasted			0.0547	-

CCN	Commodity	Recommended maximum residue level mg/kg		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
DM 0715	Cocoa powder			0.116	-
	Chocolate			0.0497	-
SM 0716	Coffee beans, roasted			0.21	-
	Instant coffee			0.94	-
	Beer (hops)			0.0355	-

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for flupyradifurone is 0–0.08 mg/kg bw. The International Daily Intakes (IEDIs) for flupyradifurone 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 2019 Extra JMPR Report.

The IEDIs ranged from 6–20% of the maximum ADI. The Meeting concluded that long-term dietary exposure to residues of flupyradifurone from uses considered by JMPR is unlikely to present a public health concern.

Acute dietary exposure

The ARfD for flupyradifurone is 0.2 mg/kg bw. The international Estimate of Short-Term Intakes (IESTIs) for flupyradifurone were calculated for the food commodities and their processes 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 2019 Extra JMPR report.

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

References

Report No Edition No. (if any)	Author(s)	Year	Title, Source, Date, etc.
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01330/M002 M-469883-02-1	Rzepka, S.	2014	Amendment No. 1 to report No: BCS- 1301V - Validation of the BCS-Method 01330/M002 for the determination of residues of flupyradifurone (BYI 02960) and its metabolite difluoroacetic acid (DFA) in/on cocoa (beans) and coffee (beans) by HPLC-MS/MS Eurofins Specht Laboratorien GmbH Bayer Date: 2013-10-29 ... amended: 2014-04-23

Report No Edition No. (if any)	Author(s)	Year	Title, Source, Date, etc.
			GLP/GEP: Yes, unpublished
10-2225 M-425351-01-1	Noss, G.; Ballmann, C.	2012	Determination of the residues of BYI 02960 in/on hop after spraying of BYI 02960 SL 200 in the field in Germany Bayer Including Trial Nos.: 10-2225-01 10-2225-02 10-2225-03 10-2225-04 Date: 2012-02-13 GLP/GEP: Yes, unpublished
10-3407 M-425311-01-1	Schulte, G.; Bauer, J.	2012	Determination of the residues of BYI 02960 in/on hop (cone, green and cone, kiln-dried) and the processed fractions (hops draff, brewer's yeast and beer) after spraying of BYI 02960 SL 200 in the field in Germany Bayer Including Trial Nos.: 10-3407-01 10-3407-02 Date: 2012-02-13 GLP/GEP: Yes, unpublished
11-2076 M-425339-01-1	Noss, G.; Ballmann, C.	2012	Determination of the residues of BYI 02960 in/on hop after spray application of BYI 02960 SL 200 in Germany Bayer Including Trial Nos.: 11-2076-01 11-2076-02 11-2076-03 11-2076-04 Date: 2012-02-13 GLP/GEP: Yes, unpublished
AAFC12-054R M-532236-01-1	Pogoda, M.	2015	BYI 02960: Magnitude of the residue on caneberry SynTech Research Laboratory Services, LLC, Stilwell, KS, USA Bayer Including Trial Nos.: AAFC12-054R-316 AAFC12-054R-317 AAFC12-054R-318 AAFC12-054R-319 AAFC12-054R-320 AAFC12-054R-321(Decline) AAFC12-054R-322 AAFC12-054R-323 AAFC12-054R-324 AAFC12-054R-335 AAFC12-054R-415 (Decline) AAFC12-054R-442 (Decline) MRID#: 49619811 Date: 2015-09-03 GLP/GEP: Yes, unpublished
I11-008 M-427469-04-1	Resende, G.	2016	Adendo 02 ao relatório final - Determinação de resíduos de BYI 02960 e seus metabólitos, na cultura do café após aplicação em

Report No Edition No. (if any)	Author(s)	Year	Title, Source, Date, etc.
			jato dirigido na base das plantas, seguida de aplicações em pulverização foliar de BYI 02960 (200 SL) no Brasil Departamento de Registro Bayer CropScience, São Paulo, Brazil Bayer Including Trial Nos.: I11-008-01 I11-008-02 I11-008-04 I11-008-05 Date: 2012-03-12 ... amended: 2016-05-06 GLP/GEP: Yes, unpublished
I12-006 M-461530-02-1	Santiago, L.	2016	Adendo 01: Determinação de resíduos de BYI 02960 e seus metabólitos, na cultura do café após aplicação em jato dirigido na base das plantas, seguida de aplicações em pulverização foliar de BYI 02960 (200 SL) em ensaios no Brasil Bayer CropScience, São Paulo, SP, Brazil Bayer Including Trial Nos.: I12-006-01 I12-006-02 I12-006-03 I12-006-04 I12-006-05 Date: 2013-07-30 ... amended: 2016-01-27 GLP/GEP: Yes, unpublished
IR-4 PR No. 10770 M-530766-02-2	Dorschner, K.	2015	BYI 02960: Magnitude of the residue on pomegranate IR-4 Project Rutgers, State University New Jersey, Princeton, NJ, USA IR4-Rutgers University Including Trial Nos.: 10770.12-CA08 10770.12-CA09 10770.12-CA10 10770.12-CA11 Date: 2015-08-12 GLP/GEP: Yes, unpublished
M-428412-03-1	Netzband, D.	2015	Storage stability of BYI 02960, difluoroacetic acid, and difluoroethyl-amino-furanone in plant matrices Bayer CropScience LP, Stilwell, KS, USA Bayer MRID#: 49619805 Date: 2012-04-03 ... amended: 2015-09-03 GLP/GEP: Yes, unpublished
M-428762-01-1	Anon.	2012	BYI02960; 200SL; coffee; Brazil; BBA Bayer S/A, Departamento de Estudos em Segurança Alimentar, Sao Paulo, Brazil Bayer Including Trial Nos.: I11-008-01 I11-008-02

Report No Edition No. (if any)	Author(s)	Year	Title, Source, Date, etc.
			I11-008-04 I11-008-05 Date: 2012-03-12 GLP/GEP: No, unpublished
M-434966-06-1	Anon	2015	Sivanto 200 SL - USA Bayer CropScience LP, RTP, NC, USA Bayer Date: 2015-09-01 GLP/GEP: n.a., unpublished
M-434966-09-1	Anon.	2018	Sivanto 200 SL - USA Bayer CropScience LP, RTP, NC, USA Bayer Date: 2018-02-16 GLP/GEP: n.a., unpublished
M-481362-01-1	Anon.	2013	BYI 02960; SL 200; coffee; Brazil; BBA Bayer CropScience Bayer Including Trial Nos.: I12-006-01 I12-006-02 I12-006-03 I12-006-04 I12-006-05 Date: 2013-07-30 GLP/GEP: Yes, unpublished
M-557163-01-1	Anon	2015	Sivanto Prime Insecticide - 1 to 1000 L - Canada Bayer CropScience Bayer Date: 2015-11-21 GLP/GEP: n.a., unpublished
M-574276-01-1	Semrau, J.	2016	Determination of residues of flupyradifurone and deltamethrin in/on fermented dry cocoa beans after four spray applications of Sivanto Energy (DLT + FPF EC 85 (10+75 g/L)) in cocoa at 2 sites in Ivory Coast and 4 sites in Ghana in 2014 Eurofins Agroscience Services GmbH, Stade, Germany Bayer Date: 2016-11-04 GLP/GEP: Yes, unpublished
M-623581-01-1	Silva, C.	2017	Rótulo e Bula AGROFIT - Sivanto Prime 200 SL Bayer S.A., Divisão Crop Science, São Paulo, SP, Brazil Bayer Date: 2017-08-17 GLP/GEP: n.a., unpublished
M-623581-01-2	Silva, C.	2017	AGROFIT - Sivanto Prime 200 SL Bayer S.A., Divisão Crop Science, São Paulo, SP, Brazil Bayer Date: 2017-08-17 GLP/GEP: n.a., unpublished
M-624026-01-1	Anon.	2018	Sivanto Energy 085 EC - 40 mL Bayer AG Bayer Date: 2018-05-15 GLP/GEP: n.a., unpublished
M-624027-01-1	Anon.	2018	Sivanto Energy 085 EC - 500 mL - Ivory Coast

Report No Edition No. (if any)	Author(s)	Year	Title, Source, Date, etc.
			Bayer West-Central Africa, Abidjan, Côte d'Ivoire Bayer Date: 2018-05-15 GLP/GEP: n.a., unpublished
M-624027-01-2	Anon.	2018	Sivanto Energy 085 EC - 500 mL - Ivory Coast Bayer West-Central Africa, Abidjan, Côte d'Ivory Bayer Date: 2018-05-15 GLP/GEP: n.a., unpublished
M-624162-01-1	Anon.	2018	Sivanto Prime - 3 L - The Netherlands - April 2018 Bayer CropScience SA-NV, Mijdrecht, Netherlands Bayer Date: 2018-04-09 GLP/GEP: n.a., unpublished
M-624162-01-2	Anon.	2018	Sivanto Prime - 3 L - The Netherlands Bayer CropScience SA-NV, Mijdrecht, Netherlands Bayer Date: 2018-04-09 GLP/GEP: n.a., unpublished
M-629682-01-1	Spiegel, K.; Reinecke, A. K.	2018	Flupyradifurone (285) - JMPR - FAO evaluation - Follow-up submission Bayer AG, Crop Science Division, Monheim, Germany Bayer Date: 2018-07-18 GLP/GEP: n.a., unpublished
M-629724-01-1	Koehler, A.	2018	Flupyradifurone (285) - JMPR - FAO evaluation - Follow-up submission - Appendix A.2.1.3: Use pattern Bayer AG, Crop Science Division, Monheim, Germany Bayer Date: 2018-07-18 GLP/GEP: n.a., unpublished
M-629725-01-1	Reinnecke, A. K.	2018	Flupyradifurone (285) - JMPR - FAO evaluation - Follow-up submission - Appendix A.2.1.4: Residue summary tables (Tier I summary tables) Bayer AG, Crop Science Division, Monheim, Germany Bayer Date: 2018-07-18 GLP/GEP: No, unpublished
RAGMN133 M-565615-02-1	Netzband, D.; Dallstream, K. A.	2016	Amendment No 1 to: Luna Privilege (fluopyram) and Sivanto 200 SL (flupyradifurone): Magnitude of the residue in hops Bayer CropScience LP, RTP, NC, USA Bayer Including Trial Nos.: GM007-15DA Date: 2016-09-14 ... amended: 2016-12-12
RARVN012 M-530915-01-1	Murphy, I.	2015	BYI 02960 200 SL - Magnitude of the residue in/on avocado Bayer CropScience LP, RTP, NC, USA Bayer Including Trial Nos.: RV006-13HA RV007-13HB RV008-13DA RV009-13HA

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			MRID#: 49619807 Date: 2015-08-18 GLP/GEP: Yes, unpublished
RARVP074-01 M-433257-02-1	Fischer, D. R.; Jerkins, E.	2018	BYI 02960 200 SL - Magnitude of the residue in/on coffee; U.S., Canada and E.U. import tolerance Bayer CropScience LP, Stilwell, KS, USA Bayer MRID#: 48843928 Date: 2012-06-27 ... amended: 2018-06-12 GLP/GEP: Yes, unpublished
RARVP075 M-433200-01-1	Hoag, R. E.	2012	BYI 02960 200 SL - Magnitude of the residue in/on processed commodities for coffee; U.S., Canada and E.U. import tolerance Bayer CropScience LP, Stilwell, KS, USA Bayer MRID#: 48843950 Date: 2012-06-26 GLP/GEP: Yes, unpublished
RARVY008 M-432695-01-1	Krolski, M. E.	2012	BYI 02960 200 SL - Magnitude of the residue in/on hops Bayer CropScience LP, Stilwell, KS, USA Bayer Including Trial Nos.: RV047-11HA RV048-11HA RV049-11HA MRID#: 48843929 Date: 2012-06-12 GLP/GEP: Yes, unpublished
S13-05059 M-493096-01-1	Amic, S	2014	Independent laboratory validation of the BCS-method-01330/M002 for the determination of residues of Residues of flupyradifurone (BYI 02960) and its metabolite difluoroacetic acid (DFA) in/on cocoa (roasted beans) and coffee (green and roasted beans) by HPLC-MS/MS Eurofins Agroscience Services, Chem SAS, Vergèze, France Bayer Date: 2014-07-11 GLP/GEP: Yes, unpublished
S15-04586 M-574274-02-1	Petrova, D.	2018	Amendment no. 1 to report no. S15-04586 / RARVN045 - Determination of residues of flupyradifurone and deltamethrin in/on fermented dry cocoa beans and processed fractions (cocoa powder, roasted beans and chocolate) after four spray applications of Sivanto Energy (DLT + FPF EC 85 (10+75 g/L)) in cocoa in Ivory Coast in 2015 Eurofins Agroscience Services GmbH, Stade, Germany Bayer Including Trial Nos.: S15-04586-01 S15-04586-02 S15-04586-03 Date: 2016-10-27 ... amended: 2017-02-15 GLP/GEP: Yes, unpublished

FOSETYL-ALUMINIUM (302)/PHOSPHONIC ACID (301)

First draft prepared by Mr D Lunn, Ministry for Primary Industries, Wellington, New Zealand

EXPLANATION

Fosetyl (and its aluminium salt) and phosphonic acid (formulated as the potassium or sodium salts) are systemic fungicides considered by the Meeting for the first time in 2017, when residue definitions and health-based guidance values were established and a number of maximum residue levels were recommended for a range of fruit, and vegetable commodities, hops and tree nuts.

The 2017 JMPR established an ADI of 0–1 mg/kg bw for fosetyl and for phosphonic acid and an ARfD was determined to be unnecessary.

The 2017 JMPR established residue definitions for plant and animal commodities:

- For compliance with MRLs and for estimation of dietary exposure for plant commodities: *sum of fosetyl, phosphonic acid and their salts, expressed as phosphonic acid*
- For compliance with MRLs and for estimation of dietary exposure for animal commodities: *Phosphonic acid*

The residue is not fat-soluble

The Fiftieth Session of the CCPR (2018) listed fosetyl-aluminium for evaluation of additional uses by the 2019 Extra JMPR. The current Meeting received new GAP information for fosetyl-Al and fosetyl on blackberries, brassica vegetables, coffee, kiwifruit, pineapples and new supporting residue information from the manufacturer.

RESIDUE ANALYSIS

Analytical methods

Analytical methods for the analysis of fosetyl-Al (and fosetyl) and for phosphonic acid in plant and animal commodities are based on either those involving GC analysis after a derivatisation step (methylation) or those involving LC-MS/MS analysis.

The SOP-90113, RE 21.82 and AR155-97 derivatisation methods used in the pineapple and kiwifruit residue studies and the 00861/M001 LC-MS/MS method used in the kiwifruit, pineapple, brassica and coffee residue studies were among those reviewed by the 2017 JMPR and considered suitable data generation methods for measuring fosetyl-Al, fosetyl and phosphonic acid in plant and animal commodities.

LC-MS/MS Method (JAOAC, 2003)

The Meeting received information on an additional LC-MS/MS method [Ref: JAOAC 86(4), 2003] used in some of the blackberry trials. In this method, homogenised samples were dispersed in water and the internal standard "diethyl phosphate" (DEP) was added. The mixture was blended, centrifuged and the filtrate was diluted with water. The analytes were separated via reversed phase HPLC and quantified by means of MS/MS detection. Two MRM transitions were monitored for fosetyl-Al (m/z 109→81 and 109→63) and phosphonic acid (m/z 81→63 and 81→79). The MRM transitions for the DEP internal standard were m/z 153→125 and 153→79.

In a validation study reported by Klose, 2012 [Ref: OG/11-2-3], apparent residues in control samples using the quantitation MRMs were found to be below 30% of the LOQ. A second MRM transition for confirmation was monitored for both analytes but results are not reported in the study. Linear correlation between the injected amount and the detector response was observed for matrix matched standards from 2.0 to 100 ng/mL, with correlation coefficients all >0.991. The LODs for both analytes were 0.01 mg/kg and the LOQs were 0.1 mg/kg.

The mean recovery rates for both analytes in blackberry fruit ranged from 82–104% with RSD values of 6.3–14%.

Table 1 Recovery results for fosetyl-Al (JAOAC 86(4), 2003) – Blackberry

		1st MRM: m/z 109→ 81			2nd MRM: m/z 109→ 63 [a]		
Fortification level [mg/kg]	N	% Recoveries	Mean [%]	RSD [%]	% Recoveries	Mean [%]	RSD [%]
0.1	5	91, 104, 81, 72, 83	86	14			
40	5	111, 109, 105, 98, 97	104	6.3			
		Overall	95	14			

[a] Results not reported in the study

Table 2 Recovery results for phosphonic acid (JAOAC 86(4), 2003) - Blackberry

		1st MRM: m/z 81→ 63			2nd MRM: m/z 81→79 [a]		
Fortification level [mg/kg]	N	% Recoveries	Mean [%]	RSD [%]	% Recoveries	Mean [%]	RSD [%]
0.1	4	81, 82, 222[b], 102, 97	90	12			
40	5	85, 89, 77, 76, 82	82	7.0			
		Overall	85	10			

[a] Results not reported in the study

[b] This value was recognized as outlier by Grubbs' test

QuPPe Method

The 2017 JMPR also concluded that the multi-residue QuPPe method is suitable for the analysis of fosetyl-Al and phosphonic acid in representative samples with a high water, high oil, high protein, high starch and high acid content. This method involves extraction in acidified methanol, centrifugation, filtration and dilution prior to LC-MS/MS analysis (using a graphitic carbon column). LOQs are 0.01 mg/kg (fosetyl-Al) and 0.1 mg/kg (phosphonic acid).

Additional validation studies were provided to the Meeting on the use of the QuPPe method for measuring residues of fosetyl-Al and phosphonic acid in blackberries.

In validation studies reported by Wilde, 2010 [Ref: P 2093 G] and Bacher, 2013 [Ref: P 2749 G], apparent residues in control samples were <30% of the LOQ except in one field trial control sample where apparent residues of fosetyl-Al were found (and the recovery values corrected accordingly). Linearity ranges were from 0.25 to 50 ng/mL for fosetyl-Al and from 0.25 to 100 ng/mL for phosphonic acid and regression correlation coefficients (r) were >0.99.

In the 2010 validation study, the LOQ for fosetyl-Al was 0.1 mg/kg and was 0.2 mg/kg for phosphonic acid and in the 2013 study the respective LOQs were 0.05 mg/kg and 0.1 mg/kg. In both studies, the results were expressed as fosetyl.

The mean recovery rates for both analytes in blackberry fruit ranged from 87–110% with RSD values of 1–17%.

Table 3 Recovery results for fosetyl-Al (QuPPe method) - Blackberry

		1st MRM: m/z 109→ 81			2nd MRM: m/z 109→ 63			Ref
Fortification [mg/kg]	N	% Recoveries	Mean [%]	RSD [%]	% Recoveries	Mean [%]	RSD [%]	
0.05	5	102, 100, 104, 100, 102	102	1.6				P 2749 G
0.1	8	90, 90, 84, 90, 92[a], 105, 107, 109	96	10	91, 90, 84, 91, 87[a], 109, 110, 112	97	12	P 2093 G
0.1	5				102, 102, 99, 100, 101	101	1.3	P 2749 G

		1st MRM: m/z 109→ 81			2nd MRM: m/z 109→ 63			Ref
Fortification [mg/kg]	N	% Recoveries	Mean [%]	RSD [%]	% Recoveries	Mean [%]	RSD [%]	
0.5	3	99, 102, 98	100	2.1				P 2749 G
1.0	3	87, 86, 90[a]	88	2.4	87, 86, 88[a]	87	1.1	P 2093 G
1.0	3				100, 101, 99	100	1.0	P 2749 G
2.0	1	104	104		104	104		P 2093 G
5.0	1	102	102		100	100		P 2093 G
5.0	2	106, 110	108					P 2749 G
10	2	105, 107	106		109, 111	110		P 2093 G
10	2				108, 110	109		P 2749 G
		Overall	99	7.9		99	9.0	

[a] Recovery corrected for residue in related untreated specimen

Table 4 Recovery results for phosphonic acid (QuPPe method) - Blackberry

		1st MRM: m/z 81→63			2nd MRM: m/z 81→79			Ref
Fortification level [mg/kg]	N	% Recoveries	Mean [%]	RSD [%]	% Recoveries	Mean [%]	RSD [%]	
0.1	5	109, 100, 82, 106, 104	100	11	107, 96, 81, 100, 96	96	9.9	P 2749 G
0.2	7	87, 112, 86, 90, 106, 112, 112	101	12	83, 116, 76, 96, 113, 117, 119	103	17	P 2093 G
1.0	3	103, 111, 105	106	3.9	102, 115, 104	107	6.5	P 2749 G
2.0	3	85, 86, 89	87	2.4	85, 87, 90	87	2.9	P 2093 G
5.0	1	101	101		103	103		P 2093 G
10	2	93, 103	98		90, 101	96		P 2749 G
100	2	99, 109	104		102, 116	109		P 2093 G
		Overall	100	9.8		100	12.7	

USE PATTERNS

Summary information on GAP for fosetyl-Al and fosetyl in over 90 countries and over 30 crops was available, with authorised fosetyl-Al or fosetyl labels available for Australia, Brazil, Central American countries (including Costa Rica and Nicaragua) and several European countries.

The following tables summarize the representative critical GAPs for fosetyl-Al and fosetyl for crops relevant to the available residue field trials.

Table 5 Representative registered uses of fosetyl-Al

Crop		Country	Application						PHI (days)	Remarks
			type	method	no	kg ai/hL (max)	water L/ha	kg ai/ha (max)		
Berries and other small fruits										
	Blackberry	Central America	F	Foliar		0.48		1.6	30	
	Blackberry	Germany [a]	F	Foliar	2		1000 max	1.73	21	RTI: 10-14 days
	Blackberry	Germany [a]	G	Foliar	2		1000 max	1.73	14	RTI: 10-14 days
	Raspberry	Central America	F	Foliar		0.48		1.6	30	
Assorted tropical and sub-tropical fruit – inedible peel										

Crop		Country	Application						PHI (days)	Remarks
			type	method	no	kg ai/hL (max)	water L/ha	kg ai/ha (max)		
	Kiwifruit	Italy	F	Soil drench	2	4.0	1-2 L/plant		40	From BBCH69, RTI: 30 days min
	Kiwifruit	Italy	F	Foliar	2	0.2	2000	4.0	40	From BBCH69, RTI: 30 days min
	Pineapple	Australia	F	Soil drench	1			3.68	After planting	
	Pineapple	Australia	F	Foliar			5000	3.68	7	RTI: 42 days, late summer to early winter
	Pineapple	Brazil	F	Seedling dip	1	0.08			Pre-plant	
	Pineapple	Brazil	F	Foliar	3	0.2	1000		20	RTI: 15 days
	Pineapple	Central America	F	Foliar		0.48		2.4	90	
	Pineapple	Costa Rica	F	Seedling dip	1	0.24			Pre-plant	
	Pineapple	Costa Rica	F	Foliar	3			3.6	90	RTI: 90 days
	Pineapple	Nicaragua	F	Foliar				3.6	90	
	Pineapple	USA	F	Seedling dip		0.24			Pre-plant	
	Pineapple	USA		Foliar	6	0.36	3740 max		90	RTI: 90 days
Seeds for beverages and sweets										
	Coffee bean	Brazil	F	Foliar	2		200-500	1.6	30	RTI: 30 days
	Coffee bean	Central Americas	F	Foliar		0.48		1.6	30	

Note: Central American countries are Belize, Dominican Republic, El Salvador, Guatemala, Honduras and Panama.

F = field use, G = glasshouse/indoor use, N = nursery use/seedling treatments

[a] In combination with fluopicolide

Table 6 Representative registered uses of fosetyl (co-formulated with propamocarb) on brassica vegetables

Crop	Country	Application						PHI (days)	Remarks
		type	method	no	kg ai/hL (max)	water L/ha	kg ai/ha (max)		
Head Brassicas									
Brussels sprouts	Sweden	G,N	Drench	2		20,000-40,000	9.3	Pre & post emergence	RTI: 10-14 days
Brussels sprouts	Hungary	G,N	Drench	2		20,000	9.3	Pre & post emergence	RTI: 7-10 days
Brussels sprouts	UK	N	Drench	2		20,000-40,000	9.3	Pre & post emergence	RTI: ~10-14 days
Cabbage, head	Sweden	G,N	Drench	2		20,000-40,000	9.3	Pre & post emergence	RTI: 10-14 days
Cabbage, head	Hungary	G,N	Drench	2		20,000	9.3	Pre & post emergence	RTI: 7-10 days
Cabbage, head	UK	N	Drench	2		20,000-40,000	9.3	Pre & post emergence	RTI: ~10-14 days
Chinese cabbage	UK	N	Drench	2		20,000-40,000	9.3	Pre & post emergence	RTI: ~10-14 days
Flowerhead Brassicas									
Broccoli	Sweden	G,N	Drench	2		20,000-40,000	9.3	Pre & post emergence	RTI: 10-14 days
Broccoli	UK	N	Drench	2		20,000-40,000	9.3	Pre & post emergence	RTI: ~10-14 days

Crop	Country	Application						PHI (days)	Remarks
		type	method	no	kg ai/hL (max)	water L/ha	kg ai/ha (max)		
Cauliflower	Sweden	G,N	Drench	2		20,000-40,000	9.3	Pre & post emergence	RTI: 10-14 days
Cauliflower	Hungary	G,N	Drench	2		20,000	9.3	Pre & post emergence	RTI: 7-10 days
Cauliflower	UK	N	Drench	2		20,000-40,000	9.3	Pre & post emergence	RTI: ~10-14 days
Brassica leafy vegetables									
Kale / Collards	UK	N	Drench	2		20,000-40,000	9.3	Pre & post emergence	RTI: ~10-14 days

G = glasshouse, N = nursery

RESULTS OF SUPERVISED RESIDUE TRIALS ON CROPS

The Meeting received information on supervised field trials involving soil or foliar treatments of fosetyl, fosetyl-Al and phosphonic acid to the following crops.

Group	Crop	Active ingredient	Region/Country	Table no
Berries and other small fruits	Blackberries	Fosetyl-Al	Europe	7
Assorted tropical and sub-tropical fruits – Inedible peel	Kiwifruit	Fosetyl-Al	Europe	8
	Pineapple	Fosetyl-Al	Central America	9
Brassica vegetables	Cabbage	Fosetyl	Europe	10
	Cauliflower			11
	Kale, curly			12
Seeds for beverages & sweets	Coffee	Fosetyl-Al	Brazil	13

The supervised trials were well documented with laboratory and field reports. Laboratory reports included procedural recoveries with spiking at residue levels similar to those occurring in samples from the supervised trials. Dates of analyses or duration of residue sample storage were also provided. Although trials included control plots, no control data are recorded in the tables unless residues in control samples exceeded the LOQ. In such cases, the residues found are noted as "c=nn mg/kg" and where these are greater than 20% of the residue in the corresponding samples from treated plots, the results are not considered suitable for estimating maximum residue levels. Residue data are recorded unadjusted for recovery unless noted.

Results from replicated field plots are presented as individual values. Residues and application rates have been reported as provided in the study reports except for finite values below the LOQ, where these have been reported as <LOQ mg/kg. The results from trials used for the estimation of maximum residue levels (underlined) have been rounded to two significant digits (or if close to the LOQ, rounded to one significant digit) in the Appraisal.

When multiple applications were made to a crop, the application rates, spray concentration and spray volumes were not always identical from one application to the next. In most trials, the actual treatment rates were within 10% of the listed 'target' application rates, but if not, the actual treatment rates are listed.

In this Evaluation, the term 'Total residues' is used to report the sum of the phosphonic acid residues and the fosetyl/fosetyl-Al residues (expressed as phosphonic acid), using the following formulae:

$$\text{Total residue (as phosphonic acid) [mg/kg]} = \frac{\text{fosetyl-Al [mg/kg]} \times MW_{\text{phosphonic acid}} \times 3}{M_{\text{fosetyl-Al}}} + \text{phosphonic acid [mg/kg]}$$

$MW_{\text{fosetyl-Al}}$: Molecular weight of fosetyl-Al = 354.1 g/mol

$MW_{phosphonic\ acid}$: Molecular weight of phosphonic acid = 82 g/mol

And

$$\text{Total residue (as phosphonic acid) [mg/kg]} = \frac{\text{fosetyl [mg/kg]} \times MW_{phosphonic\ acid}}{MW_{fosetyl}} + \text{phosphonic acid [mg/kg]}$$

$MW_{phosphonic\ acid}$: Molecular weight of phosphonic acid = 82 g/mol

$MW_{fosetyl}$: Molecular weight of fosetyl = 110 g/mol

The conversion factors are 0.695 (fosetyl-Al to phosphonic acid) and 0.745 (fosetyl to phosphonic acid).

Berries and other small fruit

Blackberry – fosetyl-Al

In supervised field (5) and greenhouse (4) trials conducted in Europe, two foliar sprays of fosetyl-Al (WG formulation) were applied 6–8 days apart to blackberry canes, between BBCH 67 and 85. Samples of berries were stored frozen for up to 320 days before extraction and LC-MS/MS analysis using either the QuPPe method (in the 2010 and 2012 trials) or method JAOAC 86(4), 2003 (in the 2011 trials) to measure residues of fosetyl-Al and phosphonic acid. Average concurrent recovery rates were 95–102% for fosetyl-Al (fortification levels of 0.05–40 mg/kg) and 85-102% for phosphonic acid (fortification levels of 0.1–100 mg/kg) and the LOQs were 0.05–0.1 mg/kg (fosetyl-Al) and 0.1–0.2 mg/kg (phosphonic acid).

In the trials conducted in 2010 and 2012, the residue results were only reported as fosetyl equivalents, and as described in the 2017 Evaluation, conversion factors of 1.073 (fosetyl to fosetyl-Al) and 0.745 (fosetyl to phosphonic acid) were used to express these results as fosetyl-Al and phosphonic acid equivalents.

Table 7 Residues in blackberries from supervised trials in Europe involving foliar applications of fosetyl-Al (WG formulations)

BLACKBERRY Country, year Location (variety) References	Application				DALA	Residues (mg/kg)			
	no	kg ai/ha	kg ai/hl	water (L/ha)		matrix	Fosetyl-Al	Phos-acid	Total residues
GAP: Germany	2	1.73		1000 max	21 (F)* 14 (G)	RTI: 10-14 days			
Germany, 2010 Koln (Loch Ness) P 2093 G-1015	2	2.0	0.2	1000	21	berries	<0.11[a] c=0.26[a]	0.97[a] c=6.1[a]	1.0[a] c=6.3[a]
Germany, 2010 Oberkirch (Loch Ness) P 2093 G-1013	2	2.0	0.2	1000	0 7 14 21 28	berries	11 7.0 1.0 0.26 <0.11	25 c=1.5 24 37 c=1.2 37 c=1.0 31	33 c=1.6 29 37 c=1.3 37 c=1.1 31

BLACKBERRY Country, year Location (variety) References	Application				DALA	Residues (mg/kg)			
	no	kg ai/ha	kg ai/hl	water (L/ha)		matrix	Fosetyl-Al	Phos-acid	Total residues
Germany, 2011 Köln (Loch Ness) OG/11-2-3 RU 1113	2	2.0	0.2	1000	0 7 14 21 28	berries	7.2 4.2 0.81 1.5 1.3	4.0 5.8 4.3 5.4 c=0.23 5.2	9.0 8.7 4.9 <u>6.4</u> c=0.3 6.2
Germany, 2011 Karlsruhe (Dirkson Thornless) OG/11-2-3 RU 1115	2	2.0	0.2	1000	14 21	berries	<0.1 <0.1	4.2 3.8	<u>4.3</u> 3.9
Germany, 2011 Köln (Loch Ness) [Greenhouse] OG/11-2-3 RU 1116	2	2.0	0.2	1000	0 7 14 21 28	berries	11 4.6 0.83 1.1 0.39	2.9 1.4 0.3 1.9 1.6	10 4.6 0.88 <u>2.6</u> 1.8
Germany, 2012 Köln (Loch Ness) [Greenhouse] P 2749 G-T1223	2	1.73	0.17	1000	0 7 14 21 28	berries	14 3.4 0.82 0.42 0.11	5.1 8.9 4.9 4.2 2.8	15 11 <u>5.5</u> 4.5 2.9
Germany, 2012 Oberkirch (Loch Tay) [Greenhouse] P 2749 G-T1224	2	1.73	0.17	1000	14 21	berries	3.0 1.6	17 c=4.2 10 c=3.1	19 c=4.3 12 c=3.2
Germany, 2011 Karlsruhe (Dirkson Thornless) [Greenhouse] 2nd season P 2749 G-T1227	2	2.0	0.2	1000	386	berries	<0.05	<0.07	<0.11
Germany, 2012 Karlsruhe (Dirkson Thornless) [Greenhouse] P 2749 G-T1225	2	1.878	0.188	1000	14 21	berries	8.0 5.0	16 9.7	<u>21</u> 13

* F – Field use; G – Glasshouse application

[a] A potential mislabelling of untreated sample and treated sample. Highest values have been selected for treated sample

Assorted tropical and sub-tropical fruits – inedible peel

Kiwifruit – fosetyl-Al

In supervised trials conducted in Europe, two foliar sprays of fosetyl-Al (WG formulations) were applied to kiwifruit vines, the first at BBCH 69 and then about 40 days before harvest (retreatment intervals of 78–110 days). Samples of whole fruit, peel and flesh were stored frozen for 113–194 days (up to 496 days in the 2008 trials) before extraction and analysis. In the 2000 and 2001 trials, samples

were analysed for fosetyl-Al and phosphonic acid using the GC-FPD method AR 155–97 with mean recovery rates of 83–95% (fosetyl-Al) and 84–90% (phosphonic acid) in samples fortified at 0.5–10 mg/kg (fosetyl-Al) and 0.5–20 mg/kg (phosphonic acid). The method LOQs were 0.5 mg/kg for each analyte. In the 2008 trials, the LC-MS/MS method 00861/M001 was used to measure residues of fosetyl-Al and phosphonic acid in peel, flesh and whole fruit, with average concurrent recovery rates of 92–96% (fosetyl-Al) and 101–108% (phosphonic acid). Fortification levels were 0.025–1.0 mg/kg (fosetyl-Al) and 0.5–20 mg/kg (phosphonic acid) and the respective LOQs were 0.025 mg/kg and 0.5 mg/kg.

Table 8 Residues in kiwifruit from supervised trials in Europe involving foliar applications of fosetyl-Al (WG formulations)

KIWIFRUIT Country, year Location (variety) References	Application				DALA	Residues (mg/kg)			
	no	kg ai/ha	kg ai/hl	water (L/ha)		matrix	Fosetyl-Al	Phos-acid	Total residues
GAP: Italy (foliar)	2	4.0 max	0.2	1500-2000	40	RTI: 30 days min			
Italy, 2000 Cesena (Forlì) (Hayward) DR00EUS159 ITA0101	2	4.0	0.267	1500	0 15 28 39 39 39	whole fruit flesh peel	6.7 2.3 1.5 0.52 <0.5 2.1	19 15 12 12 14 13	24 16 13 <u>12</u> 14 14
Italy, 2000 Bologna (Hayward) DR00EUS159 ITA0102	2	4.0	0.333	1200	0 14 28 42 42 42	whole fruit flesh peel	13 <0.5 c=3.5 <0.5 c=2.0 <0.5 c=0.56 <0.5 <0.5 c=1.7	2.8 c=8.5 3.8 c=4.3 3.5 c=11 3.0 c=11 6.1 c=14 4.6 c=18	12 c=8.8 4.1 c=6.7 3.8 c=13 3.3 c=12 6.4 c=14 4.9 c=19
Italy, 2000 Bernalda (MT) (Hayward) DR00EUS159 ITA0201	2	4.0	0.333	1200	0 41 41 41	whole fruit flesh peel	5.0 <0.5[a] <0.5 <0.5	6.6 3.6[a] 3.4 4.2	10 <u>4.0</u>[a] 3.7 4.5
Italy, 2001 Francolino (FE) (Hayward) 01R321-1	2	4.0	0.267	1500	0 40	whole fruit flesh	6.0 <0.05	46 c=0.59 50	50 c=0.94 <u>50</u>
Spain, 2001 Almussafes (Hayward) 01R321-2	2	4.0	0.267	1500	0 39	whole fruit flesh	7.2 <0.5	33 c=0.73 67	38 c=1.1 <u>67</u>
France (S), 2008 Montech (Hayward) 08-2104-01	2	4.0	1.0	400	0 40 40 40	whole fruit flesh peel	0.71 0.08 <0.025 0.24	14 c=5.0 17 c=4.7 18 c=4.4 13 c=3.3	14 c=5.0 17 c=4.7 18 c=4.4 13 c=3.3

KIWIFRUIT Country, year Location (variety) References	Application				DALA	Residues (mg/kg)			
	no	kg ai/ha	kg ai/hl	water (L/ha)		matrix	Fosetyl-Al	Phos-acid	Total residues
Italy, 2008 Brisighella (RA) (Hayward) 08-2104-02	2	4.0	0.267	1500	0 40 40 40	whole fruit flesh peel	6.5 2.6 0.23 9.0	20 30 30 26	25 <u>32</u> 30 32
Italy, 2008 Ostellato (Hayward) 08-2104-03	2	4.0	0.267	1500	0 40 40 40	whole fruit flesh peel	7.3 1.2 0.11 5.1	23 c=0.57 33 c=0.75 39 c=0.61 37 c=0.5	28 c=0.59 <u>34</u> c=0.77 39 c=0.63 41 c=0.52

[a] Calculated whole fruit residues [(peel weight × peel residues) + (flesh weight × flesh residues)] /total fruit weight

Pineapple – fosetyl-Al

In supervised trials conducted in Central America, evaluated by the 2017 JMPR, pineapple slips or suckers (ratoons) were dipped in solutions of 2.4–6.2 g ai/litre fosetyl-Al (WDG or WP formulations) immediately before planting and treated with 3-10 foliar sprays of fosetyl-Al (WDG or WP formulations) from the 9-leaf, root hair formation, start of flowering or fruit formation.

Samples of fruit (without crowns), and sub-samples of flesh and peel were stored frozen for up to 333 days before analysis using the LC-MS/MS method 00861/M001 to measure residues of fosetyl-Al and phosphonic acid, with average concurrent recovery rates of 68–109% for fosetyl-Al (fortification levels of 0.05–1.5 mg/kg) and 68–140% for phosphonic acid (fortification levels of 0.05–25 mg/kg). The LOQs were 0.05 mg/kg (fosetyl-Al) and 0.5 mg/kg (phosphonic acid).

Table 9 Residues in pineapples from supervised trials in Central America and Hawaii involving pre-plant dip and foliar applications of fosetyl-Al (WDG or WP formulations)

PINEAPPLE Country, year Location (variety) References	Application				DALA	matrix	mean residues (mg/kg) corrected for recovery		
	no	kg ai/hL	kg ai/ha	RTI (days)			Fosetyl-Al	Phos-acid	Total residues
GAP: Costa Rica	1 dip 3 foliar	0.24 0.36		90	90				
Costa Rica, 2005 Guacimo, Limon (MD2) RAFYX078 FY002-04D-TRTD1	1 dip+ 3 foliar	0.24 0.13-0.14	- 3.6-3.8	189 70 76	30 60 92 120 92 92	fruit fruit fruit fruit flesh peel	<0.05 <0.05 <0.05 <0.05 <0.05 <0.05	2.5 1.9 2.4 2.8 2.0 2.8	2.5 1.9 2.4 <u>2.8</u> 2.0 2.8
Martinique, 2005 Basse Pointe (Cayenne) RAFYX078 FY008-04D-TRTD1	1 dip + 3 foliar	0.3 0.06 0.06 0.14	- 3.6-3.8	167 181 65	27 55 88 111 88	fruit fruit fruit fruit flesh peel	<0.05 <0.05 <0.05 <0.05 <0.05 <0.05	3.2 4.7 1.0 3.8 4.2 7.0	3.2 4.7 1.0 <u>3.8</u> 4.2 7.0

PINEAPPLE Country, year Location (variety) References	Application				DALA	matrix	mean residues (mg/kg) corrected for recovery		
	no	kg ai/hL	kg ai/ha	RTI (days)			Fosetyl-Al	Phos-acid	Total residues
Costa Rica, 2014	1 dip +	0.24	-	125	90	fruit	<0.05	2.2	2.2
La Virgen de	3 foliar	0.14	3.6	148					
Sarapiquí Heredia				90	90	flesh	<0.05	1.7	1.7
(MD-2)						peel	<0.05	2.6	2.6
RAFYN023 FY001-13HA-TRTD1									
Costa Rica	1 dip +	0.24	-	128	30	fruit	<0.05	1.7	1.7
Puerto Viejo	3 foliar	0.14	3.6	138	60	fruit	<0.05	2.8	2.8
Heredia America, Middle 2014				118	89	fruit	<0.05	3.6	3.6
Pineapple MD-2					89	flesh	<0.05	2.7	2.7
(Golden)						peel	<0.05	2.6	2.6
RAFYN023 FY002-13DA-TRTD1									
Costa Rica	1 dip +	0.24	-	118	30	fruit	<0.05	9.5	9.5
Muelle Alajuela	3 foliar	0.14	3.6	100	60	fruit	<0.05	5.7	5.7
America, Middle 2014				88	84	fruit	<0.05	5.9 c=2.8	5.9 c=2.8
Pineapple MD-2					120	fruit	<0.05	6.9	6.9
(Golden)					84	flesh	<0.05	3.9 c=2.1	3.9 c=2.1
RAFYN023 FY003-13DA-TRTD1					84	peel	<0.05	7.0 c=3.9	7.0 c=3.9

Ref: Pesticide residues in food 2017. Evaluations 2017 Part 1 – Residues. FAO Plant Production and Protection Paper 233, pp 1513-1518, Table 112.

Brassica vegetables

The Meeting received information on trials with fosetyl on cabbages, cauliflower and curly kale.

Fosetyl is rapidly hydrolysed to phosphonic acid, and in vegetable seedlings, fosetyl residues decline to <LOQ within 3 weeks after treatment. For calculating total residues in brassica vegetables (harvested more than 12 weeks after treatment), where both fosetyl and phosphonic acid residues were <LOQ, the LOQ value for phosphonic acid was used to calculate total residues.

Cabbage, head - fosetyl

In supervised trials on red (1) and white (8) cabbages conducted in Europe, fosetyl (SL formulation) was applied at rates equivalent to 9.3 kg ai/ha in 20,000 litres water as soil drenches to seedling trays in greenhouses (nursery) 0–1 day after sowing and again as seedling/soil drenches 7–11 days later. After 28–54 days, the seedlings were planted out in the field. At maturity, cabbage head samples were quartered in the field and two diagonal quarter-head samples were frozen within 24 hours and stored for 62–286 days before extraction and analysis. In three trials, seedlings (without roots) were also sampled at intervals after transplanting and stored frozen for up to 373 days before extraction. Extraction and analysis for fosetyl and phosphonic acid was by the LC-MS/MS method 00861/M001 (LOQs of 0.0093 mg/kg for fosetyl and 0.2 mg/kg for phosphonic acid). Average concurrent recovery rates were 77–123% for fosetyl-Al with fortification levels of 0.01–1.0 mg/kg (3.0 mg/kg for seedlings) and 83–134% for phosphonic acid (fortification levels of 0.2–200 mg/kg).

Table 10 Residues in head cabbages from supervised trials in Europe involving soil/seedling drench applications of fosetyl (SL formulations)

CABBAGE Country, year Location (variety) References	Application				DALA	Residues (mg/kg)			
	no	kg ai/ha	kg ai/hl	water (L/ha)		matrix	Fosetyl	Phos-acid	Total residues
GAP: UK	2	9.3		20,000-40,000	Pre & post-emergence soil drench, RTI: ~10-14 days				
Belgium, 2008 Villers-Perwin (Kilaton F1) 08-2154-05	2	9.3	0.0465	20,000	23 26 30 37 105 120	seedlings without roots head	<0.0093 <0.0093 <0.0093 <0.0093 <0.0093 <0.0093	9.3 4.4 4.1 0.73 <0.2 <0.2	9.3 4.4 4.1 0.74 <u><0.2</u> <0.2
United Kingdom, 2008 Barrow (Stonehead) 08-2154-06	2	9.3	0.0465	20,000	89	head	<0.0093	<0.2	<u><0.2</u>
France, (N), 2008 Hangest en Santerre (Quintal d´Alsac) 08-2154-08	2	9.3	0.0465	20,000	102	head	<0.0093	<0.2	<u><0.2</u>
Spain, 2008 Gava (Savoy King) 08-2182-05	2	9.3	0.0465	20,000	176	head	<0.0093	<0.2	<u><0.2</u>
Italy, 2008 Andria (Charmant) 08-2182-06	2	9.3	0.0465	20,000	121	head	<0.0093	<0.2	<u><0.2</u>
Italy, 2009 Andria Bari (Charmant) 09-2025-01	2	9.3	0.0465	20,000	90 105	head	<0.0093 <0.0093	<0.2 <0.2	<0.2 <u><0.2</u>
Spain, 2009 Alginet (Redsky) [Red cabbage] 09-2025-02	2	9.3	0.0465	20,000	104 117	head	<0.0093 <0.0093	<0.2 <0.2	<0.2 <u><0.2</u>
France, (N), 2010 St. Ouen L`Aumone (nursery) Criquebeuf sur Seine (field) (Kingston) 10-2049-01	2	9.3	0.0465	20,000	29 32 36 43 129 140	seedlings without roots head	<0.0093 <0.0093 <0.0093 <0.0093 <0.0093 <0.0093	237 200 88 16 <0.2 <0.2	237 200 88 16 <0.2 <u><0.2</u>
Netherlands, 2010 Wervershoof (nursery) Zwaagdijk (field) (Candela) 10-2049-02	2	9.3	0.0465	20,000	46 49 53 60 124 145	seedlings without roots head	<0.0093 <0.0093 <0.0093 <0.0093 <0.0093 <0.0093	28 22 11 1.5 <0.2 <0.2	28 22 11 1.5 <u><0.2</u> <0.2

Cauliflower – fosetyl

In supervised trials on cauliflowers conducted in Europe, fosetyl (SL formulation) was applied at rates equivalent to 9.3 kg ai/ha in 20,000 litres water as soil drenches to seedling trays 0–1 day after sowing and again as seedling/soil drenches 7–11 days later. After 25–51 days in the nursery (greenhouse), the seedlings were planted out in the field. Curds (without green leaves) were sampled at maturity, and in some trials the curds were further sub-sampled in the field by selecting two diagonal quarter-heads. All samples were frozen within 24 hours and stored for 77-306 days before extraction and analysis. In four trials, seedlings (without roots) were also sampled at intervals after transplanting and stored frozen for up to 360 days before analysis. Extraction and analysis for fosetyl and phosphonic acid was by the LC-MS/MS method 00861/M001 (LOQs of 0.0093 mg/kg for fosetyl and 0.2 mg/kg for phosphonic acid). Average concurrent recovery rates were 89–107% for fosetyl-Al with fortification levels of 0.01–1.0 mg/kg and 91–93% for phosphonic acid (fortification levels of 0.2–20 mg/kg).

Table 11 Residues in cauliflowers from supervised trials in Europe involving soil/seedling drench applications of fosetyl (SL formulations)

CAULIFLOWER Country, year Location (variety) References	Application				DALA	Residues (mg/kg)			
	no	kg ai/ha	kg ai/hl	water (L/ha)		matrix	Fosetyl	Phos-acid	Total residues
GAP: UK	2	9.3		20,000-40,000	Pre & post-emergence soil drench, RTI: ~10-14 days				
Germany, 2008 Langenfeld (Cool) 08-2156-01	2	9.3	0.0465	20,000	18 21 25 32 86 95	seedlings without roots curd curd [a]	<0.0093 <0.0093 <0.0093 <0.0093 <0.0093 <0.0093	7.3 6.1 15 3.1 <0.2 <0.2	7.3 6.1 15 3.1 <0.2 <0.2
Netherlands, 2008 Wervershoof (Fremont) 08-2156-09	2	9.3	0.0465	20,000	33 36 40 47 101 105	seedlings without roots curd	<0.0093 <0.0093 <0.0093 <0.0093 <0.0093 <0.0093	1.7 1.9 1.6 0.39 <0.2 <0.2	1.7 1.9 1.6 0.4 <0.2 <0.2
United Kingdom, 2008 Barrow (FI Freedom) 08-2156-10	2	9.3	0.0465	20,000	26 29 34 40 95 102	seedlings without roots curd	<0.0093 <0.0093 <0.0093 <0.0093 <0.0093 <0.0093	197 175 95 36 <0.2 <0.2	197 175 95 36 <0.2 <0.2
France (N), 2008 Fondettes (Cenats F1) 08-2156-11	2	9.3	0.0465	20,000	32 35 39 46 195 216	seedlings without roots curd [a]	<0.0093 <0.0093 <0.0093 <0.0093 <0.0093 <0.0093	4.5 4.2 3.5 2.7 <0.2 <0.2	4.5 4.2 3.5 2.7 <0.2 <0.2
Germany, 2008 Werl-Westönnen (Lecanu) 08-2156-12	2	9.3	0.0465	20,000	87	curd[a]	<0.0093	<0.2	<0.2
Belgium, 2008 Villers-Perwin (Clapton F1) 08-2156-13	2	9.3	0.0465	20,000	84	curd[a]	<0.0093	<0.2	<0.2

CAULIFLOWER Country, year Location (variety) References	Application				DALA	Residues (mg/kg)			
	no	kg ai/ha	kg ai/hl	water (L/ha)		matrix	Fosetyl	Phos-acid	Total residues
Germany, 2008 Meckenbeuren (Freedom) 08-2156-14	2	9.3	0.0465	20,000	88	curd	<0.0093	<0.2	<u><0.2</u>
France (N), 2008 Hangest en Santerre (Merveille de toute saison) 08-2156-15	2	9.3	0.0465	20,000	102	curd[a]	<0.0093	<0.2	<u><0.2</u>
Spain, 2008 Gava (Pamyros) 08-2183-05	2	9.3	0.0465	20,000	183	curd[a]	<0.0093	<0.2	<u><0.2</u>
Italy, 2008 70031 Andria (Trevi) 08-2183-06	2	9.3	0.0465	20,000	133	curd	<0.0093	<0.2	<u><0.2</u>
Italy, 2012 Biancavilla (nursery) Catania (field) (Candid Charm) 12-2022-01	2	9.3	0.0465	20,000	123 130	curd	<0.0093 <0.0093	<0.2 <0.2	<0.2 <u><0.2</u>
Spain, 2012 E-08850 Gava (Casper RZ) 12-2022-02	2	9.3	0.0465	20,000	140 160	curd[a]	<0.0093 <0.0093	<0.2 <0.2	<0.2 <u><0.2</u>

[a] curds sub-sampled (2 quarters) in the field

Kale, curly – fosetyl

In supervised trials on curly kale conducted in Europe, fosetyl (SL formulation) was applied at rates equivalent to 9.3 kg ai/ha in 20,000 litres water as soil drenches to seedling trays 0–1 day after sowing and again as seedling/soil drenches 7–8 days later. After 28–47 days in the nursery (greenhouse), the seedlings were planted out in the field. Leaves were sampled at maturity, with samples frozen within 24 hours and stored for 70–337 days before extraction and analysis for fosetyl and phosphonic acid using the LC-MS/MS method 00861/M001 (LOQs of 0.0093 mg/kg for fosetyl and 0.2 mg/kg for phosphonic acid). Average concurrent recovery rates were 78–107% for fosetyl-Al with fortification levels of 0.01–0.1 mg/kg and 80-85% for phosphonic acid (fortification levels of 0.2–2.0 mg/kg).

Table 12 Residues in curly kale from supervised trials in Europe involving soil/seedling drench applications of fosetyl (SL formulations)

KALE, CURLY Country, year Location (variety) References	Application				DALA	Residues (mg/kg)			
	no	kg ai/ha	kg ai/hl	water (L/ha)		matrix	Fosetyl	Phos-acid	Total residues
GAP: UK	2	9.3		20,000-40,000	Pre & post-emergence soil drench, RTI: ~10-14 days				
Belgium, 2008 Villers-Perwin (Winnetou F1) 08-2155-05	2	9.3	0.0465	20,000	160	leaves	<0.0093	<0.2	<u><0.2</u>
France (N), 2008 37230 Fondettes (Vert Demi-Nain) 08-2155-06	2	9.3	0.0465	20,000	76	leaves	<0.0093	<0.2	<u><0.2</u>
Spain, 2008 Gava (Reflex F1) 08-2184-03	2	9.3	0.0465	20,000	152	leaves	<0.0093	<0.2	<u><0.2</u>
Italy, 2008 Manfredonia (Cavolo Nero Di Toscana) 08-2184-04	2	9.3	0.0465	20,000	104	leaves	<0.0093	<0.2	<u><0.2</u>

Seeds for beverages

Coffee – fosetyl-Al

In supervised trials conducted in Brazil, two foliar sprays of fosetyl-Al (WP formulations) were applied to coffee plants at BBCH 81 and 30 days later, at BBCH 85. Duplicate samples of coffee cherries were harvested, dried in the field (under shelter) for up to 20 days, hulled and the dried beans frozen for 224–285 days before extraction and analysis for fosetyl-Al using the LC-MS/MS method 00861/M001, with an average concurrent recovery rate of 85% in samples fortified with 0.01 mg/kg or 1.0 mg/kg fosetyl-Al. In a separate study, these samples were also analysed for phosphonic acid residues after frozen storage for 257–318 days before extraction, also using method 00861/M001, with mean recovery rates of 89–95% in samples fortified with 0.1–10 mg/kg. The LOQs were 0.01 mg/kg (fosetyl-Al) and 0.1 mg/kg (phosphonic acid).

Table 13 Residues in coffee beans from supervised trials in Brazil involving foliar applications of fosetyl-Al (WP formulations)

COFFEE Country, year Location (variety) References	Application				DALA	Residues (mg/kg)			
	No	kg ai/ha	kg ai/hL	water (L/ha)		matrix	Fosetyl	Phos-acid	Total residues (mean)
GAP: Brazil	2	1.6			30		RTI: 30 days		
Brazil, 2014 Rio Claro (Catuai) F14-019-01 F15-015	2	1.63 1.59	0.32	509 498	0 15 30 45 60	dry bean	0.025[a] <0.01, <0.01 <0.01, <0.01 <0.01, <0.01 <0.01, <0.01	5.3, 5.4 (5.4) 7.5, 7.5 (7.5) 6.9, 7.1 (7.0) 6.8, 7.1 (6.9) 8.9, 8.6 (8.8)	5.4 7.5 7.0 6.9 <u>8.8</u>

COFFEE Country, year Location (variety) References	Application				DALA	Residues (mg/kg)			
	No	kg ai/ha	kg ai/hL	water (L/ha)		matrix	Fosetyl	Phos-acid	Total residues (mean)
Brazil, 2014 Leme (Obata) F14-019-02 F15-015	2	1.63 1.61	0.32	508 502	0 15 30 45 60	dry bean	<0.01, <0.01 <0.01, <0.01 <0.01, <0.01 <0.01, <0.01 <0.01, <0.01	3.0, 2.9 (3.0) 5.5, 5.8 (5.6) 5.8, 5.9 (5.9) 8.3, 8.0 (8.2) 7.4, 7.9 (7.7)	3.0 5.6 5.9 8.2 7.7
Brazil, 2014 Campinas (Obata) F14-019-03 F15-015	2	1.67 1.61	0.32	523 502	0 15 30 45 60	dry bean	<0.01, <0.01 <0.01, <0.01 <0.01, <0.01 <0.01, <0.01 <0.01, <0.01	4.4, 3.9 (4.2) 6.0, 6.0 (6.0) 5.4, 5.3 (5.3) 5.7, 5.7 (5.7) 8.7, 8.6 (8.7)	4.2 6.0 5.3 5.7 8.7
Brazil, 2014 Pocos de Caldas (Catuai) F14-019-04 F15-015	2	1.62 1.59	0.32	505 498	30	dry bean	<0.01, <0.01	8.6, 9.0 (8.8)	8.8
Brazil, 2014 Andradas (Catuai) F14-019-05 F15-015	2	1.62 1.58	0.32	505 493	30	dry bean	<0.01, <0.01	9.1,8.7 (8.9)	8.9

[a] Mean of four replicate samples

FATE OF RESIDUES DURING PROCESSING

Pineapple dry bran

In supervised field trials conducted in USA on pineapples and evaluated by the 2017 JMPR, residues of fosetyl-Al and phosphonic acid were measures in whole fruit and in dry bran (remaining after processing).

In supervised trials conducted in Hawaii, pineapple slips or suckers (ratoons) were dipped in solutions of 2.4–6.2 g ai/litre fosetyl-Al (WDG or WP formulations) immediately before planting and treated with 3-10 foliar sprays of fosetyl-Al (WDG or WP formulations) from the 9-leaf, root hair formation, start of flowering or fruit formation.

Samples of fruit, leaves and bran (i.e. chopped peel, oven-dried to about 87% dry matter) were stored for up to 50 days before extraction and analysis using the GC-FPD method RE 21. Average concurrent recovery rates were 68-109% for fosetyl-Al (fortification levels of 0.05–1.5 mg/kg) and 68–140% for phosphonic acid (fortification levels of 0.05–25 mg/kg) and LOQs were 0.05 mg/kg (fosetyl-Al) and 0.5 mg/kg (phosphonic acid).

Processing factors (total residues in dry bran / total residues in whole fruit) were calculated from the trials involving dip plus foliar treatments and where apparent residues in untreated samples were less than 20% of the residues in treated samples.

Table 14 Processing factors calculated from residues in pineapple fruit and dry bran from supervised trials in Hawaii involving pre-plant dip and foliar applications of fosetyl-Al (WDG or WP formulations)

PINEAPPLE Country, year Location (variety) References	Application			DALA	matrix	mean residues (mg/kg) corrected for recovery			Processing factor
	no	kg ai/hL	kg ai/ha			Fosetyl-Al	Phos-acid	Total residues	
USA, 1980 Honolua Plantation, (unspecified) R000990	4 foliar	0.08	2.2	harvest	fruit	<0.05	1.0 c=0.17	1.1 c=0.20	2.8
					dry bran	<0.10	3.0 c=0.57	3.1 c=0.64	
	4 foliar	0.16	4.5	harvest	fruit	<0.05	1.8 c=0.20	1.8 c=0.23	3.2
					dry bran	<0.10	5.7 c=0.57	5.8 c=0.64	
USA, 1980 Kunia Plantation (unspecified) R000990	1 dip + 4 foliar 51-108d RTIs	0.48 0.48	- 13	9 months	fruit	<0.05	1.1 c=0.11	1.1 c=0.14	6.1
					dry bran	<0.10	6.6 c=0.31	6.7 c=0.38	
USA, 1980 Haliimaile Plantation (unspecified) R001845	1 dip + 6 foliar 90-92d RTIs	0.24 0.24	- 6.7	90	fruit	<0.05	3.1	3.1	3.9
					dry bran	<0.05	12 c=1.7	12 c=1.8	
	1 dip + 6 foliar 90-92d RTIs	0.48 0.48	- 13	90	fruit	<0.05	5.3	5.3	2.8
					dry bran	<0.05	15 c=1.7	15 c=1.8	
USA, 1982 Honolua Plantation (unspecified) R003880	1 dip + 6 foliar 78-97d RTIs	0.24 0.12	- 3.4	90	fruit	<0.05	0.71	0.74	3.0
					dry bran	<0.05	2.2 c=0.11	2.2 c=0.14	
	1 dip + 6 foliar 78-97d RTIs	0.24 0.24	- 6.7	90	fruit	<0.05	1.0	1.0	3.2
					dry bran	<0.05	3.2 c=0.11	3.2 c=0.14	
USA, 1983 Kunia Plantation (unspecified) R003880 UH8.3P37-4-A	1 dip + 6 foliar 64-93d RTIs	0.24 0.12	- 3.4	82	fruit	<0.05	4.1	4.1	3.7
					dry bran	<0.05	15	15	
	1 dip + 6 foliar 64-93d RTIs	0.24 0.24	- 6.7	82	fruit	<0.05	8.2	8.2	3.2
					dry bran	<0.05	26	26	
USA, 1983 Kunia Plantation (unspecified) R003881 UH8.3P37-4-D	1 dip + 10 foliar 84-122d RTIs	0.24 0.12	- 3.4	92	fruit	<0.05	4.0 c=0.05	4.0 c=0.085	2.2
					dry bran	<0.05	8.8 c=0.38	8.8 c=0.42	
	1 dip + 10 foliar 84-122d RTIs	0.24 0.24	- 6.7	92	fruit	<0.05	5.0 c=0.05	5.0 c=0.085	2.4
					dry bran	<0.05	12 c=0.38	12 c=0.42	

Ref: Pesticide residues in food 2017. Evaluations 2017 Part 1 – Residues. FAO Plant Production and Protection Paper 233, pp 1513-1518, Table 112.

APPRAISAL

Fosetyl-aluminium (fosetyl-Al), fosetyl and phosphonic acid are systemic protectant horticultural fungicides, rapidly absorbed through both leaves and roots and exhibit both acropetal and basipetal translocation. Their mode of action is by inhibiting germination of spores and by blocking development of mycelium, competing with phosphate as allosteric regulator of several enzymes.

Fosetyl-Al and phosphonic acid were first evaluated by the JMPR in 2017, when residue definitions and health-based guidance values were established and a number of maximum residue levels were recommended for a range of fruit, and vegetable commodities, hops and tree nuts.

The 2017 JMPR established an ADI of 0–1 mg/kg bw for fosetyl and for phosphonic acid and an ARfD was not considered necessary.

The 2017 JMPR established residue definitions for plant and animal commodities:

- For compliance with MRLs and dietary risk assessment for plant commodities: *sum of fosetyl, phosphonic acid and their salts, expressed as phosphonic acid*
- For compliance with MRLs and dietary risk assessment for animal commodities: *Phosphonic acid*

The residue is not fat-soluble.

The Fiftieth Session of the CCPR (2018) listed fosetyl-aluminium for evaluation of additional uses by the 2019 Extra JMPR. The current Meeting received new GAP information for fosetyl-Al and fosetyl on blackberries, brassica vegetables, coffee, kiwifruit, pineapples and new supporting residue information.

Methods of analysis

Analytical methods for the analysis of fosetyl-Al (and fosetyl) and for phosphonic acid in plant and animal commodities (based on either GC analysis after a derivatisation step (methylation) or LC-MS/MS analysis) and used in the new supporting residue trials were reviewed by the 2017 JMPR.

The Meeting also received information on an additional LC-MS/MS method, involving the use of diethyl phosphate (DEP) as an internal standard, reverse phase HPLC separation and MS/MS detection. This method was validated for blackberries, with an LOQ of 0.1 mg/kg for each analyte.

Additional validation information was also provided to the Meeting on the use of the 'Quick Polar Pesticide' (QuPPe) method for measuring fosetyl-Al and phosphonic acid in blackberries, with LOQs of 0.05 mg/kg (fosetyl-Al) and 0.1 mg/kg (phosphonic acid). This method was considered by the 2017 JMPR to be suitable for monitoring residues of fosetyl-Al, fosetyl and phosphonic acid in most plant commodities.

The Meeting concluded that the analytical methods used in the new supporting residue trials were suitable for measuring residues of fosetyl-Al, fosetyl and phosphonic acid in plant matrices.

Stability of pesticide residues in stored analytical samples

The 2017 JMPR concluded that while fosetyl-Al residues were not stable in high water content and high oil commodities and that residue stability was variable in high acid commodities (with residues hydrolysing to phosphonic acid), in the storage stability studies where both fosetyl-Al and phosphonic acid residue degradation was measured, the total residues of fosetyl-Al and phosphonic acid were stable over the storage intervals in the studies (6–25 months for high water content, high starch/protein content, high acid content and 29 months for high oil content).

The sample storage intervals in the new residue trials were generally less than 12 months (17 months in some of the kiwifruit trials).

Results of supervised residue trials on crops

The Meeting received GAP information and supporting residue information for fosetyl-Al on blackberries, kiwifruit, pineapple and coffee and for fosetyl on cabbage, cauliflower and curly kale.

In many trials, residues of phosphonic acid (and to a lesser extent fosetyl and fosetyl-Al) were measured in control samples. The Meeting agreed that where these residues in control samples were more than 20% of the concentrations reported in the treated samples, the values could not be used for maximum residue level estimation.

Blackberries - Fosetyl-Al

The critical GAP for fosetyl-Al on blackberries is in Germany, with up to 2 foliar sprays of 1.78 kg ai/ha, 10–14 days apart, with a PHI of 14 days for protected crops.

The properties of Fosetyl-Al are such that the outdoor and greenhouse blackberry growing conditions are not expected to be a key determinant of the residues. The residue data sets were not significantly different (Mann-Whitney). The Meeting agreed to use the results of the outdoor and greenhouse trials matching the GAP for protected crops to estimate a maximum residue level for blackberries.

In six outdoor and greenhouse trials matching the cGAP for protected blackberries, total residues were: 2.6, 4.3, 5.5, 6.4, 21 and 37 mg/kg.

The Meeting estimated a maximum residue level of 70 mg/kg and an STMR of 5.95 mg/kg for blackberries.

Kiwifruit - Fosetyl-Al

The critical GAP for fosetyl-Al on kiwifruit is in Italy, with up to 2 foliar sprays of 4.0 kg ai/ha from BBCH 69, at least 30 days apart, with a PHI of 40 days.

In trials conducted in Europe, matching this critical GAP, total residues in whole fruit were 4.0, 12, 32 and 34 mg/kg.

In these trials, total residues in flesh were 3.7, 14, 30 and 39 mg/kg. In two additional trials matching the critical GAP in Italy, total residues in kiwifruit flesh were 50 and 67 mg/kg.

Noting that total residues in kiwifruit appeared to be evenly distributed between the flesh and peel (flesh:whole fruit ratios of 0.925, 0.94, 1.15 and 1.17), the Meeting agreed that residue concentrations in flesh would also reflect concentrations in whole fruit and that the 'flesh only' results could be used to estimate a maximum residue level for kiwifruit.

For estimating a maximum residue level, the total residue data set matching the cGAP for kiwifruit is: 4.0, 12, 32, 34, 50 and 67 mg/kg.

Total residues in the flesh (edible portion) were: 3.7, 14, 30, 39, 50 and 67 mg/kg.

The Meeting estimated a maximum residue level of 150 mg/kg (whole fruit) and an STMR of 34.5 mg/kg (edible portion) for kiwifruit.

Pineapple – fosetyl-Al

GAP for fosetyl-Al on pineapples in the USA is for a pre-plant dip (0.24 kg ai/hL) followed by up to 6 foliar applications of 0.36 kg ai/hL, 90-day PHI. In trials conducted in the USA matching this GAP but with lower foliar application rates of 0.24 kg ai/hL, total residues were: 1.0, 3.1 and 8.2 mg/kg.

GAP for pineapples in Brazil is for a pre-plant dip (0.08 kg ai/hL) and up to 3 foliar applications of 0.2 kg ai/hL, 20-day PHI. No trials matching this GAP were available.

The critical GAP for pineapples in Costa Rica is for a pre-plant dip (0.24 kg ai/hL) and up to 3 foliar applications of 3.6 kg ai/ha, with a 90 day retreatment interval and a PHI of 90 days.

In five trials conducted in Central America, matching this GAP, total residues in fruit were 2.2, 2.8, 3.6, 3.8 and 6.9 mg/kg. In four of these trials, total residues in flesh (edible portion) were 1.7, 2.0, 2.7 and 4.2 mg/kg.

The Meeting estimated a maximum residue level of 15 mg/kg (whole fruit) and an STMR of 2.35 mg/kg (edible portion) for pineapple.

Head Brassicas (sub-group)

The critical GAP for fosetyl on Brussels sprouts, head cabbage and Chinese cabbage is in the UK, applying the equivalent of up to 9.3 kg ai/ha in 20,000-40,000 L/ha as a pre-emergence nursery soil drench and as a seedling drench, about 10–14 days later (before transplanting).

In nine trials conducted in Europe, matching this cGAP, total residues in cabbage heads at maturity (89–176 days after the last application) were < 0.2 (9) mg/kg.

Noting that the GAP in UK covers all commodities in the Head cabbages sub-group, the Meeting estimated a maximum residue level of 0.2 (*) mg/kg and an STMR of 0.2 mg/kg for the Subgroup of Head Brassicas.

Flowerhead Brassicas (sub-group)

The critical GAP for broccoli and cauliflower is in the UK, applying the equivalent of up to 9.3 kg ai/ha in 20,000–40,000 L/ha as a pre-emergence nursery soil drench and as a seedling drench, about 10–14 days later (before transplanting).

In twelve trials conducted in Europe, matching this cGAP, total residues in cauliflower heads at maturity (84–216 days after the last application) were < 0.2 (12) mg/kg.

Noting that the GAP in UK covers all commodities in the Flowerhead brassicas sub-group, the Meeting estimated a maximum residue level of 0.2 (*) mg/kg and an STMR of 0.2 mg/kg for the Subgroup of Flowerhead Brassicas.

Kale – fosetyl

The critical GAP for kale is in UK, applying the equivalent of up to 9.3 kg ai/ha in 20,000-40,000 L/ha as a pre-emergence nursery soil drench and as a seedling drench, about 10–14 days later (before transplanting).

In four trials conducted in Europe, matching this cGAP, total residues in curly kale leaves at maturity (76-160 days after the last application) were < 0.2 (4) mg/kg.

The Meeting estimated a maximum residue level of 0.2 (*) mg/kg and an STMR of 0.2 mg/kg for kale.

Coffee – fosetyl-Al

The critical GAP for fosetyl-Al on coffee is in Brazil, up to 2 foliar applications of 1.6 kg ai/ha, applied about 30 days apart, with a PHI of 30 days.

In trials conducted in Brazil and matching this GAP, total residues in dry coffee beans were 8.2, 8.7, 8.8, 8.8 and 8.9 mg/kg.

The Meeting estimated a maximum residue level of 30 mg/kg and an STMR of 8.8 mg/kg for coffee beans.

Fate of residues during processing

Pineapple bran – fosetyl-Al

In supervised trials conducted in the USA with fosetyl-Al on pineapples and involving dip+foliar applications, total residues were measured in whole pineapple fruit and in the dry bran remaining after

processing. Processing factors derived from these trials were 2.2, 2.4, 2.8, 2.8, 3.0, 3.2, 3.2, 3.2, 3.7, 3.9 and 6.1. The median processing factor is 3.2.

Based on this processing factor (3.2) and the median residue in whole pineapple fruit (3.6 mg/kg), the Meeting estimated a median total residue of 11.5 mg/kg for dry bran (87% dry matter).

Residues in animal commodities

Estimated maximum and mean dietary burdens of livestock

The Meeting revised the 2017 JMPR livestock dietary burden of fosetyl, phosphonic acid and their salts (expressed as phosphonic acid) in farm animals to include the additional feed items considered at this meeting (pineapple process waste – as dry bran, cabbage and kale leaves) on the basis of the diets (US/CAN, EU, Australia and Japan) listed in OECD Feed Table.

	Animal dietary burden, ppm of dry matter diet							
	US-Canada		EU		Australia		Japan	
	Max	Mean	Max	Mean	Max	Mean	Max	Mean
Beef cattle	1.6	1.6	7.8	7.8	35❶	35❸	-	-
Dairy cattle	3.8	3.8	5.7	5.7	32❷	32❹	-	-
Poultry – broiler	-	-	-	-	-	-	-	-
Poultry – layer	-	-	0.067	0.067	-	-	-	-

❶ Highest maximum beef or dairy cattle dietary burden suitable for MRL estimates for mammalian tissues

❷ Highest maximum dairy cattle dietary burden suitable for MRL estimates for mammalian milk

❸Highest mean beef or dairy cattle dietary burden suitable for STMR estimates for mammalian tissues.

❹ Highest mean dairy cattle dietary burden suitable for STMR estimates for milk.

Animal commodity maximum residue levels

Cattle

The Meeting noted that in the second cattle feeding study reviewed by the 2017 JMPR, animals in the 32 ppm dose group contained mean estimated total residues of 0.29 mg/kg (kidney), 0.22 mg/kg (liver), 0.12 mg/kg (fat), 0.07 mg/kg (muscle) and 0.05 mg/kg in milk and maximum residues were 0.3 mg/kg (kidney), 0.33 mg/kg (liver), 0.18 mg/kg (fat) and 0.089 mg/kg (muscle).

For maximum residue level estimation, the high total residues were calculated by extrapolating the maximum dietary burden (35 ppm) from the 32 ppm feeding level in the dairy cow feeding study and using the highest tissue concentrations of total residues in individual animals within the dose group.

The STMR values for the tissues were calculated by extrapolating the mean dietary burden (35 ppm) from the 32 ppm feeding level and using the mean tissue total residues from the dose group.

For milk, since both the mean and maximum diary cow dietary burdens were 32 ppm, MRL and STMR estimations were obtained directly from the mean total residues in the milk from animals in the 32 ppm dose group.

	Feed level for milk (ppm)	Residues in milk (mg/kg)	Feed level for tissues (ppm)	Residues (mg/kg)			
				Muscle	Liver	Kidney	Fat
MRL beef or dairy cattle							
Feeding study	32	0.05	32	0.089	0.33	0.3	0.18
Dietary burden/residue estimate	32❷	0.05	35❶	0.097	0.36	0.33	0.2

	Feed level for milk (ppm)	Residues in milk (mg/kg)	Feed level for tissues (ppm)	Residues (mg/kg)			
				Muscle	Liver	Kidney	Fat
STMR beef or dairy cattle							
Feeding study	32	0.05	32	0.07	0.22	0.29	0.12
Dietary burden/residue estimate	32❹	0.05	35❸	0.077	0.24	0.32	0.13

❶ Highest maximum beef or dairy cattle dietary burden suitable for MRL estimates for mammalian tissues

❷ Highest maximum dairy cattle dietary burden suitable for MRL estimates for mammalian milk

❸Highest mean beef or dairy cattle dietary burden suitable for STMR estimates for mammalian tissues.

❹ Highest mean dairy cattle dietary burden suitable for STMR estimates for milk.

The Meeting agreed that the maximum residue levels estimated by the 2017 JMPR for meat from mammals other than marine mammals (0.15 mg/kg), for edible offal, mammalian (0.5 mg/kg) and milks (0.1 mg/kg) were sufficient to accommodate the revised maximum cattle dietary burden, but estimated a higher maximum residue level of 0.3 mg/kg for mammalian fat to replace the previous recommendation of 0.2 mg/kg.

Estimated STMRs are 0.32 mg/kg (kidney), 0.24 mg/kg (liver), 0.13 mg/kg (fat), 0.077 mg/kg (muscle) and 0.05 mg/kg for milks.

Poultry

The Meeting noted that the poultry (layer) dietary burden of 0.067 ppm was based on the consumption of cabbage or kale leaves containing residues <LOQ. Since this dietary burden was about 200-fold lower than the lowest feeding level in the poultry feeding study (14 ppm), where total residues were not detected in any tissues and present at trace levels in eggs, the Meeting estimated maximum residue levels of 0.05 (*) mg/kg for poultry meat, poultry fat, poultry edible offal and eggs.

Estimated STMRs are 0 mg/kg for poultry meat, poultry fat, poultry edible offal and 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 assessment.

Definition of the residue for compliance with the MRL and dietary risk assessment for plant commodities: *Sum of fosetyl, phosphonic acid and their salts, expressed as phosphonic acid*.

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

The residue is not fat-soluble

CCN	Commodity	Recommended maximum residue level (mg/kg)		STMR or STMR-P (mg/kg)	HR or HR-P (mg/kg)	Source
		New	Previous			
FB 0264	Blackberries	70		5.95		Fosetyl-Al
FI 0341	Kiwifruit	150		34.5		Fosetyl-Al
FI 0353	Pineapple	15		2.35		Fosetyl-Al
VB 2036	Head Brassicas (sub-group)	0.2 (*)		0.2		Fosetyl
VB 0042	Flowerhead Brassicas (sub-group)	0.2 (*)		0.2		Fosetyl
VL 0480	Kale	0.2 (*)		0.2		Fosetyl
SB 0716	Coffee beans	30		8.8		Fosetyl-Al
MF 0100	Mammalian fat (except milk fats)	0.3	0.2	0.13		

CCN	Commodity	Recommended maximum residue level (mg/kg)		STMR or STMR-P (mg/kg)	HR or HR-P (mg/kg)	Source
		New	Previous			
PM 0110	Poultry meat	0.05 (*)		0		
PO 0111	Poultry, Edible offal of	0.05 (*)		0		
PF 0111	Poultry fat	0.05 (*)		0		
PE 0112	Eggs	0.05 (*)		0		
MM 0105	Edible offal (mammalian)			kidney: 0.32 liver: 0.24		
MM 0095	Meat (from mammals other than marine mammals)			fat: 0.13 muscle: 0.077		

Additional values used in estimating livestock dietary burdens

Codex classification	Commodity	Median residue (-P) (mg/kg)	Highest residue (-P) (mg/kg)	Source
-	Pineapple bran	11.5		Fosetyl-Al

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for fosetyl-aluminium is 0–1 mg/kg bw and this ADI also applies directly to phosphonic acid. The International Estimated Daily Intakes (IEDIs) for fosetyl-aluminium plus phosphonic acid 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 2019 Extra JMPR Report.

The IEDIs ranged from 1–30% of the maximum ADI. The Meeting concluded that long-term dietary exposure to residues of fosetyl-aluminium and phosphonic acid from uses considered by the JMPR is unlikely to present a public health concern.

Acute dietary exposure

The 2017 JMPR decided that an ARfD for fosetyl-aluminium and for phosphonic acid was unnecessary. The Meeting therefore concluded that the acute dietary exposure to residues of fosetyl-aluminium plus phosphonic acid from the uses considered, is unlikely to present a public health concern.

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C025403	Gloeckner, M.	2002	Residues at harvest in kiwi European Union (southern zone) 2001 Fosetyl-aluminium water dispersible granule (WG) 80%. Bayer CropScience GmbH, Frankfurt am Main, Germany. Report No.: C025403. Edition Number: M-209282-01-1. Date: 2002-08-22. GLP/GEP: Yes, unpublished	M-209282-01-1

Reference	Author(s)	Year	Title	Edition No
F14-019	Volpi, R.	2015	Adendo 01 ao relatório final - Determinação de resíduos de fosetyl na cultura de café após aplicação de Aliette em ensaios no Brasil. Bayer S.A., Bayer CropScience, São Paulo, SP, Brazil. Bayer Report No.: F14-019. Report includes Trial Nos : F14-019-01. F14-019-02. F14-019-03. F14-019-04. F14-019-05. Edition Number: M-519556-02-1. Date: 2015-04-29. ... amended: 2015-09-03. GLP/GEP: Yes, unpublished	M-519556-02-1
F15-015	Volpi, R.	2015	Determinação de resíduos de Ácido Fosfônico (metabólito do fosetyl) nas culturas de maçã, uva, citros, café e abacaxi após aplicação de Aliette em ensaios no Brasil. Bayer S.A., Bayer CropScience, São Paulo, SP, Brazil. Bayer. Report No.: F15-015. Edition Number: M-540486-01-1. Date: 2015-11-23. GLP/GEP: Yes, unpublished.	M-540486-01-1
P 2749 G	Anon.	2013	FEA+FLC; WG 71.11; blackberry ; Germany; BBA. PTRL Europe GmbH, Ulm, Germany. Bayer Report No.: P 2749 G. Report includes Trial Nos : P 2749 G-T1223. P 2749 G-T1224. P 2749 G-T1225. P 2749 G-T1227. Edition Number: M-617957-01-1. Date: 2013-04-11. GLP/GEP: No, unpublished	M-617957-01-1
P 2093 G	Wilde, N.	2011	Determination of residues of fluopicolide and its metabolite BAM and fosetyl-Al and its metabolite phosphonic acid in/on blackberry: Minor crop study 2010. PTRL Europe GmbH, Ulm, Germany. Bayer Report No.: P 2093 G. Edition Number: M-431464-01-1. Date: 2011-02-02. GLP/GEP: Yes, unpublished.	M-431464-01-1
P 2093 G	Anon.	2011	FEA+FLC; WG 71.11; blackberry; Germany; BBA. PTRL Europe GmbH, Ulm, Germany. Bayer Report No.: P 2093 G. Report includes Trial Nos : P 2093 G-1013. P 2093 G-1015. Edition Number: 603059-01-1. Date: 2011-02-02. GLP/GEP: No, unpublished	M-603059-01-1
P 2749 G	Bacher, R.	2013	Determination of residues of fluopicolide and its metabolites BAM and PCA and fosetyl and its metabolite phosphonic acid in/on blackberry, indoor and outdoor: Minor crop study 2012. PTRL Europe GmbH, Ulm, Germany. LW TechnZentr Augustenberg Report No.: P 2749 G. Edition Number: M-465390-01-1. Date: 2013-04-11. GLP/GEP: Yes, unpublished.	M-465390-01-1
JAOAC 86(4) 2003	Hernández, F.; Sancho, J.V.; Pozo, Ó.J.; Villaplana, C.; Ibáñez, M.; Grimalt, S.J	2003	Rapid Determination of Fosetyl-Aluminum Residues in Lettuce by Liquid Chromatography/Electrospray Tandem Mass Spectrometry. Journal of AOAC International, Volume 86, Number 4, July 2003, pp. 832-838(7). Published	M-431469-01-1
OG/11-2-3	Anon.	2012	FEA+FLC; WG 71.11; blackberry; Germany; BBA. Institut fuer Veterinaer-Pharmakologie u.Toxikologie (IVPT GmbH), Bernau, Germany. Bayer Field Report for Trial Nos : RU 1113. RU 1115. RU 1116. Edition Number: M-617958-01-1. Date: 2012-04-27. GLP/GEP: No, unpublished	M-617958-01-1
OG/11-2-3	Klose, J.	2012	Determination of Profiler (fluopicolide, fosetyl and its metabolite) in blackberries and blackberries (under glass). Institut fuer Veterinaer-Pharmakologie u.Toxikologie (IVPT GmbH), Bernau, Germany. Bayer Report No.: OG/11-2-3. Edition Number: M-431469-01-1. Date: 2012-04-27. GLP/GEP: Yes, unpublished.	M-431469-01-1
12-2022	Fargeix, G.	2013	Determination of the residues of fosetyl and propamocarb in/on cauliflower after drench application of Fosetyl & Propamocarb SL 840 in the greenhouse in Italy and Spain. Bayer S.A.S., Bayer CropScience, Lyon, France. Bayer Report No.: 12-2022. Report includes Trial Nos : 12-2022-01. 12-2022-02. Edition Number: M-472793-01-1. Date: 2013-12-17. GLP/GEP: Yes, unpublished	M-472793-01-1
09-2025	Uceda, L.	2010	Determination of the residues of fosetyl and propamocarb in/on cabbage, red and cabbage, white after drench of fosetyl & propamocarb SL 840 in the greenhouse in Italy and Spain. Bayer S.A.S., Bayer CropScience, Lyon, France. Bayer Report No.: 09-2025. Report includes Trial Nos : 09-2025-01. 09-2025-02. Edition Number: M-398169-01-1. Date: 2010-12-17. GLP/GEP: Yes, unpublished	M-398169-01-1

Reference	Author(s)	Year	Title	Edition No
10-2049	Uceda, L.; Meilland-Berthier, I.	2011	Determination of the residues of fosetyl and propamocarb in/on white cabbage after drench application of fosetyl & propamocarb SL 840 in the greenhouse in Northern France and the Netherlands. Bayer S.A.S., Bayer CropScience, Lyon, France. Bayer Report No.: 10-2049. Report includes Trial Nos : 10-2049-01. 10-2049-02. Edition Number: M-412638-01-1. Date: 2011-08-23. GLP/GEP: Yes, unpublished	M-412638-01-1
08-2104	Melrose, I.	2010	Determination of the residues of fosetyl-AL in/on kiwi after spraying of fosetyl-AL WG 80 in the field in France (south) and Italy. Bayer S.A.S., Bayer CropScience, Lyon, France. Bayer Report No.: 08-2104. Report includes Trial Nos : 08-2104-01. 08-2104-02. 08-2104-03. Edition Number: M-363735-01-1. Date: 2010-02-19. GLP/GEP: Yes, unpublished	M-363735-01-1
08-2154	Melrose, I.; Portet, M.	2010	Determination of the residues of fosetyl and propamocarb in/on cabbage, white after drench of fosetyl & propamocarb SL 840 in the greenhouse in Belgium, France (north), Netherlands and United Kingdom. Bayer S.A.S., Bayer CropScience, Lyon, France. Bayer Report No.: 08-2154. Report includes Trial Nos : 08-2154-05. 08-2154-06. 08-2154-07. 08-2154-08. Edition Number: M-348463-03-1. Date: 2009-06-02. ... amended: 2010-07-07. GLP/GEP: Yes, unpublished	M-348463-03-1
08-2155	Melrose, I.; Portet, M.	2009	Determination of the residues of fosetyl and propamocarb in/on kale, curly after drench of fosetyl & propamocarb SL 840 in the greenhouse in Belgium and France (North). Bayer CropScience S.A., Lyon, France. Bayer Report No.: 08-2155. Report includes Trial Nos : 08-2155-05. 08-2155-06. Edition Number: M-356365-01-1. Date: 2009-09-21. GLP/GEP: Yes, unpublished	M-356365-01-1
08-2156	Melrose, I.; Portet, M.	2009	Determination of the residues of fosetyl and propamocarb in/on cauliflower after drench of fosetyl & propamocarb SL 840 in the greenhouse in Belgium, France (North), Netherlands and United Kingdom. Bayer CropScience S.A., Lyon, France. Bayer Report No.: 08-2156. Report includes Trial Nos : 08-2156-01. 08-2156-09. 08-2156-10. 08-2156-11. 08-2156-12. 08-2156-13. 08-2156-15. Edition Number: M-348803-01-1. Date: 2009-06-05. GLP/GEP: Yes, unpublished	M-348803-01-1
08-2182	Melrose, I.; Portet, M.	2009	Determination of the residues of fosetyl and propamocarb in/on cabbage, white after drench of fosetyl & propamocarb SL 840 in the greenhouse in Italy and Spain. Bayer CropScience S.A., Lyon, France. Bayer Report No.: 08-2182. Report includes Trial Nos : 08-2182-05. 08-2182-06. Edition Number: M-356351-01-1. Date: 2009-09-22. GLP/GEP: Yes, unpublished	M-356351-01-1
08-2183	Melrose, I.; Portet, M.	2009	Determination of the residues of fosetyl and propamocarb in/on cauliflower after drench of fosetyl & propamocarb SL 840 in the greenhouse in Italy and Spain. Bayer CropScience S.A., Lyon, France. Bayer Report No.: 08-2183. Report includes Trial Nos : 08-2183-05. 08-2183-06. Edition Number: M-356341-01-1. Date: 2009-09-23. GLP/GEP: Yes, unpublished	M-356341-01-1
08-2184	Melrose, I.	2010	Determination of the residues of fosetyl and propamocarb in/on kale, curly after drench of fosetyl & propamocarb SL 840 in the greenhouse in Italy and Spain. Bayer S.A.S., Bayer CropScience, Lyon, France. Bayer Report No.: 08-2184. Report includes Trial Nos : 08-2184-02. 08-2184-03. 08-2184-04. Edition Number: M-356497-02-1. Date: 2009-09-25. . amended: 2010-06-29. GLP/GEP: Yes, unpublished	M-356497-02-1

GLYPHOSATE (158)

First draft prepared by Ms G Y Zhu, Ministry of Agriculture and Rural Affairs, Beijing, Republic of China

EXPLANATION

Glyphosate is a widely used non-selective herbicide. Glyphosate was first evaluated for toxicology and residues by the JMPR in 1986. It was further evaluated for residues on multiple occasions by the JMPR including a periodic review of residues in 2005.

The toxicology of glyphosate was re-evaluated by the 2011 JMPR which established a group ADI of 0–1 mg/kg bw for the sum of glyphosate, N-acetyl glyphosate, AMPA and N-acetyl-AMPA. The same Meeting confirmed that an ARfD was unnecessary.

Definition of the residue for compliance with MRL (for plant commodities): for soya bean, maize and rape: *sum of glyphosate and N-acetyl-glyphosate, expressed as glyphosate*, and for other crops - *glyphosate*.

Definition of the residue for compliance with MRL for animal commodities: *sum of glyphosate and N-acetyl-glyphosate, expressed as glyphosate*.

The residue definition for estimation of dietary exposure for plant and animal commodities: *glyphosate, N-acetyl-glyphosate, AMPA and N-acetyl AMPA, expressed as glyphosate*.

The residue is not fat soluble.

Glyphosate was scheduled at the Fiftieth Session of the CCPR for the evaluation of additional uses by the 2019 Extra JMPR. The current Meeting received information on analytical methods for lentil, storage stability, use patterns and supervised residue trials on conventional varieties of lentil, bean dry and tree nuts.

RESIDUE ANALYSIS

Analytical methods

The current Meeting received several concurrent method validation tests for confirming the method performance. Two analytical methods were used in trials: ME-1466-3 (2016) applied in lentils, and Method 2, reviewed by the 2005 JMPR, in tree nut, pea dry and beans dry.

In Method 2, the samples were analysed by HPLC-FLD. The LOQs were 0.05 mg/kg for both residues of glyphosate and AMPA in most plant matrices.

In method ME-1466-3, the milled matrix was weighed into 96-well tubes followed by the addition of a 0.1% formic acid solution containing both glyphosate and AMPA stable isotope labeled internal standards. The samples are capped and agitated on a high-speed shaker for extraction then centrifuged. Place plate on centrifuge and spin to clear suspended materials from the liquid column and form a solid pellet (e.g., 10 minutes at 6000 G). An aliquot of the extract is then transferred to a new 96-well plate for analysis by LC-MS/MS, using a cation exchange column and with electrospray ionization. The working range of the method without sample dilution is from 0.03 to 6.0 mg/kg, with LOQs of 0.05 mg/kg and RSDs of 0.8–4.9% for both glyphosate and AMPA.

Average recoveries at several fortification levels in the trials generally fell within the 80–120% range, and with relative standard deviations less than 10%. Information on the validation recovery rates in different commoditieis summarized below.

Table 1 Glyphosate and AMPA analytical validation recovery rate in method ME-1466-03.

Matrix	Method	Analyte (Precursor/Product Ions, m/z)	Fortification level (mg/kg)	n	Recovery (%) （Average）	RSD （%）	Reference
Soya bean	ME - 1466-03	Glyphosate (168/63) Quantitation	0.05 0.5	5	102.9-113.3 (107.1) 105.0-109.4 (108.3)	3.7 2.3	MSL0029625
		AMPA (110/63) Quantitation	0.05 0.5	5	86.3-97.5 (92.5) 96.4-98.6 (97.4)	4.7 0.9	
Canola	ME - 1466-03	Glyphosate (168/63) Quantitation	0.05 0.5	5	97.0-99.8 (98.5) 100.3-103.7 (101)	1.2 1.4	MSL0029625
		AMPA (110/63) Quantitation	0.05 0.5	5	80.0-95.8 (86.5) 93.6-97.4 (95.8)	7.2 1.6	
Soybean Oil	ME - 1466-03	Glyphosate (168/63) Quantitation	0.05 0.5	5 6	94, 99, 97, 91, 95, 97 (96) 101, 100, 99, 102, 104, 104 (102)	2.8 2.5	MSL0029625
		AMPA (110/63) Quantitation	0.05 0.5	5 6	97, 100, 94, 101, 98, 97 (98) 97, 97, 103, 100, 100, 95 (99)	2.5 2.8	
Corn Oil	ME-1466-03	Glyphosate (168/63) Quantitation	0.05 0.5	6 6	100, 95, 100, 105, 100, 100 (100) 100, 99, 103, 98, 103, 100 (101)	2.9 2.1	MSL0029625
		AMPA (110/63) Quantitation	0.05 0.5	6 6	99, 98, 99, 104, 100, 105 (101) 100, 102, 95, 103, 96, 100 (100)	2.8 3.2	
Canola Oil	ME-1466-03	Glyphosate (168/63) Quantitation	0.05 0.5	5 6	99, 95, 95, 98, 97, 104 (98) 99, 99, 100, 103, 102, 101 (101)	3.2 1.5	MSL0029625
		AMPA (110/63) Quantitation	0.05 0.5	5 6	101, 99, 102, 97, 94, 91 （97） 101, 96, 100, 98, 101, 98 （99）	4.4 1.8	
Corn Meal	ME-1466-03	Glyphosate (168/63) Quantitation	0.05 0.5	5	104, 99, 99, 95, 99, 97 (99) 96, 99, 96, 100, 100, 98 (98)	2.7 1.8	MSL0029625
		AMPA (110/63) Quantitation	0.05 0.5	5	98, 111, 101, 106, 92, 94 (101) 110, 102, 106, 98, 93, 107 (103)	7.4 6.0	
Corn Meal	ME-1466-03	Glyphosate (168/63) Quantitation	0.05 0.5	5 6	99, 96, 92, 96, 94, 96 (95) 99, 102, 97, 100, 101, 106 (101)	2.3 3.0	MSL0029625
		AMPA (110/63) Quantitation	0.05 0.5	5 6	111, 112, 103, 98, 108, 96 (105) 104, 96, 103, 102, 103, 102 (102)	6.2 2.8	
		AMPA (110/63) Quantitation	0.05 0.5	12 6	103, 92, 98, 95, 97, 103, 108, 103, 90, 106, 90, 95 (98) 106, 100, 93, 99, 93, 96 (98)	6.3 5.0	

USE PATTERNS

The Meeting received additional information on authorised uses on legume vegetables in the UK and the USA, and tree nuts in USA.

The national critical GAPs for these crops are summarized in the following table. Note that the application rates throughout this report are expressed in terms of glyphosate acid.

Table 2 Registered uses of glyphosate (water-soluble concentrate formulation)

Crop	Country	Application			Max application per season		PHI (days)	Comments
		Method	kg ai/ha (max)	Water L/ha	no	kg ai/ha		
Peas (dry), Lentils Chickpeas	USA	Pre-emergence	4.2	28 – 374				Do not graze or feed to livestock
		Pre-harvest	2.5	93-187	1		7	
Beans (dry)	USA	Pre-emergence	4.2	28-374				Do not graze or feed to livestock
		Pre-harvest	0.87	28-187	1		7	
Beans (field)	UK	Pre-emergence	0.54		1			
		Pre-harvest	1.44	80-250	1		7	
Tree nuts	USA	Directed*	4.2	28-234		8.8	3	14 day PHI for coconut
		Broadcast	1.7	28-234			21	Suppression of grasses.

* Directed spray between and within rows.

Label for tree nuts covers: Almond; Beechnut; Betelnut; Brazil nut; Butternut; Cashew; Chestnut; Chinquapin; Coconut; Filbert (hazelnut); Hickory nut; Macadamia; Pecan; Pine nut; Pistachio; Walnut (black, English)

RESULTS OF SUPERVISED RESIDUE TRIALS ON CROPS

The Meeting received information on supervised field trials involving foliar treatments of glyphosate to lentil, peas dry, beans dry, almond, pecan, and walnut.

Group		Crop	Countries	Table no
015B	Subgroup of dry peas	Lentil (dry)	Canada and USA	3
015A	Subgroup of dry beans	Beans (dry)	USA	4
022	Tree nuts	Almond	USA	5
022	Tree nuts	Pecan	USA.	6
022	Tree nuts	Walnut	USA	6

Results from replicated field plots are listed and mean values are calculated. The results from trials used for the estimation of maximum residue levels (underlined) have been rounded to two significant digits. Residue values were selected for estimating maximum residue levels and for dietary exposure assessment at longer PHI instead of that at the GAP, if those values were found to be higher. The highest residue was selected from trials which were considered to be not independent.

Pulses

Lentil

In eleven lentil trials, two applications of glyphosate (SL) were applied, the first as a pre-emergence application and the second as a pre-harvest application. Samples of seed were stored frozen for up to 5 months before analysis using method ME 1466-03. Concurrent recovery rates in samples spiked with 0.05-20 mg/kg glyphosate or AMPA ranged from 96 -111% (glyphosate) and 93 -113% (AMPA), and the LOQ for both analytes was 0.05 mg/kg.

Table 3 Residues in lentils from supervised trials in Canada and the USA in 2011 involving one pre-emergence and one pre-harvest application of glyphosate (SL formulation).

LENTILS Location (Variety)	Application				Growth Stage	Matrix	DALA	Residues (mg/kg)			Reference & Comments
	N	kg ai/ha		water (L/ha)				glyphosate (mean)	AMPA (mean)	Total	
USA GAP: 1×4.2 kg ai/ha pre-emergence and 1×2.5kg ai/ha pre-harvest, PHI 7 days											
Canada, 2011 Carberry, MB (CDC Imax)	1+ 12	4.25 2.44	4.78 2.77	89 88	pre-emergence BBCH 85–87	Seed	7	2.04 1.96 (2.0)	<0.05 <0.05 (<0.05)	2.0	MSL0029625 Trial-12MB
Canada,2011 Dundurn,SK (CDC Maxim)	1+2 1	4.32 2.51	4.73 2.78	91.4 90.4	pre-emergence BBCH 85–86	Seed	7	0.44 0.39 (0.41)	<0.05 <0.05 (<0.05)	0.41	MSL0029625 Trial-03SK
Canada,2011 Hanley, SK (CDC Maxim)	1+ 1 2	4.20 2.50	4.72 2.78	89 90	pre-emergence BBCH 87	Seed	7	5.04 5.54 (5.3)	<0.05 <0.05 (<0.05)	5.3	MSL0029625 Trial-04SK
Canada,2011 Kenaston, SK (CDC Maxim)	1+ 1 1+ 1	4.25 2.53	4.72 2.78	90 91	pre-emergence BBCH 87–88	Seed	7	2.33 2.43 1.23 1.30 (1.8)	<0.05 <0.05 <0.05 <0.05 (<0.05)	1.8	MSL0029625 Trial-05SK
Canada,2011 Delisle, SK (CDC Maxim)	1+ 1	4.28 2.40	4.70 2.79	91 86	BBCH 80	Seed	7	0.53 0.23 (0.37)	<0.05 <0.05 (<0.05)	0.37	MSL0029625 Trial-06SK
Canada,2011 Harris, ID (CDC Maxim)	1+ 1	4.35 2.56	4.73 2.78	92 92	pre-emergence BBCH 82	Seed	7	1.08 0.72 (0.90)	<0.05 <0.05 (<0.05)	0.90	MSL0029625 Trial-
Canada,2011 Alvena, SK (CDC Maxim)	1+ 1	4.38 2.38	4.28 2.50	102.4 95.2	pre-emergence BBCH 81	Seed	7	3.70 4.08 1.05 2.77 (2.9)	<0.05 <0.05 <0.05 <0.05 (<0.05)	2.9	MSL0029625 Trial-11SK
USA,2011 Velva, ND (CDC Impala)	1+ 1	4.28 2.53	4.73 2.66	91.4 95.1	pre-emergence R6	Seed	7	5.29 7.26 (6.3)	<0.05 0.06 (0.05)	6.4	MSL0029625 Trial-02ND
USA,2011 Payette, ID (Crimson)	1+ 1	4.42 2.55	4.62 2.68	95.6 95.2	pre-emergence 70% of pods ripe (hard)	Seed	7	2.07 1.70 (1.9)	<0.05 <0.05 (<0.05)	1. 9	MSL0029625 Trial-08ID
USA,2011 Jerome, ID (small browns)	1+ 1	4.26 2.52	4.89 2.93	87.1 85.9	pre-emergence BBCH 88	Seed	7	1.95 1.68 1.21 1.50 (1.6)	<0.05 <0.05 <0.05 <0.05 (<0.05)	1.6	MSL0029625 Trial-09ID
USA,2011 Ephrata, WA (Pardina)	1+ 1	4.28 2.51	6.14 3.57	69.7 70.4	pre-emergence BBCH 88	Seed	7	0.39 0.40 3.38 0.94 (1.3)	<0.05 <0.05 <0.05 <0.05 (<0.05)	1.3	MSL0029625 Trial-10WA

The results from 5 (previously submitted) supervised trials on peas dry in the USA were provided to the Meeting.

Peas dry

In peas dry, the Meeting did not receive new data. In data previously evaluated by the 2011 JMPR glyphosate residues (glyphosate only) in peas dry treated with one pre-emergence application of ca. 2.5 kg/ha and one pre-harvest application of ca. 2.5 kg ai/ha glyphosate (SL) with a 7-day PHI were (n=5): 0.70, 0.77, 1.1, 3.4, and 4.2 mg/kg. .

In five trials on dry peas evaluated by the 2011 JMPR, two applications of glyphosate (SL) were applied, the first as a pre-emergence application and the second as a pre-harvest application. Samples of pea seed were stored frozen for up to 7 months before analysis using Method 2. Concurrent recovery rates in samples spiked with 0.05-10 mg/kg glyphosate ranged from 85–118% (glyphosate) and the LOQ was 0.05 mg/kg.

Table 5 Residues in peas dry from supervised trials in the USA in 1998 involving one pre-emergence and one pre-harvest application of glyphosate (SL formulation)

Trial, Location State; country, year (variety)	Form (g ae/L)[a]	No.[b]	Interval (d)	kg ae/ha[a]	kg ae/hL[a]	date of last treatment, timing	PHI (days)	residues, mg/kg[c] glyphosate	Reference
WA*35, Prosser, Washington, USA, 1998 (Columbian)	SL 360	1 + 1	91	2.45 2.52	1.06 1.81	July 13, 80-85% mature pods, crop height 80-90 cm	7 7 7 13 13 13 21 21 21	0.66 0.73 0.70 0.98 1.1 1.0 1.0 1.2 1.1	IR-4 PR No. A6139 Volume 2 of 2
WA*36, Prosser, Washington, USA, 1998 (Columbian)	SL 360	1 + 1	91	2.49 2.48	1.06 1.80	July 13, 80-85% mature pods, crop height 80-90 cm	7 7 7	0.59 0.81 0.70	IR-4 PR No. A6139 Volume 2 of 2
WA*37, Prosser, Washington, USA, 1998 (Columbian)	SL 360	1 + 1	91	2.54 2.48	1.06 1.80	July 13, 80-85% mature pods, crop height 80-90 cm	7 7 7	0.74 0.80 0.77	IR-4 PR No. A6139 Volume 2 of 2
ND07, Fargo, North Dakota, USA, 1998 (Profi)	SL 360	1 + 1	83	2.42 2.63	2.27 2.28	July 21, mature 85% yellow pods, crop height 80-90 cm	7 7 7 14 14 14 21 21 21	3.6 3.3 3.4 2.9 3.0 3.0 2.8 3.7 3.3	IR-4 PR No. A6139 Volume 2 of 2
ND25, Carrington, North Dakota, USA, 1998 (Grande)	SL 360	1[d]	na[d]	2.48	1.52	August 7, 80% commercially mature, crop height 80-90 cm	7 7 7	6.1[e] 2.2[f] 4.2	IR-4 PR No. A6139 Volume 2 of 2

[a] The active ingredient and all residues are reported as glyphosate free acid equivalents (ae).

[b] The number of applications includes the pre-emergence applications + the post emergence applications as x + y, respectively.

[c] Individual replicate values are shown followed by average of replicates in bold font.

[d] Trial ND25 was performed without the pre-plant soil application

[e] Average of triplicate analysis of single field sample.

[f] Average of duplicate analysis of single field sample.

[Barney, 2005, IR-4 PR No. A6139]. No unusual weather conditions. Treated plot size 31-223 m^2. ATV mounted spray boom with spray volume 107-240 l/ha. Plants were swathed with sickle mower, windrowed and allowed to dry in the field for two days. Plants were collected and trashed. Seed were run through seed clipper. Seeds (10-35 unit not given) were sampled at harvest (BBCH not stated).

Samples were stored frozen for a maximum of 221 days. Samples were analysed using a Chelex® 100 resin extraction followed by HPLC analysis with o-phthalaldehyde (OPA) post column reactor with fluorescence detector. Individual recoveries seed were 85-118%.

Subgroup of dry beans

The results from 13 supervised trials on dry beans in the USA were provided to the Meeting.

Beans dry

In thirteen dry beans trials, two applications of glyphosate (SL) were applied, the first as a pre-emergence application and the second as a pre-harvest application. Samples of seed were stored frozen for up to 6 months before analysis using Method 2. Concurrent recovery rates in samples spiked with 0.05–10 mg/kg glyphosate or AMPA ranged from 85–103% (glyphosate) and 64–98% (AMPA), and the LOQs for both analytes were 0.05 mg/kg.

Table 4 Residues in bean dry from supervised trials in the USA in 2001 involving one pre-emergence and one pre-harvest application of glyphosate (SL formulation)

DRY BEAN Country, year Location (Variety)	Application				Growth Stage	Matrix	DAT	Residues (mg/kg)			Reference & Comments
	N	kg ai/ha	kg ai/hL	water (L/ha)				Glyphosate (mean)	AMPA (mean)	Total (mean)	
USA GAP: 1×4.2 kg ai/ha pre-emergence and 1×2.5kg ai/ha pre-harvest, PHI 7 days											
USA, 2001 Wayne County, NY (Kidney) (Montcalm)	1+ 1	4.20 1.71	4.49 1.79	93 95	Pre-emergence Mature pods	Beans	7	0.19	<0.05	0.19	MSL17194
USA, 2001 Kent County, MI (Cranberry)	1+ 1	4.20 1.68	4.87 2.08	86 81	Pre-emergence Mature pods	Beans	7	<0.05	<0.05	<0.1	MSL17194 Trial- MI-1
USA, 2001 Ottawa County, MI (Navy Avanti)	1+ 1	4.20 1.66	5.13 2.03	82 82	Pre-emergence Mature pods	Beans	7	0.21	<0.05	0.21	MSL17194 Trial- MI-2
USA, 2001, Freeborn County ,MN (Navy Norstar)	1+ 1	4.28 1.66	4.05 1.65	106 101	Pre-emergence Mature podsR8	Beans	7	0.19	<0.05	0.19	Report: MSL17194
USA, 2001 York County, NE (Navy Great Northern)	1+ 1	4.17 1.65	3.94 1.56	106 106	Pre-emergence Mature pods 7days prior to maturity	Beans	1 2 7 13 20	0.96 0.43 0.52 1.75 1.65	<0.05 <0.05 <0.05 <0.05 <0.05	0.96 0.43 0.52 1.75 1.65	MSL17194 Trial- NE-1
USA, 2001 Hall County, NE (Navy Great Northern)	1+ 1	4.23 1.68	3.96 1.64	107 103	Pre-emergence 80% Maturity	Beans	7	10.5	0.12	10.7	MSL17194 Trial- NE-2
USA, 2001 Foster County, ND (Pinto Maverick)	1+ 1	4.21 1.68	5.34 1.60	79 105	Pre-emergence R8, 80% Maturity	Beans	7	0.53	<0.05	0.53	MSL17194 Trial- ND-1
USA, 2001 Eddy County, ND (Pinto Othello)	1+ 1	4.21 1.65	5.41 1.59	78 104	Pre-emergence R8, 80% Maturity pods	Beans	7	0.63	<0.05	0.63	MSL17194 Trial- ND-2
USA, 2001 McHenry County, ND (Pinto Othello)	1+ 1	4.23 1.67	5.29 1.59	80 105	Pre-emergence R8, 80% Maturity pods	Beans	7	0.32	<0.05	0.32	MSL17194 Trial- ND-3

DRY BEAN Country, year Location (Variety)	Application				Growth Stage	Matrix	DAT	Residues (mg/kg)			Reference & Comments
	N	kg ai/ha	kg ai/hL	water (L/ha)				Glyphosate (mean)	AMPA (mean)	Total (mean)	
USA , 2001 Weld County, CO (Pinto Montrose)	1+ 1	4.28 1.68	4.04 1.64	106 103	Pre-emergence Mature pods	Beans	7	2.6	<0.05	2.6	MSL17194
USA, 2001 Cache County, UT (Pinto Montrose)	1+ 1	4.18 1.73	4.48 1.89	93 91	Pre-emergence Mature pods	Beans	7	0.20	<0.05	0.20	MSL17194
USA, 2001 Tulare County, CA (California Blackeye#5)	1+ 1	4.22 1.68	4.47 1.82	94 92	Pre-emergence Mature pods	Beans	7	0.80	<0.05	0.80	MSL17194
USA, 2001 Payette County, ID (Pinto Othello)	1+ 1	4.20 1.73	4.05 1.61	104 107	Pre-emergence Mature pods	Beans	7	0.06	<0.05	0.06	MSL17194

Tree nuts

The results from 11 trials on tree nuts (previously submitted to the 2005 JMPR) conducted in the USA were provided to the Meeting.

Almond, pecan, and walnut

In five almond, three pecan, and three walnut trials, one application of glyphosate (SL) was applied as a directed spray between and within the tree rolls. Samples of tree nut were taken from the ground. The tree nuts were raked into piles and placed in plastic lined buckets for transport to the facility next door for separation into hull and nutmeat samples. The hull and nutmeat samples were placed in a freezer within 24 hours of sampling. Samples were stored frozen for up to 5 months before analysis using Method 2. Concurrent recovery rates in samples spiked with 0.05–1 mg/kg glyphosate or AMPA ranged from 65–112% (glyphosate) and 60–99% (AMPA), and the LOQs for both analytes were 0.05 mg/kg.

Table 5 Residues in tree nuts from supervised trials in the USA in 1989 involving one directed application of glyphosate (SL formulation)

TREE NUTS Country, year Location (Variety)	Application			Growth Stage	Residues (mg/kg) Matrix	DALA	Residues(mg/kg)			Reference & Comments
	no	kg ai/ha	water (L/ha)				Glyphosate	AMPA	Total	
GAP: 1×0.43 – 4.2 kg ai/ha, up to 8.8 kg ai/ha, PHI 3 days										
USA,1989 Fresno, California Almond (Mission)	1	8.91 3.81	280	Mature Trees	Nutmeats Hulls	3/10	0.10/0.07 17.6/7.2	<0.05/<0.05 0.06/0.06	0.1/0.07 17.7/7.3	Report: MSL 11022/11519
USA,1989 Hughson, CA, California Almond (Thompson)	1	8.91 7.43	120	Mature Trees	Nutmeats Hulls	3/10	<0.05/<0.05 0.7/0.8	<0.05/<0.05 0.06/0.06	<0.05/<0.05 0.8/0.9*	Report: MSL 11022/11519
USA,1989 Popular, CA, California Almond (Mission)	1	8.91 3.71	240	Mature Trees	Nutmeats Hulls	3/10	0.58/0.5 12.9/14.9	<0.05/<0.05 <0.05	0.58/0.5 12.9/14.9*	MSL 11022/11519
USA,1989 Porterville, CA Almond (Non Pareil)	1	8.91 3.43	260	Mature Trees	Nutmeats Hulls	3/10	<0.05/0.15 0.6/2.9	<0.05/<0.05 <0.05/0.08	<0.05/0.15* 0.6/3.0*	MSL 11022/11519
USA,1989 Turlock, CA Almond (Thompson)	1	8.91 6.85	130	Mature Trees	Nutmeats Hulls	3/10	0.07/0.05 2.6/1.5	<0.05/<0.05 <0.05/<0.05	0.07/0.05 2.6/1.5	MSL 11022/11519

TREE NUTS Country, year Location (Variety)	Application			Growth Stage	Residues (mg/kg) Matrix	DALA	Residues(mg/kg)			Reference & Comments
	no	kg ai/ha	water (L/ha)				Glyphosate	AMPA	Total	
USA,1989 Hawkinsville, GA Pecan (Stuart)	1	8.91 4.69	190	Mature Trees	Nutmeats	3/10	0.15/0.05	<0.05/<0.05	0.15/0.05	MSL 11022/11519
USA,1989 College Station, Station Pecan (Desirable)	1	8.91 4.69	190	Mature Trees	Nutmeats	3/10	<0.05/<0.05	<0.05/<0.05	<0.05/<0.05	MSL 11022/11519
USA,1989 Messilla, NM Pecan (Berton)	1	8.91 4.69	190	Mature Trees	Nutmeats	3/10	<0.05/<0.05	<0.05/<0.05	<0.05/<0.05	MSL 11022/11519
USA,1989 Fresno, CA Walnut (Franqutte)	1	8.91 3.18	280	Mature Trees	Nutmeats	3/10	0.06/<0.05	<0.05/<0.05	0.06/<0.05	MSL 11022/11519
USA,1989 Hughson, CA Walnut (Hartley)	1	8.91 7.43	120	Mature Trees	Nutmeats	3/10	0.69/0.08	<0.05<0.05	0.69/<0.08	MSL 11022/11519
USA,1989 Popular, CA Walnut (Franqutte)	1	8.91 3.71	240	Mature Trees	Nutmeats	3/10	0.45/0.20	<0.05/<0.05	0.45/0.20	MSL 11022/11519

*The residues at the 10-day PHI are higher.

APPRAISAL

Glyphosate is a widely used non-selective herbicide. Glyphosate was first evaluated for toxicology and residues by the JMPR in 1986. It was further evaluated for residues on multiple occasions by the JMPR including a periodic review of residues in 2005.

The 2011 JMPR established a group ADI of 0–1 mg/kg bw for the sum of glyphosate, N-acetyl glyphosate, AMPA and N-acetyl-AMPA. The same Meeting confirmed that an ARfD was unnecessary.

Definition of the residue for compliance with the MRL (for plant commodities): *for soya bean, maize and rape - sum of glyphosate and N-acetyl-glyphosate, expressed as glyphosate*; and for other crops - *glyphosate*.

Definition of the residue for compliance with the MRL (for animal commodities): *sum of glyphosate and N-acetyl-glyphosate, expressed as glyphosate*.

The residue definition for dietary risk assessment (for plant and animal commodities): *glyphosate, N-acetyl-glyphosate, AMPA and N-acetyl AMPA, expressed as glyphosate.*

The residue is not fat soluble.

Glyphosate was scheduled at the Fiftieth Session of the CCPR for the evaluation of additional uses at the Extra 2019 JMPR. The current Meeting received information on analytical method for lentil, storage stability, use pattern and supervised residue trials on conventional varieties of lentil, bean dry and tree nuts.

Methods of analysis

An HPLC-FLD analytical method used for determining residues of glyphosate and AMPA in pea dry, bean dry, and tree nuts was previously evaluated by the 2005 JMPR. A new analytical method for lentils along with validation data was received by the Meeting. The residues in lentil were extracted with a 0.1% formic acid solution, centrifuged and analysed by LC-MS/MS. The method was validated with LOQs of 0.05 mg/kg for both glyphosate and AMPA in lentils.

Storage stability of residues

In 2005, JMPR confirmed that the glyphosate residues were stable under frozen storage conditions (-20 °C) in/on the following commodities (storage interval in parentheses): beans, rape and linseed (at least 18 months), and soya bean seed (at least 6 months).

All samples in new residue trials were stored frozen for less than 5 months before extraction and analysis.

Results of supervised residue trials on crops

The Meeting received supervised trial data for foliar applications of glyphosate on lentils, bean dry, almond, pecan and walnut.

To calculate the sum of glyphosate and AMPA, expressed as parent equivalents (total residues), the Meeting used the approach agreed at the 2005 JMPR.

"When glyphosate and AMPA were summed, AMPA was converted to glyphosate equivalents (AMPA mg/kg × 1.523). All numerical figures for glyphosate application rates (kg ae/ha) or residue levels (mg/kg) are expressed as glyphosate acid equivalents (molecular weight 169 amu), and do not include any mass amounts for the salt cation (e.g., isopropylamine)."

"If AMPA residues are < 0.05 mg/kg, they are not summed with glyphosate, because they are typically much less than glyphosate residues. If both glyphosate and AMPA are < LOQ, then sum is < LOQ of glyphosate. The exception is where there is evidence that AMPA residues are comparable to glyphosate residues such as for soya beans in which case the residues are summed and if both glyphosate and AMPA residues are < LOQ, the sum is less than the combined LOQs for glyphosate and AMPA. "

The Meeting noted that soya bean is a representative crop for metabolism of pulses and decided extend this approach to pulses.

The table below describes how total residues were calculated for each trial.

Glyphosate (mg/kg)	AMPA (mg/kg)	Total (mg/kg)
<0.05	<0.05	<0.05
<0.05	<0.05	<0.1 (Pulses)
0.05	<0.05	0.05
0.05	0.05	0.13 (0.05+(0.05×1.523))

Dry peas, subgroup of

The critical GAP for dry peas, lentils and chickpeas in the USA is 2 applications of 4.2 kg ai/ha pre-emergence and 2.5 kg ai/ha pre-harvest with a PHI of 7 days.

Trials available for the current Meeting were conducted on lentils (4 from USA and 7 from Canada) approximating GAP in the USA.

Glyphosate residues were (n=11) 0.37, 0.41, 0.90, 1.3, 1.6, 1.8, 1.9, 2.0, 2.9, 5.3, and 6.3 mg/kg. Total residues from these 11 trials in ranked order were (n=11) 0.37, 0.41, 0.90, 1.3, 1.6, 1.8, 1.9, 2.0, 2. 9, 5.3, and 6.4 mg/kg (express as glyphosate).

In 2011, JMPR received five additional field trials on conventional peas (dry) performed in the USA in 1998, matching the US GAP. Glyphosate residues (glyphosate only) in seeds in rank of order

were (n=5): 0.70, 0.77, 1.1, 3.4, and 4.2 mg/kg at DALA 7 days. As the residue of AMPA was below 0.05 mg/kg even when glyphosate residue is 5.3 mg/kg, the Meeting concluded that the residue of AMPA in pea dry were below 0.05 mg/kg.

As the US GAP covers the subgroup of dry peas, the Meeting decided to recommend a maximum residue level for subgroup of dry peas. The data on lentils and peas, dry, were not significantly different according to the Mann-Whitney U test. The Meeting decided to combine the datasets.

Combined residues of glyphosate were: (n=16) 0.37, 0.41, 0.70, 0.77, 0.90, 1.1, 1.3, 1.6, 1.8, 1. 9, 2.0, 2.9, 3.4, 4.2, 5.3 and 6.3 mg/kg. The total residues were: (n=16) 0.37, 0.41, 0.70, 0.77, 0.90, 1.1, 1.3, 1.6, 1.8, 1.9, 2.0, 2.9, 3.4, 4.2, 5.3 and 6.4 mg/kg.

The Meeting estimated a maximum residue level for the subgroup of dry peas at 10 mg/kg, and an STMR at 1.7 mg/kg, and withdrew the previous maximum residue level recommendations for pea dry and lentil of 5 mg/kg.

Dry beans, except soya bean

The critical GAP for dry beans in the UK is one application at 1.44 kg ai/ha pre-harvest with a PHI of 7 days.

Thirteen trials in beans, dry were conducted in the USA at an application rate of 4.20 kg ai/ha pre-emergence and an application rate of 1.71 kg ai/ha pre-harvest with harvest 7 DALA. The Meeting considered that the pre-emergence applications did not contribute significantly to the residue level at harvest.

The data of the glyphosate residues in these trials were (n=13): <0.05, 0.06, 0.19 (2), 0.20, 0.21, 0.32, 0.53, 0.63, 0.80, 1.8, 2.6 and 10 mg/kg. The total residues of glyphosate residues were (n=13): <0.1, 0.06, 0.19(2), 0.20, 0.21, 0.32, 0.53, 0.63, 0.80, 1.8, 2.6 and 11 mg/kg.

The Meeting noted that dry bean is the representative commodity of subgroup of dry beans, and estimated a maximum residue level of 15 mg/kg and a STMR of 0.32 mg/kg for glyphosate on dry beans subgroup (except soya bean). The Meeting withdrew its previous recommendation of 2 mg/kg for beans, dry.

Tree nuts

The critical GAP for tree nuts in the USA is for one or more ground directed applications of 4.2 kg ai/ha up to a total seasonal rate of 8.8 kg ai/ha and a PHI of 3 days.

The 2005 JMPR received trial data for glyphosate on almond, pecan, and walnut from the USA, which included one directed application of 8.9 kg ai/ha with harvest 3 DALA. The residue trials submitted did not match the GAP.

The current Meeting did not receive new residue data. The Meeting concluded that the proportionality approach could not be applied to the available data, thus an estimate of a maximum residue level could not be performed.

Animalfeed commodities

The maximum dietary burdens calculated by the 2005 JMPR for cattle and poultry were 381 ppm for cattle and 22.7 ppm for poultry. The current Meeting calculated the additional contribution to the dietary burdens for cattle and poultry from the residues in pea dry and bean dry represented a minor portion (up to 0.79 ppm) of the dietary burdens calculated by the 2005 JMPR. The Meeting confirmed its previous recommendations for animal commodities.

RECOMMENDATIONS

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

Definition of the residue for compliance with the MRL (for plant commodities): for soya bean, maize and rape - sum of glyphosate and N-acetyl-glyphosate, expressed as glyphosate, and for other crops - glyphosate.

Definition of the residue for compliance with the MRL (for animal commodities): sum of glyphosate and N-acetyl-glyphosate, expressed as glyphosate.

Definition of the residue for dietary risk assessment (for plant and animal commodities): glyphosate, N-acetyl-glyphosate, AMPA and N-acetyl AMPA, expressed as glyphosate.

The residue is not fat soluble.

CCN	Commodity Name	Recommended Maximum residue level (mg/kg)		STMR or STMR-P (mg/kg)
		New	Previous	
VD 2066	Subgroup of Dry Peas	10	-	1.7
VD 0072	Peas (dry)	W	5	
VD 0533	Lentils (dry)	W	5	
VD 2065	Subgroup of Dry beans, except soya bean	15	-	0.32
VD 0071	Bean, (dry)	W	2	

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for glyphosate is 0–1 mg/kg bw. The International Estimated Daily Intakes (IEDIs) for glyphosate 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 2019 Extra JMPR Report.

The IEDIs ranged from 1–4% of the maximum ADI. The Meeting concluded that long-term dietary exposure to residues of glyphosate from uses considered by the JMPR is unlikely to present a public health concern.

Acute dietary exposure

The 2011 JMPR decided that an ARfD for glyphosate was unnecessary. The Meeting therefore concluded that the acute dietary exposure to residues of glyphosate from the uses considered is unlikely to present a public health concern.

REFERENCES

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Reference	Author(s)	Year	Title
MSL0011022	Manning, M.J.	2001	Glyphosate Residues in Almond, Pecan, and Walnut Raw Agricultural Commodities Following Pre-harvest Application of Roundup Herbicide. Monsanto Company, Report MSL0011022. Unpublished
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MSL0029625	Mueth, M., Allan, J.M., Kaur, G.	2018	Amended from MSL0023812, Magnitude of Glyphosate Residues in Lentil Raw Agricultural Commodities Following Applications of a Glyphosate-Based Formulation. U. S. and Canadian Trials. Monsanto Company, Report MSL0029625. Unpublished
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MESOTRIONE (277)

First draft prepared by Dr R Scrivens, Health and Safety Executive, York, United Kingdom

EXPLANATION

Mesotrione belongs to the benzoylcyclohexanedione group of herbicides. It is a systemic pre-emergence and post-emergence herbicide for selective contact and residual control of broadleaf weeds. It is rapidly absorbed by green plant tissue or taken up via the soil, and is distributed within plants by both acropetal and basipetal movement. Mesotrione acts as an inhibitor of the enzyme 4-hydroxyphenylpyruvate dioxygenase (HPPD), thereby disrupting carotenoid biosynthesis and maintenance of chlorophyll in sensitive plants resulting in a bleaching effect.

Mesotrione was first evaluated by the JMPR in 2014, when an ADI of 0–0.5 mg/kg bw was established (an ARfD was unnecessary) and maximum residue levels were recommended for a number of commodities.

The residue definition established by the 2014 JMPR for plant and animal commodities, for both compliance with MRLs and dietary risk assessment, is: mesotrione. The residue is not fat soluble.

At the Fiftieth Session of the CCPR (2018), mesotrione was scheduled for evaluation of additional uses by the 2019 Extra JMPR. The current Meeting received GAP information, residue data and processing studies for citrus fruit, pome fruit, stone fruit and tree nuts.

RESIDUE ANALYSIS

Analytical methods

Analytical methods were evaluated by the 2014 JMPR. Method RAM 366/01 was among those considered fit for purpose to determine mesotrione alone or in combination with MNBA in plant and animal commodities at a LOQ of 0.01 mg/kg. A modified version of RAM 366/01 was used for all of the new residues studies submitted, and the Meeting received new validation data and procedural recovery data for mesotrione using this method.

Method RAM 366/01-Rev

In this method, residues of mesotrione and MNBA were extracted from crop samples with acetonitrile/water containing 10 g/L sodium chloride (50:50), centrifuged and diluted with methanol/water (10:90) before reverse phase HPLC-MS/MS analysis (m/z 338→291 for quantitation, no confirmation ion transition used). The main modification to the earlier reference method (evaluated by JMPR, 2014) was the exclusion of the additional SPE clean up and methylene chloride partitioning steps.

The validation data (Table 1) for the newly considered commodities for the modified method involved determination of three recoveries at each fortification level. Mean recovery rates ranged from 73–114% and RSDs were 0.7–13.7%. The LOQ was 0.01 mg/kg in all the crop matrices investigated. The method was also validated for the metabolite MNBA with an LOQ of 0.01 mg/kg.

Table 1 Recovery data for mesotrione by LC-MS/MS (negative ion mode; m/z 338→291) for the modified method of RAM 366/01

Matrix	Fortification Level (mg/kg)	Range (%)	Mean (%)	n	RSD (%)	Report Reference
Orange (Fruit)	0.01	76, 92, 98	89	3	12.6	TK0003124
	1.0	98, 99, 101	99	3	1.2	
Orange (Dried pulp)	0.01	85, 106, 112	101	3	13.7	
	1.0	98, 100, 100	99	3	1.0	
Orange (Juice)	0.01	83, 90, 100	91	3	9.6	
	1.0	101, 102, 104	102	3	1.8	

Matrix	Fortification Level (mg/kg)	Range (%)	Mean (%)	n	RSD (%)	Report Reference
Orange (Citrus oil)	0.01	90, 98, 104	97	3	7.0	
	1.0	96, 100, 102	99	3	3.1	
Apple (Fruit)	0.01	71, 73, 74	73	3	1.8	TK0003122
	1.0	83, 102, 104	97	3	12.3	
Apple (Wet pomace)	0.01	88, 89, 100	92	3	7.5	
	1.0	95, 96, 97	96	3	0.7	
Apple (Juice)	0.01	93, 103, 104	100	3	6.0	
	1.0	111, 115, 115	114	3	2.1	
Dried prune	0.01	83, 88, 95	88	3	6.8	TK0003121
	1.0	96, 97, 102	98	3	3.2	
Plum (Fruit)	0.01	83, 87, 89	86	3	3.5	
	1.0	94, 96, 99	96	3	2.4	
Almond (Nutmeat)	0.01	85, 87, 93	89	3	5.0	TK0003120
	1.0	94, 96, 98	96	3	2.2	
Almond (Hull)	0.01	84, 93, 97	91	3	7.8	
	1.0	95, 96, 99	97	3	1.7	

Results of the concurrent recoveries in the field trials are summarized below in the evaluation of the residues trials.

Stability of pesticide residues in stored analytical samples

The freezer storage stability of residues of mesotrione has been assessed previously by the JMPR in 2014. JMPR concluded that in analytical samples stored under frozen conditions, residues of mesotrione were stable for at least 32 months in maize commodities, radish root and soya bean seed and stable for at least 13 months in blueberry, asparagus, sugar cane and okra.

The 2019 Extra JMPR evaluated storage stability data for mesotrione in lettuce leaf, orange (fruit and juice) and almond nutmeat, in samples stored over a period of 24 months. Homogenised samples were fortified with mesotrione (and MNBA as a mixed fortification solution) at 0.1 mg/kg (Report 2K13-901-TK0061099-001). Duplicate samples were prepared and stored under frozen conditions (-20 °C) and analysed at intervals up to 24 months. Mesotrione residues were quantified using the method RAM 366/01-Rev with a LOQ of 0.01 mg/kg.

Table 2 Storage of mesotrione residues in frozen plant matrices, fortified at 0.1 mg/kg mesotrione

Matrix	Sample storage interval (months)	Fortification level (mg/kg)	Residues remaining (%, uncorrected for procedural recoveries)	Procedural recoveries (%)
Leaf lettuce	0	0.1	86, 83 (85)	76, 78
	3		99, 102 (101)	92, 102
	6		94, 95 (95)	112, 107
	12		100, 101 (101)	106, 105
	18		96, 96 (96)	105, 106
	24		111, 110 (111)	105, 114
Orange Fruit	0	0.1	83, 82 (83)	88, 75
	3		82, 96 (89)	89, 88
	6		99, 89 (94)	111, 107
	12		101, 103 (102)	107, 104
	18		104, 104 (104)	105, 105
	24		109, 106 (108)	105, 117
Orange juice	0	0.1	80, 81 (81)	80, 80
	3		86, 95 (91)	106, 93
	6		96, 99 (98)	118, 120
	12		100, 103 (102)	108, 107
	18		97, 95 (96)	108, 107
	24		105, 106 (106)	115, 108

Matrix	Sample storage interval (months)	Fortification level (mg/kg)	Residues remaining (%, uncorrected for procedural recoveries)	Procedural recoveries (%)
Almond nutmeat	0	0.1	79, 79 (79)	77, 74
	3		90, 93 (92)	93, 87
	6		86, 87 (87)	100, 96
	12		102, 101 (102)	103, 101
	18		105, 112 (109)	115, 112
	24		111, 108 (110)	111, 110

Values in parentheses = mean recovery of stored samples

USE PATTERN

Mesotrione is a systemic herbicide used pre-emergence and post-emergence for selective control of annual broad-leaved weeds. Mesotrione is registered for use in a wide range of crops in many countries. Table 3 represents a summary of the additional GAP information provided to the Meeting.

Table 3 List of additional uses of mesotrione (479 g ai/L SC formulation)

Crop	Country	Method of application	No	RTI (weeks)	Timing of 1st application	Rate (g ai/ha)	Water (L/ha)	Rate – max. per season (g ai/ha)	PHI (days)
Citrus fruit [b]	USA	Post-emergence [a]	2	20	Late fall/early winter or spring	210	93.5-374	421	1
Pome fruit [c]	USA	Post-emergence [a]	2	20	Late fall/early winter or spring	210	93.5-374	421	30
Stone fruit [d]	USA	Post-emergence [a]	2	20	Late fall/early winter or spring	210	93.5-374	421	30
Tree nuts [e]	USA	Post-emergence [a]	2	20	Late fall/early winter or spring	210	93.5-374	421	30

[a] Directed or shielded spray to orchard floor, avoiding contact with trunks, fruit or crop foliage

[b] Citrus fruit = 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, sour orange, sweet orange, pummelo, Russell River lime, Satsuma mandarin, sweet lime, Tachibana orange, Tahiti lime, tangelo, tangerine (Mandarin), tangor, trifoliate orange, unique fruit, cultivars, varieties and/or hybrids of these

[c] Pome fruit = apple, azarole, crab apple, loquat, mayhaw, medlar, pear, Asian pear, quince, Chinese quince, Japanese quince, tejocote, cultivars, varieties and/or hybrids of these

[d] Stone fruit = apricot, Japanese apricot, capulin, black cherry, Nanking cherry, sweet cherry, tart cherry, Chinese jujube, nectarine, peach, plum, American plum, beach plum, Canada plum, cherry plum, Chickasaw plum, Damson plum, Japanese plum, Klamath plum, prune plum, plumcot, sloe, cultivars, varieties and/or hybrids of these

[e] Tree nuts = African nut-tree, almond, beech nut, Brazil nut, Brazilian pine, bunya, bur oak, butternut, Cajou nut, candlenut, cashew, chestnut, chinquapin, coconut, Coquito nut, Dika nut, ginkgo, Guiana chestnut, hazelnut (filbert), heartnut, hickory nut, Japanese horse-chestnut, macadamia nut, Mongongo nut, monkey-pot, monkey puzzle nut, Okari nut, Pachira nut, peach palm nut, pecan, pequi, pili nut, pine nut, pistachio, Sapucaia nut, tropical almond, black walnut, English walnut, yellowhorn, cultivars, varieties and/or hybrids of these

RESULTS OF SUPERVISED RESIDUE TRIALS ON CROPS

The Meeting reviewed the supervised field trial information for the following crops.

Crop Group	Commodity	Region	Table No.
Citrus	Orange	North America	4
	Grapefruit	North America	5
	Lemon	North America	6
Pome fruit	Apples	North America	7
	Pears	North America	8

Crop Group	Commodity	Region	Table No.
Stone fruit	Cherries	North America	9
	Peaches	North America	10
	Plums	North America	11
Tree nuts	Almonds	North America	12
	Pecans	North America	13
	Almond hulls (animal feed)	North America	14

The supervised trials were well documented with laboratory and field reports. In addition to the description and details of the field trials and analytical methods, 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 as all residues in controls were < 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.

Results from replicated field plots are presented as individual values and have not been corrected for concurrent method recoveries. When residues were not detected they are shown as ND and when detected but below the LOQ they are reported as <0.01 mg/kg. Residues have been rounded to two significant digits. Average values have been calculated from the residue results prior to rounding and the results from trials conducted according to the maximum GAP and used for the estimation of maximum residue levels have been (underlined). Where the results of duplicate analyses are available, the highest individual value has been selected as the HR for dietary exposure estimation. Residues of MNBA were sought but not found (above the LOQ of 0.01 mg/kg) in all the residues trials and are not reported in the tables.

Citrus fruits

Twenty three residue trials were conducted in citrus fruits; twelve in orange, six in grapefruit, and five in lemon the USA in 2011.

Two broadcast applications to the orchard floor were made using an SC formulation at a nominal rate of 211 g ai/ha with an application interval of around 31 days. An adjuvant and a spray additive, the latter which was included in most trials not all, were applied in each tank mix.

Fruit samples were collected at normal commercial harvest at 1 day after last application. Additionally one trial for orange and one trial for grapefruit were conducted as decline trials where samples over time (ranging from a PHI of 0 to 10 days) were taken.

Samples were immediately frozen and maintained in frozen storage for periods of up to 277 days prior to extraction and analysis.

Residues of mesotrione (and MNBA) in oranges, grapefruits and lemons were determined simultaneously using Method RAM 366/01-Rev. Procedural recoveries were conducted at fortification levels of 0.01 mg/kg and 0.1 mg/kg with recoveries in the range of 79–114%.

Table 4 Residues in oranges from supervised trials in the USA involving two broadcast applications (applied to the orchard floor) of mesotrione (SC formulation)

Location, Country Year, Crop/Variety	No	Rate (g ai/ha)	Interval (days)	Growth stage at application	PHI (days)	Crop part	Mesotrione (mg/kg)	Reference & Comments
GAP USA	2	210	20 weeks	late fall/early winter or spring	1			
Oak Hill, Florida, USA 2011 Orange / Hamlin	2	208 208	29	BBCH 79 BBCH 83	0 1 3 7 10	Fruit	ND, <0.01 (<0.01) ND, ND (ND) <0.01, <0.01 (<u><0.01</u>) ND, <0.01 (<0.01) <0.01, ND (<0.01)	TK0003124-01 Applied Oct-Nov
Clermont, Florida, USA 2011 Orange / Mid sweet	2	202 201	29	BBCH 81 BBCH 83	1	Fruit	ND, <0.01 (<u><0.01</u>)	TK0003124-02 Applied Oct-Nov
Clermont, Florida, USA 2011 Orange / Hamlin	2	206 206	29	BBCH 81 BBCH 83	1	Fruit	ND, ND (ND)	TK0003124-03 Applied Oct-Nov Only above trial underlined, as this trial is the same apart from variety only
Oviedo, Florida, USA 2011 Orange / Navel	2	211 212	29	BBCH 81 BBCH 83	1	Fruit	ND, ND (<u>ND</u>)	TK0003124-04 Applied Oct-Nov
Chuluota, Florida, USA 2011 Orange / Hamlin	2	205 207	29	BBCH 81 BBCH 83	1	Fruit	ND, ND (<u>ND</u>)	TK0003124-05 Applied Oct-Nov
Bithlo, Florida, USA 2011 Orange / Valencia	2	213 211	30	BBCH 85 BBCH 89	1	Fruit	<0.01, ND (<u><0.01</u>)	TK0003124-06 Applied Apr-May
Clermont, Florida, USA 2011 Orange / Valencia	2	210 213	30	BBCH 85 BBCH 89	1	Fruit	<0.01, <0.01 (<u><0.01</u>)	TK0003124-07 Applied Apr-May

Location, Country Year, Crop/Variety	No	Rate (g ai/ha)	Interval (days)	Growth stage at application	PHI (days)	Crop part	Mesotrione (mg/kg)	Reference & Comments
Oviedo, Florida, USA 2011 Orange / Valencia	2	211 211	30	BBCH 85 BBCH 89	1	Fruit	ND, ND (ND)	TK0003124-08 Applied Apr-May
Raymondville, Texas, USA 2011 Orange / N33- Navel	2	228 216	28	BBCH 83 BBCH 85	1	Fruit	<0.01, <0.01 (<0.01)	TK0003124-09 Applied Nov-Dec
Porterville, California, USA 2011 Orange / Valencia	2	212 214	31	BBCH 85 BBCH 89	1	Fruit	<0.01, <0.01 (<0.01)	TK0003124-10 Applied Jun-Jul
Porterville, California, USA 2011 Orange / Washington	2	213 211	30	BBCH 81 BBCH 85	1	Fruit	ND, ND (ND)	TK0003124-11 Applied Nov-Dec
Richgrove, California, 2011 Orange / Atwoods	2	210 210	29	BBCH 81 BBCH 85	1	Fruit	<0.01, <0.01 (<0.01)	TK0003124-12 Applied Nov-Dec

Values in parentheses = mean of two independent representative treated samples taken at the trial site

Table 5 Residues in grapefruits from supervised trials in USA involving two broadcast applications (applied to the orchard floor) of mesotrione (SC formulation)

Location, Country Year, Crop/Variety	No	Rate (g ai/ha)	Interval (days)	Growth stage at application	PHI (days)	Crop part	Mesotrione (mg/kg)	Reference & Comments
GAP USA	2	210	20 weeks	late fall/early winter or spring	1			
Oak Hill, Florida, USA 2011 Grapefruit / Rio Red	2	206 211	29	BBCH 79 BBCH 83	0 1 3 7 10	Fruit	ND, ND (ND) ND, ND (ND) ND, ND (ND) <0.01, <0.01 (<0.01) ND, ND (ND)	TK0003124-13 Applied Oct-Nov
Clermont, Florida, USA 2011 Grapefruit / Ray	2	207 205	29	BBCH 79 BBCH 83	1	Fruit	ND, ND (ND)	TK0003124-14 Applied Oct-Nov

Location, Country Year, Crop/Variety	No	Rate (g ai/ha)	Interval (days)	Growth stage at application	PHI (days)	Crop part	Mesotrione (mg/kg)	Reference & Comments
Mims, Florida, USA 2011 Grapefruit / Marsh White	2	211 211	29	BBCH 81 BBCH 83	1	Fruit	<0.01, ND (<u><0.01</u>)	TK0003124-15 Applied Nov-Dec
Raymondville, Texas, USA 2011 Grapefruit / Rio Red	2	225 217	28	BBCH 83 BBCH 85	1	Fruit	<0.01, <0.01 (<u><0.01</u>)	TK0003124-16 Applied Nov-Dec
Porterville, California, USA 2011 Grapefruit / Mellogold	2	211 210	30	BBCH 75 BBCH 89	1	Fruit	ND, <0.01 (<u><0.01</u>)	TK0003124-17 Applied Oct-Nov
Lindsay, California, USA 2011 Grapefruit / Mellogold	2	212 212	29	BBCH 79 BBCH 85	1	Fruit	ND, <0.01 (<u><0.01</u>)	TK0003124-18 Applied Nov-Dec

Values in parentheses = mean of two independent representative treated samples taken at the trial site

Table 6 Residues in lemons from supervised trials in USA involving two broadcast applications (applied to the orchard floor) of mesotrione (SC formulation)

Location, Country Year, Crop/Variety	No	Rate (g ai/ha)	Interval (days)	Growth stage at application	PHI (days)	Crop part	Mesotrione (mg/kg)	Reference & Comments
GAP USA	2	210	20 weeks	late fall/early winter or spring	1			
Clermont, Florida, USA 2011 Lemon / Bearss	2	212 211	30	BBCH 76 BBCH 79	1	Fruit	ND, ND (<u>ND</u>)	TK0003124-19 Applied Jul-Aug
Porterville, California, USA 2011 Lemon / Pryor	2	203 211	30	BBCH 75 BBCH 78	1	Fruit	ND, ND (<u>ND</u>)	TK0003124-20 Applied Oct-Nov
Filmore, California, USA 2011 Lemon / Allen/Mac	2	211 213	30	BBCH 87 BBCH 89	1	Fruit	ND, ND (<u>ND</u>)	TK0003124-21 Applied Nov-Dec

Location, Country Year, Crop/Variety	No	Rate (g ai/ha)	Interval (days)	Growth stage at application	PHI (days)	Crop part	Mesotrione (mg/kg)	Reference & Comments
Somis, California, USA 2011 Lemon / Eureka	2	211 208	29	BBCH 74 BBCH 89	1	Fruit	ND, <0.01 (<u><0.01</u>)	TK0003124-22 Applied Sep-Oct
Lindsay, California, USA 2011 Lemon / Lisbon	2	210 211	30	BBCH 85 BBCH 89	1	Fruit	ND, ND (<u>ND</u>)	TK0003124-23 Applied Nov-Dec

Values in parentheses = mean of two independent representative treated samples taken at the trial site

Pome fruits

Eighteen residue trials were conducted in pome fruits; twelve in apples and six in pears, in the USA in 2011.

Two broadcast applications to the orchard floor were made using an SC formulation at a nominal rate of 211 g ai/ha with an application interval of around 30 days. An adjuvant and a spray additive, the latter which was included in most trials not all, were applied in each tank mix.

Fruit samples were collected at normal commercial harvest (close to anticipated PHI of 30 days). Additionally one trial for apple and one trial for pear were conducted as decline trials where samples over time (ranging from a PHI of 27 to 39 days) were taken.

Samples were immediately frozen and maintained in frozen storage for periods of up to 152 days prior to extraction and analysis.

Residues of mesotrione (and MNBA) in apples and pears were determined simultaneously using the modification to the analytical method RAM 366/01-Rev. Procedural recoveries were conducted at fortification levels of 0.01 mg/kg and 0.1 mg/kg with recoveries in the range of 89–119%.

Table 7 Residues in apples from supervised trials in the USA involving two broadcast applications (applied to the orchard floor) of mesotrione (SC formulation)

Location, Country Year, Crop/Variety	No	Rate (g ai/ha)	Interval (days)	Growth stage at application	PHI (days)	Crop part	Mesotrione (mg/kg)	Reference & Comments
GAP USA	2	210	20 weeks	late fall/early winter or spring	30			
Alton, New York, USA 2011 Apple / Granny Smith	2	212 212	30	BBCH 76 BBCH 77	30	Fruit	<0.01, <0.01 (<u><0.01</u>)	TK0003122-01 Applied Aug-Sep
North Rose, New York, USA 2011 Apple / Empire	2	211 208	30	BBCH 77 BBCH 81	30	Fruit	<0.01, <0.01 (<u><0.01</u>)	TK0003122-02 Applied Aug-Sep

Location, Country Year, Crop/Variety	No	Rate (g ai/ha)	Interval (days)	Growth stage at application	PHI (days)	Crop part	Mesotrione (mg/kg)	Reference & Comments
Hereford, Pennsylvania, USA 2011 Apple / Starkrimson Red Delicious	2	214 211	29	BBCH 75 BBCH 79	31	Fruit	<0.01, <0.01 (<u>≤0.01</u>)	TK0003122-03 Applied Jul-Aug
Cana, Virginia, USA 2011 Apple / Rome	2	211 208	30	BBCH 76 BBCH 81	31	Fruit	<0.01, <0.01 (<u>≤0.01</u>)	TK0003122-04 Applied Jul-Aug
Conklin, Michigan, USA 2011 Apple / Red Delicious	2	210 211	30	BBCH 75 BBCH 81	30	Fruit	<0.01, <0.01 (<u>≤0.01</u>)	TK0003122-05 Applied Jul-Aug
Marengo, Illinois, USA 2011 Apple / Red Chief	2	211 212	30	BBCH 74 BBCH 80	29	Fruit	<0.01, <0.01 (<u>≤0.01</u>)	TK0003122-06 Applied Jul-Aug
Perry, Utah, USA 2011 Apple / Gala	2	214 212	32	BBCH 74 BBCH 78	31	Fruit	<0.01, <0.01 (<u>≤0.01</u>)	TK0003122-07 Applied Jul-Aug
Porterville, California, USA 2011 Apple / Granny Smith	2	213 214	30	BBCH 75 BBCH 78	31	Fruit	<0.01, <0.01 (<u>≤0.01</u>)	TK0003122-08 Applied Jul-Aug
Ephrata, Washington, USA 2011 Apple / Red Delicious	2	211 208	29	BBCH 75 BBCH 84	31	Fruit	<0.01, <0.01 (<u>≤0.01</u>)	TK0003122-09 Applied Aug-Sep
Ephrata, Washington, USA 2011 Apple / Gala	2	214 211	30	BBCH 72 BBCH 77	30	Fruit	<0.01, <0.01 (<u>≤0.01</u>)	TK0003122-10 Applied Jul-Aug

Location, Country Year, Crop/Variety	No	Rate (g ai/ha)	Interval (days)	Growth stage at application	PHI (days)	Crop part	Mesotrione (mg/kg)	Reference & Comments
Weiser, Idaho, USA 2011 Apple / Law Rome	2	208 211	30	BBCH 76 BBCH 79	27 30 33 36 38	Fruit	<0.01, <0.01 (<0.01) <0.01, <0.01 (<u>0.01</u>) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	TK0003122-11 Applied Aug-Sep
Caldwell, Idaho, USA 2011 Apple / Jonathan	2	217 219	31	BBCH 75 BBCH 78	29	Fruit	<0.01, <0.01 (<u>0.01</u>)	TK0003122-12 Applied Jul-Aug

Table 8 Residues in pears from supervised trials in USA involving two broadcast applications (applied to the orchard floor) of mesotrione (SC formulation)

Location, Country Year, Crop/Variety	No	Rate (g ai/ha)	Interval (days)	Growth stage at application	PHI (days)	Crop part	Mesotrione (mg/kg)	Reference & Comments
GAP USA	2	210	20 weeks	late fall/early winter or spring	30			
Alton, New York, USA 2011 Pear / Bartlett	2	212 212	30	BBCH 73 BBCH 75	30	Fruit	<0.01, <0.01 (<u>0.01</u>)	TK0003122-13 Applied Jun-Jul
Lindsay, California, USA 2011 Pear / Olympic	2	212 212	30	BBCH 75 BBCH 77	27 30 33 36 39	Fruit	<0.01, <0.01 (<0.01) <0.01, <0.01 (<u>0.01</u>) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	TK0003122-14 Applied Jul-Aug
Porterville, California, USA 2011 Pear / Olympic	2	211 212	30	BBCH 75 BBCH 79	30	Fruit	<0.01, <0.01 (<u>0.01</u>)	TK0003122-15 Applied Jul-Aug
Ephrata, Washington, USA 2011 Pear / Bartlett	2	214 208	30	BBCH 72 BBCH 78	30	Fruit	<0.01, <0.01 (<u>0.01</u>)	TK0003122-16 Applied Jul-Aug

Location, Country Year, Crop/Variety	No	Rate (g ai/ha)	Interval (days)	Growth stage at application	PHI (days)	Crop part	Mesotrione (mg/kg)	Reference & Comments
Ephrata, Washington, USA 2011 Pear / Concorde	2	211 211	30	BBCH 76 BBCH 85	30	Fruit	<0.01, <0.01 (<u><0.01</u>)	TK0003122-17 Applied Jul-Aug
Payette, Idaho, USA 2011 Pear / Bartlett	2	208 206	30	BBCH 75 BBCH 77	30	Fruit	<0.01, 0.01 [a] (<u><0.01</u>)	TK0003122-18 Applied Jul-Aug

Values in parentheses = mean of two independent representative treated samples taken at the trial site

[a] Rounded from 0.0100 mg/kg, An estimated 0.0057 mg/kg was found in the control sample. Taking account of the other trials results that are all <0.01 mg/kg, the average result for this trial is concluded as <0.01 mg/kg.

Stone fruits

Twenty one residue trials were conducted in stone fruits; six in cherries, nine in peaches, and six in plum in the USA in 2011.

Two broadcast applications to the orchard floor were made using an SC formulation at a nominal rate of 211 g ai/ha with an application interval of around 30 days. An adjuvant and a spray additive, the latter which was included in most trials not all, were applied in each tank mix.

Fruit samples were collected at normal commercial harvest (close to anticipated PHI of 30 days). Additionally one trial for cherry and one trial for peach were conducted as decline trials where samples over time (ranging from a PHI of 27 to 39 days) were taken.

Samples were immediately frozen and maintained in frozen storage for periods of up to 256 days prior to extraction and analysis.

Residues of mesotrione (and MNBA) in cherries, peaches and plums were determined simultaneously using the modification to the analytical method RAM 366/01-Rev. Procedural recoveries were conducted at fortification levels of 0.01 mg/kg and 1.0 mg/kg with recoveries in the range of 92–117%.

Table 9 Residues in cherries from supervised trials in the USA involving two broadcast applications (applied to the orchard floor) of mesotrione (SC formulation)

Location, Country Year, Crop/Variety	No	Rate (g ai/ha)	Interval (days)	Growth stage at application	PHI (days)	Crop part	Mesotrione (mg/kg)	Reference & Comments
GAP USA	2	210	20 weeks	late fall/early winter or spring	30			
Alton, New York, USA 2011 Sour Cherries / Montmorency	2	213 213	30	BBCH 65 BBCH 75	27 30 33 37 39	Fruit	<0.01, <0.01 (<0.01) <0.01, <0.01 (<u><0.01</u>) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	TK0003121-01 Applied May-Jun

Location, Country Year, Crop/Variety	No	Rate (g ai/ha)	Interval (days)	Growth stage at application	PHI (days)	Crop part	Mesotrione (mg/kg)	Reference & Comments
GAP USA	2	210	20 weeks	late fall/early winter or spring	30			
Conklin, Michigan, USA 2011 Sour Cherries / Montmorency	2	211 210	30	BBCH 66 BBCH 78	30	Fruit	<0.01, <0.01 (<u>≤0.01</u>)	TK0003121-02 Applied May-Jun
Merengo, Illinois, USA 2011 Sour Cherries / North Star	2	208 215	30	BBCH 65 BBCH 73	30	Fruit	<0.01, <0.01 (<u>≤0.01</u>)	TK0003121-03 Applied May-Jun
Plainview, California, USA 2011 Sweet cherries / Tulare	2	212 212	30	BBCH 61 BBCH 71	30	Fruit	<0.01, <0.01 (<u>≤0.01</u>)	TK0003121-04 Applied Mar-Apr
Ephrata, Washington, USA 2011 Sweet cherries / Bing	2	213 214	30	BBCH 69 BBCH 75	30	Fruit	<0.01, <0.01 (<u>≤0.01</u>)	TK0003121-05 Applied May-Jun
Weiser, Idaho, USA 2011 Sweet cherries / Benton	2	208 216	30	BBCH 65 BBCH 73	30	Fruit	<0.01, <0.01 (<u>≤0.01</u>)	TK0003121-06 Applied May-Jun

Table 10 Residues in peaches from supervised trials in the USA involving two broadcast applications (applied to the orchard floor) of mesotrione (SC formulation)

Location, Country Year, Crop/Variety	No	Rate (g ai/ha)	Interval (days)	Growth stage at application	PHI (days)	Crop part	Mesotrione (mg/kg)	Reference & Comments
GAP USA	2	210	20 weeks	late fall/early winter or spring	30			
Alton, New York, USA 2011 Peach / Red Haven	2	224 211	30	BBCH 73 BBCH 75	31	Fruit	<0.01, <0.01 (<u>≤0.01</u>)	TK0003121-07 Applied Jun-Jul

Location, Country Year, Crop/Variety	No	Rate (g ai/ha)	Interval (days)	Growth stage at application	PHI (days)	Crop part	Mesotrione (mg/kg)	Reference & Comments
GAP USA	2	210	20 weeks	late fall/early winter or spring	30			
Monetta, South Carolina, USA 2011 Peach / Big Red	2	212 210	30	BBCH 75 BBCH 77	29	Fruit	<0.01, <0.01 (<u>≤0.01</u>)	TK0003121-08 Applied Jun-Jul
Chula, Georgia, USA 2011 Peach / Hawthorne	2	205 219	30	30% fruit formation BBCH 75	30	Fruit	<0.01, <0.01 (<u>≤0.01</u>)	TK0003121-09 Applied Apr-May
Morven, Georgia, 2011 Peach / Gala	2	216 211	30	30% fruit formation BBCH 75	30	Fruit	<0.01, <0.01 (<u>≤0.01</u>)	TK0003121-10 Applied Apr-May
Conklin, Michigan, USA 2011 Peach / Red Haven	2	208 211	29	BBCH 73 BBCH 75	30	Fruit	<0.01, <0.01 (<u>≤0.01</u>)	TK0003121-11 Applied Jun-Jul
D'Hanis, Texas, USA 2011 Peach / La Feliciana	2	212 205	30	BBCH 73 BBCH 79	30	Fruit	<0.01, <0.01 (<u>≤0.01</u>)	TK0003121-12 Applied Apr-May
Kingsburg, California, USA 2011 Peach / Cling	2	215 214	30	BBCH 75 BBCH 78	27 30 33 36 39	Fruit	<0.01, <0.01 (<0.01) <0.01, <0.01 (<u>≤0.01</u>) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	TK0003121-13 Applied May-Jun
Porterville, California, USA 2011 Peach / Fay Alberta	2	208 211	30	BBCH 75 BBCH 78	30	Fruit	<0.01, <0.01 (<u>≤0.01</u>)	TK0003121-14 Applied Jun-Jul
Dinuba, California, USA 2011 Peach / Princess Tyme	2	214 215	30	BBCH 74 BBCH 75	30	Fruit	<0.01, <0.01 (<u>≤0.01</u>)	TK0003121-15 Applied Apr-May

Table 11 Residues in plums from supervised trials in the USA involving two broadcast applications (applied to the orchard floor) of mesotrione (SC formulation)

Location, Country Year, Crop/Variety	No	Rate (g ai/ha)	Interval (days)	Growth stage at application	PHI (days)	Crop part	Mesotrione (mg/kg)	Reference & Comments
GAP USA	2	210	20 weeks	late fall/early winter or spring	30			
Conklin, Michigan, USA 2011 Plum / Stanley	2	211 211	30	BBCH 75 BBCH 78-81	30	Fruit	<0.01, <0.01 (<u>≤0.01</u>)	TK0003121-16 Applied Jun-Jul
Poplar, California, USA 2011 Plum / French Prunes	2	210 211	30	BBCH 71 BBCH 81	30	Fruit	<0.01, <0.01 (<u>≤0.01</u>)	TK0003121-17 Applied Jun-Jul
Woodville, California, USA 2011 Plum / French Prunes	2	210 211	30	BBCH 77 BBCH 81	30	Fruit	<0.01, <0.01 (<u>≤0.01</u>)	TK0003121-18 Applied Jun-Jul
Dinuba, California, USA 2011 Plum / Friar	2	210 212	30	BBCH 75 BBCH 76	30	Fruit	<0.01, <0.01 (<u>≤0.01</u>)	TK0003121-19 Applied May-Jun
Lindsay, California, USA 2011 Plum / Angelina's	2	206 210	30	BBCH 79 BBCH 81	30	Fruit	<0.01, <0.01 (<u>≤0.01</u>)	TK0003121-20 Applied Jun-Jul
Newberg, Oregon, USA 2011 Plum / Italian	2	210 211	30	BBCH 74 BBCH 78	31	Fruit	<0.01, <0.01 (<u>≤0.01</u>)	TK0003121-21 Applied Jul-Aug

Values in parentheses = mean of two independent representative treated samples taken at the trial site

Tree nuts

Ten residue trials were conducted in tree nuts; five in almonds and five in pecans, in the USA in 2011.

Two broadcast applications to the orchard floor were made using an SC formulation at a nominal rate of 211 g ai/ha with an application interval of around 30 days. An adjuvant and a spray additive, the latter which was included in most trials not all, were applied in each tank mix.

Samples of nutmeat (almond and pecan) and hulls (almonds only) were collected at normal commercial harvest (close to anticipated PHI of 30 days). Additionally one trial for pecan and one trial

for almonds was conducted as a decline trial where samples over time (ranging from PHI of 27 to 39 days) were taken.

Samples were immediately frozen and maintained in frozen storage for periods of up to 174 days prior to extraction and analysis.

Residues of mesotrione (and MNBA) in almonds and pecans were determined simultaneously using the modification to the analytical method RAM 366/01-Rev. Procedural recoveries were conducted at fortification levels of 0.01 mg/kg and 0.1 mg/kg with recoveries in the range of 89–115%.

Table 12 Residues in almonds from supervised trials in the USA involving two broadcast applications (applied to the orchard floor) of mesotrione (SC formulation). Please refer to Table 14 for data on almonds hulls.

Location, Country Year, Crop/Variety	No	Rate (g ai/ha)	Interval (days)	Growth stage at application	PHI (days)	Crop part	Mesotrione (mg/kg)	Reference & Comments
GAP USA	2	210	20 weeks	late fall/early winter or spring	30			
Terra Bella, California, USA 2011 Almond / Carmel	2	210 212	32	BBCH 79 BBCH 87	28	Nutmeat	<0.01, <0.01 (<u><0.01</u>)	TK0003120-01 Applied Jun-Jul
Wasco, California, USA 2011 Almond / Price	2	208 211	32	BBCH 79 BBCH 87	28	Nutmeat	<0.01, <0.01 (<u><0.01</u>)	TK0003120-02 Applied Jun-Jul
Buttonwillow, California, USA 2011 Almond / Monterey	2	212 211	31	BBCH 79 BBCH 87	29	Nutmeat	<0.01, <0.01 (<u><0.01</u>)	TK0003120-03 Applied Jul-Aug
Dinuba, California, USA 2011 Almond / Carmel	2	208 213	26	BBCH 79 BBCH 86	30	Nutmeat	<0.01, ND (<u><0.01</u>)	TK0003120-04 Applied Jun-Jul
Strathmore, California, USA 2011 Almond / Nonpareil	2	213 213	30	BBCH 79 BBCH 81	27 30 32 35 39	Nutmeat	<0.01, <0.01 (<0.01) <0.01, <0.01 (<u><0.01</u>) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	TK0003120-05 Applied Jun-Jul

Table 13 Residues in pecans from supervised trials in the USA involving two broadcast applications (applied to the orchard floor) of mesotrione (SC formulation)

Location, Country Year, Crop/Variety	No	Rate (g ai/ha)	Interval (days)	Growth stage at application	PHI (days)	Crop part	Mesotrione (mg/kg)	Reference & Comments
GAP USA	2	210	20 weeks	late fall/early winter or spring	30			
Girard, Georgia, USA 2011 Pecan / Desirables	2	213 211	30	BBCH 76 BBCH 80	29	Nutmeat	<0.01, <0.01 (<u><0.01</u>)	TK0003120-06 Applied Sep-Oct
Mystic, Georgia, USA 2011 Pecan / Sumner	2	211 206	29	BBCH 73 BBCH 79	31	Nutmeat	<0.01, <0.01 (<u><0.01</u>)	TK0003120-07 Applied Sep-Oct
Alexandria, Louisiana, USA 2011 Pecan/ Creek	2	216 203	29	Early dough Beginning shuck split	31	Nutmeat	<0.01, <0.01 (<u><0.01</u>)	TK0003120-08 Applied Sep-Oct
Pearsall, Texas, USA 2011 Pecan/ Cheyenne	2	210 207	30	BBCH 79 BBCH 87	27 29 33 36 39	Nutmeat	<0.01, <0.01 (<0.01) <0.01, <0.01 (<u><0.01</u>) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	TK0003120-09 Applied Aug-Sep
Anton, Texas, USA 2011 Pecan / Western Schley	2	211 204	30	Green shuck Green shuck	30	Nutmeat	<0.01, <0.01 (<u><0.01</u>)	TK0003120-10 Applied Oct-Nov

Values in parentheses = mean of two independent representative treated samples taken at the trial site

Animal Feed- Almond hulls

Five residue trials, as reported in the evaluation above for tree nuts, were conducted in almonds in the USA in 2011 providing data on almond hulls.

Table 14 Residues in almonds from supervised trials in the USA involving two broadcast applications (applied to the orchard floor) of mesotrione (SC formulation)

Location, Country Year, Crop/Variety	No	Rate (g ai/ha)	Interval (days)	Growth stage at application	PHI (days)	Crop part	Mesotrione (mg/kg)	Reference & Comments
GAP USA	2	210	20 weeks	late fall/early winter or spring	30			
Terra Bella, California, USA 2011 Almond / Carmel	2	210 212	32	BBCH 79 BBCH 87	28	Hull	<0.01, <0.01 (<u>≤0.01</u>)	TK0003120-01 Applied Jun-Jul
Wasco, California, USA 2011 Almond / Price	2	208 211	32	BBCH 79 BBCH 87	28	Hull	<0.01, <0.01 (<u>≤0.01</u>)	TK0003120-02 Applied Jun-Jul
Buttonwillow, California, USA 2011 Almond / Monterey	2	212 211	31	BBCH 79 BBCH 87	29	Hull	<0.01, <0.01 (<u>≤0.01</u>)	TK0003120-03 Applied Jul-Aug
Dinuba, California, USA 2011 Almond / Carmel	2	208 213	26	BBCH 79 BBCH 86	30	Hull	<0.01, <0.01 (<u>≤0.01</u>)	TK0003120-04 Applied Jun-Jul
Strathmore, California, USA 2011 Almond / Nonpareil	2	213 213	30	BBCH 79 BBCH 81	27 30 32 35 39	Hull	0.051, 0.058 (0.055) 0.014, <0.01 (0.012) 0.024, 0.025 (<u>0.025</u>) 0.025, 0.013 (0.019) 0.012, 0.034 (0.023)	TK0003120-05 Applied Jun-Jul

FATE OF RESIDUES DURING PROCESSING

In processing-effect on the residue level

For the current meeting new data was received on the fate of mesotrione residues on the processing of citrus (orange), pome fruits (apple), and stone fruit (plums).

The validated analytical method (RAM 366/01-Rev) was used to measure residues of mesotrione with concurrent recoveries for the processed fractions of 87–116% (at fortification levels at the LOQ of 0.01 mg/kg and at 0.1 mg/kg). Residues of MNBA were sought but not found (above the LOQ of 0.01 mg/kg) in all the processing trials and are not reported below.

Citrus (orange)

In two of the supervised field trials on oranges, an additional plot was treated with an exaggerated rate (3×) of 632 g ai/ha as a directed orchard floor spray. The application interval was 30–31 days. Whole orange fruit samples were harvested 1 day after the last application.

Fruit samples were processed via representative processes to dried pulp, orange oil and juice. The processes used were to simulate commercial operations. The effect of peeling was not investigated as part of this study.

The residues in the raw agricultural commodity (RAC) and the processed fractions are presented in Table 14.

Pome fruit (apple)

In two of the supervised field trials on apples, an additional plot was treated with an exaggerated rate (3×) of 632 g or 645 g ai/ha as a directed orchard-floor spray. The application interval was 29–30 days. Whole apple fruit samples were harvested 30 or 31 days after the last application.

Fruit samples were processed via representative processes to wet pomace and apple juice. The processes used were to simulate commercial operations.

The residues in the raw agricultural commodity (RAC) and the processed fractions are presented in Table 14.

Stone fruit (plum)

In two of the supervised field trials on plums, an additional plot was treated with an exaggerated rate (3×) of 632 g as a directed orchard-floor spray. The application interval was 30 days. Whole plum fruit samples were harvested 30 days after the last application.

Fruit samples were processed via representative processes to dried prunes. The processes used were to simulate commercial operations.

The residues in the raw agricultural commodity (RAC) and the processed fractions are presented in Table 15.

Table 15 Residues of mesotrione in processed fractions of oranges, apples and plums

Crop	Trial Details	Commodity	Residue of mesotrione (mg/kg)	Processing Factor (PF) [a]
Oranges	Oveido, FL (variety Valencia) 2 × 632-637 g ai/ha 187 L water/ha 30-day RTI 1-day PHI TK0003124-08	Orange whole fruit (RAC)	<0.01, <0.01 (<0.01)	–
		Juice	<0.01, <0.01 (<0.01)	n.d.
		Orange oil	<0.01, <0.01 (<0.01)	n.d.
		Dried pulp	<0.01, <0.01 (<0.01)	n.d.
	Porterville, CA (variety Valencia) 2 × 631-634 g ai/ha 262 L water/ha 31-day RTI 1-day PHI TK0003124-10	Orange whole fruit (RAC)	<0.01, <0.01 (<0.01)	–
		Juice	<0.01, 0.01 [b] (<0.01)	n.d.
		Orange oil	<0.01, <0.01 (<0.01)	n.d.
		Dried pulp	<0.01, <0.01 (<0.01)	n.d.
Apples	Alton, NY (variety Granny Smith) 2 × 630-633 g ai/ha 374 L water/ha 30-day RTI 30-day PHI TK0003122-01	Apple whole fruit (RAC)	<0.01, <0.01 (<0.01)	–
		Juice	<0.01, <0.01 (<0.01)	n.d.
		Wet pomace	<0.01, <0.01 (<0.01)	n.d.
	Ephrata, WA (variety Red Delicious)	Apple whole fruit (RAC)	<0.01, <0.01 (<0.01)	–
		Juice	ND, ND ND)	n.d.

Crop	Trial Details	Commodity	Residue of mesotrione (mg/kg)	Processing Factor (PF) [a]
	2 × 649-642 g ai/ha 281 L water/ha 29-day RTI 31-day PHI TK0003122-09	Wet pomace	<0.01, <0.01 (<0.01)	n.d.
Plums	Poplar, CA (variety French prunes) 2 × 631-632 g ai/ha 262 L water/ha 30-day RTI 30-day PHI TK0003121-17	Plum whole fruit (RAC)	<0.01, <0.01 (<0.01)	–
		Dried prunes	<0.01, <0.01 (<0.01)	n.d.
	Woodville, CA (variety French Prunes) 2 × 631-633 g ai/ha 262 L water/ha 30-day RTI 30-day PHI TK0003121-18	Plum whole fruit (RAC)	<0.01, <0.01 (<0.01)	–
		Dried prunes	<0.01, <0.01 (<0.01)	n.d.

Values in parentheses = mean of two independent representative treated samples taken at the trial site

RAC = raw agricultural commodity

[a] Processing factor = residue in processed commodity (mg/kg) / residue in RAC (mg/kg). Since average residues for each trial were <LOQ (<0.01 mg/kg), it is not possible to give a numeric estimation of a processing factor. It is not possible to conclude whether residues decline or concentrate over processing.

[b] Rounded from 0.0105 mg/kg, An estimated 0.0042 mg/kg was found in the control juice sample. The average result for this trial is concluded as <0.01 mg/kg.

Due to residues being concluded as <LOQ in all the processed and unprocessed (RAC) fractions, it is not possible to conclude on whether there is any concentration or reduction in residues over processing, and processing factors could not be derived.

APPRAISAL

Mesotrione, a herbicide, was firstly evaluated by the JMPR in 2014, when an ADI of 0–0.5 mg/kg bw was established, and an ARfD was unnecessary. The residue definition for plant and animal commodities, for both compliance with MRLs and dietary risk assessment is: *mesotrione*. The residue is not fat soluble.

At the Fiftieth Session of the CCPR (2018), mesotrione was scheduled for evaluation of additional uses by the 2019 Extra JMPR. The current Meeting received GAP information, residue data and processing studies for citrus fruit, pome fruit, stone fruit and tree nuts.

Methods of analysis

Residues were determined in the crops with a method involving extraction with acetonitrile/water containing sodium chloride, with a final determination, following dilution with methanol/water, using HPLC-MS/MS. The Meeting concluded that suitable methods are available for the determination of residues of mesotrione with a LOQ of 0.01 mg/kg in the commodities under consideration.

Stability of residues in stored analytical samples

The stability of residues has been assessed previously by the JMPR for a range of crop matrices. In this meeting stability data were provided for lettuce leaf, orange (fruit and juice) and almond nutmeat indicating that residues of mesotrione were stable in these commodities for at least 24 months of frozen storage. The maximum length of storage of commodities considered by the current meeting was up to 277 days. All trial samples and processed commodities were analysed within acceptable storage intervals.

Results of supervised residue trials on crops

Citrus fruits

The critical GAP in the USA for citrus is two applications at 210 g ai/ha with a PHI of 1 day. Eleven residue trials in orange, six in grapefruit, and five in lemon, approximating the GAP but with a shorter application interval were received.

Residues in citrus fruits (oranges, grapefruits and lemons) were all $\underline{< 0.01}$ mg/kg (n=22).

Whilst trial data are not available for the subgroup mandarins, the other various citrus crop data that are available show that residues would not be expected in mandarins following the GAP for citrus fruits. The Meeting agreed to include mandarins in the recommendation.

Six trials across various fruit tree crops including citrus fruits conducted at an exaggerated rate (3×) for the purpose of studying processing showed residues < 0.01 mg/kg. Furthermore, mesotrione is applied to the ground at the base of the trees and not directed to the crop.

The Meeting estimated a maximum residue level of 0.01(*) mg/kg and a STMR of 0 mg/kg for the citrus fruits group.

Pome fruits

The critical GAP in the USA for pome fruit is two applications at 210 g ai/ha with a PHI of 30 days. Twelve residue trials in apples and six in pears, approximating the GAP but with a shorter application interval were received.

Residues in pome fruits (apples and pears) were all $\underline{< 0.01}$ mg/kg (n=18).

Six trials across various fruit tree crops including pome fruits conducted at an exaggerated rate (3×) for the purpose of studying processing showed residues <0.01 mg/kg. Furthermore, mesotrione is applied to the ground at the base of the trees and not directed to the crop.

The Meeting estimated a maximum residue level of 0.01(*) mg/kg and a STMR of 0 mg/kg for the pome fruits group.

Stone fruits

The critical GAP in the USA for stone fruits is two applications at 210 g ai/ha with a PHI of 30 days. Six residue trials in cherries, nine in peaches and six in plums, approximating the GAP but with a shorter application interval, were received.

Residues in stone fruits (cherries, peaches and plums) were all $\underline{< 0.01}$ mg/kg (n=21).

Six trials across various fruit tree crops including stone fruits conducted at an exaggerated rate (3×) for the purpose of studying processing showed residues < 0.01 mg/kg. Furthermore, mesotrione is applied to the ground at the base of the trees and not directed to the crop.

The Meeting estimated a maximum residue level of 0.01(*) mg/kg and a STMR of 0 mg/kg for stone fruits group.

Tree nuts

The critical GAP in the USA for tree nuts is two applications at 210 g ai/ha with a PHI of 30 days. Five residue trials in almonds and five in pecans, approximating the GAP but with a shorter application interval were received.

Residues in tree nuts (almonds and pecans) were all < 0.01 mg/kg (n=10).

The Meeting estimated a maximum residue level of 0.01(*) mg/kg and a STMR of 0.01 mg/kg for tree nuts group.

Animal feed commodities

Almond hulls

The critical GAP is for the USA which is two applications at 210 g ai/ha with a PHI of 30 days. Five residue trials in almonds, approximating the GAP but with a shorter application interval were available.

Residues in almond hulls were: < 0.01 (4) and 0.025 mg/kg.

The Meeting estimated a maximum residue level of 0.04 mg/kg (dry weight basis) and a median of 0.01 mg/kg for almond hulls.

Residues in processed commodities

The current meeting received residue data on the magnitude of residues over processing for mesotrione on citrus fruits (orange), pome fruits (apple), and stone fruits (plums). Two trials for each commodity were conducted at an exaggerated rate (3×); residues were below the LOQ (< 0.01 mg/kg) in both raw and processed fractions and it was not possible to derive processing factors.

Residues in animal commodities

Dietary burden calculations, incorporating almond hulls and the other feed items considered by the JMPR in 2014, have been undertaken. Estimation by the present meeting does not impact on the previous (2014) level of the dietary burden. The Meeting confirmed the previous recommendations for mesotrione 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 assessment.

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

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

The residue is not fat-soluble.

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
FC 0001	Citrus fruits	0.01*	-	0	
FP 0009	Pome fruits	0.01*	-	0	
FS 0012	Stone fruits	0.01*	-	0	
TN 0085	Tree nuts	0.01*	-	0.01	

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
AM 0660	Almond hulls	0.04 (dw)	-	Median: 0.01 (as)	

(as) – as received; (dw) – dry weight

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for mesotrione is 0–0.5 mg/kg bw. The International Estimated Daily Intakes (IEDIs) for mesotrione 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 2019 Extra JMPR Report.

The IEDIs were 0% of the maximum ADI. The Meeting concluded that long-term dietary exposure to residues of mesotrione from uses considered by the JMPR is unlikley to present a public health concern.

Acute dietary exposure

The 2014 JMPR decided that an ARfD for mesotrione was unnecessary. The Meeting therefore concluded that the acute dietary exposure to residues of mesotrione from the uses considered is unlikely to present a public health concern.

REFERENCES

Author	Report No./Trial ID	Year	Title, Institute
Wyatt, D.R.	TK0003124	2012	Mesotrione: Mesotrione SC (A12738A) – Magnitude of the Residues in or on Orange, Grapefruit, and Lemon as Representative Crops of Citrus Fruits, Group 10 – USA, 2011. Report No. TK0003124. Syngenta File No. A12738A_50053
Wyatt, D.R.	TK0003122	2012	Mesotrione: Mesotrione SC (A12738A) – Magnitude of the Residues in or on Apple and Pear as Representative Crops of Pome Fruits, Group 11 – USA, 2011. Report No. TK0003122. Syngenta File No. A12738A_50050
Wyatt, D.R.	TK0003121	2012	Mesotrione: Mesotrione SC (A12738A) – Magnitude of the Residues in or on Cherry, Peach, and Plum as Representative Crops of Stone Fruits, Group 12 – USA, 2011. Report No. TK0003121. Syngenta File No. A12738A_50046
Wyatt, D.R.	TK0003120	2012	Mesotrione: Mesotrione SC (A12738A) - Magnitude of the Residues in or on Almond and Pecan as Representative Crops of Tree Nuts, Group 14 - USA, 2011. Report No. TK0003120. Syngenta File No. A12738A_50043
Perez, R, Patel, D, Perez, S	2K13-901-TK0061099-001	2013	Stability of Mesotrione and MNBA Residues in Diverse Crop Commodities Stored Under Freezer Conditions. Report No. 2K13-901-TK0061099-001. Syngenta File No. ZA1296_50735

METAFLUMIZONE (236)

First draft prepared by Dr G Ye, Ministry of Agriculture and Rural Affairs, Beijing, Republic of China

EXPLANATION

Metaflumizone is a broad-spectrum semicarbazone insecticide composed of two optical isomers in the ratio E:Z of 90:10. Metaflumizone was first evaluated for residues and toxicology by the JMPR in 2009. An ADI of 0–0.1 mg/kg bw was established and that an ARfD was unnecessary.

The residue definition for compliance with MRLs and dietary risk assessment for plants and animals is: *Metaflumizone, sum of E-isomer and Z-isomer*.

The residue is fat-soluble.

Metaflumizone was scheduled at the Fiftieth Session of the CCPR for evaluation of additional uses for residues by the 2019 Extra JMPR. The Meeting received information from the manufacturer on environmental fate in soil, stability in stored analytical samples, use patterns, supervised residue trials, and the fate of the residues during storage and processing.

Fate and behaviour in the environment

One study on the degradation of metaflumizone under aerobic conditions in Brazilian soil was received (Tornisielo A. 2010, BASF DocID 2018/3001301). [^{14}C]-Metaflumizone (specific activity 6.42 MBq/mg, Radiochemical purity: 97.5%) was applied at a concentration of 0.640 mg/kg, corresponding to the maximum agronomic rate of 240 g ai/ha in four different Brazilian soil types (typical Aluminium-enriched Melanic Gleysol (GM), typical Dystrophic Red Latosol (LVd), Typical Orthic Quartzarenic Neosol (RQ) and chernozemic Eutroferric Red Argisol (PV)).

The treated soils were maintained at 40% of the maximum water holding capacity under dark conditions, at a temperature of 20 ± 2 °C for a period of 118 days. In the periods of 0, 7, 14, 30, 61, 89 and 118 days after treatment (DAT), production of ^{14}CO2, metabolism, bound-residue formation and volatilization of ^{14}C- Metaflumizone (BAS 320 I), were assessed. The microbial activity in the soils (biomass) was checked at 14 and 118 days after application.

The samples were extracted with acetonitrile, followed by a mixture of acetonitrile/water and finally with acetone. The combined and concentrated extracts were resuspended in acetonitrile/water and analysed by Radio-HPLC. The mass balance (extract + non-extractable residue + volatiles) varied between 88.7% and 102.0% of TAR for all soils with mean values between 92.7% and 96.8% of TAR. For all the incubated soils, the extractable radioactivity decreased from 101.3% of TAR at 0 DAT to 40.2% of TAR after 118 days of incubation. The non-extractable radioactivity (bound residue) for all four soils increased from 0.2% of TAR at 0 DAT to 25.7% of TAR after 118 days of incubation. The formation of accumulated CO2 was observed in all four soils reaching maximum values between 10.5 and 29.2% of TAR after 118 days of incubation. The formation of accumulated organic volatile products was observed in the LVd, RQ and PV soils reaching maximum values between 0.1 and 1.5% of TAR after 118 days of incubation.

Table 1 Characteristics of soils used for rate of degradation of ^{14}C-Metaflumizone (BAS 320 I) in Brazilian soils (2018/3001301)

Soil	GM	LVd	RQ	PV
Soil Taxonomy (EUA, 1999)	Humaquept	Hapludox	Quartzipsamment	Mollic Hapludalf
Brazilian System of Soil Classification (Embrapa 2006)	Typic Aluminium-enriched Melanic Gleysol	Typic Dystrophic Red Latosol	Typic Orthic Quartzarenic Neosol	Chernozemic Eutroferric Red Argisol
pH (water)	4.6	4.6	4.8	5.0
Organic carbon (g/kg)	62.8	19.2	6.4	30.8
Nitrogen (mg/kg)	4900	1820	840	2380

Soil	GM	LVd	RQ	PV
Soil Taxonomy (EUA, 1999)	Humaquept	Hapludox	Quartzipsamment	Mollic Hapludalf
Brazilian System of Soil Classification (Embrapa 2006)	Typic Aluminium-enriched Melanic Gleysol	Typic Dystrophic Red Latosol	Typic Orthic Quartzarenic Neosol	Chernozemic Eutroferric Red Argisol
Clay (g/kg)	640	630	80	460
Silt (g/kg)	280	120	20	220
Sand (g/kg)	80	250	900	320
MWHC1/ (g H2O/100g dry soil)	143	62	29	41
Microbial biomass (mg C/ 100 g soil)	185	94	25	136

[a] Maximum Water Holding Capacity

Metaflumizone was found to degrade in the four different Brazilian soils under aerobic conditions with half-lives ranging from 61 days to 205 days. ^{14}C- Metaflumizone (BAS 320 I) dropped from 98.4%, 96.6%, 100.1% and 98.7% of the total applied radioactivity (TAR) at 0 DAT to 53.8%, 35.7%, 67.8% and 56.0% in the GM, LVd, RQ and PV soils, respectively, after 118 days of aerobic incubation. For the GM, RQ and PV soils the maximum quantities of the degradation products M320I04 and M320I23 found during the incubation period were less than 10% of the TAR and for the LVd soil alone the maximum quantities of the degradation products M320I04 and M320I23 found after 118 days of incubation were 20.8% and 5.8% of the TAR, respectively. The half-life of ^{14}C- Metaflumizone (BAS 320 I) was 145 days in the GM soil, 61 days in the LVd soil, 205 days in the RQ soil and 155 days in the PV soil.

Table 2 Recovery and distribution of radioactivity during degradation of ^{14}C-Metaflumizone in GM soil and LVd (2018/3001301)

DAT	GM soil [% TAR]				LVd soil [% TAR]			
	ERR	NER	Volatiles*	Mass Balance	ERR	NER	Volatiles*	Mass Balance
0 (rep 1)	99.2	0.8	n.d.	99.9	101.2	0.6	n.d.	101.8
0 (rep 2)	99.3	0.7	n.d.	100.1	97.7	0.6	n.d.	98.2
0 (mean)	99.2	0.8	n.d.	100.0	99.4	0.6	n.d.	100.0
7	94.4	0.3	1.9	96.6	95.0	0.4	2.0	97.4
14	89.1	3.9	3.8	96.8	82.2	6.5	5.5	94.2
30	83.7	8.6	4.0	96.2	70.3	17.7	13.2	101.2
61 (rep1)	74.7	11.3	5.6	91.6	55.8	20.6	18.6	95.0
61 (rep2)	74.1	10.9	5.5	90.4	55.1	21.3	17.8	94.2
61 (mean)	74.4	11.1	5.5	91.0	55.4	21.0	18.2	94.6
89	69.4	13.9	7.8	91.1	46.1	20.9	23.9	90.9
118 (rep1)	64.6	15.9	10.5	91.0	41.6	25.3	30.7	97.6
118 (rep2)	64.0	16.3	10.5	90.9	40.2	25.7	30.7	96.7
118 (mean)	64.3	16.1	10.5	90.9	40.9	25.5	30.7	97.2

TAR: total applied radioactivity

DAT : days after treatment

ERR : extracted residual radioactivity

NER :non-extractable radioactivity (bound residue)

n.d.: not determined

rep: replicate

*: accumulated values

Table 3 Recovery and distribution of radioactivity during degradation of ^{14}C-Metaflumizone in RQ soil and PV soil (2018/3001301)

DAT	RQ soil [% TAR]				PV soil [% TAR]			
	ERR	NER	Volatiles*	Mass Balance	ERR	NER	Volatiles*	Mass Balance
0 (rep 1)	100.6	0.3	n.d.	100.9	97.2	0.8	n.d.	98.0
0 (rep 2)	98.9	0.2	n.d.	99.1	101.3	0.7	n.d.	102.0
0 (mean)	99.8	0.2	n.d.	100.0	99.3	0.7	n.d.	100.0
7	99.2	0.2	0.0	99.3	93.1	1.1	0.2	94.3
14	93.7	0.4	0.5	94.6	88.6	2.7	0.6	92.0
30	89.2	2.8	4.5	96.5	83.5	7.4	3.4	94.3
61 (rep1)	80.2	2.3	9.4	91.8	74.5	9.3	5.9	89.7
61 (rep2)	79.9	3.3	8.8	92.1	72.8	10.1	6.1	89.0
61 (mean)	80.1	2.8	9.1	92.0	73.6	9.7	6.0	89.3
89	75.3	3.9	12.9	92.0	67.7	12.0	9.4	89.1
118 (rep1)	69.5	13.0	15.5	98.1	59.7	18.3	11.4	89.4
118 (rep2)	69.0	11.9	15.5	96.4	56.9	20.4	11.3	88.7
118 (mean)	69.3	12.4	15.5	97.2	58.3	19.3	11.4	89.0

TAR: total applied radioactivity

DAT : days after treatment

ERR : extracted residual radioactivity

NER :non-extractable radioactivity (bound residue)

n.d.: not determined

rep: replicate

*: accumulated values

Table 4 Biotransformation of ^{14}C-Metaflumizone in soils (2018/3001301)

Day After Treatment	TAR[mg/kg]	%TAR						
		Total Extracted	M 320 I 04 tR ~ 43.8	M 320 I 23 tR ~ 45.0	Metaflumizone			Unknown compound
					(Z-Isomer) tR ~ 58.5	(E-Isomer) tR ~ 61.9	(Z) + (E)	
soil GM, (20 °C)								
0 (rep 1)	0.578	99.2	2.0	-	8.6	88.5	97.2	-
0 (rep 2)		99.3	0.9	-	8.0	90.4	98.4	-
0 (mean)		99.2	1.5	-	8.3	89.4	97.8	-
7		94.4	2.2	-	3.6	88.6	92.2	-
14		89.1	3.5	-	6.9	78.7	85.6	-
30		83.7	3.1	1.1	2.9	76.6	79.5	-
61(rep1)		74.7	5.6	0.3	2.7	66.1	68.8	-
61(rep2)		74.1	6.9	0.2	0.3	66.7	67.0	-
61(mean)		74.4	6.3	0.2	1.5	66.4	67.9	-
89		69.4	4.3	0.5	1.7	62.5	64.1	0.4
118(rep1)		64.6	-	6.4	0.8	56.9	57.8	0.5
118(rep2)		64.0	-	9.5	0.6	53.1	53.8	0.7
118(mean)		64.3	-	8.0	0.7	55.0	55.8	0.6
soil LVd, (20 °C)								
0 (rep 1)	0.590	101.2	4.6	-	9.0	87.6	96.6	-
0 (rep 2)		97.7	4.0	-	8.6	85.0	93.7	-
0 (mean)		99.4	4.3	-	8.8	86.3	95.2	-
7		95.0	3.9	-	2.3	88.8	91.0	-
14		82.2	5.8	-	4.3	72.1	76.4	-
30		70.3	15.8	1.0	0.9	52.7	53.6	-
61(rep1)		55.8	19.7	2.4	0.6	32.4	33.0	0.7
61(rep2)		55.1	20.8	0.6	1.2	32.5	33.7	-
61(mean)		55.4	20.2	1.5	0.9	32.4	33.3	0.3

Day After Treatment	TAR[mg/kg]	%TAR						
		Total Extracted	M 320 I 04 tR ~ 43.8	M 320 I 23 tR ~ 45.0	Metaflumizone			Unknown compound
					(Z-Isomer) tR ~ 58.5	(E-Isomer) tR ~ 61.9	(Z) + (E)	
89		46.1	3.9	1.9	2.7	37.6	40.3	-
118(rep1)		41.6	-	5.8	0.3	35.4	35.7	0.2
118(rep2)		40.2	1.0	1.1	1.3	36.3	37.6	0.6
118(mean)		40.9	0.5	3.4	0.8	35.9	36.6	0.4
soil RQ, (20 °C)								
0 (rep 1)	0.602	100.6	0.6	-	8.0	92.0	100.1	-
0 (rep 2)		98.9	1.5	-	8.2	89.1	97.4	-
0 (mean)		99.8	1.1	-	8.1	90.6	98.7	-
7		99.2	0.6	-	8.0	90.6	98.6	-
14		93.7	0.5	0.4	8.6	84.2	92.8	-
30		89.2	0.4	-	6.6	82.2	88.8	-
61(rep1)		80.2	0.5	0.3	3.0	76.3	79.3	-
61(rep2)		79.9	0.8	0.2	3.4	75.6	79.0	-
61(mean)		80.1	0.7	0.3	3.2	75.9	79.1	-
89		75.3	5.4	0.6	1.3	68.0	69.3	-
118(rep1)		69.5	0.2	0.2	2.3	66.6	68.9	0.3
118(rep2)		69.0	0.6	0.5	0.6	67.3	67.8	0.0
118(mean)		69.3	0.4	0.4	1.4	66.9	68.4	0.1
soil PV, (20 °C)								
0 (rep 1)	0.611	97.2	1.0	-	7.1	89.2	96.3	-
0 (rep 2)		101.3	2.7	-	6.6	92.0	98.7	-
0 (mean)		99.3	1.8	-	6.9	90.6	97.5	-
7		93.1	0.8	-	6.6	84.0	90.6	1.7
14		88.6	2.0	0.3	6.7	79.1	85.7	0.6
30		83.5	-	1.4	4.0	77.6	81.7	0.4
61(rep1)		74.5	1.0	4.2	1.6	66.8	68.4	0.8
61(rep2)		72.8	0.3	1.6	2.8	67.7	70.5	0.3
61(mean)		73.6	0.7	2.9	2.2	67.3	69.5	0.6
89		67.7	-	3.3	2.0	62.4	64.4	-
118(rep1)		59.7	0.2	0.5	0.6	58.5	59.1	-
118(rep2)		56.9	0.2	0.8	0.5	55.4	56.0	-
118(mean)		58.3	0.2	0.6	0.6	56.9	57.5	-

TAR: Total Applied Radioactivity

tR： retention time [min]

rep: replicate

mean: mean of replicate values

-: means no detected peak

Stability of pesticide residues in stored analytical samples

A storage stability study was conducted to determine the stability of metaflumizone residues in cucumber, sunflower seed, snap bean (succulent seed), potato tuber and strawberry plant samples, following field treatment with a 240 g/L SC and, stored under frozen conditions (Stewart J., 2012 a 2010/7013133).

For each crop, one treated plot and one untreated control plot were established. The treated plot received three broadcast applications of metaflumizone at a rate of 1.2 kg ai/ha per application, with a 6–10 day retreatment interval, for an exaggerated total seasonal rate of 3.7 kg ai/ha. The applications were typical foliar sprays except for potato, for which the applications were made to mature tubers placed on the soil surface in order to ensure obtaining residues in the samples. All applications were made in a spray volume of 183–191 L/ha of water, with an adjuvant at a rate targeting 1% v/v in the spray mixture. The crop RAC samples (cucumber (fruit), sunflower (seed), snap bean (succulent seed), potato (tuber), and strawberry (berry) were harvested (by hand) 2 and 7 days after the last application. Duplicate samples were collected from the untreated plot at the sample interval corresponding to 2

DALA, and two independent treated samples were collected from the treated plot at each sampling interval. Each RAC sample was commercially acceptable and weighed a minimum of 0.45 kg. All samples were received frozen from the field and were stored in a freezer (<-5 °C) prior to homogenization and analysis.

The data indicate that residues of metaflumizone are stable at <-5 °C for at least 729 to 971 days (24–32 months) in field-treated cucumber (fruit), sunflower (seed), snap bean (succulent seed), potato (tuber), and strawberry (berry) samples.

Table 5 Storage stability of metaflumizone (E-isomer) in plant matrices

A: in stored samples, % remaining of residues B: procedural, in freshly spiked sample										
	A	B	A	B	A	B	A	B	A	B
Day	Cucumber (fruit)		Sunflower (seed)		Snap bean (succulent seed)		Potato (tuber)		Strawberry (berry)	
0, 2	100	87	100	83	100	87	100	97	100	90
20-66	88	89	-	-	111	114	101	87	99	91
77-91	-	-	74	75	90	87	116	107	77	76
140-210	111	89	85	91	92	89	114	94	85	97
331-417	82	102	94	99	113	106	85	94	92	102
525-545	97	100	-	-	103	98	-	-	98	111
729-971	91	99	86	79	102	98	95	105	84	94

Table 6 Storage stability of metaflumizone (Z-isomer) in plant matrices

A: in stored samples, % remaining of residues B: procedural, in freshly spiked sample										
	A	B	A	B	A	B	A	B	A	B
Day	Cucumber (fruit)		Sunflower (seed)		Snap bean (succulent seed)		Potato (tuber)		Strawberry (berry)	
0, 2	100	84	100	82	100	97	100	91	100	85
20-31	-	-	-	-	101	95	98	98	89	83
40-66	79	91	-	-	-	-	-	-	109	77
77-91	-	-	84	81	90	92	100	91	94	73
140-210	104	87	80	99	90	88	107	94	100	92
331-417	86	98	90	98	89	96	90	99	90	87
525-545	92	103	-	-	90	100	-	-	84	98
729-971	94	102	55	65	88	91	94	88	80	94

Table 7 Storage stability of M320I04 in plant matrices

A: in stored samples, % remaining of residues B: procedural, in freshly spiked sample										
	A	B	A	B	A	B	A	B	A	B
Day	Cucumber (fruit)		Sunflower (seed)		Snap bean (succulent seed)		Potato (tuber)		Strawberry (berry)	
0, 2	100	80	100	62	100	94	100	86	100	84
20-31	-	-	-	-	-	78	71	88	126	82
34	-	-	-	-	-	-	-	-	182	65
40-66	-	73	-	-	-	-	-	-	168	73
77-91	-	-	-	91	-	71	69	105	182	86
140-207	-	95	-	108	-	106	93	86	227	82
210	-	-	-	-	-	-	-	-	172	87
331-417	-	97	-	93	-	81	82	82	161	81
525-545	-	99	-	-	-	81	-	-	223	86

A: in stored samples, % remaining of residues B: procedural, in freshly spiked sample										
	A	B	A	B	A	B	A	B	A	B
Day	Cucumber (fruit)		Sunflower (seed)		Snap bean (succulent seed)		Potato (tuber)		Strawberry (berry)	
729-971	-	94	-	75	-	91	88	88	214	88

Table 8 Storage stability of M320I23 in plant matrices

A: in stored samples, % remaining of residues B: procedural, in freshly spiked sample										
	A	B	A	B	A	B	A	B	A	B
Day	Cucumber (fruit)		Sunflower (seed)		Snap bean (succulent seed)		Potato (tuber)		Strawberry (berry)	
0, 2	100	107	100	84	100	111	100	96	100	92
20-31	-	-	-	-	-	89	117	98	-	88
34	-	-	-	-	-	-	-	-	-	67
40-66	-	97	-	-	-	-	-	-	-	97
77-91	-	-	-	86	-	103	138	118	-	81
140-210	-	-	-	83	-	124	102	103	-	89
331-417	-	107	-	105	-	100	82	109	-	90
525-545	-	100	-	-	-	92	-	-	-	100
729-971	-	94	-	116	-	107	86	95	-	96

USE PATTERN

Metaflumizone is registered in many countries for use in fruits, vegetables, cereals and tree nuts. The information considered by the Meeting on registered uses is summarized in Table 9.

Table 9 Registered uses of metaflumizone

Crop	Country	Formulation		Application				PHI (day)	Remarks
		g ai/L	type	Method	Rate (kg ai/ha)	Appl. interval	No. per season		
Citrus fruits (Group 001)									
Citrus fruits FC 0001	Brazil	240	CS	spraying	0.384-0.48	7 days	3	7	Water volume 2000L/ha
Pome fruits (Group 002)									
Apple FP 0226	Brazil	240	CS	spraying	0.192-0.24	7 days	4	3	Water volume 1000L/ha
Apple FP 0226	Korea	240	CS	spraying	0.16-0.32	10 days	3	14	Water volume 1-2000L/ha
Other small fruited berries (Subgroup 004C)									
Grape FB 0269	Brazil	240	CS	spraying	0.144-0.24	7 days	3	3	Water volume 1000L/ha
Cucurbit-inedible peel (Subgroup 011B)									
Melon, except watermelon VC0046	Brazil	240	CS	spraying	0.154-0.192	7 days	5	3	Water volume 800L/ha
Watermelon VC0432	Brazil	240	CS	spraying	0.154-0.192	7 days	5	3	Water volume 800L/ha
Pulses (Subgroup 015)									
Soybean VD 0541	Brazil	240	CS	spraying	0.192-0.24	7 days	3	14	Water volume 200L/ha
Cereal grains (Subgroup 020)									
Maize GC 0645	Brazil	240	CS	spraying	0.12-0.24	7 days	5	14	Water volume 200L/ha
Grasses for sugar or syrup production (Subgroup 021)									
Sugarcane GS 0659	Brazil	240	CS	In furrow at planting	0.192-0.24	Not applicable	1	Not defined (due to mode of application)	Water volume 200L/ha
Seed for beverage and sweet (Subgroup 024)									
Coffee beans SB0716	Brazil	240	CS	spraying	0.36-0.48	30 days	2	45	Water volume 200-400L/ha

RESULTS OF SUPERVISED RESIDUE TRIALS ON CROPS

Supervised trials were provided to support the estimation of maximum residue levels for metaflumizone when used for foliar application on citrus fruits, apple, grape, melon, watermelon, maize, soya bean and coffee, and as a furrow use in sugarcane.

Supervised field trials were conducted in Brazil. Each trial consisted of one treated and one control plot. A metaflumizone 240 g/L SC formulation was used for the foliar applications. All samples were stored at -20°C for periods less than the intervals of demonstrated storage stability for metaflumizone E-isomer and metaflumizone Z-isomer. The residues of metaflumizone E-isomer and

Z-isomer were determined using LC-MS/MS with method BASF 531/1. The method was previously reviewed as suitable for all commodities with LOQs of 0.01 mg/kg for each analyte.

Citrus fruits

The field trials were conducted on citrus fruits (orange and lemon) in Brazil during the 2012 and 2013 growing season. Each trial consisted of one treated and one control plot. Metaflumizone 240 g/L SC formulation was foliar applied three times at rates of 0.48 kg ai/ha in spray volumes of 2000 L/ha. Control and treated samples were harvested 7 days after the last treatment (DALA) for the harvest trials and at 0, 7, 14 and 21 DALA in the decline trials. Samples were kept at -20 °C until analysis. All orange and lemon samples were stored for less than 395 days. The residues of metaflumizone E-isomer and Z-isomer in citrus fruits were determined using LC-MS/MS method BASF 531/1 with LOQs of 0.01 mg/kg for each analyte.

Oranges

Table 10 Residues of metaflumizone in orange after foliar application of 240g /L SC

Country, Year, Location, Variety, Trial No.	Application				Portion analysed	Residue mg/kg			Study Reference
	Method	No (int, days)	Rate kg ai/ha	DALA		E-isomer	Z-isomer	Total	
Brazil, 2011, Sao Paulo Aguai, Natal, G100279	foliar	3 (10,9)	0.48	0	Whole Fruit	0.83	0.86	1.69	2012/3003761
				7	Whole Fruit	0.49	0.86	1.35	
				14	Whole Fruit	0.41	0.69	1.1	
0Brazil, 2011, Sao Paulo Santo Antonio de Posse, Pera-coroa, G100679	foliar	3 (10,9)	0.48	0	Whole Fruit	0.62	0.47	1.09	2012/3003761
				7	Whole Fruit	0.43	0.58	1.01	
				14	Whole Fruit	0.29	0.45	0.74	
Brazil, 2011, Parana Londrina, Pera-Rio, G100680	foliar	3 (11,10)	0.48	7	Whole Fruit	0.32	0.34	0.66	2012/3003761
Brazil, 2011, Sao Paulo Jaboticabal, Pera, G100681	foliar	3 (10,11)	0.48	7	Whole Fruit	0.08	0.14	0.22	2012/3003761
Brazil, 2012, Sao Paulo Santo Antonio de Posse, Hamelin, G110286	foliar	3 (13,7)	0.12	0	Whole Fruit	0.18	0.26	0.44	2014/3000341
				7	Whole Fruit	0.12	0.25	0.37	
				14	Whole Fruit	<0.01	<0.01	<0.02	
				21	Whole Fruit	0.06	0.13	0.19	
Brazil, 2012, Sao Paulo Santo Antonio de Posse, Hamelin, G110286	foliar	3 (10,11)	0.12	7	Peel Pulp	0.6 <0.01	1.66 <0.01	2.26 <0.02	2014/3000341
Brazil, 2012, Sao Paulo Jaboticabal, Pera, G110287	foliar	3 (11,10)	0.48	0	Whole Fruit	0.18	0.36	0.53	2014/3000341
				7	Whole Fruit	0.12	0.3	0.42	
				14	Whole Fruit	0.09	0.29	0.38	
				21	Whole Fruit	0.09	0.25	0.34	

Country, Year, Location, Variety, Trial No.	Application				Portion analysed	Residue mg/kg			Study Reference
	Method	No (int, days)	Rate kg ai/ha	DALA		E-isomer	Z-isomer	Total	
Brazil, 2012, Sao Paulo Jaboticabal, Pera, G110287	foliar	3 (9,11)	0.48	7	Peel Pulp	0.79 <0.01	2.34 0.02	3.13 <0.03	2014/3000341
Brazil, 2012, Sao Paulo Aguai, Westin, G110288	foliar	3 (9,11)	0.48	7	Whole Fruit	0.35	0.86	1.21	2014/3000341
Brazil, 2013, Parana Tamarana, Pera Rio, G110292	foliar	3 (10,10)	0.48	7	Whole Fruit	0.32	0.52	0.84	2014/3000341
Brazil, 2013, Parana Londrina, Pera Rio, G110293	foliar	3 (10,9)	0.48	7	Whole Fruit	0.17	0.25	0.42	2014/3000341
Brazil, 2012, Sao Paulo Rio Claro, Laranja Pera, G110294	foliar	3 (10,8)	0.48	7	Whole Fruit	0.24	0.47	0.71	2014/3000341
Brazil, 2013, Parana Jataizinho, Pera Rio, G110295	foliar	3 (10,10)	0.48	7	Whole Fruit Peel Pulp	0.09 0.62 <0.01	0.13 0.96 <0.01	0.22 1.58 <0.02	2014/3000341
Brazil, 2013, Sao Paulo Mogi Mirim, Pera Coroa, G110355	foliar	3 (10,9)	0.48	0	Whole Fruit	0.27	0.24	0.51	2014/3000341
				7	Whole Fruit	0.08	0.13	0.21	
				14	Whole Fruit	0.13	0.21	0.34	
				21	Whole Fruit	0.1	0.13	0.23	

Lemon

Table 11 Residues of metaflumizone in lemon after foliar application of a 240 g /L SC formulation

Country, Year, Location, Variety, Trial No.	Application				Portion analysed	Residue mg/kg			Study Reference
	Method	No (Int, days)	Rate kg ai/ha	DALA		E-ISOMER	Z-ISOMER	Total	
Brazil, 2012, Sao Paulo Limeira, Tahiti, G110289	foliar	3 (9,11))	0.48	0	Whole Fruit	0.19	0.11	0.3	2014/3000341
				7	Whole Fruit	0.06	0.11	0.17	
				14	Whole Fruit	0.06	0.16	0.22	
				21	Whole Fruit	0.09	0.21	0.3	
Brazil, 2013, Parana Cambe, Tahiti, G110290	foliar	3 (10,10)	0.48	0	Whole Fruit	0.11	0.07	0.18	2014/3000341
				7	Whole Fruit	0.11	0.14	0.25	
				14	Whole Fruit	0.1	0.15	0.25	
				21	Whole Fruit	0.11	0.16	0.27	
	foliar	3	0.48	0	Whole Fruit	0.35	0.19	0.54	2014/3000341

Country, Year, Location, Variety, Trial No.	Application				Portion analysed	Residue mg/kg			Study Reference
	Method	No (Int, days)	Rate kg ai/ha	DALA		E-ISOMER	Z-ISOMER	Total	
Brazil, 2013, Parana Cornelio Procopio, Tahiti, G110291		(10,10)		7	Whole Fruit	0.19	0.22	0.41	
				14	Whole Fruit	0.21	0.31	0.52	
				21	Whole Fruit	0.08	0.1	0.18	
Brazil, 2012, Sao Paulo Itapolis, Tahiti, G110296	foliar	3 (10,10)	0.48	7	Whole Fruit Peel Pulp	0.32 1.06 0.04	0.59 2.35 0.05	0.91 3.41 0.09	2014/3000341
Brazil, 2012, Sao Paulo Pirangi, Tahiti, G110297	foliar	3 (10,10)	0.48	7	Whole Fruit Peel Pulp	0.35 1.03 0.01	0.71 2.84 0.02	1.06 3.87 0.03	2014/3000341

Pome fruits

Apple

The field trials were conducted on apple in Brazil during the 2012 and 2013 growing season. Each trial consisted of one treated and one control plot. Metaflumizone 240 g/L SC formulation was foliar applied four times at rates of 0.24 kg ai/ha in spray volumes of 1000 L/ha. Control and treated samples were harvested at 3 DALA and additionally at 0, 1, 7 and 10 DALA in the decline trials. Samples were kept at or below -20°C until analysis. The apple samples were stored for up to 351 days prior to analysis. The residues of metaflumizone E-isomer and Z-isomer in apple were determined using LC-MS/MS method BASF 531/1 with LOQs of 0.01 mg/kg for each analyte.

Table 12 Residues of metaflumizone in apple after foliar application of a 240g /L SC formulation

Country, Year, Location, Variety, Trial No.	Application				Portion analysed	Residue mg/kg			
	Method	No (int. days)	Rate kg ai/ha	DALA		E-ISOMER	Z-ISOMER	Total	Study Reference
Brazil, 2009, Parana, Campo do Tenente, Imperial Gala, G090291	foliar	4 (7,7,7)	0.24	0	whole fruit	0.18	0.17	0.35	2013/1043077
				1	whole fruit	0.13	0.19	0.32	
				3	whole fruit	0.13	0.2	0.33	
				7	whole fruit	0.1	0.15	0.25	
				10	whole fruit	0.06	0.1	0.16	
Brazil, 2009, Parana, Ponto Amazonas, Gala Royall, G090292	foliar	4 (7,7,7)	0.24	0	whole fruit	0.17	0.17	0.34	2013/1043077
				1	whole fruit	0.17	0.21	0.38	
				3	whole fruit	0.11	0.19	0.3	
				7	whole fruit	0.09	0.16	0.25	
				10	whole fruit	0.06	0.09	0.15	
Brazil, 2009, Santa Catarina, Farburgo, Max Gala, G090293	foliar	4 (7,7,8)	0.24	3	whole fruit	0.1	0.14	0.24	2013/1043077

Country, Year, Location, Variety, Trial No.	Application				Portion analysed	Residue mg/kg			
	Method	No (int. days)	Rate kg ai/ha	DALA		E-ISOMER	Z-ISOMER	Total	Study Reference
Brazil, 2010, Santa Catarina, São Joaquim, Fugi, G090434	foliar	4 (7,7,7)	0.24	3	whole fruit	0.09	0.16	0.25	2013/1043077
Brazil, 2011, Parana, Campo Tenente, Gala, G110160	foliar	4 (7,7,7)	0.24	0	whole fruit	0.1	0.11	0.21	2013/3012922
				1	whole fruit	0.07	0.09	0.16	
				3	whole fruit	0.06	0.1	0.16	
				7	whole fruit	0.03	0.06	0.09	
				10	whole fruit	0.02	0.04	0.06	
Brazil, 2011, Santa Catarina, Fraiburgo, Gala, G110161	foliar	4 (7,7,7)	0.24	3	whole fruit	0.07	0.12	0.19	2013/3012922
Brazil, 2012, Parana, Guaragi, Eva, G110335	foliar	4 (7,7,7)	0.24	0	whole fruit	0.14	0.12	0.26	2013/3012922
				1	whole fruit	0.09	0.15	0.24	
				3	whole fruit	0.06	0.11	0.17	
				7	whole fruit	0.06	0.1	0.16	
				10	whole fruit	0.05	0.09	0.14	
Brazil, 2012, Parana, Urai, Eva, G110336	foliar	4 (7,7,7)	0.24	0	whole fruit	0.29	0.27	0.56	2013/3012922
				1	whole fruit	0.23	0.3	0.53	
				3	whole fruit	0.15	0.28	0.43	
				7	whole fruit	0.08	0.15	0.23	
				10	whole fruit	0.07	0.14	0.21	
Brazil, 2012, Parana, Campo Tenente, Gala, G110337	foliar	4 (7,7,7)	0.24	3	whole fruit	0.09	0.13	0.22	2013/3012922
Brazil, 2012, Santa Catarina, Fraiburgo, Gala, G110338	foliar	4 (7,7,7)	0.24	3	whole fruit	0.22	0.32	0.54	2013/3012922
Brazil, 2012, Parana, Ibipora, Eva, G110339	foliar	4 (7,7,7)	0.24	3	whole fruit	0.17	0.31	0.48	2013/3012922
Brazil, 2012, Santa Catarina, Sao Joaquim, Fuji, G110351	foliar	4 (8,8,6)	0.24	0	whole fruit	0.24	0.26	0.5	2013/3012922
				1	whole fruit	0.22	0.21	0.43	
				3	whole fruit	0.16	0.36	0.52	
				7	whole fruit	0.08	0.21	0.29	
				10	whole fruit	0.1	0.24	0.34	

Grape

The field trials were conducted on grapes in Brazil during the 2011 and 2012 growing seasons. Each trial consisted of one treated and one control plot. Three foliar applications of a metaflumizone 240 g/L SC formulation were made at rates of 0.24 kg ai/ha in spray volumes of 1000 L/ha. Control and treated samples were harvested at 3 DALA and additionally at 0, 7, 14 and 21 DALA in the decline trials. Samples were kept at or below -20 °C until analysis. Grape samples were stored for up to 450 days prior to analysis. The residues of metaflumizone E-isomer and Z-isomer in grapes were determined using LC-MS/MS method BASF 531/1 with LOQs of 0.01 mg/kg for each analyte.

Table 13 Residues of metaflumizone in grapes after foliar application of a 240g /L SC formulation

CROP, Country, Year, Location, Variety, Trial No.	Application			DALA	Portion analysed	Residue mg/kg			
	Method	No (int. days)	Rate kg ai/ha			E-ISOMER	Z-ISOMER	Total	Study Reference
Brazil, 2010, Parana, Ponta Grossa, Niagara Branca, G100206	foliar	3 (7,7)	0.24	0	fruit	1.15	0.78	1.93	2012/3003762
				3	fruit	0.64	0.76	1.4	
				7	fruit	0.55	0.76	1.31	
Brazil, 2011, Parana, Londrina, Benitaka, G100207	foliar	3 (7,7)	0.24	0	fruit	0.98	0.57	1.55	2012/3003762
				3	fruit	0.73	0.72	1.45	
				7	fruit	0.83	0.89	1.72	
Brazil, 2010, Santa Catarina, Videira, Italia, G100208	foliar	3 (7,8)	0.24	3	fruit	0.3	0.33	0.63	2012/3003762
Brazil, 2011, Sao Paulo, Jundiai, Niagara Rosada, G100209	foliar	3 (7,8)	0.24	3	fruit	0.12	0.15	0.27	2012/3003762
Brazil, 2011, Parana, Ponta Grossa, Niagara Branca, G110162	foliar	3 (8,6)	0.24	0	fruit	1.76	0.89	2.65	2013/3014221
				3	fruit	1.47	1.24	2.71	
				7	fruit	1.04	1.2	2.24	
				14	fruit	0.81	1.14	1.95	
				21	fruit	0.71	1.1	1.81	
Brazil, 2011, Sao Paulo, Jundiai, Niagara Rosada, G110163	foliar	3 (7,7)	0.24	0	fruit	0.47	0.29	0.76	2013/3014221
				3	fruit	0.23	0.28	0.51	
				7	fruit	0.2	0.23	0.43	
				14	fruit	0.15	0.18	0.33	
				21	fruit	0.18	0.26	0.44	
Brazil, 2012, Pernambuco, Petrolina, Italia, G110164	foliar	3 (7,7)	0.24	3	fruit	0.58	0.81	1.39	2013/3014221
Brazil, 2012, Parana, Rolandia, Benitaka, G110329	foliar	3 (7,7)	0.24	0	fruit	0.62	0.39	1.01	2013/3014221
				3	fruit	0.38	0.38	0.76	
				7	fruit	0.56	0.65	1.21	
				14	fruit	0.3	0.57	0.87	

CROP, Country, Year, Location, Variety, Trial No.	Application			DALA	Portion analysed	Residue mg/kg			
	Method	No (int. days)	Rate kg ai/ha			E-ISOMER	Z-ISOMER	Total	Study Reference
				21	fruit	0.19	0.29	0.48	
Brazil, 2012, Sao Paulo, Taiacu, Niagara Rosada, G110330	foliar	3 (7,7)	0.24	0	fruit	1.16	0.66	1.82	2013/3014221
				3	fruit	1.08	0.76	1.84	
				7	fruit	0.71	0.74	1.45	
				14	fruit	0.75	0.91	1.66	
				21	fruit	0.46	0.61	1.07	
Brazil, 2012, Parana, Urai, Rubi, G110331	foliar	3 (7,7)	0.24	3	fruit	0.38	0.37	0.75	2013/3014221
Brazil, 2012, Parana, Cambe, Niagara, G110332	foliar	3 (7,7)	0.24	3	fruit	0.08	0.07	0.15	2013/3014221
Brazil, 2012, Sao Paulo, Indaiatuba, Niagara, G110333	foliar	3 (7,7)	0.24	3	fruit	0.35	0.29	0.64	2013/3014221

Melon

The field trials were conducted on melons in Brazil during the 2012 and 2013 growing seasons. Five foliar applications of a metaflumizone 240 g/L SC were made at rates of 0.24 kg ai/ha, in spray volumes of 1000 L/ha. Control and treated samples were harvested at 3 DALA and additionally at 0, 1, 7 and 10 DALA in decline trials. For the 3 DALA samples, the fruits were cut in longitudinal and transverse sections, and the two equidistant sides were sampled as whole fruit and the remaining two sides were sampled as peel and pulp. All samples were double bagged and placed in a freezer on the date of collection. Samples were kept at or below -20 °C until analysis. Melon samples were stored for up to 299 days prior to analysis. The residues of metaflumizone E-isomer and Z-isomer in melon were determined using LC-MS/MS method BASF 531/1 with LOQs of 0.01 mg/kg for each analyte.

Table 14 Residues of metaflumizone in melon after foliar application of a 240g /L SC formulation

CROP, Country, Year, Location, Variety, Trial No.	Application			DALA	Portion analysed	Residue mg/kg			Study Reference
	Method	No (int, days)	Rate kg ai/ha			E-ISOMER	Z-ISOMER	Total	
Brazil, 2011, Parana, Ibipora, Louis, G090307	foliar	5 (7,7,7,7)	0.192	0	Whole fruit	0.11	0.1	0.21	2012/3003764
				1	Whole fruit	0.13	0.14	0.27	
				3	Whole fruit	0.06	0.08	0.14	
				7	Whole fruit	0.02	0.03	0.05	
				10	Whole fruit	0.04	0.05	0.09	
Brazil, 2010, Goias, Senador Canedo,	foliar	5 (7,7,7,7)	0.24	0	Whole fruit	0.04	0.08	0.12	2012/3003764
				1	Whole fruit	0.04	0.07	0.11	
				3	Whole fruit	0.03	0.07	0.1	

CROP, Country, Year, Location, Variety, Trial No.	Application			DALA	Portion analysed	Residue mg/kg			Study Reference
	Method	No (int, days)	Rate kg ai/ha			E-ISOMER	Z-ISOMER	Total	
Gaucho, G090308				7	Whole fruit	0.01	0.03	0.04	
				10	Whole fruit	<0.01	0.02	<0.03	
Brazil, 2010, Sao Paulo, Santo Antonio de Posse, Sunrise, G090309	foliar	5 (7,7,7,7)	0.24	3	Whole fruit	0.11	0.18	0.29	2012/3003764
Brazil, 2010, Rio Grande do Norte, Mossoro, Colderx, G090310	foliar	5 (7,7,7,7)	0.24	3	Whole fruit	0.03	0.04	0.07	2012/3003764
Brazil, 2011, Parana, Londrina, Louis, G090311	foliar	5 (7,7,7,7)	0.24	3	Whole fruit	0.28	0.33	0.61	2012/3003764
Brazil, 2012, Pernambuco, Petrolina, Amarelo, G120078	foliar	5 (7,8,6,7)	0.24	0	Whole fruit	0.02	0.02	0.04	2013/3014222
				1	Whole fruit	0.01	0.02	0.03	
				3	Whole fruit	<0.01	<0.01	<0.02	
				7	Whole fruit	<0.01	<0.01	<0.02	
				10	Whole fruit	<0.01	<0.01	<0.02	
Brazil, 2012, Pernambuco, Petrolina, Amarelo, G120078	foliar	5 (7,8,6,7)	0.24	3	Peel Pulp	0.02 <0.01	0.06 <0.01	0.08 <0.02	2013/3014222
Brazil, 2012, Pernambuco, Assai, Louis, G120080	foliar	5 (7,7,7,7)	0.24	0	Whole fruit	0.12	0.12	0.24	2013/3014222
				1	Whole fruit	0.09	0.12	0.21	
				3	Whole fruit	0.08	0.12	0.2	
				7	Whole fruit	0.06	0.11	0.17	
				10	Whole fruit	0.07	0.12	0.19	
Brazil, 2012, Pernambuco, Assai, Louis, G120080	foliar	5 (7,7,7,7)	0.24	3	Peel Pulp	0.29 <0.01	0.6 <0.01	0.89 <0.02	2013/3014222
Brazil, 2012, Bahia, Sobradinho, Pele de Sapo, G120081	foliar	5 (7,7,7,7)	0.24	3	Peel Pulp Whole Fruit	0.02 <0.01 <0.01	0.02 <0.01 <0.01	0.04 <0.02 <0.02	2013/3014222

Soya bean

The field trials were conducted on soya bean in Brazil during the 2010 and 2011 growing seasons. Each trial consisted of one treated and one control plot. Three foliar applications of a metaflumizone 240 g/L SC were made at rates of 0.24 kg ai/ha in spray volumes of 200 L/ha. Control and treated soya bean seed were harvested at 14 DALA (BBCH 83–89) and additionally at 0, 7 and 21 DALA in decline trials. Samples were kept at or below -20 °C until analysis. Soya bean grain samples were stored for up to 365 days prior to analysis. The residues of metaflumizone E-isomer and Z-isomer in soya bean were determined using LC-MS/MS method BASF 531/1 with LOQs of 0.01 mg/kg for each analyte.

Table 15 Residues of metaflumizone in soya bean seeds after foliar application of a 240 g/L SC formulation

CROP, Country, Year, Location, Variety, Trial No.	Application			DALA	Portion analysed	Residue mg/kg			Study Reference
	Method	No (int. days)	Rate kg ai/ha			E-ISOMER	Z-ISOMER	Total	
Brazil, 2010, Sao Paulo, Santo Antonio de Pesse, Monsoy, G090261	foliar	3 (9,11)	0.24	0	Seed	0.13	0.15	0.28	2013/1043078
				6	Seed	0.07	0.09	0.16	
				14	Seed	0.03	0.04	0.07	
				21	Seed	0.01	0.02	0.03	
				28	Seed	0.02	0.02	0.04	
Brazil, 2010, Parana, Ponta Grossa, BRS-232, G090262	foliar	3 (10,10)	0.24	0	Seed	0.02	0.02	0.04	2013/1043078
				7	Seed	<0.01	<0.01	<0.02	
				14	Seed	<0.01	<0.01	<0.02	
				21	Seed	<0.01	<0.01	<0.02	
				28	Seed	<0.01	<0.01	<0.02	
Brazil, 2010, Goias, Ardpolis, M-SOY RR 7908, G090263	foliar	3 (10,9)	0.24	14	Seed	<0.01	0.01	0.02	2013/1043078
Brazil, 2010, Goias, Senader Canedo, M-SOY RR 7908, G090264	foliar	3 (10,10)	0.24	14	Seed	<0.01	0.02	0.03	2013/1043078
Brazil, 2011, Parana, Ponta Grossa, Innox, G100563	foliar	3 (9,10)	0.24	0	Seed	0.03	0.04	0.07	2014/3002726
				7	Seed	0.02	0.03	0.05	
				14	Seed	<0.01	0.01	0.02	
				21	Seed	<0.01	<0.01	<0.02	
Brazil, 2010, Goias, Senador Canedo, BRSGO7560, G100564	foliar	3 (9,10)	0.24	0	Seed	0.06	0.05	0.11	2014/3002726
				7	Seed	0.02	0.02	0.04	
				14	Seed	<0.01	<0.01	<0.02	
				21	Seed	<0.01	<0.01	<0.02	
Brazil, 2011, Goias, Anapolis, BRSGO7560, G100565	foliar	3 (6,10)	0.24	14	Seed	<0.01	<0.01	<0.02	2014/3002726

CROP, Country, Year, Location, Variety, Trial No.	Application			DALA	Portion analysed	Residue mg/kg			Study Reference
	Method	No (int. days)	Rate kg ai/ha			E-ISOMER	Z-ISOMER	Total	
Brazil, 2011, Sao Paulo, Santo Antonio de Posse, Innox, G100566	foliar	3 (10,10)	0.24	14	Seed	0.03	0.08	0.11	2014/3002726

Cereal Grain

Maize (field)

The field trials were conducted on maize in Brazil during the 2010 and 2011 growing seasons. Each trial consisted of one treated and one control plot. Five foliar applications of a metaflumizone 240 g/L SC were made at rates of 0.24 kg ai/ha in spray volumes of 300 L/ha. Control and treated samples were harvested at 14 DALA and additionally at 0, 7 and 21 DALA in decline trials. Samples were kept at or below -20 °C until analysis. Maize grain samples were stored for up to 160 days prior to analysis. The residues of metaflumizone E-isomer and Z-isomer in maize were determined using a modified version of LC-MS/MS method BASF 531/1 with LOQs of 0.01 mg/kg for each analyte.

Table 16 Residues of metaflumizone in maize grains after foliar application of a 240g /L SC formulation

CROP, Country, Year, Location, Variety, Trial No.	Application			DALA	Portion analysed	Residue mg/kg			Study Reference
	Method	No (int. days)	Rate kg ai/ha			E-ISOMER	Z-ISOMER	Total	
Brazil, 2010, Sao Paulo, Santo Antonio de Posse, Ag 700 Gieldgard, G090273	foliar	5 (7,8,6,7)	0.24	0	Grain	<0.01	<0.01	<0.02	2012/3003401
				7	Grain	<0.01	<0.01	<0.02	
				14	Grain	<0.01	<0.01	<0.02	
				21	Grain	<0.01	<0.01	<0.02	
				28	Grain	<0.01	<0.01	<0.02	
Brazil, 2010, Parana, Ponta Grossa, 2A 120, G090274	foliar	5 (7,7,7,7)	0.24	0	Grain	<0.01	<0.01	<0.02	2012/3003401
				7	Grain	<0.01	<0.01	<0.02	
				14	Grain	<0.01	<0.01	<0.02	
				21	Grain	<0.01	<0.01	<0.02	
				28	Grain	<0.01	<0.01	<0.02	
Brazil, 2010, Goias, Senador Canedo , Engopa 501, G090275	foliar	5 (7,7,7,8)	0.24	14	Grain	<0.01	<0.01	<0.02	2012/3003401
Brazil, 2010, Goias, Anapolis , BRS 1030, G090276	foliar	5 (7,7,7,7)	0.24	14	Grain	<0.01	<0.01	<0.02	2012/3003401
Brazil, 2011, Goias, Senador Canedo, Yielogard, G100567	foliar	5 (7,7,7,6)	0.24	0	Grain	<0.01	<0.01	<0.02	2012/3003763
				7	Grain	<0.01	<0.01	<0.02	
				14	Grain	<0.01	<0.01	<0.02	
				21	Grain	<0.01	<0.01	<0.02	

CROP, Country, Year, Location, Variety, Trial No.	Application			DALA	Portion analysed	Residue mg/kg			Study Reference
	Method	No (int. days)	Rate kg ai/ha			E-ISOMER	Z-ISOMER	Total	
Brazil, 2011, Sao Paulo, Santo Antonio de Posse, Dow 2B710CL, G100568	foliar	5 (7,7,7,6)	0.24	14	Grain	<0.01	<0.01	<0.02	2012/3003763
Brazil, 2011, Parana, Cambe, Cargo, G100677	foliar	5 (7,7,7,7)	0.24	0	Grain	0.06	<0.01	0.07	2012/3003763
				7	Grain	0.04	<0.01	0.05	
				14	Grain	0.01	<0.01	0.02	
				21	Grain	<0.01	<0.01	<0.02	
Brazil, 2011, Parana, Ibipora, Cargo, G100678	foliar	5 (7,7,7,7)	0.24	14	Grain	<0.01	<0.01	<0.02	2012/3003763

Sugarcane

Field trials on sugar cane were conducted in Brazil during the 2012 and 2013 growing seasons. Each trial consisted of one treated and one control plot. A metaflumizone 240 g/L SC was applied once in-furrow at a rate of 1.2 kg ai/ha (5 times the label rate) in a spray volume of 150 L/ha. Control and treated samples were harvested at 500, 510 and 520 DALA in decline trials. In the other trials, the sample timing was not defined due to the application mode. Samples were kept at or below -20°C until analysis. Sugar cane samples were stored for up to 256 days prior to analysis. The residues of metaflumizone E-isomer and Z-isomer in sugar cane were determined using LC-MS/MS method BASF 531/1 with LOQs of 0.01 mg/kg for each analyte.

Table 17 Residues of metaflumizone in sugar cane after foliar application of a 240 g/L SC formulation

Country, Year, Location, Variety, Trial No.	Application			DALA	Portion analysed	Residue mg/kg			Study Reference
	Method	No	Rate kg ai/ha			E-ISOMER	Z-ISOMER	Total	
Brazil, 2008, Sao Paulo, Santo Antonio de Posse, SP 801816, G080385	in furrow	1	1.2	302	Stalks	<0.01	<0.01	<0.02	2013/1043079
Brazil, 2009, Minas Gerais, Uberlandia, G080386	in furrow	1	1.2	301	Stalks	<0.01	<0.01	<0.02	2013/1043079
Brazil, 2012, Sao Paulo, Jaboticabal, IAC-SP955094, G110266	in furrow	1	1.2	500	Stalks	<0.01	<0.01	<0.02	2014/3000342
				510	Stalks	<0.01	<0.01	<0.02	
				520	Stalks	<0.01	<0.01	<0.02	

Country, Year, Location, Variety, Trial No.	Application			DALA	Portion analysed	Residue mg/kg			Study Reference
	Method	No	Rate kg ai/ha			E-ISOMER	Z-ISOMER	Total	
Brazil, 2012, Goias, Senador Canedo, RB867515, G110344	in furrow	1	1.2	449	Stalks	<0.01	<0.01	<0.02	2014/3000342
Brazil, 2012, Minas Gerais, Uberlandia, RB867515, G110345	in furrow	1	1.2	464	Stalks	<0.01	<0.01	<0.02	2014/3000342
Brazil, 2012, Sao Paulo, Santo Antonio de Posse, SP801816, G110346	in furrow	1	1.03	500	Stalks	<0.01	<0.01	<0.02	2014/3000342
				510	Stalks	<0.01	<0.01	<0.02	
				520	Stalks	<0.01	<0.01	<0.02	

Coffee

Field trials were conducted on coffee beans in Brazil during the 2014 and 2016 growing seasons. Each trial consisted of one control and eight treated plots. Metaflumizone 240 g/L SC was applied twice as a foliar spray at rates of 0.36 kg ai/ha and 0.48 kg ai/ha in spray volumes of 400 L/ha. Control and treated samples were harvested at 45, 60, 75 and 90 DALA. The cherry coffee was sampled by hand, and dried in the field processing shed at ambient temperatures. After drying, the coffee cherries passed through the pulping process, with the aid of a manual pulper, in order to separate the grains (beans) from the husk. Samples were kept at or below -20 °C until analysis. Coffee bean samples were stored for up to 174 days prior to analysis. The residues of metaflumizone E-isomer and Z-isomer in coffee beans were determined using LC-MS/MS method BASF 531/1 with LOQs of 0.01 mg/kg for each analyte.

Table 18 Residues of metaflumizone in coffee bean after foliar application of a 240g /L SC formulation

Year, Location, Variety, Trial No.	Application			DALA	Portion analysed	Residue mg/kg			Study Reference
	Method	No (int.days)	Rate kg ai/ha			E-ISOMER	Z-ISOMER	Total	
Brazil, 2014, Sao Paulo, Santo Antônio do Jardim, Obatã, G130169	foliar	2 (42)	0.48	0	Bean	0.05	0.08	0.13	2014/3021341
				44	Bean	0.02	0.06	0.08	
				60	Bean	0.03	0.06	0.09	
				75	Bean	0.02	0.05	0.07	
				90	Bean	0.02	0.03	0.05	
Brazil, 2014, Parana, Jaguapitã, Tupi, G130170	foliar	2 (30)	0.48	0	Bean	<0.01	<0.01	<0.02	2014/3021341
				45	Bean	<0.01	<0.01	<0.02	
				60	Bean	<0.01	<0.01	<0.02	
				75	Bean	<0.01	<0.01	<0.02	
				90	Bean	<0.01	<0.01	<0.02	
Brazil, 2014, Minas Gerais, Araguari,	foliar	2 (31)	0.48	0	Bean	<0.01	0.02	<0.03	2014/3021341
				45	Bean	<0.01	<0.01	<0.02	
				60	Bean	<0.01	<0.01	<0.02	

Year, Location, Variety, Trial No.	Application					Residue mg/kg			
	Method	No (int.days)	Rate kg ai/ha	DALA	Portion analysed	E-ISOMER	Z-ISOMER	Total	Study Reference
Catuaí, G130171				75	Bean	<0.01	<0.01	<0.02	
				90	Bean	<0.01	<0.01	<0.02	
Brazil, 2014, Minas Gerais, Indianápolis, Catuaí, G130172	foliar	2 (31)	0.48	0	Bean	0.01	0.02	0.03	2014/3021341
				45	Bean	<0.01	<0.01	<0.02	
				60	Bean	<0.01	<0.01	<0.02	
				75	Bean	<0.01	<0.01	<0.02	
				90	Bean	<0.01	<0.01	<0.02	
Brazil, 2014, Parana, Cambé, IPR 103, G130173	foliar	2 (30)	0.48	0	Bean	0.04	0.05	0.09	2014/3021341
				45	Bean	<0.01	<0.01	<0.02	
				60	Bean	<0.01	<0.01	<0.02	
				75	Bean	<0.01	<0.01	<0.02	
				90	Bean	<0.01	<0.01	<0.02	
Brazil, 2014, Minas Gerais, Iraí de Minas, IAPAR 59, G130243	foliar	2 (30)	0.24	0	Bean	<0.01	0.01	0.02	2014/3021341
				45	Bean	<0.01	<0.01	<0.02	
				60	Bean	<0.01	<0.01	<0.02	
				75	Bean	<0.01	<0.01	<0.02	
				90	Bean	<0.01	<0.01	<0.02	
Brazil, 2014, Minas Gerais, Iraí de Minas, IAPAR 59, G130243	foliar	2 (30)	0.36	0	Bean	0.02	0.04	0.06	2014/3021341
				45	Bean	<0.01	<0.01	<0.02	
				60	Bean	<0.01	<0.01	<0.02	
				75	Bean	<0.01	0.01	0.02	
				90	Bean	<0.01	0.01	0.02	
Brazil, 2014, Minas Gerais, Araguari, Mundo Novo, G130244	foliar	2 (30)	0.24	0	Bean	0.03	0.06	0.09	2014/3021341
				45	Bean	0.01	0.02	0.03	
				60	Bean	0.01	0.02	0.03	
				75	Bean	<0.01	<0.01	<0.02	
				90	Bean	<0.01	<0.01	<0.02	
Brazil, 2014, Minas Gerais, Araguari, Mundo Novo, G130244	foliar	2 (30)	0.36	0	Bean	0.08	0.17	0.25	2014/3021341
				45	Bean	0.02	0.04	0.06	
				60	Bean	0.02	0.04	0.06	
				75	Bean	0.01	0.02	0.03	
				90	Bean	<0.01	0.01	0.02	
Brazil, 2014, Parana, Jaguapitã, Tupi, G130245	foliar	2 (29)	0.24	0	Bean	<0.01	<0.01	<0.02	2014/3021341
				45	Bean	<0.01	<0.01	<0.02	
				60	Bean	<0.01	<0.01	<0.02	
				75	Bean	<0.01	0.01	0.02	
				90	Bean	<0.01	<0.01	<0.02	
Brazil, 2014, Parana, Jaguapitã, Tupi, G130245	foliar	2 (29)	0.36	0	Bean	0.01	0.02	0.03	2014/3021341
				45	Bean	<0.01	<0.01	<0.02	
				60	Bean	<0.01	<0.01	<0.02	
				75	Bean	<0.01	<0.01	<0.02	
				90	Bean	<0.01	<0.01	<0.02	
Brazil, 2014, Parana,	foliar	2 (29)	0.24	0	Bean	<0.01	<0.01	<0.02	2014/3021341
				45	Bean	<0.01	<0.01	<0.02	

Year, Location, Variety, Trial No.	Application					Residue mg/kg			
	Method	No (int.days)	Rate kg ai/ha	DALA	Portion analysed	E-ISOMER	Z-ISOMER	Total	Study Reference
Cambé, IPR 103, G130246				60	Bean	<0.01	<0.01	<0.02	
				75	Bean	<0.01	<0.01	<0.02	
				90	Bean	<0.01	<0.01	<0.02	
Brazil, 2014, Parana, Cambé, IPR 103, G130246	foliar	2 (29)	0.36	0	Bean	<0.01	<0.01	<0.02	2014/3021341
				45	Bean	0.04	0.02	0.06	
				60	Bean	<0.01	<0.01	<0.02	
				75	Bean	<0.01	<0.01	<0.02	
				90	Bean	<0.01	<0.01	<0.02	
Brazil, 2016, Minas Gerais, Indianopolis, Catuai, G150229	foliar	2 (29)	0.36	45	Bean	<0.01	<0.01	<0.02	2017/3001462
				60	Bean	<0.01	<0.01	<0.02	
				75	Bean	<0.01	<0.01	<0.02	
				90	Bean	<0.01	<0.01	<0.02	
Brazil, 2016, Minas Gerais, Indianopolis, Catuai, G150229	foliar	2 (29)	0.48	45	Bean	<0.01	0.01	0.02	2017/3001462
				60	Bean	<0.01	<0.01	<0.02	
				75	Bean	<0.01	<0.01	<0.02	
				90	Bean	<0.01	<0.01	<0.02	
Brazil, 2016, Sao Paulo, Campinas, Catuai amarelo, G150230	foliar	2 (28)	0.36	45	Bean	<0.01	<0.01	<0.02	2017/3001462
				60	Bean	0.02	0.03	0.05	
				75	Bean	<0.01	<0.01	<0.02	
				90	Bean	<0.01	<0.01	<0.02	
Brazil, 2016, Sao Paulo, Campinas, Catuai amarelo, G150230	foliar	2 (28)	0.48	45	Bean	<0.01	0.01	0.02	2017/3001462
				60	Bean	0.01	0.02	0.03	
				75	Bean	<0.01	<0.01	<0.02	
				90	Bean	<0.01	<0.01	<0.02	
Brazil, 2016, Sao Paulo, Leme, Obata, G150231	foliar	2 (28)	0.36	45	Bean	<0.01	<0.01	<0.02	2017/3001462
				60	Bean	0.01	0.02	0.03	
				75	Bean	<0.01	<0.01	<0.02	
				90	Bean	<0.01	<0.01	<0.02	
Brazil, 2016, Sao Paulo, Leme, Obata, G150231	foliar	2 (28)	0.48	45	Bean	0.01	0.04	0.05	2017/3001462
				60	Bean	<0.01	0.02	0.03	
				75	Bean	0.01	0.03	0.04	
				90	Bean	<0.01	<0.01	<0.02	

FATE OF RESIDUES DURING PROCESSING

Oranges

The Meeting received processing studies for oranges (Guimarães S.F., 2014 d 2014/3004081, and Guimarães S.F., 2018 b 2018/3000482). Three field trials were conducted in Brazil in 2013 to investigate the residue behaviour of metaflumizone in oranges (whole fruits) and its processed fractions, i.e., dried pulp, juice and oil. Metaflumizone was applied three times as a broadcast foliar spray at 2.4 kg ai/ha in 2000 L/ha of water (5× the maximum label rate) at BBCH 89 with a 10 day interval between each application. Samples (minimum 2.0 kg) were harvested at 7 DALA. For processing, around 250 kg citrus fruit per sample were washed, peeled, and fruit as well as oil from juice and dried pulp were separated. Samples of orange fruit, dried pulp, juice and oil were frozen and packed in separate plastic

bags to be stored in a frozen at ≤-20 °C. The maximum storage interval from harvest till analysis was 119 days. The residues of metaflumizone E-isomer and Z-isomer in oranges and processed commodity fractions were determined using LC-MS/MS method BASF 531/1 with LOQs of 0.01 mg/kg for each analyte. The processing procedures for peeled fruits and juice are described below.

Orange processing: For processing around 250 kg of orange fruit, per sample, were washed using an industrial water bath and rotary brush cleaner (Barana machine with 13 brushed axels, water bath and two rows of nozzles). Cleaned fruits were transferred to an industrial extractor (JBT HP 391 at standard configuration HP 2H2L (NFC), using 2 'cups') for separation of peel, fruit and oil from juice and dried pulp. During crushing the of the fruit, water was sprayed onto the fruit and 'cups'. The resulting waste water (yellow water) was recovered as a mixture of citrus oil and water. The peel-juice mixture was passed through a finisher (JBT UCF35, with 0.01"mesh", set at 27 to 28 psi) to separate juice from dried pulp. The 'yellow-water' was decanted and centrifuged to obtain oil. Juice and dried pulp samples were taken at the finisher.

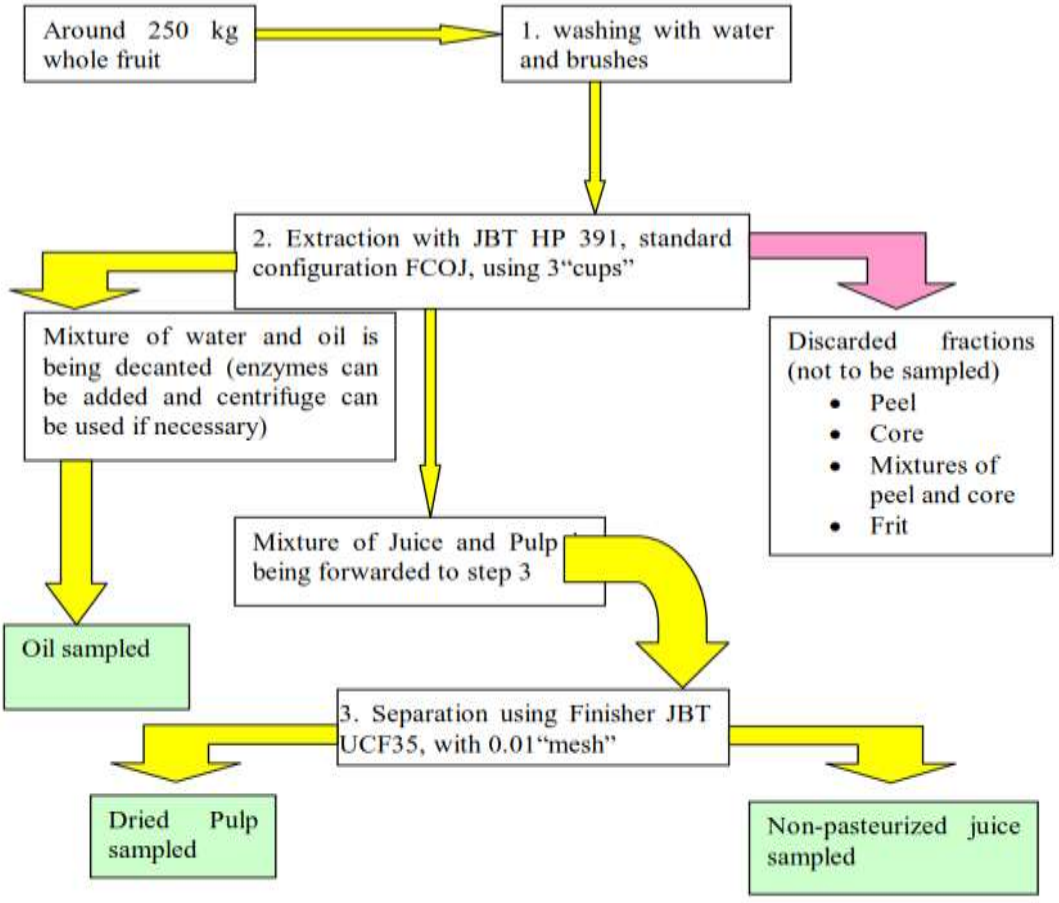

Figure 1 Orange processing flow chart

Table 19 Residues of total metaflumizone (E- and Z-isomer) in oranges after application of BAS 320 00 I

Matrix	Trail no.	Residues [mg/kg] [a]				Sum of residues of metaflumizone (E and Z) [b] [mg/kg]
		Metaflumizone (E)	Metaflumizone (Z)	M320I04 (parent eq)	M320I23 (parent eq)	
Orange, whole fruit	G130175	2.92	4.33	0.3151	0.0487 [c]	7.25
	G130176	1.60	2.71	0.2626	0.0389	4.13

	G130177	2.19	3.23	0.2626	0.0195	5.42
Orange, dried pulp	G130175	0.02	0.03	<0.0175	<0.0097	0.05
	G120080	0.02	0.03	<0.0175	<0.0097	0.05
	G130177	0.02	0.03	<0.0175	<0.0097	0.05
Orange, juice	G130175	0.03	0.02	<0.0175	<0.0097	0.05
	G130176	0.04	0.03	0.0350	<0.0097	0.07
	G130177	0.05	0.03	0.0350	<0.0097	0.08
Orange, oil	G130175	126.94	19.10	1.4355	0.7688	146.04
	G130176	140.10	11.38	1.7857	0.7590	151.48
	G130177	177.66	11.30	4.6568	0.4379	188.96

[a] All residues expressed in terms of parent BAS 320 I. The validated LOQ for each analyte is 0.01 mg/kg (expressed as parent equivalents, 0.0097 mg/kg for M320I23 and 0.0175 mg/kg for M320I04).

[b] Residues values of below LOQ were considered 0.01 mg/kg for calculating the total metaflumizone residues (sum of E and Z isomers).

[c] Mean of results

Table 20 Summary of total metaflumizone (E- and Z-isomer) and transfer factors in orange and its processed fractions after application of metaflumizone

Matrix	Residue total Metaflumizone mg/kg			Transfer factor [a] Metaflumizone			
Trial (application rate)	G120078	G120080	G120081	G120078	G120080	G120081	Median
Orange, whole fruit (3× 2.4 kg ai/ha)	7.25	4.31	5.42	-	-	-	-
Orange, dried pulp (3× 2.4 kg ai/ha)	0.05	0.05	0.05	0.01	0.01	0.01	0.01
Orange, juice (3× 2.4 kg ai/ha)	0.05	0.07	0.08	0.01	0.02	0.01	0.01
Orange, oil (3× 2.4 kg ai/ha)	146.04	151.48	188.96	20.14	35.15	34.86	34.86

[a] Transfer factor = total metaflumizone (E- and Z-isomer) in processed fraction / total metaflumizone (E- and Z-isomer) in whole fruit.

Apples

The Meeting received apple processing studies from the USA (Wyatt D.R., 2015 b, 2014/7002590). Three field trials were conducted on apples in the USA in 2013 to investigate the residue behaviour of metaflumizone in apples and the processed fractions apple sauce, canned apples, dried apples, dried pomace, fruit syrup, juice, wash water, washed apples and wet pomace. Metaflumizone 240 g/L SC was applied four times as a foliar spray at an exaggerated rate of 1.2 kg ai/ha in 935–1412 L/ha of water (5× the maximum label rate) between BBCH 76–89 and the intervals between each application were 6–8 days. The fruit were sampled at normal crop maturity on the day of the last application (0 DALA), i.e., 24 fruits, about 5 kg and for processing a minimum of 150 kg bulk samples. Prior to processing, a representative unwashed apple whole fruit RAC sample was collected and placed in frozen storage.

Apple processing: The apples were washed in a stainless steel wash cart using a ratio of 2 kg of cold water to each 1 kg of fruit for 5 minutes. The washed apples were then fed into the Suntech fruit press hammermill and reduced to crushed apple pulp. The crushed apple pulp was transferred to the 35 L Swept Surface steam Jacketed kettle and heated with low-pressure steam until the temperature of the apple pulp reached 45–50 °C, 1.5 g of pectin enzyme per kg of apple pulp was then added and mixed for approximately 2 minutes. The enzyme treated pulp was permitted to react for approximately 2 hours, then pressed using the Suntech fruit press. The wet pomace was removed, and dried at 70–83 °C, the dried pulp was milled, the fresh juice was filtered to remove any coarse solids. The fresh juice for apple syrup was combined with sugar, lemon juice and pectin and boiled at 100 ºC for 2 minutes.

All samples were stored frozen at ≤-20°C until analysis. The maximum storage interval from harvest until analysis was 357 days. The residues of metaflumizone E- and Z-isomer in apple and

processed commodity samples were determined using LC-MS/MS method with an LOQ of 0.02 mg/kg for the two isomers.

Table 20 Residues of total metaflumizone (E- and Z-isomer) in apple and its processed fractions

Matrix	Trial no.	Residues [mg/kg][a]			Sum of residues (E and Z)[b] [mg/kg]
		metaflumizone (E)	metaflumizone (Z)	M320I04 (parent eq.)	
Apple, whole fruit	R130332	0.690	0.720	0.140	1.410
	R130333	0.820	0.480	0.310	1.300
	R130334	0.450	0.340	0.070	0.790
Apple sauce	R130332	<0.02	<0.02	<0.035	<0.04
	R130333	<0.02	<0.02	<0.035	<0.04
	R130334	<0.02	<0.02	<0.035	<0.04
Canned apples	R130332	<0.02	<0.02	<0.035	<0.04
	R130333	<0.02	<0.02	<0.035	<0.04
	R130334	<0.02	<0.02	<0.035	<0.04
Dried apples	R130332	0.034	<0.02	<0.035	0.054
	R130333	0.033	<0.02	<0.035	0.053
	R130334	0.025	<0.02	<0.035	0.045
Dried pomace	R130332	15.000	8.100	3.100	23.100
	R130333	12.000	4.900	2.700	16.900
	R130334	9.000	4.600	1.200	13.600
Fruit syrup	R130332	0.027	<0.02	<0.035	0.047
	R130333	0.051	<0.02	0.050	0.071
	R130334	0.020	<0.02	<0.035	0.040
Juice	R130332	0.065	0.025	<0.035	0.090
	R130333	0.340	0.200	0.040	0.540
	R130334	0.042	0.020	<0.035	0.062
Wet pomace	R130332	3.100	3.300	0.770	6.400
	R130333	2.600	1.900	0.650	4.500
	R130334	1.500	1.100	0.240	2.600
Washed apples	R130332	0.120	0.240	0.073	0.360
	R130333	0.250	0.320	0.190	0.570
	R130334	0.090	0.120	<0.035	0.210
Wash water	R130332	0.038	0.030	<0.035	0.068
	R130333	0.150	0.079	<0.035	0.229
	R130334	<0.02	<0.02	<0.035	<0.04

[a] All residues expressed in terms of parent BAS 320 I. The validated LOQ for each analyte is 0.02 mg/kg (expressed as parent equivalents, 0.035 mg/kg for M320I04).

[b] Residues values of below LOQ were considered 0.02 mg/kg for calculating the total metaflumizone residues (sum of E and Z isomers).

Table 721 Summary of total metaflumizone (E- and Z-isomer) and transfer factors in apple and its processed fractions after application of METAFLUMIZONE

Matrix	Residue total Metaflumizone mg/kg			Transfer factor [a] Metaflumizone			
Trial	R130332	R130333	R130334	R130332	R130333	R130334	Median
Apple, whole fruit	1.410	1.300	0.790	-	-	-	-
Apple sauce	<0.04	<0.04	<0.04	0.03	0.03	0.05	0.03
Canned apples	<0.04	<0.04	<0.04	0.03	0.03	0.05	0.03
Dried apples	0.054	0.053	0.045	0.04	0.04	0.06	0.04
Dried pomace	23.100	16.900	13.600	16.38	13.00	17.22	16.38
Fruit syrup	0.047	0.071	0.040	0.03	0.05	0.05	0.05
Juice	0.090	0.540	0.062	0.06	0.42	0.08	0.08
Wet pomace	6.400	4.500	2.600	4.54	3.46	3.29	3.46
Washed apples	0.360	0.570	0.210	0.26	0.44	0.27	0.27
Wash water	0.068	0.229	<0.04	0.05	0.18	0.05	0.05

[a] Transfer factor = total metaflumizone (E- and Z-isomer) in processed fraction / total metaflumizone (E- and Z-isomer) in whole fruit.

Grapes

The Meeting received grapes processing studies from the USA (Wyatt D.R., 2015a, 2014/7002591). Three field trials were conducted on grapes in the USA in 2013 to investigate the residue behaviour of metaflumizone in grapes and its processed fractions whole fruit, raisins, stalks (raisins), crush, must deposit, must naturally cloudy, must separated, pasteurized juice, pomace, red and rose wine, stalks and yeast deposit. Metaflumizone 240 g/L SC was applied three times as a foliar spray at an exaggerated rate of 1.2 kg ai/ha in 1048–1786 L/ha of water (5× the maximum label rate) between BBCH 83–89 and the intervals between each application were 7 days. Samples of RAC (12 bunches, minimum 2 kg) and samples for processing (minimum 100 kg) were harvested at normal crop maturity (BBCH 89) on the day of the last application (0 DALA). The bulk samples for raisin generation were dried at each field site to produce at least 12.1 kg of dried fruit (including stems). Grapes were processed into crush (red wine production), must deposit, must naturally cloudy and must separated (red and rose wine making), pasteurized juice (red and rose), pomace (red and rose), red wine, rose wine, stalks (red wine making) and yeast deposit (red and rose). All samples were stored frozen at ≤-20 °C until analysis. The maximum storage interval from harvest until analysis was 362 days. The residues of metaflumizone E- and Z-isomer in grapes and processed commodity samples were determined using an LC-MS/MS method with a LOQ of 0.02 mg/kg for each parent isomer.

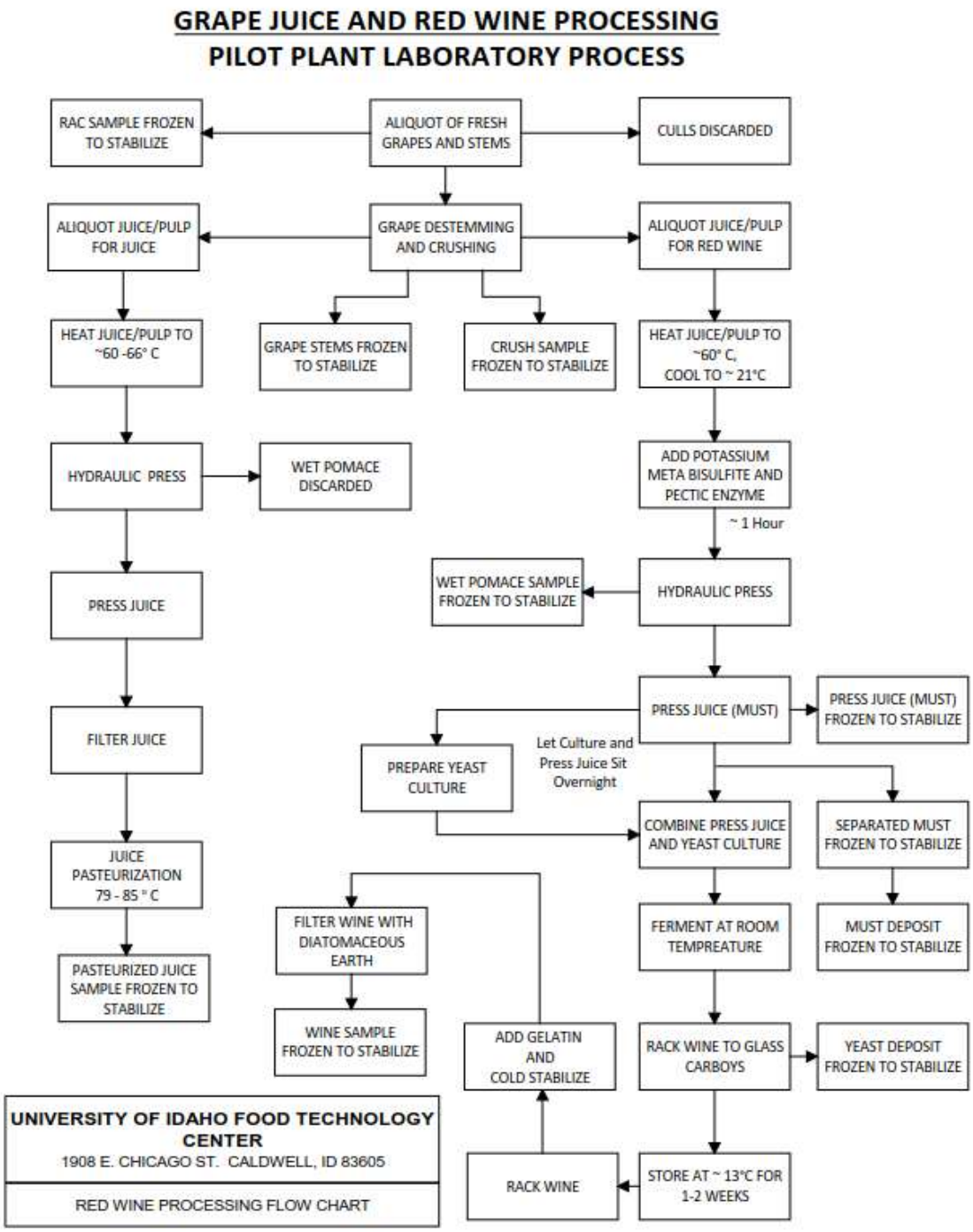

Figure 2 Flow chart for grape juice and red wine processing

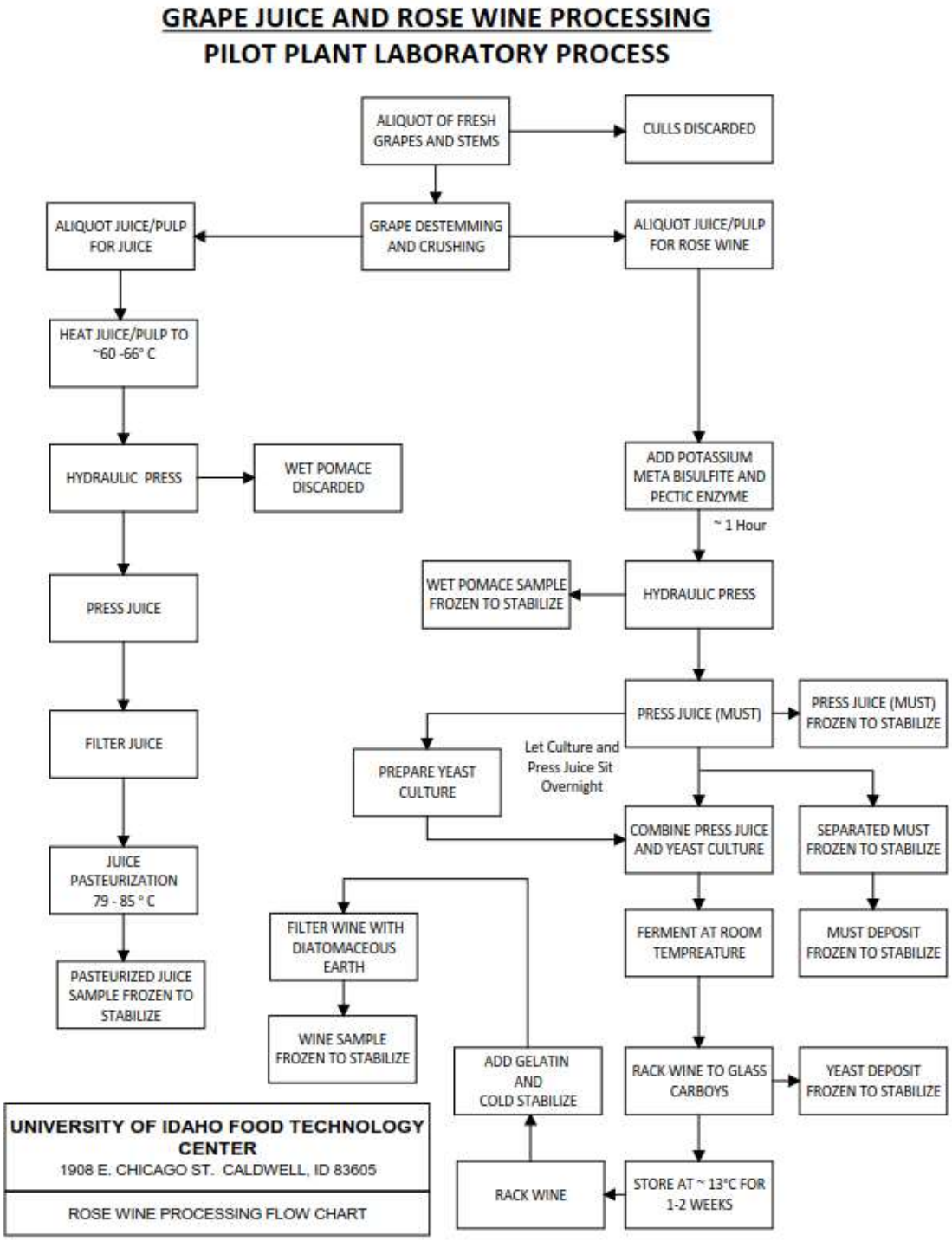

Figure 3 Flow chart of grape juice and Rose wine processing

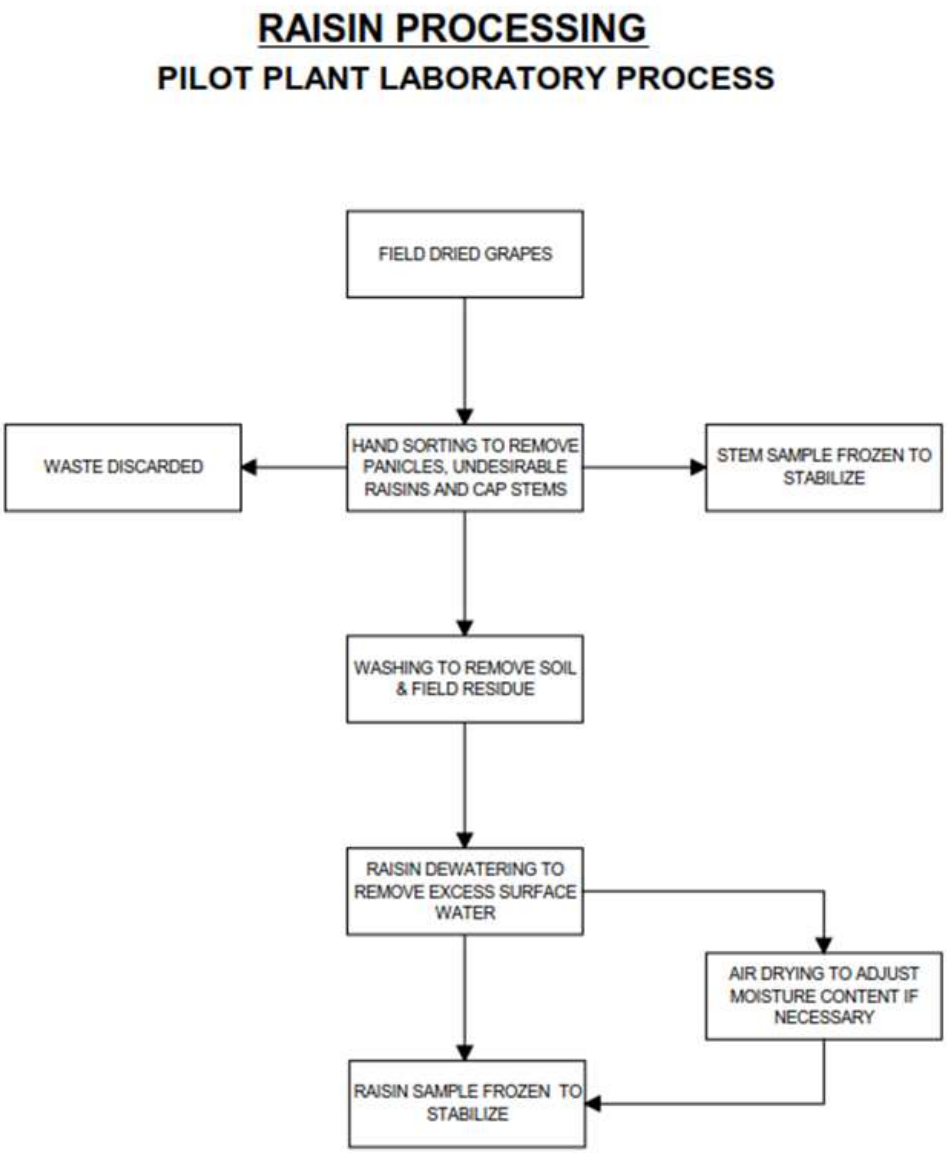

Figure 4 Flow chart of raisin processing

Table 22 Residues of total metaflumizone (E and Z-isomer) in grape and its processed fractions

Matrix	Trial no	Residues [mg/kg]1			Sum of residues of (E and Z)2 [mg/kg]
		metaflumizone (E)	metaflumizone(Z)	M320I04 (parent eq.)	
Grape, whole fruit	R130335	2.8	1.9	0.19	4.7
	R130336	0.26	0.12	<0.035	0.38
	R130337	0.3	0.2	0.053	0.5
Crush (red wine)	R130335	3.8	2	0.14	5.8
	R130336	0.3503	0.1703	0.0463	0.521
	R130337	0.55	0.25	0.054	0.8
Must deposit (red wine)	R130335	0.33	0.1	0.075	0.43
	R130336	0.41	0.11	<0.035	0.52

Matrix	Trial no	Residues [mg/kg]1			Sum of residues of (E and Z)2 [mg/kg]
		metaflumizone (E)	metaflumizone(Z)	M320I04 (parent eq.)	
	R130337	0.98	0.27	0.071	1.25
Must deposit (rose wine)	R130335	0.089	0.022	<0.035	0.111
	R130336	0.31	0.092	<0.035	0.402
	R130337	0.19	0.062	<0.035	0.252
Must naturally cloudy (red wine)	R130335	0.39	0.15	<0.035	0.54
	R130336	0.55	0.18	<0.035	0.73
	R130337	0.67	0.22	0.061	0.89
Must naturally cloudy (rose wine)	R130335	0.055	<0.02	<0.035	0.075
	R130336	0.31	0.094	<0.035	0.404
	R130337	0.67	0.24	<0.035	0.91
Must separated (red wine)	R130335	0.068	<0.02	<0.035	0.088
	R130336	0.077	0.023	<0.035	0.1
	R130337	0.66	0.23	<0.035	0.89
Must separated (rose wine)	R130335	0.041	<0.02	<0.035	0.061
	R130336	0.043	<0.02	<0.035	0.063
	R130337	0.062	<0.02	<0.035	0.082
Pasteurized juice (red wine)	R130335	0.57	0.08	0.15	0.65
	R130336	0.46	0.087	0.038	0.547
	R130337	0.39	0.048	0.054	0.438
Pasteurized juice (rose wine)	R130335	0.83	0.14	0.24	0.97
	R130336	0.94	0.12	0.073	1.04
	R130337	0.56	0.063	0.052	0.623
Pomace (red wine)	R130335	9	4.7	1.2	13.7
	R130336	0.68	0.29	0.22	0.97
	R130337	0.7	0.37	0.27	1.07
Pomace (rose wine)	R130335	9.8	5.7	0.54	15.5
	R130336	0.67	0.26	0.1	0.93
	R130337	0.36	0.23	0.067	0.59
Raisins	R130335	11	1.2	2.1	12.2
	R130336	0.46	0.62	0.12	1.08
	R130337	0.26	0.37	0.08	0.63
Red wine	R130335	<0.02	<0.02	<0.035	<0.04
	R130336	<0.02	<0.02	<0.035	<0.04
	R130337	<0.02	<0.02	<0.035	<0.04
Rose wine	R130335	<0.02	<0.02	<0.035	<0.04
	R130336	<0.02	<0.02	<0.035	<0.04
	R130337	<0.02	<0.02	<0.035	<0.04
Stalks (raisins)	R130335	72	18	1.6	90
	R130336	2	2.3	0.27	4.3
	R130337	1.9	2.2	0.38	4.1
Stalks (red wine)	R130335	6.6	2.8	0.18	9.4
	R130336	0.57	0.22	0.055	0.79
	R130337	0.76	0.34	0.11	1.1
Yeast deposit (red wine)	R130335	17	1.8	0.58	18.8
	R130336	9.4	1.1	0.26	10.5

Matrix	Trial no	Residues [mg/kg]1			Sum of residues of (E and Z)2 [mg/kg]
		metaflumizone (E)	metaflumizone(Z)	M320I04 (parent eq.)	
	R130337	22	2.9	0.43	24.9
Yeast deposit (rose wine)	R130335	5.1	0.46	0.15	5.56
	R130336	17	1.5	0.36	18.5
	R130337	15	1.1	0.26	16.1

[a] Transfer factor = total metaflumizone (E- and Z-isomer) in processed fraction / total metaflumizone (E- and Z-isomer) in whole fruit.

Sugar cane

The Meeting received a sugarcane processing study (Guimarães S.F., 2014c 2014/3000343). During the growing seasons of 2012 and 2013, two field trials were conducted on sugar cane in Brazil to investigate the residue behaviour of metaflumizone (BAS 320 I) in sugar cane and its processed fractions after treatment with metaflumizone (240 g/L SC). The test item was applied once in-furrow at exaggerated rates between 5.4–6.0 kg ai/ha in 150 L/ha of water at BBCH 00. Duplicate treated raw agricultural commodity (RAC) samples (minimum 2 kg) and bulk samples for processing (minimum 150 kg) were harvested at BBCH 49. Leaves and straws were separated from the sugarcane stalks. No residues were detected in the RAC samples (stalks) above the limit of quantitation of 0.01 mg/kg in the treated samples; therefore, it was not necessary to process and analyse the processing fractions.

Coffee beans

The Meeting received a coffee bean processing study (Guimarães S.F., 2017a 2017/3001463). Four field trials were conducted on coffee in Brazil in 2016 to investigate the residue behaviour of metaflumizone in coffee beans, dried coffee cherry and the processed fractions roasted and ground beans, concentrated liquor and instant coffee. Metaflumizone 240 g/L SC was applied twice as a foliar spray at an exaggerated rate of 1.8 kg ai/ha in 400 L/ha of water (3.75× the maximum label rate) between BBCH 77–85 with a 30 day spray application interval. Samples of cherry coffee (180 kg from 58 plants) were harvested at 45 DALA (BBCH 85–89). Samples were first kept frozen (at ≤-20 °C) until processing. The maximum storage interval from harvest till analysis was 146 days. The residues of metaflumizone E- and Z-isomer in coffee beans and processed commodity samples were determined using a LC-MS/MS method with LOQs of 0.01 mg/kg for each analyte.

Roasting: A 2 kg sample of frozen coffee beans were separated and kept at room temperature to defrost. A 1.0 kg sample of the defrosted coffee beans was roasted in fractions of 200–300 g in roasters to generate samples of roasted and grounded beans. After roasting, the coffee beans were stored at room temperature for a maximum of 18 hours to expel CO_2 generated during the process and to equalize moisture levels. The equipment was kept in operation at 250 °C for 10 minutes, cleaned with hot water and ethanol, between samples to eliminate any potential pesticide residue contaminants.

Grinding: After equalizing, the roasted coffee beans were ground in a cone mill. After the grinding of each fraction, an aliquot of 100 g was taken and its particle size classification was determined by the equipment Produtest with rheostat on 8 for 30 minutes. Roasted and grounded coffee produced was stored in high density polyethylene containers (packed in double plastic bags) at -20 °C.

Concentrated Liquor: Roasted coffee beans were ground in a cone mill and then sieved to remove fines. The coffee was weighed, separated in fractions of 2.5 kg and stored in plastic bags at 5 °C until processing in the extraction columns. The water used for extraction was heated by a water bath kept at (90 ± 5) ºC with an immersed resistance coil. The residence time of the water in each column was 17±1 minutes. The extract was collected with DOR (Draw of Ratio) of 0.8–1.0 from the column. The extract was stored in high density polyethylene containers of 250–330 mL and kept in freezer at -20 °C.

Instant Coffee: The stored extract was dried by a Spray Dryer (B191, Buchi) for production of instant coffee using a air flow sprayer.

Table 24 Residues of total metaflumizone (E- and Z-isomer) in coffee and its processed fractions after application of BAS 320 00 I

Matrix	Trial no.	Residues [mg/kg][a]				Sum of residues of E and Z [b] [mg/kg]
		metaflumizone (E)	metaflumizone(Z)	M320I04	M320I23	
Coffee beans	G150166	<0.01	<0.01	<0.0175	<0.0097	<0.02
	G150167	<0.01	<0.01	<0.0175	<0.0097	<0.02
	G150168	<0.01	<0.01	<0.0175	<0.0097	<0.02
	G150169	0.031	0.057	<0.0175	<0.0097	0.088
Dried coffee cherry	G150166	0.500	0.810	0.0595	0.0185	1.310
	G150167	0.320	0.580	0.0368	0.0127	0.900
	G150168	0.330	0.530	0.0350	0.0117	0.860
	G150169	1.500	2.500	0.1287	0.0311	4.000
Roasted and ground beans	G150169	<0.01	<0.01	<0.0175	<0.0097	<0.02
Concentrated liquor	G150169	<0.01	<0.01	<0.0175	<0.0097	<0.02
Instant coffee	G150169	<0.01	<0.01	n.a.[c]	<0.0097	<0.02

[a] All residues expressed in terms of parent BAS 320 I. The validated LOQ for each analyte is 0.01 mg/kg (expressed as parent equivalents, 0.0097 mg/kg for M320I23 and 0.0175 mg/kg for M320I04).

[b] Residues values of below LOQ were considered 0.01 mg/kg for calculating the total metaflumizone residues (sum of E and Z isomers).

[c] This matrix was not analysed for M320I04 due to a high interference of matrix in the recovery. Even when using matrix-matched standards, the results were not satisfactory.

Table 25 Summary of total metaflumizone (E- and Z-isomer) and transfer factors in coffee and its processed fractions after application of metaflumizone

Matrix	Residue total Metaflumizone mg/kg				Transfer factor1 Metaflumizone				
Trial	G150166	G150167	G150168	G150169	G150166	G150167	G150168	G150169	Mean
Coffee beans	<0.02	<0.02	<0.02	0.088	-	-	-	-	-
Dried coffee cherry	1.310	0.900	0.860	4.000	n.a.	n.a.	n.a.	45.45	n.a.
Roasted and ground beans	-	-	-	<0.02	-	-	-	0.23	n.a.
Concentrated liquor	-	-	-	<0.02	-	-	-	0.23	n.a.
Instant coffee	-	-	-	<0.02	-	-	-	0.23	n.a.

[a] Transfer factor = total metaflumizone (E- and Z-isomer) in processed fraction / total metaflumizone (E- and Z-isomer) in whole fruit.

n.a. = not applicable

APPRAISAL

Metabolism in plants

Metaflumizone is a broad-spectrum semicarbazone insecticide composed of two optical isomers in the ratio E: Z of 90: 10. Metaflumizone was first evaluated for residues and toxicology in JMPR 2009, and ADI of 0–0.1mg/kg bw was established and the ARfD was unnecessary. The residue definition for compliance with MRLs and estimation of dietary intake for plants and animals: metaflumizone, sum of E-isomer and Z-isomer. The residue is fat-soluble.

Metaflumizone was scheduled at the 50th session of the CCPR for additional uses for residues by the 2019 JMPR extra meeting. The Meeting received information on environmental fate in soil, storage stability, use patterns, supervised residue trials, fate of residue during processing.

Environmental fate

The Meeting received one study of metaflumizone on degradation under aerobic condition in Brazilian soil. The half-lives of Metaflumizone applied at rate of 240 g ai/ha in four different soils were 61–205 days, the M320I04 was the major degradation product up to 21% of total applied radioactivity (61 days after application). The study confirmed the conclusion of previous evaluation.

Stability of residues in stored analytical samples

The Meeting received one storage stability study. The incurred residues of metaflumizone are stable at <-5°C for at least 729 to 971 days (24–32 months) in cucumber, sunflower seed, snap bean (succulent seed), potato, and strawberry.

Results of supervised residue trials on crops

Supervised residue trial data were available for metaflumizone on citrus fruits, apples, grapes, melons, soya bean, maize, sugarcane and coffee bean.

Citrus fruits

The critical GAP for citrus fruits in Brazil is for 3 foliar applications at rate of 0.48 kg ai/ha, with a retreatment interval of 7 days and a PHI of 7 days. The Meeting received supervised residue trial data for metaflumizone on oranges and lemon conducted in Brazil.

In 11 trials conducted approximating the Brazilian GAP, the residues of metaflumizone in orange fruits were: 0.22(2), 0.34, 0.42(2), 0.66, 0.71, 0.84, 1.01, 1.21 and 1.35 mg/kg (n=11).

The Meeting estimated a maximum residue level of 3 mg/kg, and an STMR of 0.66 mg/kg for oranges, and agreed to extrapolate to the Oranges, Sweet, Sour sub group (including Orange-like hybrids, FC 0004).

In five trials conducted approximating the Brazilian GAP, residues of metaflumizone in lemon fruits were: 0.27, 0.3, 0.52, 0.91and 1.06 mg/kg (n=5).

The Meeting estimated a maximum residue level of 2 mg/kg, and an STMR of 0.52 mg/kg for lemons, and agreed to extrapolate to the Lemons and limes subgroup (including citron, FC 0002).

Apples

The critical GAP for apples in Brazil is 4 foliar applications at a rate of 0.24 kg ai/ha, with retreatment interval of 7 days and a PHI of 3 days. The Meeting received supervised residue trial data for metaflumizone on apples conducted in Brazil.

In 12 trials conducted approximating the critical GAP in Brazil, the residues of metaflumizone in apples were: 0.16, 0.17, 0.19, 0.22, 0.24, 0.25, 0.3, 0.33, 0.43, 0.48, 0.52 and 0.54 mg/kg (n=12).

The Meeting estimated a maximum residue level of 0.9 mg/kg and an STMR of 0.275 mg/kg for apples.

Grapes

The critical GAP for grapes in Brazil is 3 foliar applications at rate of 0.24 kg ai/ha, with a retreatment interval of 7 days and a PHI of 3 days. The Meeting received supervised residue trial data for metaflumizone on grapes conducted in Brazil.

In trials conducted approximating Brazilian GAP, the residues of metaflumizone in grapes were: 0.15, 0.27, 0.51, 0.63, 0.64, 0.75, 1.21, 1.39, 1.4, 1.84, 1.72 and 2.71 mg/kg (n=12).

The Meeting estimated a maximum residue level of 5 mg/kg and an STMR of 0.98 mg/kg for grapes.

Melons, except Watermelons

The critical GAP for melons in Brazil is 5 foliar applications at rate of 0.192 kg ai/ha, with a retreatment interval of 7 days and a PHI of 3 days. The Meeting received supervised residue trial data for metaflumizone on melons conducted in Brazil.

In trials conducted approximating GAP, the residues of metaflumizone in melons were: < 0.02(2), 0.07, 0.1, 0.14, 0.2, 0.29 and 0.61 mg/kg (n=8), the residues of metaflumizone in pulp were < 0.02 (n=3).

The Meeting estimated a maximum residue level of 1 mg/kg and an STMR of 0.02 mg/kg for melons, except watermelon.

Soya bean

The critical GAP for soya bean in Brazil is 3 foliar applications at rate of 0.24 kg ai/ha, with a retreatment interval of 7 days and a PHI of 14 days. The Meeting received supervised residue trial data for metaflumizone on soya beans conducted in Brazil.

In trials conducted approximating Brazilian GAP, the residues of metaflumizone in soya beans were: < 0.02(3), 0.02(2), 0.03, 0.07 and 0.11 mg/kg (n=8).

The Meeting estimated a maximum residue level of 0.2 mg/kg and an STMR of 0.02 mg/kg for soya beans.

Maize

The critical GAP for maize in the Brazil is 5 foliar applications at rate of 0.24 kg ai/ha, with a retreatment interval of 7 days and a PHI of 14 days. The Meeting received supervised residue trial data for metaflumizone on maize conducted in Brazil.

In trials conducted approximating Brazilian GAP, the residues of metaflumizone in maize grains were: < 0.02(7), 0.02 mg/kg (n=8).

The Meeting estimated a maximum residue level of 0.04 mg/kg and an STMR of 0.02 mg/kg for maize grains.

Sugarcane

The critical GAP for sugarcane in Brazil is one application at rate of 0.48 kg ai/ha as an in-furrow treatment at planting. The Meeting received supervised residue trial data for metaflumizone on sugarcane conducted in Brazil.

In trials conducted at an exaggerated rate of 1.2 kg ai/ha, the residues of metaflumizone in sugarcane were: < 0.02(6) mg/kg.

The Meeting estimated a maximum residue level of 0.02(*) mg/kg and an STMR of 0 mg/kg for sugarcane considering all residues were less than LOQ after application at 3 times the GAP rate as an in-furrow at planting treatment.

Coffee bean

The critical GAP for coffee in Brazil is 2 foliar applications at rate of 0.48 kg ai/ha, with a retreatment interval of 30 days and a PHI of 45 days. The Meeting received supervised residue trial data for metaflumizone on coffee conducted in Brazil.

In trials conducted approximating Brazilian GAP, the residues of metaflumizone in coffee beans were: < 0.02(6), 0.02(2), 0.05(2), 0.06(2), 0.09 mg/kg (n=13).

The Meeting estimated a maximum residue level of 0.15 mg/kg and an STMR of 0.02 mg/kg for coffee beans

Fate of residues during processing

The Meeting received processing studies on orange, apple, grape and coffee. A summary of the processing factors is provided below.

Commodity	Processed Fraction	Processing Factor	Best estimate PF	RAC STMR or STMR-P or median residues
Orange	Fruits (RAC)			0.66
	Juice	0.01, 0.01, 0.02	0.01	0.0066
	Dry pulp	0.01, 0.01, 0.01	0.01	0.0066
	Oil	20.14, 34.86, 35.15	34.86	23
Coffee				0.02
	Roasted and ground beans	0.23	0.23	0.046
	Instant coffee	0.23	0.23	0.046
Apple				0.275
	Juice	0.06, 0.08, 0.42	0.08	0.022
	Apple sauce	<0.03, <0.03, <0.05	<0.03	<0.00825
	Canned apples	<0.03, <0.03, <0.05,	<0.03	<0.00825
	Dried apples	0.04, 0.04, 0.06	0.04	0.011
	Dried pomace	13.00, 16.38, 17.22	16.38	4.5
	Wet pomace	3.29, 3.46, 4.54	3.46	0.95
Grape				0.98
	Must separated	0.01, 0.02, 0.16, 0.17, 0.26, 1.78	0.165	0.16
	Must naturally cloudy	0.02, 0.11, 1.06, 1.78, 1.82, 1.92	1.42	1.39
	Pasteurized juice	0.14, 0.21, 0.88, 1.25, 1.44, 2.74	1.065	1.04
	Pomace	1.18, 2.14, 2.45, 2.55, 2.91, 3.30	2.5	2.45
	Raisins	1.26, 2.60, 2.84	2.60	2.55
	Wine	<0.01, <0.1, <0.08, <0.08, <0.11, <0.11	<0.08	0.078

The residues of Metaflumizone concentrated in orange oil, and raisins, the Meeting estimated a maximum residue level of 100 mg/kg (3 × 35) for orange oil, 13 mg/kg (5 × 2.6) for grape raisin.

Residues in animal commodities

Estimation of livestock dietary burdens

Dietary burdens were calculated for beef cattle, dairy cattle, broilers and laying poultry based on feed items evaluated by the JMPR. Potential cattle feed items include: citrus pulp, apple pomace, grape pomace, tomato pomace, maize grain and soya bean seed. The dietary burdens, estimated using the OECD diets listed in Appendix IX of the 2016 edition of the FAO manual, are presented in Annex 6 and summarized below.

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

	US-Canada		EU		Australia		Japan	
	Max	Mean	Max	mean	max	Mean	max	Mean
Beef cattle	0.02	0.02	0.503	0.503	3.28	3.28	0.02	0.02
Dairy cattle	0.255	0.255	0.252	0.252	3.28 [A B]	3.28 [C D]	0.02	0.02
Broilers	0.022	0.02	0.02	0.02	0.003	0.003	0.016	0.016
Layers	0.022[E]	0.022[F]	0.019	0.019	0.0034	0.0034	0.0182	0.0182

[A] Highest maximum beef or dairy cattle dietary burden suitable for maximum residue level estimates for mammalian meat.

[B] Highest maximum dairy cattle dietary burden suitable for maximum residue level estimates for mammalian milk.

[C] Highest mean beef or dairy cattle dietary burden suitable for STMR estimates for mammalian meat.

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

[E] Highest maximum poultry dietary burden suitable for maximum residue level estimates for poultry meat and eggs.

[F] Highest mean poultry dietary burden suitable for STMR estimates for poultry meat and eggs.

Animal commodity maximum residue levels

The calculations used to estimate maximum residue levels, STMR values for cattle matrices are shown below.

	Feed level (ppm) for milk residues	Residues (mg/kg) in milk	Residues (mg/kg) in cream	Feed level (ppm) for tissue residues	Residues of metaflumizone (mg/kg)			
					Muscle	liver	Kidney	Fat
MRL (mg/kg), beef or dairy cattle								
Feeding study	1.0	<0.01	0.0519	1.0	<0.02	<0.02	<0.02	0.0429
	5.5	0.0286	0.242	5.5	<0.02	<0.02	<0.02	0.182
Dietary burden and high residue estimation	3.28	0.019	0.148	3.28	<0.02	<0.02	<0.02	0.115
STMR (mg/kg), beef or dairy cattle								
Feeding study	1.0	<0.01	0.0473	1.0	<0.02	<0.02	<0.02	0.0191
Dietary burden and median residue estimated	5.5	<0.01	0.117	5.5	<0.02	<0.02	<0.02	0.163
Dietary burden and median residue estimation	3.28	<0.01	0.083	3.28	<0.02	<0.02	<0.02	0.092

The maximum dietary burden calculated for cattle is 3.35 ppm for beef cattle and 3.34 ppm for dairy cattle. The mean dietary burden calculated for cattle is 3.35 ppm for beef cattle and 3.34 ppm for dairy cattle.

The Meeting estimated a maximum residue level of 0.02 mg/kg for milk, 0.6 mg/kg for milk fat (0.131x4, cream containing 25% fat) and 0.02*(fat) mg/kg for meat from mammals other than marine mammals, 0.02* mg/kg for edible offal (mammalian), and 0.15 mg/kg for mammalian fat except milk fat. The Meeting estimated STMRs of 0.01 mg/kg for milk, 0.33 mg/kg for milk fat, 0.02 mg/kg for meat from mammals other than marine mammals and edible offal (mammalian), and 0.092 mg/kg for mammalian fat. The Meeting decided to withdraw the previous recommendation.

The calculations used to estimate maximum residue levels, STMR values for poultry matrices are shown below.

	Feed level (ppm) for egg residues	Residues (mg/kg) in egg	Feed level (ppm) for tissue residues	Residues of metaflumizone (mg/kg)		
				Muscle	liver	Fat
MRL (mg/kg), broiler or layer poultry						
Feeding study	0.1	0.061	0.1	0.021	0.033	0.338
Dietary burden and high residue estimation	0.022	0.013	0.022	0.0046	0.0073	0.074
STMR (mg/kg), broiler or layer poultry						
Feeding study	0.1	0.035	0.1	0.01	0.031	0.315
Dietary burden and median residue estimation	0.022	0.0077	0.022	0.0022	0.00688	0.0693

The maximum and mean dietary burdens calculated for poultry (layers and broiler) are 0.022 ppm.

The Meeting estimated maximum residue levels of 0.02 mg/kg for egg, 0.02(*)(fat) mg/kg for poultry meat, 0.08 mg/kg for poultry fat and 0.02*mg/kg for poultry edible offal. The Meeting estimated

STMRs of 0.0077 mg/kg for eggs, 0.0022 mg/kg for poultry meat, 0.0068mg/kg for poultry edible offal, and 0.069 mg/kg for poultry fat.

RECOMMENDATIONS

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

The residue definition for compliance with MRLs and estimation of dietary intake for plants and animals: *metaflumizone, sum of E-isomer and Z-isomer*.

The residue is fat-soluble.

Commodity		Recommended maximum residue levels (mg/kg)		STMR or STMR-P, median residue (mg/kg)	
CCN	Name	New	Previous	New	Previous
FP 0226	Apple	0.9		0.275	
SB 0716	Coffee bean	0.15		0.02	
MO 0105	Edible offal (mammalian)	0.02*	0.02*(w)	0.02	0.013(w)
DF 0269	Dried grapes (=currants, Raisins and Sultanas)	13		2.55	
PE 0112	Eggs	0.02		0.0077	
FB 0269	Grape	5		0.98	
FC 0002	Lemons and limes, Sub group of	2		0.52	
GC 0645	Maize	0.04		0.02	
MF 0100	Mammalian fats (except milk fats)	0.6	0.02*(w)	0.092	0.013(w)
MM 0095	Meat (from mammals other than marine mammals)	0.02*(fat)	0.02*(w)	0.02	0.013(w)
VC 0046	Melon	1		0.02	
	Milk fat	0.7	0.02(w)	0.33	0.013(w)
ML 0106	Milks	0.02	0.01(w)	0.01	0.007(w)
	Orange oil	100		23	
FC 0004	Orange, sweet, sour, Sub group of	3		0.66	
PO 0111	Poultry edible offal	0.02*		0.0068	
PF 0111	Poultry fat	0.08		0.069	
PM 0110	Poultry meat	0.02*(fat)		0.0022	
VD 0541	Soya bean	0.2		0.02	
GS 0659	Sugar cane	0.02*		0	
For dietary estimation					
	Orange juice			0.0066	
	Orange dry pulp			0.0066	
	Roasted and ground beans			0.046	
	Instant coffee			0.046	
	Apple juice			0.022	
	Apple sauce			0.00825	
	Canned apples			0.00825	
	Dried apples			0.011	
	Apple, wet pomace			0.95	

Commodity		Recommended maximum residue levels (mg/kg)		STMR or STMR-P, median residue (mg/kg)	
CCN	Name	New	Previous	New	Previous
	Grape, must, naturally cloudy			1.39	
	Grape, must, separated			0.16	
	Grape, pasteurized juice			1.04	
	Grape, pomace			2.45	
	Grape, wine			0.078	

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for metaflumizone is 0–0.1 mg/kg bw. The International Estimated Daily Intakes (IEDIs) for metaflumizone 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 2019 Extra JMPR Report. The IEDIs ranged 1–4% of the maximum ADI. The Meeting concluded that long-term dietary exposure to residues of metaflumizone from uses considered by the JMPR is unlikely to present a public health concern.

Acute dietary exposure

The 2009 JMPR decided that an ARfD for metaflumizone was unnecessary. The Meeting therefore concluded that the acute dietary exposure to residues of metaflumizone from the considered uses is unlikely to present a public health concern.

Threshold of toxicological concern (TTC) approach for metabolites

The metabolites M320I04, M320I06 and M320I29 are unlikely to be genotoxic and could be assessed using the TTC approach (Cramer Class III threshold of 1.5 µg/kg bw per day).

The metabolite M320I04 was found in plant metabolism studies, present at 11-22% of the metaflumizone (E+Z) residues in cabbage and tomato; and 45% in cotton seed but at a low level (0.059 mg/kg). In all field trials, the residues of M320I04 did not exceed 20% of the metaflumizone (E+Z) residues. M320I04 was the major degradation product under baking, brewing, boiling simulation and represented up to 26% of applied radioactivity. The maximum IEDI (Annex 3) calculated for metaflumizone is 3.83 µg/kg bw. Based on the highest ratio between the metabolite and parent of 0.26 (simulated hydrolysis), the estimated maximum IEDI is 1.0 µg/kg bw.

The residues of M320I06 in the plant metabolism studies were much lower than M320I04. M320I06 was not found in either processing studies or supervised trials. M320I029 was only found in soil and not expected in plant commodities.

Therefore, the Meeting concluded that dietary exposure to residues of M320I04, M320I06 and M320I29 from uses considered by the JMPR would not be expected to be a safety concern.

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Guimaraes S.F.	2018	Addendum no. 01 to Final Report - Study of Metaflumizone residues in citrus (fruits and processed fractions) after treatment with BAS 320 00 I under field conditions in Brasil, BASF SA, Guaratingueta, Brazil, 2018/3000482, unpublished
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Guimaraes S.F.	2014	Study of Metaflumizone residues in sugarcane (stalks and processed fractions) after treatment with BAS 320 00 I under field conditions in Brazil, BASF SA, Guaratingueta, Brazil, 2014/3000343, unpublished
Guimaraes S.F.	2017	Residue study of Metaflumizone in coffee (dried coffee cherry, beans and processed fractions) after treatment with BAS 320 00 I under field conditions in Brazil, BASF SA, Guaratingueta, Brazil, 2017/3001463, unpublished

METHOPRENE (147)

First draft prepared by Dr R Scrivens, Health and Safety Executive, York, United Kingdom

EXPLANATION

Methoprene is an insect growth regulator classified as a juvenile hormone mimic. It has insecticidal activity against a variety of insect species. Methoprene is used to control infestations in post-harvest stored cereal grain commodities and other stored commodities (sunflower and peanuts).

Methoprene was first evaluated by the JMPR in 1984 and re-evaluated for residues several times. The most recent residues evaluation was conducted in 2016. The ADI of 0–0.09 mg/kg bw was established for racemic methoprene (R and S enantiomers in ratio 1:1); a separate ADI of 0–0.05 mg/kg bw was established for S-methoprene (2001). An ARfD was unnecessary. The residue definition for methoprene and for S-methoprene for plant and animal commodities, for both compliance with MRLs and dietary risk assessment is methoprene. The residue is fat soluble.

At the Fiftieth Session of the CCPR (2018), methoprene was scheduled for evaluation of additional use patterns by the 2019 Extra JMPR. The current Meeting received residue data for post-harvest use on stored peanuts.

RESIDUE ANALYSIS

Analytical methods

The Meeting received recovery data (generated concurrently to the analysis of the residue trial samples) for the analytical method employed in the analysis of stored peanut commodities (CAP 427.05). This method was previously evaluated by the 2016 JMPR, validated for the determination of methoprene in sunflower seeds by reverse-phase HPLC with UV detection at 264 nm. Prior to analysis, samples were extracted with 100 mL methanol by shaking the unshelled peanut samples for a minimum of 5 hours. Samples were allowed to sit or shake for 19 additional hours, after which 5 mL of dibutyl phthalate (DBP) was added as an internal standard. Mean procedural recoveries for unshelled peanuts analysed using CAP 427.05 were approximately 100% with a relative standard deviation of approximately 6% (Table 1), and a lowest limit of method validation of 1.3 mg/kg.

Table 1 Procedural recovery data for method CAP 427.05

Matrix	Analyte	Fortification level (approx.) [mg/kg]	Individual recoveries [%]	Range of recoveries [%]	Mean recovery [%]	RSD [%]
Unshelled peanuts	Methoprene	1.3	100, 107, 114, 115, 118	100-118	111	6.6
		2.7	100, 104, 104, 106, 109, 111, 111, 114, 115, 119,	100-119	109	5.4

Stability of pesticide residues in stored analytical samples

No new storage stability data were submitted to the current Meeting.

USE PATTERN

The additional (peanut) GAP submitted for consideration in the current Meeting is summarized in Table 2. Whilst the formulation may be diluted in water or oil for other stored commodities, for peanuts the label states to dilute with water only.

Table 2 List of additional uses of S-methoprene submitted in 2019

Crop	Country	Formulation		Application		WHP [a] (days)
		Type	Conc.	Rate	No.	
Peanuts	USA	EC	288 g/L	max 34.6 g ai/1000 bushels [b] (up to 4.5 g ai/t)	ns	ns

[a] WHP=withholding period

[b] 1000 bushels (USA) 7.7 t is the weight/volume (t/1000 bushels) for Virginia type and 9.5 t is for south-eastern runners (unshelled) peanuts

ns: not stated

RESULTS OF SUPERVISED RESIDUE TRIALS ON CROPS

Peanuts

Five residue trials were conducted in the USA in 2015.

Commercially grown unshelled peanuts from three different farm locations were harvested, bagged, and sent to the testing facility for treatment and residue analysis. Each farm location submitted 13 to 45 kg unshelled peanuts. One of the farms provided three different varieties for testing. Each of the trials consisted of two untreated controls and two treated samples of 2.3 kg size. At a single location, the formulation containing S-methoprene was applied at 7.5 mL product/US ton which equates to 2.4 g ai/t by admixture to the peanuts while being turned in a cement mixer, this occurred for each trial. Water was used as the diluent in line with the label recommendation for peanuts. The cement mixer was used to simulate peanuts flowing through a grain auger. Prior to the application, excess dust was collected from the cement mixer using a dust collection device. Although the intended product can be used with dust-controlling oils this physical removal of dust is not usual label recommended practice prior to application with S-methoprene. Despite this, the intention was to remove excess dust to prevent S-methoprene adhering to dust, and is therefore likely to be worst case (in terms of residue levels on the target peanut lots). Treated samples were taken one day after treatment and placed into frozen storage where they were maintained frozen for periods of up to 149 days prior to extraction and analysis.

Residues following application of S-methoprene were determined as methoprene in peanuts following the method CAP 427.05 using reverse-phase HPLC with UV detection. Procedural recoveries from spiking at similar levels to those occurring in the samples from the supervised trials were reported (see Table 1).

The results of the supervised trials are presented in Table 3. No residues were found in the untreated controls (< 0.01 mg/kg). Residue results have been presented uncorrected for recovery. Residue values which have been used for the estimation of maximum residue levels and STMRs are underlined.

These trials can be regarded as independent trials, as the application of S-methoprene was made separately for each trial, and the trials themselves did not involve storing the peanuts for a period of storage under normal commercial food handling conditions. The aim being to analyse a situation in which the highest likely residues from label use might arise, i.e., where no pre-harvest interval is specified. As a result residues of methoprene in peanuts were determined shortly after treatment (after one day) for all trials.

Table 3 Residues in Peanuts from supervised trials in the USA involving S-methoprene as a post-harvest application

Year, Variety (source of peanuts)	Application			DALA days	Residues determined as methoprene (mg/kg)	Reference
	Form.	g ai/1000 bushels (g ai/t) unshelled peanuts	no.			
GAP USA: Peanuts	EC	up to4.5 g ai/t 34.6 g ai/1000 bushels	ns ns	ns ns		
2015 Peanuts (unshelled)/ GA 06G Peanuts sourced from Newton, Alabama, USA	EC	22.1 (2.4)	1	1	2.2, 1.9 mean = 2.0 (LOD = 0.007)	5189 Haas and Witte, 2016
2015 Peanuts (unshelled)/ Spanish (Organic) Peanuts sourced from Wellman, Texas, USA	EC	26.2 (2.4)	1	1	2.2, 2.1 mean = 2.1 (LOD = 0.006)	
2015 Peanuts (unshelled)/ Runner Peanuts sourced from Wellman, Texas, USA	EC	23.8 (2.4)	1	1	1.7, 1.9 mean=1.8 (LOD = 0.004)	
2015 Peanuts (unshelled)/ Virginia Peanuts sourced from Wellman, Texas, USA	EC	22.8 (2.4)	1	1	2.0, 2.0 mean=2.0 (LOD = 0.003)	
2015 Peanuts (unshelled)/ OG6 Peanuts sourced from Ashburn, Georgia, USA	EC	26.2 (2.4)	1	1	1.9, 2.1 mean=2.0 (LOD = 0.002)	

FATE OF RESIDUES DURING PROCESSING

No new data were received on the fate of S-methoprene residues on processing.

APPRAISAL

Methoprene, an insect growth regulator, was first evaluated by the JMPR in 1984 and evaluated for residues several times. The most recent residues evaluation was conducted in 2016. The ADI of 0–

0.09 mg/kg bw was established for racemic methoprene (R and S enantiomers in ratio 1:1); a separate ADI of 0–0.05 mg/kg bw was established for S-methoprene (2001). An ARfD was unnecessary. The residue definition for methoprene and for S-methoprene for plant and animal commodities, for both compliance with MRLs and dietary risk assessment, is methoprene. The residue is fat soluble.

At the Fiftieth Session of the CCPR (2018), methoprene was scheduled for evaluation of additional use patterns by the 2019 Extra JMPR. The current Meeting received residue data for post-harvest use on stored peanuts.

Methods of analysis

Residues of methoprene were determined in peanuts using an HPLC-UV analytical method that was previously evaluated by the 2016 JMPR. New data validating the method for peanuts was received by the Meeting with the lower and upper levels of fortification validated being 1.3 and 2.7 mg/kg. Based on the residue levels found in the trials, the Meeting concluded that the available validation data are adequate to ensure the validity of the results.

Stability of residues in stored analytical samples

The 2005 Meeting concluded that "numerous laboratory and field trials have shown long term stability of methoprene in stored grain, not only at -20 °C but even at room temperature". Noting that residues of methoprene in wheat grain trials evaluated by the JMPR in 2005 remained stable over 180 days of ambient storage, the Meeting concluded that residues of methoprene in samples from the peanut supervised trials would be stable over the periods of frozen storage of up to 149 days.

Results of supervised residue trials on crops

Peanut

The critical GAP in the USA is application of S-methoprene at up to 36.4 g ai/1000 bushels (corresponding to up to 4.5 g ai/t) with no withholding period specified. Five residue trials from the USA at dose rates (2.4 g ai/t; 64, 66, 69, 76 and 76% of GAP rate in g ai/1000 bushels) below the critical GAP were provided to the Meeting.

Residues in peanuts in rank order (n=5) were: 1.8, 2.0 (3), and 2.1 mg/kg.

As in the trials, where S-methoprene was applied separately to different peanut lots simulating commercial application practice, the results reflected a high recovery of applied methoprene (75 to 88% of the 2.4 g ai/t 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.

Based on the GAP, and with an anticipated variation in weights of different peanut varieties per 1000 bushels (the label expression reflecting amount of S-methoprene applied to 1000 bushels of peanuts), the Meeting considered that residues of up to about 4.5 mg/kg can be anticipated.

The Meeting estimated a maximum residue level of 5 (Po) mg/kg and a STMR of 5 mg/kg.

Residues in animal commodities

Peanut meal can be fed to livestock. The 2016 JMPR evaluated residues of methoprene in cereal grains and oilseeds (except for peanuts). Estimation by the present Meeting, now including peanuts, does not significantly increase the previously estimated (2016) 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 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.

CCN	Commodity	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
SO 0703	Peanut whole	5 Po	-	5	-

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 2019 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 unlikley 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.

REFERENCES

Author	Report No./Trial ID	Year	Title, Institute
Haas, K.L. and Witte, J.	5189	2016	(S)-Methoprene: Residues on Peanuts The above report contains methods of analyses CAP 414 and CAP 427.05 as appendices

PENDIMETHALIN (292)

First draft prepared by Ms G Y Zhu, Ministry of Agriculture and Rural Affairs, Beijing, Republic of China

EXPLANATION

Pendimethalin is a selective herbicide used to control most annual grasses and certain broad leaf weeds in various crops, such as fruits and vegetables, cereals, pulses and oilseeds, root crops and ornamentals. The compound has an ADI of 0–0.1 mg/kg bw, and an ARfD of 1 mg/kg bw. The residue definition for plant and animal commodities for compliance with the MRL and dietary risk assessment is pendimethalin. The residue is fat soluble.

Pendimethalin was first evaluated for toxicology and residues by the 2016 JMPR. It was scheduled at the Fiftieth Session of the CCPR for the evaluation of additional uses by the 2019 Extra JMPR. The current Meeting received information on GAP and supervised residue trials and storage stability study for berries and herbs.

RESIDUE ANALYSIS

Analytical methods

In 2016, the Meeting received a number of analytical methods (LC-MS/MS, GC-MS and GC-NPD) for determination of pendimethalin and M455H025 in plant and animal matrices. They were considered suitable for measuring pendimethalin and M455H025 with a LOQ of 0.01 mg/kg in all plant matrices.

The current Meeting received data on the validation of Method D0203 (LC-MS/MS) for pendimethalin in cane berries and blueberry. The pendimethalin analytical validation recoveries are shown below.

Table 1 Pendimethalin analytical validation recovery rate in supervised trials

Matrix	Method	Analyte	Fortification level (mg/kg)	n	Recovery (%) （Average）	RSD （%）
Cane berry	D0203	Pendimethalin	0.05 0.5	3 3	79, 80, 81 (80) 88, 91, 92 (90)	1.3 2.3
Blueberry	D0203	Pendimethalin	0.05 0.5	3 3	80, 82, 83 (82) 87, 89, 90 (89)	1.9 1.7

USE PATTERNS

The Meeting received information on authorised uses on small berries and herbs in Ireland, the United Kingdom and the USA.

The use patterns in these countries on these crops is summarized in the following table.

Table 2 Registered uses of pendimethalin on berries and mint

Crop	Country	Form	Max application		Water L/ha	RTI(days)	PHI (day)	Note
			no	kg ai/ha				
Cane berries [a]								
Cane berries	USA	38.7% CS (360g/L)	3	6.7 (per application) 6.7 (per year)	28–374	30	30	Soil appl., Ground boom sprayer, Fixed wing, Chemigation Irrigation system

Crop	Country	Form	Max application		Water L/ha	RTI(days)	PHI (day)	Note
			no	kg ai/ha				
Bush berries [b]								
Bush berries	USA	38.7%CS	3	6.7 (per application), 6.7 (per year)		30	30	Soil appl., Ground boom sprayer, Fixed wing, Chemigation Irrigation system
Low growing berries [c]								
Low growing berries	USA	38.7%CS		3.2 (per application) 3.2 (per year)		n.a.	35	Soil appl., Ground boom sprayer, Fixed wing, Chemigation Irrigation system
Strawberry								
Strawberry	IRL	445g ae/L CS	1	1.3	100-200		-	Soil application Fixed by approved use / latest time of application: after flower initiation but before flower truss emergence
Strawberry	GBR	445g ae/L CS	1	1.3	100-200		-	Soil application Fixed by approved use / latest time of application: after flower initiation but before flower truss emergence
Strawberry	USA	38.7%SC	2	3.2 (per application) 3.2 (per year)		n.a.	35	Soil appl., Ground boom sprayer, Fixed wing, Chemigation Irrigation system
Mint								
Mint	USA	38.7%SC	1	2.24 (per application) 2.24 (per year)			90	Soil app., Ground boom sprayer, Fixed wing

[a] According to US crop grouping, Caneberry subgroup includes blackberry; loganberry; raspberry, black and red; wild raspberry; cultivars, varieties, and/or hybrids of these.

[b] According to US crop grouping, Bushberry subgroup includes 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; cultivars, varieties, and/or hybrids of these

[c] According to US crop grouping, Low growing berries (subgroup, includes bearberry; bilberry; blueberry, lowbush; cloudberry; cranberry; lingonberry; muntries; partridgeberry; strawberry; cultivars, varieties, and/or hybrids of these.

RESULTS OF SUPERVISED RESIDUE TRIALS ON CROPS

The Meeting received information on supervised field trials involving soil treatments of pendimethalin on blackberry, raspberry, blueberry, strawberry and mint.

Group		Crop	Countries	Table no
004A	Subgroup of cane berries	Blackberry, raspberry	USA	3
004B	Subgroup of bush berries	Blueberry	USA	4
004E	Subgroup of low growing berries	Strawberry	Greece, Italy, Spain, UK, USA	5
027	Group of herbs	Mint	USA	6

Cane berries, Subgroup

The Meeting received seven cane berry trials on blackberry (4) and raspberry (3). Pendimethalin (360 g/L CS) was applied once to the soil at 6.46–6.95 kg ai/ha in broadcast spray volumes of 243–337 L/ha. Control and treated samples were harvested 28–35 days after the treatment.

The residues of pendimethalin were determined with method D0203 (LC-MS/MS). The LOQ was 0.05 mg/kg. Average concurrent recovery rates at fortification levels of 0.05 mg/kg were 73–89%. The RSD value was 7.4%.

Table 3 Residues in cane berries from supervised trials in the USA in 2011 involving one soil application of pendimethalin (SC formulation)

Location (Variety)	Application			Growth Stage	DALA	Residues (mg/kg)		Reference & Comments
	N	kg ai/ha	water (L/ha)			Pendimethalin	mean	
CA132 Parlier, CA Blackberry (Ouachita)	1	6.5	271.3	Blooming/fruiting	28	<0.05 <0.05	<u><0.05</u>	IR-4 PR No. 09840
MI48 Holt,MI Blackberry (Illini)	1	6.5	271.3	Fruiting	30	<0.05 <0.05	<u><0.05</u>	IR-4 PR No. 09840
NC34 Jackson Springs, NC Blackberry (Kiowa)	1	6.7	243.2	Late flowering/green fruit	29	<0.05 <0.05	<u><0.05</u>	IR-4 PR No. 09840
OR30 Aurora, OR Blackberry (Marion)	1	6.5	280.6	Fruiting/flowering	34	<0.05 <0.05	<u><0.05</u>	IR-4 PR No. 09840
NY29 Ithaca, NY Raspberry (Royalty)	1	6.9	280.6	Fruiting	28	<0.05 <0.05	<u><0.05</u>	IR-4 PR No. 09840
OR28 Aurora, OR Raspberry (Willamette)	1	6.9	336.7	Fruiting/flowering	30	<0.05 <0.05	<u><0.05</u>	IR-4 PR No. 09840
OR29 Aurora, OR Raspberry (Willamette)	1	6.5	243.2	Fruiting	35	<0.05 <0.05	<0.05	IR-4 PR No. 09840

Blueberries

The results from seven supervised trials on blueberries in the USA were provided to the Meeting.

In the blueberry trials, one foliar application of 6.67–7.07 kg ai/ha pendimethalin (360g/L SC) was applied as a broadcast spray to the soil.

The residues of pendimethalin were determined with method D0203 (LC-MS/MS). The LOQ was 0.05 mg/kg. Average concurrent recovery rates at fortification levels of 0.05 mg/kg were 80–83%. The RSD value was 1.7%.

Table 4 Residues in blueberry from 7 supervised trials in the USA in 2011 involving one soil application of pendimethalin

Location (Variety)	Application			DAT	Residues (mg/kg)		Reference & Comments
	N	kg ai/ha	water (L/ha)		pendimethalin	mean	
GA*17 Alapaha, GA （TH667）	1	6.9	318	31	<0.05 <0.05	<u><0.05</u>	Report: IR-4 PR No. 10181
MI49 Fennville, MI （Jersey）	1	6.9	187	28	<0.05 <0.05	<u><0.05</u>	Report: IR-4 PR No. 10181

Location (Variety)	Application			DAT	Residues (mg/kg)		Reference & Comments
	N	kg ai/ha	water (L/ha)		pendimethalin	mean	
MI50 Holt, MI （Jersey）	1	6.8	280	28	<0.05 <0.05	<0.05	Report: IR-4 PR No. 10181
NC35 Castle Hayne, NC (Croatan）	1	6.7	205	30	<0.05 <0.05	<0.05	Report: IR-4 PR No. 10181
NJ16 Cream Ridge, NJ (Duke）	1	6.8	271	29	<0.05 <0.05	<0.05	Report: IR-4 PR No. 10181
OR31 Aurora, OR (Bluecrop)	1	7.1	289	35	<0.05 <0.05	<0.05	Report: IR-4 PR No. 10181
OR41 Aurora, OR (Bluecrop）	1	7.0	290	28	<0.05 <0.05	<0.05	Report: IR-4 PR No. 10181

Strawberry

The results from 22 supervised trials on strawberries in Europe and the USA were provided to the Meeting.

In twelve strawberry trials from the USA, two applications of pendimethalin (38% EC) were applied, involving 1 application pre-transplanting, and 1 further application at 24 to 34 days prior to harvest.

In ten strawberry trials in Europe, one application of pendimethalin (445 g/L CS) was applied either before planting of strawberries or shortly before/after vegetative re-growth of strawberries.

The residues of pendimethalin were determined with method SOP 2M1930.01 (GC-NPD) in the USA. The LOQ was 0.05 mg/kg in strawberries. Average concurrent recovery rates at fortification levels of 0.05, 0.5 mg/kg were 80–83%. The RSD values were 1.2–6.5%. Another method L0163/01 (LC-MS/MS) was uesed in Europe to determine the residue of pendimethalin in strawberries. The LOQ was 0.01 mg/kg. Average concurrent recovery rates at fortification levels of 0.01, 0.1 and 1 mg/kg were 73–91%. The RSD values were 0.9–1.9%.

Table 5 Residues in strawberry from 22 supervised trials in Europe and the USA involving one or two soil applications of pendimethalin

Country, year Location (Variety)	Application			Growth Stage	DAT	Residues (mg/kg)		Reference & Comments
	N	kg ai/ha	water (L/ha)			pendimethalin	mean	
USA,1996 NY11 Freeville, NY (Honeoye)	2	2×1.68 RTI:375days	320.71 15 320.09 24	blooming	24	<0.05 <0.05	<0.05	Study code:02739, DOC:2005 7002525
USA,1996 WI10 Arlington, WI (Midway)	2	2×1.68 RTI:385days	187 227	Pre-bloom	29	<0.05 <0.05	<0.05	Study code:02739, DOC:2005 7002525
USA,1995 WA*37 Prosser, WA (Sumas)	1	1.68	120	Late bud stage	34	<0.05 <0.05	<0.05	Study code:02739, DOC:2005 7002525

Country, year Location (Variety)	Application			Growth Stage	DAT	Residues (mg/kg)		Reference & Comments
	N	kg ai/ha	water (L/ha)			pendimethalin	mean	
USA,1995 WA40 Mt. Vernon, WA (Totern)	1	1.68	536	Start of bloom	31	<0.05 <0.05	<0.05	Study code:02739, DOC:2005 7002525
USA,2005 L07719.05-JN17 Bridgeon, NJ (Avaion)	2	2×1.84 and 1.69=3.53 RTI:43days	238 239	Dormant and Blooming	27	<0.05	<0.05	Study code:851924 DOC:2018/7004894
USA,2005 CA*84 Watsonville, CA (Camarosa)	2	2×1.74 and 1.70=3.42 RTI:209days	234 267	Not provided and Mature plant	29	<0.05	<0.05	Study code:851924 DOC:2018/7004894
USA,2005 CA*85 Salinas, CA (Diamante)	2	2×1.72 and 1.68=3.37 RTI:191days	100 408	Not provided and Mature plant	26	<0.05	<0.05	Study code:851924 DOC:2018/7004894
USA,2005 CA86 Irvine, CA (Camarosa)	2	2×1.72 and 1.72=3.43 RTI:129days	238 286	Not provided and Fruiting	28	<0.05	<0.05	Study code:851924 DOC:2018/7004894
USA,2005 FL32 Wimauma (Festival)	2	2×1.72 and 1.70=3.42 RTI:60days	288 239	Not provided and Mature plant	30	<0.05	<0.05	Study code:851924 DOC:2018/7004894
USA,2005 MI18 Holt, MI (Darselect)	2	2×1.78 and 1.81=3.59 RTI:41days	198 201	Not provided and Blooming	29	<0.05	<0.05	Study code:851924 DOC:2018/7004894
USA,2005 NC15 Clinton , NC (Chandler)	2	2×1.67 and 1.68=3.35 RTI:181days	207 258	Not provided and Blooming	28	<0.05	<0.05	Study code:851924 DOC:2018/7004894
USA,2005 OR13 Aurora, OR (Totem)	2	2×1.74 and 1.70=3.42	281 580	Not provided and Mature plant	29	<0.05	<0.05	Study code:851924 DOC:2018/7004894
Germany,2016-2017 L160440 Ingelheim (Clery)	1	1.0	300	00(plot 2) 00(plot 3)	83 83	<0.01 <0.01	<0.01	Study code:766507, DOC:2017/1192651
Germany,2016-2017 L160441 Offenbach/ Queich (Malwina)	1	1.0	300	00(plot 2) 00(plot 3)	105 105	<0.01 <0.01	<0.01	Study code:766507, DOC:2017/1192651
Nertherlands,2016-2017 L160442 5584AR Stevensbeek (Allegro)	1	1.0	300	10-11(plot 2) 10-11(plot 3)	81 81	<0.01 <0.01	<0.01	Study code:766507, DOC:2017/1192651
U.K,2016-2017 L160443 GL54PB Winchcombe (Elsanta)	1	1.0	300	00-10(plot 2) 00-10(plot 3)	116 116	<0.01 <0.01	<0.01	Study code:766507, DOC:2017/1192651
Spain,2016-2017 L160444 46800 Xativa (Camarosa)	1	1.0	300	00-10	198	<0.01	<0.01	Study code:766507, DOC:2017/1192651
Italy,2016-2017 L160445 Albosaggia (Elsanta)	1	1.0	300	12-13	49	<0.01	<0.01	Study code:766507, DOC:2017/1192651

Country, year Location (Variety)	Application			Growth Stage	DAT	Residues (mg/kg)		Reference & Comments
	N	kg ai/ha	water (L/ha)			pendimethalin	mean	
Italy,2012-2013 L120127 Berbenno Di Valtellina (Selva)	1	1.6	200	00	58	0.016	0.016	Study code:766507, DOC:2017/1192651
Greece,2012-2013 L120128 Svoronos (Kamarosa)	1	1.6	200	00	115	<0.01	<0.01	Study code:766507, DOC:2017/1192651
Italy,2012-2013 L120129 Albosaggia (Monterrey)	1	1.6	200	Not reported	63	0.011	0.011	Study code:766507, DOC:2017/1192651
Spain,2012-2013 L120130 Quatretonda (Camarosa)	1	1.6	200	Not reported	228	<0.01	<0.01	Study code:766507, DOC:2017/1192651

Herbs

The results from five trials on mints (peppermint and spearmint) and the processing to mint oil on pendimethalin residues were provided to the Meeting.

Mints

In the mint trials, a single soil application of 2.15–10.35 kg ai/ha pendimethalin (330g/L EC) was applied.

The residues of pendimethalin were determined with method SOP 2M1930.01 (GC-NPD). The LOQ was 0.1 mg/kg in mint, average concurrent recovery rates at fortification levels of 0.1, 1, 10 mg/kg were 67–142%. The RSD values were 0.7–19%.

Table 6 Residues in mints from supervised trials in the USA in 1997 following 1 soil application of pendimethalin (EC formulation)

Location (Variety)	Application			Growth Stage	Matrix	DAT	Residues (mg/kg)		Reference & Comments
	no	kg ai/ha	water (L/ha)				pendimethalin	Mean	
WI17, Portage, WI (Spearmint)	1	2.15	`186	Dormant	Foliage Foliage	84	<0.1 <0.1	<u><0.1</u>	Study code:A3888 DOC:2001/7002774
WI18 Portage, WI (Murray peppermint)	1	2.2	190	Dormant	Foliage Foliage	91	<0.1 <0.1	<0.1	Study code:A3888 DOC:2001/7002774
		10.1	193	Dormant	Foliage Foliage	91	0.219 0.103	0.161	Study code:A3888 DOC:2001/7002774
WA*49 Mabton, WA (Black Mitchum Peppermint)	1	2.21	291	Dormant	Foliage Foliage	90	<0.1 <0.1	<u><0.1</u>	Study code:A3888 DOC:2001/7002774
WA*50 Mabton, WA (Black Mitchum Peppermint)	1	2.21	291	Dormant	Foliage Foliage	90	<0.1 <0.1	<0.1	Study code:A3888 DOC:2001/7002774
WA*51 Mabton, WA (Black Mitchum Peppermint)	1	2.13	282	Dormant	Foliage Foliage	90	<0.1 <0.1	<0.1	Study code:A3888 DOC:2001/7002774
		10.35	295	Dormant	Foliage Foliage	90	<0.1 <0.1	<0.1	Study code:A3888 DOC:2001/7002774

FATE OF RESIDUES DURING PROCESSING

Six processing studies completed in the USA in 1994 and 1997 were available for mints (peppermint and spearmint).

Table 7 The estimated processing factors with the respective recommendations of pendimethalin are shown in the following table

Field Trial, Year Location Various	Applications			PHI	Residue in mint (mg/kg)	Residue in mint oil (mg/kg)	Transfer factor	Reference
	No	kg ai/ha	Method					
5523.94, 1994 WA*04 Spearmint	1	2.24	Soil broadcast	124	0.054	<0.05	/	IR4-study5523
5523.94, 1994 WA*04 Spearmint	1	11.20	Soil broadcast	124	<0.05	1.21	/	IR4-study5523
5523.94, 1994 OR07 Black Mitchum	1	2.24	Soil broadcast	145	<0.05	0.05 (maximum of 0.06 mg/kg in control sample)	/	IR4-study5523
5523.94, 1994 OR07 Black Mitchum	1	11.20	Soil broadcast	145	0.076	1.88	24.7	IR4-study5523
WI18, 1997 Portage, WI (Murray peppermint)	1	2.2	Soil broadcast	91	<0.1	0.61	/	Study code:A3888 DOC:2001/7002774
	1	10.1			0.219	7.84	35.8	
WA*51, 1997 Mabton, WA (Black Mitchum Peppermint)	1	2.13	Soil broadcast	90	<0.1	0.51	/	Study code:A3888 DOC:2001/7002774
	1	10.35			<0.1	3.84	/	

APPRAISAL

Methods of analysis

Pendimethalin is a meristematic inhibitor herbicide that interferes with plant cellular division or mitosis. Pendimethalin was first evaluated for toxicology and residues by the JMPR in 2016. The compound has an ADI of 0–0.1 mg/kg bw and an ARfD of 1 mg/kg bw. The residue definition for both plant and animal commodities for compliance with the MRL and dietary risk assessment is pendimethalin. The residue is fat soluble.

It was scheduled at the Fiftieth Session of the CCPR for the evaluation of additional uses by the 2019 Extra JMPR. The current Meeting received information on storage stability, use patterns and supervised residue trials for berries and herbs.

Storage stability of residues

The 2016 JMPR confirmed that pendimethalin residues in high water, high starch and high acid content matrices were stable for at least 24 months. In soya bean and almond nutmeat, pendimethalin was stable for up to 18 and 12 months, respectively. The frozen storage periods of samples in the trials submitted to the current Meeting were less than 18 and 24 months after sampling for berries and herbs, respectively.

Results of supervised residue trials on crops

The Meeting received supervised residue trial data for soil applications of pendimethalin on cane berries, blue berries, strawberries and mint.

Cane berries, subgroup of

The critical GAP for pendimethalin on cane berries in the USA is one soil application at a rate of 6.7 kg ai/ha and a PHI of 30 days.

Six supervised field trials were conducted on cane berries in the USA matching the critical GAP for soil application.

Residues of pendimethalin in blackberry were (n=4): < 0.05 (4) mg/kg.

Residues of pendimethalin in raspberry were (n=2): < 0.05 (2) mg/kg.

Noting that the US GAP covers the cane berries subgroup, the Meeting decided to estimate a maximum residue level of 0.05(*) mg/kg, STMR of 0.05 mg/kg and HR of 0.05 mg/kg for the cane berries subgroup.

Bush berries, subgroup of

The critical GAP for pendimethalin on bush berry in the USA is one soil application at a rate of 6.7 kg ai/ha and a PHI of 30 days.

Seven trials on blueberries were conducted in the USA matching the GAP.

In blueberries, residues of pendimethalin in these trials were (n=7): < 0.05 (7) mg/kg.

The Meeting noted that the US GAP is for bush berries, and decided to estimate a maximum residue level of 0.05(*) mg/kg, STMR of 0.05 mg/kg and HR of 0.05 mg/kg for the bush berries subgroup.

Strawberry

The critical GAP in Ireland and UK is one soil application at 1.3 kg ai/ha after flower initiation but before flower truss emergence. In six European trials at 1 kg ai/ha, residues of pendimethalin were <0.01 (6) mg/kg. In four other trials, with higher application rates of 1.6 kg ai/ha, residues were found from <0.01 to 0.016 mg/kg.

The critical GAP for pendimethalin in low growing berries including strawberry in the USA is 1 soil application at 3.2 kg ai/ha and a PHI of 35 days. In eight trials approximating the US GAP conducted in the USA, residues of pendimethalin were < 0.05 (8) mg/kg.

The Meeting decided to estimate a maximum residue level of 0.05(*) mg/kg, an STMR of 0.05 mg/kg, and an HR of 0.05 mg/kg for strawberry on basis of the trial data from the USA.

Mint

The critical GAP for pendimethalin on mint in the USA is 1 soil application of 2.24 kg ai/ha and a PHI of 90 days.

In four independent trials conducted in the USA on mint approximating the US GAP, residues of pendimethalin were (n=4): <0.05, 0.054, <0.1 and <0.1 mg/kg.

The Meeting decided to estimate a maximum residue level of 0.2 mg/kg, STMR of 0.077 mg/kg and HR of 0.1 mg/kg for mint.

Fate of residues during processing

Four studies were submitted on processing of mint to mint oil. In two trials with finite residue in mint leaves, residues in mint leaves were 0.076 and 0.219 mg/kg, and the residues in mint oil were 1.88 and 7.84 mg/kg. Processing factors were calculated to be 24.7 and 35.8. The best estimation of processing factor was 30.

The Meeting estimated a maximum residue level of 6 mg/kg and an STMR-P of 2.3 mg/kg for mint oil.

Residues in animal commodities

None of the commodities or their by-products for which supervised trial data were submitted to the current Meeting are fed to animals. The Meeting confirmed its previous recommendations for animal commodities.

RECOMMENDATIONS

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

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

The residue is fat soluble.

	Commodity	Recommended Maximum residue level (mg/kg)		STMR or STMR-P (mg/kg)	HR (mg/kg)
CCN	Name	New	Previous		
FB 2005	Cane berries, subgroup of	0.05*	-	0.05	0.05
FB 2006	Bush berries, subgroup of	0.05*	-	0.05	0.05
FB 0275	Strawberries	0.05*	-	0.05	0.05
HH 0738	Mints	0.2	-	0.077	0.1
OR 0738	Peppermint Oil, edible	6	-	2.3	

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for pendimethalin is 0–0.1 mg/kg bw. The International Estimated Daily Intakes (IEDIs) for pendimethalin 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 2019 Extra JMPR Report.

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

Acute dietary exposure

The ARfD for pendimethalin is 1 mg/kg bw. The International Estimate of Short Term Intakes (IESTIs) for pendimethalin 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 2019 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 pendimethalin from uses considered by the present Meeting is unlikely to present a public health concern.

Dietary risk of metabolites previously evaluated by the Meeting against their threshold of toxicological concern

The 2016 JMPR concluded that the dietary exposure to the metabolites M455H025, M455H029 and M455H030 are below the threshold of toxicological concern (TTC) of 1.5 µg/kg bw per day for a Cramer Class III compound.

Based on the uses evaluated by the current Meeting, the estimated dietary exposure to M455H025 increased from 1.30 to 1.32 µg/kg bw per day while the estimated dietary exposures to M455H029 (found in animal commodities) and M455H030 (found in rotated root crops only) remained unchanged.

The Meeting confirmed its previous conclusion that dietary exposure to the metabolites M455H025, M455H029 and M455H030 are unlikely to present a public health concern.

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Doc-ID No	Author	Year	Title
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Doc. No. 2018/7004894	Cheryl B. Cleveland	2018	Report summary for Pendimethalin magnitude of the residue on strawberry conducted by the US IR-4 Project. Unpublished
815209	Harald Weber Christian Gollner	2018	Storage Stability of Pendimethalin and its Metabolite M455H025 in Dried Peas, Carrots, Potatoes and Wheat Forage under Deep Frozen Caonditions. Unpublished
1997/7004981	Rose A.Sweeney Daneil L. Lunkel	1997	Volume 2 of 2 IR-4 Minor use submission in support of a tolerance for pendimethalin in or on mint. Unpublished
2001/7002774	Frederick P.Salzman, Ph.D	2001	Volume 2 of 2 Pendimethalin: Magnitude of the residue on mint. Unpublished

SPIROTETRAMAT (234)

First draft prepared by Dr C Anagnostopolous, Benaki Phytopathological Institute, Athens, Greece

EXPLANATION

Spirotetramat is a systemic insecticide for the control of a broad spectrum of sucking insects. It was first evaluated by JMPR in 2008 (T, R). The most recent residue evaluation was conducted in 2015 (R).

The 2008 JMPR established an ADI for spirotetramat of 0–0.05 mg/kg bw and an ARfD of 1 mg/kg bw. The residue definition for compliance with the MRL for plant commodities is spirotetramat plus spirotetramat enol, expressed as spirotetramat. The residue definition for estimation of dietary exposure for plant commodities is spirotetramat plus the metabolites enol, ketohydroxy, enol glucoside, and monohydroxy, expressed as spirotetramat. The residue definition for compliance with the MRL and dietary exposure for animal commodities is spirotetramat enol, expressed as spirotetramat. The residue is not fat soluble.

It was scheduled at the Fiftieth Session of the CCPR for the evaluation of additional uses by the 2019 Extra JMPR. New supervised trial data in three commodities (strawberries, carrot and sugar beet), new data on storage stability and processing studies in sugar beets were provided to the present meeting.

RESIDUE ANALYSIS

Analytical methods

Several analytical methods were developed for the residue analysis of spirotetramat (STM) in different matrices. In the framework of the current submission the analytical method 00857 was used and its modifications 00857/M005 and FN-007- P08-01. An overview of the use of the analytical methods is presented in the following Table 1.

Table 1 Overview of the analytical methods used under the current submission

Study used	Matrix	Analyte	Method No.	LOQ (mg/kg) [a]	Previous evaluation	Study code
M-475140-01-1 M-487160-01-1 M-358802-01-1 M-486221-01-1	**Carrots** **Sugar beet:** roots pulp dry molasses refined sugar	STM STM cis-enol, STM cis-ketohydroxy STM monohydroxy STM enol-glucoside (Glc)	00857	0.01	JMPR 2008	M-253112-03-01
M-610814-01-1	bean dry seed kiwi fruit		00857 (M005)	0.01	No	
M-487160-01-1 M-486221-01-1	**Sugar beet:** roots leaves		FN-007- P08-01	0.01	JMPR 2013	FN-007- P08-01

[a] (metabolites given as STM equivalents)

The analytical method 00857 was evaluated by the 2008 JMPR for the analysis of residues of spirotetramat (STM) and its metabolites, STM -enol, STM -ketohydroxy, STM -mono-hydroxy and STM -enol-Glc in sugar beet root and molasses. Percent recoveries (mean ± SD) were: 79 ± 9 at 0.01 mg/kg and 91 ± 5 at 1 mg/kg (root), and 90 ± 3 at 0.01 mg/kg and 107 ± 5 at 1 mg/kg (molasses).

The analytical method 00857 (M005) was applied for determination of residues in dry beans and kiwi fruit. Residues were extracted with an acidic acetonitrile/water mixture (4/1,v/v) filtered and quantitated by LC/MS/MS using stable isotopically labelled internal standards. The LOQ for each

analyte was 0.01 mg/kg (expressed as parent equivalents) thus the LOQ for the total residue is calculated to 0.05 mg/kg. The recoveries obtained during the validation of the method are summarized in Tables 2 and 3.

Table 2 Recoveries for STM, STM-enol, STM-ketohydroxy, STM-mono-hydroxy and STM-enol-Glc in/on dry bean seed

Study Trial No. Year	STM, metabolite	n	Spike level (mg/kg)	Recovery (%)				
				Individual recoveries	Min	Max	Mean	RSD
P642160506 2016	STM	3	0.01	102;107;100	100	107	103.0	3.5
	STM	3	0.1	101;97;99	97	101	99.0	2.0
	STM-enol	3	0.01	97;95;99	95	99	97.0	2.1
	STM-enol	3	0.1	93;98;99	93	99	96.7	3.3
	STM-ketohydroxy	3	0.01	90;88;100	88	100	92.7	6.9
	STM-ketohydroxy	3	0.1	82;84;86	82	86	84.0	2.4
	STM-mono-hydroxy	3	0.01	92;101;99	92	101	97.3	4.9
	STM-mono-hydroxy	3	0.1	99;96;101	96	101	98.7	2.6
	STM-enol-Glc	3	0.01	102;101;94	94	102	99.0	4.4
	STM-enol-Glc	3	0.1	96;97;100	96	100	97.7	2.1

Table 3 Recoveries for STM, STM-enol, STM-ketohydroxy, STM-mono-hydroxy and STM-enol-Glc in/on kiwi fruit

Study Trial No. Year	STM, metabolite	n	Spike level (mg/kg)	Recovery (%)				
				Individual recoveries	Min	Max	Mean	RSD
P642160506 2016	STM	3	0.01	89;90;109	89	109	96.0	11.7
	STM	3	0.1	94;93;87	87	94	91.3	4.1
	STM-enol	3	0.01	77;81;92	77	92	83.3	9.3
	STM-enol	3	0.1	87;93;86	86	93	88.7	4.3
	STM-ketohydroxy	3	0.01	90;106;109	90	109	101.7	10.0
	STM-ketohydroxy	3	0.1	97;97;99	97	99	97.7	1.2
	STM-mono-hydroxy	3	0.01	87;94;98	87	98	93.0	6.0
	STM-mono-hydroxy	3	0.1	94;91;93	91	94	92.7	1.6
	STM-enol-Glc	3	0.01	90;86;97	86	97	91.0	6.1
	STM-enol-Glc	3	0.1	91;86;89	86	91	88.7	2.8

The analytical method FN-007- P08-01 which is a modification 00857 was applied to determination of residues in sugar beet leaves and roots. The recoveries obtained during the validation of the method are summarized in Tables 4 and 5.

Table 4 Recoveries for STM, STM-enol, STM-ketohydroxy, STM-mono-hydroxy and STM-enol-Glc in/on sugar beet leaves

Study Trial No. Year	STM, metabolite	n	Spike level (mg/kg)	Recovery (%)				
				Individual recoveries	Min	Max	Mean	RSD
RAFNP073, FN-007-P08-01	STM	8	0.01	93;89;89	86	98	91.6	4.9
	STM	1	0.1	94	94	94	94.0	-
	STM	5	2	93;94;93	92	96	93.6	1.6
	STM-enol	8	0.01	96;89;95	87	98	93.6	4.3
	STM-enol	1	0.1	89	89	89	89.0	-
	STM-enol	5	2	94;98;93	93	98	95.2	2.0
	STM-enol-Glc	8	0.01	93;100;87	87	112	97.8	9.9
	STM-enol-Glc	1	0.1	83	83	83	83.0	-

Study Trial No. Year	STM, metabolite	n	Spike level (mg/kg)	Recovery (%)				
				Individual recoveries	Min	Max	Mean	RSD
	STM-enol-Glc	5	2	89;87;79	79	89	83.2	5.5
	STM-ketohydroxy	8	0.01	119;97;104	84	119	106.4	10.7
	STM-ketohydroxy	1	0.1	97	97	97	97.0	-
	STM-ketohydroxy	5	2	99;102;99	99	102	100.4	1.5
	STM-mono-hydroxy	8	0.01	106;89;107	86	107	94.8	8.2
	STM-mono-hydroxy	1	0.1	92	92	92	92.0	-
	STM-mono-hydroxy	5	2	93;95;93	90	95	92.4	2.1

Table 5 Recoveries for STM, STM-enol, STM-ketohydroxy, STM-mono-hydroxy and STM-enol-Glc in/on sugar beet roots

Study Trial No. Year	STM, metabolite	n	Spike level (mg/kg)	Recovery (%)				
				Individual recoveries	Min	Max	Mean	RSD
RAFNP073, FN-007-P08-01	STM	8	0.01	93;97;94	75	97	90.4	7.8
	STM	1	0.1	101	101	101	101.0	-
	STM	4	2	88;91;91	88	94	91.0	2.7
	STM-enol	8	0.01	90;96;89	85	103	92.5	6.9
	STM-enol	1	0.1	98	98	98	98.0	-
	STM-enol	4	2	98;92;94	92	98	94.5	2.7
	STM-enol-Glc	8	0.01	83;88;78	73	94	83.8	7.5
	STM-enol-Glc	1	0.1	89	89	89	89.0	-
	STM-enol-Glc	4	2	86;87;85	81	87	84.8	3.1
	STM-ketohydroxy	8	0.01	81;96;99	81	99	92.4	6.8
	STM-ketohydroxy	1	0.1	99	99	99	99.0	-
	STM-ketohydroxy	4	2	102;109;103	101	109	103.8	3.5
	STM-mono-hydroxy	8	0.01	90;101;93	83	104	92.5	7.7
	STM-mono-hydroxy	1	0.1	96	96	96	96.0	-
	STM-mono-hydroxy	4	2	101;92;89	89	101	95.8	6.5

Stability of pesticide residues in stored analytical samples

In the residue studies (field residue, processing of field samples) submitted to the current Meeting, samples were stored for up to approximately 7 months (carrot), 9 months (strawberry) or 14 months (sugar beet root; Table 6).

Table 6 Storage stability period of samples from residue field and processing trials

Matrix	Category	Longest storage duration (d)	Study Report No.
Carrot (roots)	high starch	226	IR-4 10788
Sugar beet (root)		412	RAFNP073
		372	RAFNP074
Strawberry (fruits)	high acid	287	08-2146

The Meeting received a storage stability study (M-610814-01-1) for dry beans and kiwi fruit. Samples of dry beans and kiwi fruits were spiked with 0.1 mg/kg of each analyte (STM, STM-enol, STM-ketohydroxy, STM-mono-hydroxy, STM-enol-Glc) separately and stored at -18 °C for approximately 30, 60, 90, 180, 370 and 540 days. Samples were analysed by LC-MS/MS method 00857/M005 using internal standards. Adequate method validation data were provided and reported above in section “Methods for residue analysis”. Samples spiked with spirotetramat were analysed for

all analytes and the total residues were expressed as spirotetramat equivalents. The results of the study are summarized in Table 7 for dry beans and Table 8 for kiwi fruit.

Table 7 Storage stability and procedural recovery data (fortified at 0.1 mg/kg) for spirotetramat and its related metabolites in dry beans.

Analyte	Storage period (days)	Residue levels in stored spiked samples			Single procedural recoveries [%]
		mg/kg (expressed as parent equivalents)	% of nominal spiking level	% remaining	
STM	0	0.093;0.103;0.1;0.103;0.098	93;103;100;103;99	99.6	103;99
	29	0.078;0.093;0.086	78;93;86	85.7	94;109
	90	0.08;0.085;0.08	80;85;81	82.0	102;106
	176	0.077;0.088;0.083	77;88;83	82.7	101;109
	367	0.075;0.079;0.076	75;79;76	76.7	101;110
	548	0.0631;0.0812;0.0763	63;81;76	73.3	105;93
STM-enol	0	0.086;0.087;0.092;0.089;0.095	86;88;92;89;95	90.0	88;90
	29	0.082;0.09;0.092	82;90;92	88.0	86;99
	90	0.083;0.094;0.091	83;94;91	89.3	99;101
	176	0.07;0.072;0.076	70;72;76	72.7	94;101
	367	0.09;0.086;0.085	90;86;85	87.0	101;109
	548	0.081;0.093;0.095	81;93;95	89.7	105;95
STM-ketohydroxy	0	0.096;0.107;0.111;0.106;0.104	96;107;111;106;104	104.8	94;95
	29	0.088;0.098;0.099	88;98;99	95.0	89;101
	90	0.097;0.097;0.102	97;97;102	98.7	96;101
	176	0.084;0.096;0.101	84;96;101	93.7	88;97
	367	0.09;0.093;0.088	90;93;89	90.7	93;102
	548	0.098;0.099;0.098	98;99;98	98.3	102;88
STM-mono-hydroxy	0	0.086;0.094;0.093;0.103;0.103	87;94;93;103;103	96.0	91;96
	29	0.09;0.087;0.088	90;87;88	88.3	90;95
	90	0.089;0.096;0.098	89;96;98	94.3	93;99
	176	0.086;0.1;0.102	86;100;102	96.0	91;101
	367	0.09;0.091;0.091	90;91;91	90.7	94;103
	548	0.097;0.092;0.103	97;92;103	97.3	104;92
STM-enol-Glc	0	0.089;0.102;0.086;0.108;0.103	90;102;86;108;103	97.8	96;89
	29	0.106;0.094;0.102	106;94;103	101.0	88;112
	90	0.091;0.101;0.099	91;101;99	97.0	95;101
	176	0.093;0.092;0.106	93;92;106	97.0	107;96
	367	0.101;0.103;0.098	101;103;98	100.7	96;108
	548	0.105;0.091;0.114	105;91;114	103.3	104;94

Table 8 Storage stability and procedural recovery data (fortified at 0.1 mg/kg) for spirotetramat and its related metabolites in kiwi fruit.

Analyte	Storage period (days)	Residue level in stored spiked samples			Single procedural recoveries [%]
		mg/kg (expressed as parent equivalents)	% of nominal spiking level	% remaining	
STM	0	0.099;0.097;0.095;0.093;0.081	99;97;95;93;81	93.0	92;103
	30	0.09;0.092;0.09	90;93;90	91.0	91;93
	90	0.1;0.1;0.085	100;100;85	95.0	106;97
	171	0.113;0.109;0.112	113;109;112	111.3	104;103
	364	0.111;0.101;0.103	111;101;104	105.3	107;111
	545	0.107;0.107;0.115	107;107;115	109.7	115;116[b]

Analyte	Storage period (days)	Residue level in stored spiked samples			Single procedural recoveries [%]
		mg/kg (expressed as parent equivalents)	% of nominal spiking level	% remaining	
STM-enol	0	0.081;0.081;0.084;0.08;0.074	81;82;84	80.2	76;82
	30	0.086;0.089;0.089	86;89;89	88.0	82;84
	90	0.09;0.108;0.093	90;108;93	97.0	102;90
	171	0.082;0.092;0.097	82;92;97	90.3	86;89
	364	0.096;0.1;0.099	96;100;99	98.3	102;105
	545	0.103;0.102;0.101	103;102;101	102.0	102;108
STM-ketohydroxy	0	0.094;0.093;0.099;0.093;0.098	94;93;99	95.4	88;88
	30	0.09;0.094;0.088	90;94;88	90.7	92;92
	90	0.085;0.087;0.086	85;88;86	86.3	97;86
	171	0.099;0.099;0.104	99;99;104	100.7	85;87
	364	0.098;0.104;0.105	98;104;105	102.3	97;101
	545	0.107;0.106;0.096 [c]	107;106;96	103.0	102;105;88[c]
STM-mono-hydroxy	0	0.088;0.089;0.092;0.094;0.083	88;89;92	89.2	84;96
	30	0.087;0.087;0.085	87;87;85	86.3	88;90
	90	0.075;0.077;0.089	75;77;89	80.3	89;84
	171	0.087;0.1;0.094	87;100;94	93.7	88;93
	364	0.106;0.106;0.1	106;106;100	104.0	97;103
	545	0.103;0.104;0.106	103;105;106	104.7	98;105
STM-enol-Glc	0	0.106;0.099;0.097;0.102;0.106	106;99;97	102.0	87;93
	30	0.095;0.096;0.095	95;96;95	95.3	82;98
	90	0.073;0.088;0.09	73;88;90	83.7	93;90
	171	0.095;0.085;0.08	95;85;80	86.7	78;83
	364	0.09;0.083;0.102	90;83;102	91.7	88;94
	545	0.087;0.083;0.081	87;83;81	83.7	85;76

Spirotetramat and its metabolites STM-enol. STM-ketohydroxy. STM-mono-hydroxy. STM-enol-Glc are stable in the different matrix types (*high acid* and *high protein*) for at least 18 months (kiwi fruit 545 days. bean dry 548 days) when stored at ≤ -18 °C. Overall, these results validate the residue values reported in all supervised field trials and processing studies with respect to storage stability of samples frozen prior to analysis.

USE PATTERN

The use patterns relevant to the residue data submitted for evaluation by the present JMPR meeting are summarized in Table 9. Spirotetramat 240 SC and 100 SC are suspension concentrate (SC) formulations containing 240 g ai/L and 100 g ai/L, respectively.

Table 9 Registered uses of spirotetramat 240 SC and 100 SC formulations on carrots. strawberries and sugar beets

Crop	Country	Application							PHI (days)
		method	No. max	Interval (min)	kg ai/hL min/max	Water L/ha min/max	kg ai/ha max	Total/season. kg ai/ha (max)	
Carrot	Canada (outdoor)	Foliar/ ground	2	7		Min 200	0.09	0.18	1
Carrot	USA (outdoor)	Foliar/ Ground or aerial	2	7		140.3[a] 46.8[b]	0.09	0.18	1 (application at infestation)

Crop	Country	Application							PHI (days)
		method	No. max	Interval (min)	kg ai/hL min/ max	Water L/ha min/ max	kg ai/ha max	Total/season. kg ai/ha (max)	
Sugar beet	Canada (outdoor)	Foliar/ground	2	14		Min 200	0.09	0.31	28
Sugar beet	USA (outdoor)	Foliar/ Ground or aerial	2	14		93.5 [a] 46.8 [b]	0.157	0.31	28
Strawberry	Spain (indoor)	Foliar	2	14	0.05 - 0.1	300-1000	0.1	0.2	at infestation (up to BBCH 13-56)

[a] min water rate for ground applications

[b] min water rate for aerial applications

RESULTS OF SUPERVISED RESIDUE TRIALS ON CROPS

The residue trials were conducted with two formulations containing spirotetramat: OD 150 (150 g ai/L) and SC 240 (240 g ai/L). Trials were generally well documented with laboratory and field reports. Laboratory reports included method validation with procedural recoveries from spiking at residue levels similar to those occurring in samples from the supervised trials. Dates of analyses or duration of residue sample storage were also provided. Although trials included control plots, no control data are recorded in the tables except where residues in control samples exceeded the LOQ. Unless stated otherwise, residue data are recorded unadjusted for recovery.

All residues presented for the metabolites are expressed as parent equivalents. Where a component is reported as <'value', the <'value' is added into the calculation of the total equivalents.

Strawberry (FB 0275)

Eight supervised field residue trials were conducted with Spirotetramat 100 SC on indoor strawberries in Europe (Belgium, France, Germany, Italy, the Netherlands and Spain) in the growing seasons of 2008 and 2009 (M-358802-01-1).

Spirotetramat 100 SC was applied to strawberries twice at the nominal and actual application rate of 0.1 kg ai/ha with re-treatment intervals of 11 to 14 days. Strawberry fruits were collected 14 to 63 days after the final application. No decline study was submitted. The maximum storage period of deep-frozen samples before analysis was 287 days (approximately 10 months).

Residues of STM and its four metabolites were analysed using method 00857. Procedural recoveries for spirotetramat and its metabolites in strawberry fruits were performed at 3 spiking levels (0.01, 0.1, and 1 mg/kg). Recoveries were calculated for the parent and each metabolite separately. The average recoveries were 91–99%. The RSD was ≤ 9.8%. The full dataset on strawberries is presented in Table 10.

Table 10 Results of residue trials conducted with SC 100 formulation on indoor strawberries fruits in Europe in 2008

Location (Variety)	Application				PHI (days)	Residues (mg/kg) as parent equivalent							Reference
	Rate (kg ai/ha)	Volume (L/ha)	No. (RTI. days)	BBCH		STM	STM-enol	STM-ketohydroxy	STM-enol-Glc	STM-mono-hydroxy	Sum of STM and STM-enol	Total residue of STM	
Spain critical GAP: 2 x 0.1 kg ai/ha. BBCH 13-56. 14 days interval. n.a. PHI													
Belgium (Elsanta)	0.1 0.1	650 650	2 (13)	55-57	32	0.01	0.04	0.01	<0.01	<0.01	0.05	0.09	08-2146-01-T
France (Ronde)	0.1 0.1	800 800	2 (14)	55-56	39	<0.01	0.04	<0.01	<0.01	<0.01	0.05	0.08	08-2146-02-T
Netherlands (Elsanta)	0.1 0.1	600 600	2 (14)	55-56	22	0.03	0.12	0.02	<0.01	<0.01	0.15	0.19	08-2146-03-T
Italy (Candanga)	0.1 0.1	1000 1000	2 (14)	55-56	14	0.05	0.10	0.02	<0.01	<0.01	0.15	0.19	08-2146-04-T
Spain (Ventana)	0.1 0.1	1000 1000	2 (14)	14-57	63	<0.01	0.02	<0.01	<0.01	<0.01	0.03	0.06	08-2146-05-T
France (Pajaro)	0.1 0.1	1000 1000	2 (14)	55-56	47	<0.01	0.04	<0.01	<0.01	<0.01	0.05	0.08	08-2146-06-T
Germany (Darselect)	0.1 0.1	300 300	2 (14)	55-56	50	<0.01	0.03	0.02	<0.01	<0.01	0.04	0.07	08-2146-07-T
Germany (Darselect)	0.1 0.1	400 400	2 (11)	55-56	34	<0.01	0.01	<0.01	<0.01	<0.01	0.02	0.05	08-2146-08-T

Carrot (VR 0577)

Eight supervised field residue trials were conducted with Spirotetramat SC 240 on carrots in the USA during the growing seasons of 2012 and 2013 (M-475140-01-1). Spirotetramat 240 SC was applied to carrot two times at the nominal application rate of 0.09 kg ai/ha (actual application rates ranged from 0.87 to 0.94 kg ai/ha) with re-treatment intervals of seven to eight days.

Carrot roots were collected 1 to 2 days after the final application. One decline trial (10788.12-WA) was conducted with harvest occurring 0. 1, 3, 7 and 14 days after the last application.

The maximum storage period of deep-frozen samples before analysis was 226 days.

Residues of STM and its four metabolites were analysed using method 00857. including minor modifications. Procedural recoveries for spirotetramat and its metabolites in carrots were performed at 3 spiking levels (0.01. 0.1. 1 mg/kg). Recoveries were calculated for the parent and each metabolite separately. The average recoveries were [put in actual range of recoveries]. The RSD was ≤14.2%. The full dataset on carrots is presented in Table 11.

Table 11 Results of residue trials conducted with SC 240 formulations on carrots in USA

Location (Variety)	Application			PHI (days)	Residues (mg/kg)							Reference
	Rate (g ai/ha)	Volume (L/ha)	No. (RTI. days)		STM	STM-enol	STM-ketohydroxy	STM-enol-Glc	STM-mono-hydroxy	Sum of STM and STM-enol	Total residue of STM	
Canada and USA critical GAP: 2 x 0.09 kg ai/ha. at infestation. 7 days interval. PHI of 1 day												
USA	0.087	543	2 (7)	1	<0.01	0.019	<0.01	<0.01	<0.01	0.029	0.059	10788.12-CA*36
2012	0.087	664		1	<0.01	0.019	<0.01	<0.01	<0.01	0.029	0.059	
(Laguna)				Mean:	<0.01	0.019	<0.01	<0.01	<0.01	0.029	0.059	
USA	0.092	384	2 (7)	1	<0.01	<0.01	0.026	<0.01	<0.01	<0.02	0.066	10788.12-CA34
2012	0.094	393		1	<0.01	<0.01	0.034	<0.01	<0.01	<0.02	0.074	
(Danvers 126)				Mean:	<0.01	<0.01	0.03	<0.01	<0.01	<0.02	0.07	
USA	0.090	234	2 (8)	2	<0.01	<0.01	<0.01	<0.01	<0.01	<0.02	<0.05	10788.12-CA35
2013	0.090	234		2	<0.01	<0.01	<0.01	<0.01	<0.01	<0.02	<0.05	
(Enterprise)				Mean:	<0.01	<0.01	<0.01	<0.01	<0.01	<0.02	<0.05	
USA	0.091	309	2 (7)	1	<0.01	<0.01	<0.01	<0.01	<0.01	<0.02	<0.05	10788.12-GA*03
2012	0.089	299		1	<0.01	<0.01	<0.01	<0.01	<0.01	<0.02	<0.05	
(Imperator 58)				Mean:	<0.01	<0.01	<0.01	<0.01	<0.01	<0.02	<0.05	
USA	0.087	786	2 (7)	1	<0.01	<0.01	0.074	<0.01	<0.01	<0.02	0.114	10788.12-NM03
2012	0.089	786		1	<0.01	<0.01	0.055	<0.01	<0.01	<0.02	0.095	
(Danvers 126)				Mean:	<0.01	<0.01	0.064	<0.01	<0.01	<0.02	0.1	
USA	0.089	374	2 (7)	1	<0.01	<0.01	<0.01	<0.01	<0.01	<0.02	<0.05	10788.12-OH*04
2012	0.090	402		1	<0.01	<0.01	<0.01	<0.01	<0.01	<0.02	<0.05	
(Maverick)				Mean:	<0.01	<0.01	<0.01	<0.01	<0.01	<0.02	<0.05	
USA	0.090	253	2 (7)	1	<0.01	0.019	<0.01	<0.01	<0.01	0.029	0.059	10788.12-TX05
2013	0.090	253		1	<0.01	0.020	<0.01	<0.01	<0.01	0.03	0.06	
(Sugar Snax 54)				Mean:	<0.01	0.02	<0.01	<0.01	<0.01	0.03	0.06	
USA	0.092	496	2 (7)	0	<0.01	<0.01	<0.01	<0.01	<0.01	<0.02	<0.05	10788.12-WA*10
2012	0.090	477		0	<0.01	<0.01	<0.01	<0.01	<0.01	<0.02	<0.05	
(Hilmar)				1	<0.01	<0.01	<0.01	<0.01	<0.01	<0.02	<0.05	
				1	<0.01	<0.01	<0.01	<0.01	<0.01	<0.02	<0.05	
				Mean:	<0.01	<0.01	<0.01	<0.01	<0.01	<0.02	<0.05	
				3	<0.01	<0.01	<0.01	<0.01	<0.01	<0.02	<0.05	
				3	<0.01	<0.01	<0.01	<0.01	<0.01	<0.02	<0.05	
				7	<0.01	<0.01	<0.01	<0.01	<0.01	<0.02	<0.05	
				7	<0.01	<0.01	<0.01	<0.01	<0.01	<0.02	<0.05	
				14	<0.01	<0.01	<0.01	<0.01	<0.01	<0.02	<0.05	
				14	<0.01	<0.01	<0.01	<0.01	<0.01	<0.02	<0.05	

Sugar beet

Seventeen supervised field residue trials were conducted using Spirotetramat SC 240 and OD 150 on sugar beets grown in Canada and the USA during the growing seasons of 2012 and 2013 (M-487160-01-1). From these trials fifteen were considered independent.

Spirotetramat 240 SC or OD 150 were applied to sugar beets two times at the nominal application rate of 0.158 kg ai/ha. For the formulation 240 SC, the actual application rates ranged from 0.156 to 0.161 kg ai/ha per application, with re- treatment intervals of 12–14 days. For the 150 OD, application rates ranged from 0.155–0.165 kg ai/ha per application, re- treatment intervals of 12-15 days.

Sugar beet leaves and roots were collected 28 to 33 days after the final application. In three additional decline trials (FN026-12DA- TRTDO. FN035-12DA- TRTDO and FN037-12DA- TRTDO). samples were collected at 25, 30 (33), 35, 42 and 49 days after the last application. The maximum storage period of deep-frozen samples before analysis was 412 days (approx. 14 months).

Residues of STM and its four metabolites were analysed using methods 00857 and FN-007-P08-01. Procedural recoveries for spirotetramat and its metabolites in sugar beet roots and leaves were performed at 3 spiking levels (0.01, 0.1 and 2 mg/kg). Recoveries were calculated for the parent and each metabolite separately. The average recoveries were 85–106%. The RSD was ≤10.7%. The full dataset on sugar beets is presented in Table 11.

Table 11 Results of residue trials conducted with SC 240 and OD 150 formulations on sugar beet roots in Canada and the USA.

Location Year (Variety)	Form	Application			PHI (days)	Residues (mg/kg)							Reference
		Rate (kg ai/ha)	Vol (L/ha)	No. (RTI. days)		STM	STM-enol	STM-ketohydroxy	STM-enol-Glc	STM-mono-hydroxy	Sum of STM and STM-enol	Total residue of STM	
Canada and USA critical GAP: 2 x 0.157 kg ai/ha. 14 days interval. PHI of 28 day													
USA 2012 (variety not stated)	OD Surf.	0.158 0.159	187 187	2 (12)	29 29 Mean:	<0.01 <0.01 <0.01	0.012 0.012 0.012	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	0.022 0.022 0.022	0.052 0.052 0.052	FN022-(4022RR) 12HA-TRTDO (OD formulation) 12HA-TRTDS (SD formulation)
	SC Surf.	0.158 0.159	187 187	2 (14)	29 29 Mean:	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.02 <0.02 <0.02	<0.05 <0.05 <0.05	
USA 2012 (variety not stated)	OD Surf.	0.159 0.155	141 138	2 (14)	31 31 Mean:	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.02 <0.02 <0.02	<0.05 <0.05 <0.05	FN023-(RZ07RR08) 12HA-TRTDO (OD formulation) 12HA-TRTDS (SD formulation)
	SC Surf.	0.159 0.158	141 141	2 (14)	31 31 Mean:	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.02 <0.02 <0.02	<0.05 <0.05 <0.05	
USA 2012 (variety not stated)	OD Surf.	0.160 0.159	119 118	2 (14)	30 30 Mean:	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.02 <0.02 <0.02	<0.05 <0.05 <0.05	FN024-12HA-TRTDO
USA 2012 (Poncho Beta)	OD Surf.	0.161 0.160	143 142	2 (15)	31 31 Mean:	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.02 <0.02 <0.02	<0.05 <0.05 <0.05	FN025-12HA-TRTDO
USA 2012 (Poncho Beta)	OD Surf.	0.160 0.159	142 142	2 (14)	25 25 30 30 Mean: 35 35 42 42 49 49	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01	0.014 0.012 <0.01 0.010 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01 <0.01<0.01 <0.01 <0.01 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01	0.024 0.022 <0.02 0.02 0.02 <0.02 <0.02 <0.02 <0.02 <0.02 <0.02	0.054 0.052 <0.05 0.05 0.05 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05	FN026-12DA-TRTDO
Canada 2012 (BTS-47RR75-Proso)	OD Surf.	0.161 0.163	153 154	2 (14)	30 30 Mean	<0.01 <0.01 <0.01	0.016 0.018 0.018	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	0.026 0.028 0.027	0.056 0.058 0.057	FN027-12HA-TRTDO
Canada 2012 (Hilleshog	OD Surf.	0.159 0.160	100 101	2 (13)	33 33 Mean:	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.02 <0.02 <0.02	<0.05 <0.05 <0.05	FN028-12HA-TRTDO

Location Year (Variety)	Form	Application			PHI (days)	Residues (mg/kg)							Reference
		Rate (kg ai/ha)	Vol (L/ha)	No. (RTI. days)		STM	STM-enol	STM-ketohydroxy	STM-enol-Glc	STM-mono-hydroxy	Sum of STM and STM-enol	Total residue of STM	
Canada and USA critical GAP: 2 x 0.157 kg ai/ha. 14 days interval. PHI of 28 day													
HM7211RZ)													(OD formulation)
	SC Surf.	0.160 161	100 102	2 (13)	33 33 Mean:	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.02 <0.02 <u><0.02</u>	<0.05 <0.05 <u><0.05</u>	12HA-TRTDS (SC formulation)
Canada k 2012 (Hilleshog HM7211RZ) *[Trial was conducted in similar location as FN028-12HA-TRTDO]*	OD Surf.	0.159 0.158	100 100	2 (14)	28 28 Mean:	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.02 <0.02 <0.02	<0.05 <0.05 <0.05	F FN029-12HA-TRTDO
Canada 2013 (Hilleshog HM7211RZ)	OD Surf.	0.157 0.160	99 101	2 (14)	29 29 Mean	<0.01 <0.01 <0.01	0.016 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	0.026 <0.02 <u>0.023</u>	0.056 <0.05 <u>0.053</u>	FN030-12HA-TRTDO
Canada 2013 (BTS-47RR75-Proso) *[Trial was conducted in similar location as FN032-12HA-TRTDO]*	OD Surf.	0.161 0.160	81 81	2 (14)	29 29 Mean:	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.02 <0.02 <0.02	<0.05 <0.05 <0.05	FN031-12HA-TRTDO
Canada 2013 (Beta 49RR33) *[Trial was conducted in similar location as FN031-12HA-TRTDO]*	OD Surf.	0.165 0.165	187 186	2 (16)	28 28 Mean:	<0.01 <0.01 <0.01	0.016 0.017 0.016	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	0.026 0.027 <u>0.027</u>	0.056 0.057 <u>0.057</u>	FN032-12HA-TRTDO
USA 2012 (Phoenix)	OD Surf.	0.161 0.164	179 183	2 (13)	34 34 Mean	<0.01 <0.01 <0.01	0.011 0.016 0.014	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	0.021 0.026 <u>0.024</u>	0.051 0.056 <u>0.054</u>	FN033-12HA-TRTDO
USA Jerome 2012 (Crystal RR876)	OD Surf.	0.159 0.159	177 171	2 (14)	30 30 Mean:	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.02 <0.02 <0.02	<0.05 <0.05 <0.05	FN034-12HA-TRTDO (OD formulation) 12HA-TRTDS (SC formulation)
	SC Surf.	0.160 0.161	178 173	2 (14)	30 30 Mean:	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.01 <0.01 <0.01	<0.02 <0.02 <u><0.02</u>	<0.05 <0.05 <u><0.05</u>	
TRTDO USA Sanger	OD Surf.	0.157 0.161	139 144	2 (12)	25 33	<0.01 <0.01	0.018 0.011	<0.01 <0.01	<0.01 <0.01	<0.01 <0.01	0.028 0.021	0.058 0.051	FN035-12DA-

Location Year (Variety)	Form	Application			PHI (days)	Residues (mg/kg)							Reference
		Rate (kg ai/ha)	Vol (L/ha)	No. (RTI. days)		STM	STM-enol	STM-ketohydroxy	STM-enol-Glc	STM-mono-hydroxy	Sum of STM and STM-enol	Total residue of STM	
Canada and USA critical GAP: 2 x 0.157 kg ai/ha. 14 days interval. PHI of 28 day													
2012 (*variety not stated)*					33	<0.01	0.017	<0.01	<0.01	<0.01	0.027	0.057	
					35	<0.01	0.012	<0.01	<0.01	<0.01	0.022	0.052	
					35	<0.01	0.018	<0.01	<0.01	<0.01	0.028	0.058	
					Mean	<0.01	0.015	<0.01	<0.01	<0.01	0.025	0.055	
					42	<0.01	0.016	<0.01	<0.01	<0.01	0.026	0.056	
					42	<0.01	0.011	<0.01	<0.01	<0.01	0.021	0.051	
					49	<0.01	<0.01	<0.01	<0.01	<0.01	<0.02	<0.05	
					49	<0.01	<0.01	<0.01	<0.01	<0.01	<0.02	<0.05	
USA Porterville 2012 (Phoenix)	OD Surf.	0.155	163	2 (14)	30	<0.01	<0.01	<0.01	<0.01	<0.01	<0.02	<0.05	FN036-12HA-TRTDO (OD formulation) 12HA-TRTDS (SC formulation)
		0.159	166		30	<0.01	0.011	<0.01	<0.01	<0.01	0.021	0.051	
					Mean	<0.01	0,011	<0.01	<0.01	<0.01	0.021	0.051	
	SC Surf.	0.158	166	2 (14)	30	<0.01	<0.01	<0.01	<0.01	<0.01	<0.02	<0.05	
		0.159	165		30	<0.01	0.011	<0.01	<0.01	<0.01	<0.02	<0.05	
					Mean	<0.01	0.011	<0.01	<0.01	<0.01	<0.02	<0.05	
USA Minidoka 2012 (Crystal RR929)	OD Surf.	0.162	157	2 (14)	25	<0.01	0.015	<0.01	<0.01	<0.01	0.025	0.055	FN037-12DA-TRTDO
		0.169	125		25	<0.01	0.018	<0.01	<0.01	<0.01	0.028	0.058	
					30	<0.01	0.020	<0.01	<0.01	<0.01	0.030	0.060	
					30	<0.01	0.020	<0.01	<0.01	<0.01	0.030	0.060	
					Mean:	<0.01	0.02	<0.01	<0.01	<0.01	0.030	0.060	
					35	<0.01	0.016	<0.01	<0.01	<0.01	0.026	0.056	
					35	<0.01	0.014	<0.01	<0.01	<0.01	0.024	0.054	
					42	<0.01	0.011	<0.01	<0.01	<0.01	0.021	0.051	
					42	<0.01	0.014	<0.01	<0.01	<0.01	0.024	0.054	
					49	<0.01	<0.01	<0.01	<0.01	<0.01	<0.02	<0.05	
					49	<0.01	<0.01	<0.01	<0.01	<0.01	<0.02	<0.05	
USA 2012 (BTS28RR4N)	OD Surf.	0.162	173	2 (13)	28	<0.01	0.032	<0.01	<0.01	<0.01	0.042	0.072	FN038 12HA-TRTDO (OD formulation) 12HA-TRTDS (SC formulation)
		0.160	170		28	<0.01	0.032	<0.01	<0.01	<0.01	0.042	0.072	
					Mean:	<0.01	0.032	<0.01	<0.01	<0.01	0.042	0.072	
	SC Surf.	0.159	170	2 (14)	28	<0.01	0.012	<0.01	<0.01	<0.01	0.022	0.052	
		0.156	167		28	<0.01	<0.01	<0.01	<0.01	<0.01	<0.02	<0.05	
					Mean:	<0.01	0.012	<0.01	<0.01	<0.01	0.022	0.052	

Table 12 Results of residue trials conducted with SC 240 and OD 150 formulations on sugar beets leaves in Canada and the USA.

Location Year (Variety)	Form	Application			PHI (days)	Residues (mg/kg)							Reference
		Rate (kg ai/ha)	Volume (L/ha)	No. (RTI. days)		STM	STM-enol	STM-ketohydroxy	STM-enol-Glc	STM-mono-hydroxy	Sum of STM and STM-enol	Total residue of STM	
Canada and the USA critical GAP: 2 x 0.157 kg ai/ha. 17 days interval. PHI of 28 day													
USA 2012 (variety not stated)	OD Surf.	0.158	187	2	29	0.31	0.12	0.15	0.02	<0.01	0.43	0.61	FN022-12HA-TRTDO
		0.159	187	(12)	29	0.42	0.13	0.18	0.02	<0.01	0.55	0.77	
					Mean						0.49	0.69	

Location Year (Variety)	Form	Application			PHI (days)	Residues (mg/kg)							Reference
		Rate (kg ai/ha)	Volume (L/ha)	No. (RTI. days)		STM	STM-enol	STM-ketohydroxy	STM-enol-Glc	STM-mono-hydroxy	Sum of STM and STM-enol	Total residue of STM	
Canada and the USA critical GAP: 2 x 0.157 kg ai/ha. 17 days interval. PHI of 28 day													
	SC Surf.	0.158 0.159	187 187	2 (14)	29 29 Mean	0.39 0.34	0.10 0.12	0.15 0.13	<0.01 0.012	<0.01 <0.01	0.49 0.46 0.48	0.67 0.61 0.64	(OD formulation) 12HA- TRTDS (SD formulation)
USA 2012 (variety not stated)	OD Surf.	0.159 0.155	141 138	2 (14) 2	31 31 Mean	0.1 0.12	0.085 0.085	0.061 0.037	0.065 0.064	<0.01 <0.01	0.18 0.19 0.19	0.32 0.30 0.31	FN023-(RZ07RR08) 12HA-TRTDO (OD formulation) 12HA- TRTDS (SD formulation)
	SC Surf.	0.159 0.158	141 141	2 (14)	31 31 Mean	0.12 0.17	0.071 0.084	0.041 0.048	0.015 0.019	<0.01 <0.01	0.19 0.25 0.22	0.26 0.33 0.30	
USA 2012 (variety not stated)	OD Surf.	0.160 0.159	119 118	2 (14)	30 30 Mean	0.015 0.010	0.010 <0.01	0.011 <0.01	0.067 0.049	<0.01 <0.01	0.025 0.02 0.023	0.11 0.089 0.10	FN024-12HA- TRTDO
USA 2012 (Poncho Beta)	OD Surf.	0.161 0.160	143 142	2 (15)	31 31 Mean	0.42 0.53	0.130 0.18	0.12 0.085	0.043 0.067	<0.01 <0.01	0.57 0.71 0.64	0.72 0.87 0.80	FN025-12HA-TRTDO
USA 2012 (Poncho Beta)	OD Surf.	0.160 0.159	142 142	2 (14)	25 25 30 30 Mean 35 35 42 42 49 49	0.19 0.12 0.096 0.085 0.066 0.072 0.047 0.052 0.073 0.042	0.12 0.21 0.16 0.14 0.12 0.15 0.077 0.080 0.11 0.069	0.075 0.099 0.078 0.090 0.097 0.069 0.061 0.088 0.10 0.071	0.019 0.023 0.018 0.018 0.019 0.021 0.015 0.024 0.020 0.013	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01	0.3 0.33 0.25 0.23 0.24 0.19 0.22 0.12 0.13 0.18 0.11	0.4 0.46 0.36 0.34 0.25 0.31 0.32 0.21 0.25 0.31 0.21	FN026-12DA-TRTDO
Canada 2012 (BTS-47RR75-Proso)	OD Surf.	0.161 0.163	153 154	2 (14)	30 30 Mean	0.18/ 0.015 ** 0.120	0.23 0.2	0.051 0.045	0.053 0.056	<0.01 <0.01	0.41 0.32 0.37	0.52 0.43 0.48	FN027-12HA-TRTDO
Canada 2012 (Hilleshog HM7211RZ)	SC Surf.	0.160 0.161	100 102	2 (13)	33 33 Mean	0.056 0.056	0.023 0.029	0.027 0.027	0.013 0.016	<0.01 <0.01	0.079 0.085 0.082	0.13 0.14 0.14	FN028-12HA-TRTDO (OD formulation) 12HA- TRTDS (SC formulation)
	OD Surf.	0.159 0.160	100 101	2 (13)	33 33 Mean	0.064 0.12	0.037 0.060	0.024 0.038	0.033 0.037	<0.01 <0.01	0.1 0.1 0.14	0.18 0.27 0.23	
Canada k 2012 (Hilleshog HM7211RZ) *[Trial was conducted in similar location as FN028-12HA-TRTDO]*	OD Surf.	0.159 0.158	100 100	2 (14)	28 28 Mean	0.052 0.042	0.029 0.037	0.018 0.023	0.023 0.025	<0.01 <0.01	0.081 0.079 0.080	0.13 0.14 0.14	F FN029-12HA-TRTDO
Canada 2013 (Hilleshog HM7211RZ)	OD Surf.	0.157 0.160	99 101	2 (14)	29 29 Mean	0.076 0.048	0.072 0.054	0.030 0.016	0.080 0.061	<0.01 <0.01	0.15 0.10 0.13	0.27 0.19 0.23	FN030-12HA-TRTDO

Location Year (Variety)	Form	Application			PHI (days)	Residues (mg/kg)							Reference
		Rate (kg ai/ha)	Volume (L/ha)	No. (RTI. days)		STM	STM-enol	STM-ketohydroxy	STM-enol-Glc	STM-mono-hydroxy	Sum of STM and STM-enol	Total residue of STM	
Canada and the USA critical GAP: 2 x 0.157 kg ai/ha. 17 days interval. PHI of 28 day													
Canada 2013 (BTS-47RR75-Proso) *[Trial was conducted in similar location as FN032-12HA-TRTDO]*	OD	0.161	81	2	29	<0.01	0.020	<0.01	0.030	<0.01	0.030	0.080	FN031-12HA-TRTDO
	Surf.	0.160	81	(14)	29	<0.01	0.017	<0.01	0.028	<0.01	0.027	0.075	
					Mean						0.028	0.078	
Canada 2013 (Beta 49RR33) *[Trial was conducted in similar location as FN031-12HA-TRTDO]*	OD	0.165	187	2	28	0.13	0.11	0.043	0.021	<0.01	0.23	0.31	FN032-12HA-TRTDO
	Surf.	0.165	186	(16)	28	0.14	0.095	0.040	0.027	<0.01	0.21	0.29	
					Mean						0.22	0.30	
USA 2012 (Phoenix)	OD	0.161	179	2	30	0.016	0.034	0.052	0.052	<0.01	0.050	0.16	FN033-12HA-TRTDO
	Surf.	0.164	183	(13)	30	0.036	0.049	0.090	0.073	<0.01	0.085	0.26	
					Mean						0.068	0.21	
USA Jerome 2012 (Crystal RR876)	OD	0.159	177	2	30	0.013	0.028	0.018	0.023	<0.01	0.041	0.092	FN034-12HA-TRTDO (OD formulation) 12HA- TRTDS (SC formulation)
	Surf.	0.159	171	(14)	30	<0.01	0.015	0.017	0.018	<0.01	0.025	0.070	
					Mean						0.033	0.081	
	SC	0.160	178	2	30	<0.01	<0.01	0.013	<0.01	<0.01	<0.02	0.053	
	Surf.	0.161	173	(14)	30	<0.01	<0.01	<0.01	<0.01	<0.01	<0.02	<0.05	
					Mean						<0.02	0.052	
TRTDO USA Sanger 2012 (*variety not stated)*	OD	0.157	139	2	25	0.33	0.	0.15	0.066	<0.01	0.48	0.71	FN035-12DA-TRTDO
	Surf.	0.161	144	(12)	33	0.38	0.16	0.16	0.080	<0.01	0.55	0.79	
					33	0.36	0.15	0.099	0.086	<0.01	0.51	0.79	
					Mean						0.53	0.75	
					35	0.18	0.074	0.063	0.037	<0.01	0.25	0.36	
					35	0.19	0.086	0.057	0.075	<0.01	0.28	0.42	
					42	0.061	0.093	0.13	0.046	<0.01	0.15	0.34	
					42	0.027	0.030	0.037	0.036	<0.01	0.057	0.14	
					49	0.011	0.012	0.011	0.027	<0.01	0.023	0.071	
					49	<0.01	0.012	0.014	0.022	<0.01	0.022	0.068	
USA Porterville 2012 (Phoenix)	OD	0.155	163	2	30	<0.01	<0.01	<0.01	0.047	<0.01	<0.02	0.087	FN036-12HA-TRTDO (OD formulation) 12HA- TRTDS (SC formulation)
	Surf.	0.159	166	(14)	30	<0.01	<0.01	0.012	0.014	<0.01	<0.02	0.056	
					Mean						<0.02	0.072	
	SC	0.158	166	2	30	<0.01	<0.01	<0.01	0.017	<0.01	<0.02	0.057	
	Surf.	0.159	165	(14)	30	<0.01	<0.01	<0.01	0.022	<0.01	<0.02	0.062	
											<0.02	0.060	
USA Minidoka 2012 (Crystal RR929)	OD	0.162	157	2	25	0.019	0.052	0.038	0.029	<0.01	0.071	0.15	FN037-12DA-TRTDO
	Surf.	0.169	125	(14)	25	0.035	0.055	0.044	0.028	<0.01	0.090	0.17	
					30	0.019	0.043	0.032	0.035	<0.01	0.062	0.14	
					30	0.017	0.035	0.031	0.029	<0.01	0.052	0.12	
					Mean						0.057	0.13	
					35	0.013	0.032	0.023	0.048	<0.01	0.045	0.17	
					35	0.018	0.028	0.029	0.032	<0.01	0.046	0.10	
					42	<0.01	0.025	0.030	0.029	<0.01	0.035	0.1	
					42	<0.01	0.023	0.027	0.040	<0.01	0.033	0.11	
					49	<0.01	0.027	0.028	0.055	<0.01	0.037	0.13	
					49	<0.01	0.024	0.017	0.042	<0.01	0.034	0.1	
USA	OD	0.162	173	2	28	0.79	0.32	0.19	0.05	<0.01	1.1	1.4	
	Surf.	0.160	170	(13)	28	0.92	0.3	0.17	0.062	<0.01	1.2	1.5	FN038

Location Year (Variety)	Form	Application			PHI (days)	Residues (mg/kg)							Reference
		Rate (kg ai/ha)	Volume (L/ha)	No. (RTI. days)		STM	STM-enol	STM-ketohydroxy	STM-enol-Glc	STM-mono-hydroxy	Sum of STM and STM-enol	Total residue of STM	
Canada and the USA critical GAP: 2 x 0.157 kg ai/ha. 17 days interval. PHI of 28 day													
2012 (BTS28RR4N)					mean						1.2	1.5	12HA-TRTDO (OD formulation) 12HA- TRTDS (SC formulation)
	SC	0.159	170	2	28	1.03	0.28	0.19	0.015	<0.01	1.3	1.5	
	Surf.	0.156	167	(14)	28	1.2	0.4	0.22	0.014	<0.01	1.6	1.8	
					Mean						1.4	1.7	

FATE OF RESIDUES DURING PROCESSING

Sugar beet (refined sugar. dried pulp. and molasses)

Two processing studies on sugar beets were carried out in USA (M-486221-01-1). Two supervised field residue trials were carried about in Minnesota USA with Spirotetramat SC 240 on sugar beets. The trial plot received two foliar broadcast applications with the test substance (spirotetramat 240 SC) 12 or 16 days apart. The application rates were in the range 0.79–0.81 kg ai/ha per application.

Sugar beet roots (RAC) were collected 29 or 31 days after the final application. Triplicate sub-samples of the RAC were removed and the remaining sugar beet root samples were processed into dried pulp, molasses and refined sugar.

Samples were weighed and cleaned. During cleaning. heavy deposits of soil were removed from the roots. Loose leaves and foreign matter were also removed. Cleaned beets were chopped into cossettes. During diffusion. cossesstes were exposed successively to water bathes at 88–92 °C for 30–45 seconds and 68–74 °C five times for 9 minutes. After diffusion, the raw juice was filtered and the diffused cossetes were dewatered with a hydraulic press and then dried in an oven at 54–71 °C to a final moisture content of 15% or less.

The juice from dewatering and diffusion was combined and 2 stages of phosphatisation were performed. During the 1st stage. juice was heated to 80–85 °C. adjusted to pH 10.5 (calcium oxide) and centrifuged. At the 2nd stage. centrifuged juice was re-heated to 80–85 °C. adjusted to pH 9.1–9.3 (3M phosphoric acid). re-centrifuged and filtrated. The thin juice that was produced was heated again to 80–85 °C. adjusted to pH to 8.8–9.0 (sodium bisulfite) and cooled overnight. After overnight cooling. thick juice was obtained by evaporation of thin juice until 50–60 brix. Thick juice was then filtered and evaporated until a 70–80 brix. Crystallisation was started by adding a small amount of sugar. After cooling and crystallisation. the sugar and molasses were separated by centrifugation. Sugar was then dried in an oven at 54–71 °C to final moisture of 1%.

The maximum storage period of frozen samples (< 0 °C) before analysis was 372 days (approx. 12 months).

Residues of STM and its four metabolites were analysed using methods 00857 and FN-007-P08-01. Procedural recoveries for spirotetramat and its metabolites in sugar beet roots. dried pulp. refined sugar and molasses were performed at 2 spiking levels (0.01 and 2 mg/kg). Recoveries were calculated for the parent and each metabolite separately. The average recoveries were 76–103%. The RSD was ≤ 10.5%.

After two foliar spray applications at the rate of 0.79–0.81 kg ai/ha (5×-dose) the residues of parent spirotetramat at harvest were up to 0.022 mg/kg in sugar beet roots. The residues of the metabolites STM cis-enol and STM cis-ketohydroxy amounted to 0.01–0.037 mg/kg and < 0.01–0.071 mg/kg. respectively. whereas the residues of STM enol-glucoside and monohydroxy were not detected above LOQ. The sum of STM and STM cis-enol ranged from 0.02 to 0.059 mg/kg and total residue of STM 08330 from 0.05 to 0.15 mg/kg.

Table 13 Residues on sugar beet processed fractions from the foliar application of spirotetramat (Freeseman, P.L.: Lenz, C. 2014)

Location Year	portion analysed	Residue (mg/kg) expressed as spirotetramat equivalents							Processing Factor		
		STM	STM-enol	STM-ketohydroxy	STM-enol-Glc	STM-mono-hydroxy	Sum of STM and STM-enol	Total residue of STM	Enforcement	Risk assessment	reference
USA 2012	root[a]	0.022	0.037	0.071	<0.01	<0.01	0.059	0.15	-	-	FN039-12PA-TRT5X
	pulp, dry	<0.01	0.035	0.040	<0.01	<0.01	0.045	0.11	0.8	0.7	
	molasses	<0.01	0.15	<0.01	<0.01	<0.01	0.16	0.19	1.3	2.7	
	refined sugar	<0.01	<0.01	<0.01	<0.01	<0.01	<0.02	<0.05	<0.3	<0.3	
USA 2012	root[a]	<0.01	0.01	<0.01	<0.01	<0.01	0.02	0.05	-	-	FN040-12PA-TRT5X
	pulp, dry	<0.01	0.015	<0.01	<0.01	<0.01	0.025	0.055	1.3	1.1	
	molasses	<0.01	0.09	<0.01	<0.01	<0.01	0.1	0.13	2.6	5	
	refined sugar	<0.01	<0.01	<0.01	<0.01	<0.01	<0.02	<0.05	<1	<1	

[a] Mean from 3 replicate analyses.

APPRAISAL

Spirotetramat is a systemic insecticide for the control of a broad spectrum of sucking insects. It was first evaluated by JMPR in 2008 (T, R). The latest residue evaluation was conducted in 2015 (R).

The 2008 JMPR established an ADI for spirotetramat of 0–0.05 mg/kg bw and an ARfD of 1 mg/kg bw.

The residue definition for compliance with the MRL for plant commodities is *spirotetramat plus spirotetramat enol, expressed as spirotetramat*.

The residue definition for estimation of dietary exposure for plant commodities is *spirotetramat plus the metabolites enol, ketohydroxy, enol glucoside, and monohydroxy, expressed as spirotetramat*.

The residue definition for compliance with the MRL and dietary exposure for animal commodities is *spirotetramat enol, expressed as spirotetramat*.

The residue is not fat soluble.

It was scheduled at the Fiftieth Session of the CCPR for the evaluation of additional uses by the 2019 Extra JMPR. New supervised trial data in 3 commodities (strawberries, carrot and sugar beet), new data on storage stability and processing studies in sugar beets were provided to the present meeting.

Methods of analysis

Analytical methods used in raw agricultural commodities from field trials were suitable for quantifying spirotetramat residues including the metabolites spirotetramat enol, spirotetramat ketohydroxy, spirotetramat monohydroxy and spirotetramat enol glucoside in the various plant commodities. The methods were based on LC-MS/MS and the reference method used was evaluated by the Meeting in 2008 and 2013. The limits of quantitation (LOQ) for the raw commodities are 0.01 mg/kg (expressed as parent equivalents) for each analyte and 0.05 mg/kg for total spirotetramat equivalents.

For the determination of residues in dry beans and kiwi fruit a modification M005 of the analytical method 00857 was applied. The limit of quantification was 0.01 mg/kg for individual residues. The residues of individual analytes were expressed as spirotetramat equivalents and summed up to yield the total residue of spirotetramat plus enol (LOQ 0.02 mg/kg) and spirotetramat plus 4 metabolites (LOQ 0.05 mg/kg). The recoveries for individual residue components were tested at 0.01 and 0.1 mg/kg for dry beans and kiwi fruit, and their relative standard deviations were within an acceptable range.

In addition, the analytical method FN-007-P08-01 which is a modification 00857, was applied to determination of residues in sugar beet leaves and roots. The residues of individual analytes were expressed as spirotetramat equivalents and summed up to yield the total residue of spirotetramat plus enol (LOQ 0.02 mg/kg) and spirotetramat plus 4 metabolites (LOQ 0.05 mg/kg). The recoveries for individual residue components were tested at 0.01, 0.1 and 2 mg/kg for both leaves and roots and their relative standard deviations were within an acceptable range.

Stability of pesticides in stored analytical samples

Individual data on storage stability of spirotetramat and its metabolites were evaluated by the JMPR in 2008. The Meeting concluded that spirotetramat including its enol metabolite was stable (≥80% remaining) for up to 2 years in tomato, lettuce, climbing French beans, tomato paste (*high water*), potato (*high starch*) and almond nutmeat (*high oil*) stored frozen for intervals typical of storage prior to analysis.

An additional storage stability study on dry beans (*high protein*) and kiwi fruit (*high acid*) was submitted (M-610814-01-1). Spirotetramat and its metabolites STM-enol, STM-ketohydroxy, STM-mono-hydroxy and STM-enol-Glc are stable for at least 18 months (kiwi fruit 545 days, bean dry 548 days) when stored at ≤ -18 °C.

Results of supervised residue trials on crops

The Meeting received supervised residue trial data for the foliar application of spirotetramat as a suspension concentrate (SC) or oil dispersion (OD) formulation to carrots, sugar beets and strawberries.

In the discussions below, spirotetramat plus enol residues are considered first for the estimation of maximum residue levels followed by total residues (spirotetramat plus the metabolites enol, ketohydroxy, monohydroxy, and enol glucoside, expressed as spirotetramat) for estimation of STMR and HR values for the dietary risk assessments.

All residues presented for the metabolites are expressed as parent equivalents. Where a component is reported as <'value', the <'value' is added into the calculation of the total equivalents.

Strawberry

In Spain, spirotetramat is registered for indoor use on strawberries at a rate of 2×0.1 kg ai/ha, with a 14-day retreatment interval. No explicit PHI was indicated as the last application is growth stage specific, i.e., up to BBCH 56 (inflorescence elongating). Eight residue trials were conducted in the EU approximating the Spanish GAP.

Residues of the *sum of spirotetramat and spirotetramat -enol* from the trials were (n=8): 0.02, 0.03, 0.04, 0.05(3) and 0.15(2) mg/kg.

Total residues of spirotetramat from the trials were (n=8): 0.05, 0.06, 0.07, 0.08(2), 0.09 and 0.19(2) mg/kg.

The Meeting estimated a maximum residue level of 0.3 mg/kg and an STMR of 0.08 mg/kg and an HR of 0.19 mg/kg for strawberries.

Carrot

The critical GAP is from the registration in USA on carrots, at a rate of 2x0.09 kg ai/ha, a 7-day retreatment interval and a 1-day PHI. Eight residue trials were conducted in the USA approximating the critical GAP.

Residues of the sum of spirotetramat and spirotetramat -enol from the trials were (n=8): < 0.02(6), 0.029 and 0.030 mg/kg in roots.

Total residues of spirotetramat from the trials were (n=8): < 0.05(4), 0.059, 0.060, 0.07, and 0.1 (highest individual residue of 0.114) mg/kg in roots.

The Meeting estimated a maximum residue level of 0.04 mg/kg and an STMR of 0.0545 mg/kg and an HR of 0.114 mg/kg for carrots.

Sugar beet, roots

In Canada and the USA, spirotetramat is registered for the use on sugar beets at a rate of 2×0.16 kg ai/ha, a 14-day retreatment interval with a 28 day PHI. Seventeen residue trials were conducted in Canada (six trials) and the USA (11 trials) approximating the Canadian and US GAPs. From these only fifteen trials were considered independent.

Residues of the sum of spirotetramat and spirotetramat -enol from the trials were (n=15): < 0.02(5), 0.02, 0.021, 0.022, 0.023, 0.024, 0.025, 0.027(2), 0.030 and 0.042 mg/kg.

Total residues of spirotetramat from the trials were (n=15): <0.05(5), 0.05, 0.051, 0.052, 0.053, 0.054, 0.055, 0.057(2), 0.06 and 0.072 mg/kg.

The Meeting estimated a maximum residue level of 0.06 mg/kg, an STMR of 0.052 mg/kg and a highest residue of 0.072 mg/kg for sugar beet roots

Animal feedstuffs

Sugar beet, leaves and tops

In the USA and Canada, spirotetramat is registered for the use on sugar beets at a rate of 2×0.16 kg ai/ha, a 14-day retreatment interval with a 28 day PHI. Seventeen residue trials were conducted in Canada (six trials) and the USA (11 trials) approximating the Canadian and US GAPs. From the above only fifteen trials were considered independent.

Residues of sum of spirotetramat and spirotetramat -enol from the trials were (n=15): < 0.02, 0.023, 0.033, 0.057, 0.068, 0.13, 0.14, 0.22 (2), 0.24, 0.37, 0.49, 0.53, 0.64 and 1.45 mg/kg in sugar beet leaves or tops (as received).

Total residues of spirotetramat from the trials were (n=15): 0.072, 0.081, 0.10, 0.13, 0.21, 0.23 (2), 0.25, 0.3, 0.31, 0.48, 0.69, 0.75, 0.8 and 1.7 mg/kg in sugar beet leaves or tops (as received).

The Meeting estimated a maximum residue level of 8 mg/kg [expressed on dry weight basis (23% DM content)] and a median residue of 0.25 mg/kg and an highest residue of 1.7 mg/kg for sugar beet leaves or tops (as received)

Fate of residues during processing

The processing factors derived from the processing studies and the resulting recommendations for STMR-Ps, HR-Ps, and/or maximum residue levels are summarized in the table below.

RAC	Processed Commodity	Processing Factor (mean)	RAC MRL	Processed Commodity MRL	RAC STMR	Processed Commodity STMR-P
Sugar beet (roots)	dried pulp	*Risk assessment:* 0.7, 1.1 (0.9) *Enforcement:* 0.8, 1.3 (1.05)	0.06	-	0.052	0.047
	molasses	*Risk assessment:* 1.3, 2.6 (1.95) *Enforcement:* 2.7, 5 (3.85)	0.06	0.3	0.052	0.1
	refined sugar	*Risk assessment/ Enforcement:* <0.3, <1 (<0.65)	0.06	-	0.052	0.034

Each value represents a separate study. The factor is the ratio of the total residue in the processed item divided by the total residue in the RAC. The total residue is the parent spirotetramat plus four metabolites, calculated as spirotetramat.

In cases were residues in the processing item was <LOQ, the LOQ value (in this case was 0.02 for sum of spirotetramat and spirotetramat -enol and 0.05 mg/kg for total residues of spirotetramat) was used and the PF included the "<"

symbol.

Residues in animal commodities

Estimated maximum and mean dietary burdens of livestock

Dietary burdens were calculated for beef cattle, dairy cattle, broilers and laying poultry based on the feed items evaluated by the current (carrots, sugar beet tops, pulp, and molasses) and previous Meetings. The calculations were made according to the animal diets listed in Appendix IX of the 2016 edition of the FAO manual.

Animal dietary burden, spirotetramat total residue, mg/kg of dry matter diet					
		US-Canada	EU	Australia	Japan
Beef cattle	max	1.4	6.53	40[a]	0.52
	mean	0.65	3.37	19.0[b]	0.52
Dairy cattle	max	10.2	7.2	22.3	0.47
	mean	5.1	3.37	10.8	0.47
Poultry Broiler	max	0.27	0.63	0.39	0.24
	mean	0.27	0.46	0.39	0.24
Poultry Layer	max	0.27	4.9	0.39	0.24
	mean	0.27	2.3	0.39	0.24

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

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

The spirotetramat dietary burden for animal commodities reached a level of 42.4 ppm for cattle and of 0.6 ppm for poultry burdens. These results are only slightly higher than the previous cattle livestock dietary burden calculations performed in the 2011 JMPR (highest maximum beef or dairy cattle dietary burden of 40 ppm) and below the levels for poultry (4.8 ppm). The meeting confirmed its previous recommendations for animal commodities.

RECOMMENDATIONS

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

The residue definition for compliance with the MRL for plant commodities is spirotetramat plus spirotetramat enol, expressed as spirotetramat.

The residue definition for estimation of dietary exposure for plant commodities is spirotetramat plus the metabolites enol, ketohydroxy, enol glucoside, and monohydroxy, expressed as spirotetramat.

The residue definition for compliance with the MRL and dietary exposure for animal commodities is spirotetramat enol, expressed as spirotetramat.

The residue is not fat soluble

CCN	Commodity name	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
FB 0275	Strawberry	0.3	-	0.08	0.19
VR 0577	Carrot	0.04	-	0.0545	0.114
VR 0596	Sugar beet roots	0.06	-	0.052	0.072

CCN	Commodity name	Recommended Maximum residue level (mg/kg)		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
					(highest residue)
AV 0596	Sugar beet leaves or tops (dry)	8 (dw)	-	0.25 (median residue)	1.7 (highest residue)
DM 0596	Sugar beet molasses	0.3	-	0.1	-
-	Sugar	-	-	0.034	-

Additional values used in estimating livestock dietary burdens

CCN	Commodity name	Median residue (-P) mg/kg	highest residue (-P) mg/kg
AB 0596	Sugar beet pulp, Dry	0.047	-

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for spirotetramat is 0–0.05 mg/kg bw. The International Estimated Daily Intakes (IEDIs) for spirotetramat 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 2019 Extra JMPR Report.

The IEDIs ranged from 2-20% of the maximum ADI. The Meeting concluded that long-term dietary exposure to residues of spirotetramat from uses considered by the JMPR is unlikely to present a public health concern.

Acute dietary exposure

The ARfD for spirotetramat is 1 mg/kg bw. The International Estimate of Short Term Intakes (IESTIs) for spirotetramat 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 2019 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 spirotetramat from uses considered by the present Meeting is unlikely to present a public health concern.

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Report number	Author(s)	Year	Title. Source. Company name. Report No.. Date. GLP status published or not
			GLP/GEP: Yes. unpublished
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RAFNP074	Freeseman. P. L.; Lenz. C.	2014	Spirotetramat 240 SC - Magnitude of the residue in/on sugar beet processed commodities Bayer CropScience LP. RTP. NC. USA Bayer Report includes Trial Nos.: FN039-12PA FN040-12PA Edition Number: M-486221-01-1 MRID#: 49879109 Date: 2014-05-07 GLP/GEP: Yes. unpublished

TEBUCONAZOLE (189)

First draft prepared by Dr C Anagnostopolous, Benaki Phytopathological Institute, Athens, Greece

EXPLANATION

Tebuconazole is a triazole fungicide in the DMI (demethylation inhibitor) class. Tebuconazole was first evaluated by JMPR in 1994 (T, R). The latest residue evaluation was conducted in 2017 (R).

The 2010 JMPR review of tebuconazole reaffirmed an ADI of 0–0.03 mg/kg bw and established an ARfD of 0.3 mg/kg bw. The residue definition for compliance with the MRL and for estimation of dietary exposure for plant and animal commodities is parent tebuconazole. The residue is not fat soluble.

Tebuconazole was scheduled at the Fiftieth Session of the CCPR for the evaluation of additional uses by the 2019 Extra JMPR. The meeting received residue studies to support the uses in citrus fruits.

RESIDUE ANALYSIS

Analytical methods

The analytical method HW-002-P09-01 (HW-002-P09-01) was used to measure residues of tebuconazole in orange fruit, pulp, oil and juice. Tebuconazole residues are extracted by adding 3:1 v/v acetone:water to an aliquot of plant matrix followed by blending. An isotopic internal standard (IS) is added to the extract and the sample is capped and mixed. The sample was then filtered and vialed. An aliquot of the sample was analysed by LC-MS/MS using a C18 column and the determination was performed in ESI positive MRM mode, however only one MRM transition was monitored instead of two. Since the method was used for data collection in the residue trials, the confirmation of the target analyte which is tebuconazole is not questioned due to the absence of a 2nd transition. Validation was evaluated by recovery from spiked samples. The limit of quantitation was 0.01 mg/kg. Mean recovery values for the individual sample materials and spiking levels (spiking level 0.01–10 mg/kg) were in the range of 80–118% (relative standard deviations 0.8–13.6%). The recoveries obtained during the validation of the method for all 4 matrices are summarized in Table 1 below.

Table 1 Recoveries for tebuconazole in/on various orange commodities (method HW-002-P09-01)

Sample /method	Spike level [mg/kg)	No of tests	Mean recovery [%]	Recovery range [%]	RSD [%]
Orange, fruit	0.01	3	109	107-111	2.2
	0.10	3	104	99-106	3.9
	10.0	3	106	103-110	3.5
Orange, dried pulp	0.01	3	106	106-108	0.8
	0.10	3	105	105-107	1.0
	50.0	3	114	112-118	2.9
Orange, oil	0.01	3	111	98-119	10.7
	0.10	3	85	80-94	8.5
	150	3	103	100-105	2.6
Orange, juice	0.01	3	114	113-116	1.1
	0.1	3	109	106-113	3.4

USE PATTERN

The use pattern relevant for the residue data submitted for evaluation by the present JMPR meeting are summarized in Table 2. Tebuconazole EW 25 is an oil-in-water emulsion (EW) formulation containing 250 g ai/L of the active substance.

Table 2 Registered use of tebuconazole on citrus

Crop	Country	Formulation Type	Application method	No.	Interval (min)	Growth stage	Kg ai/hL	PHI days
Citrus	ES	EW	Post-harvest (drench spray)	1	-	Fruits	0.1	0

RESULTS OF SUPERVISED RESIDUE TRIALS ON CROPS

Citrus fruits

Eight residue trials were conducted in Germany in 1996 (RA-2076/96), with Tebuconazole 250 EW to determine the residues of tebuconazole on mandarin (4) and oranges (4) following one post-harvest spray application. The harvested fruit were at growth stage BBCH 79–89 at the time of application.

Fruits were placed in eight crates in single layers. Each crate contained between 233 to 280 mandarins or 135 to 207 oranges. Tebuconazole 250 EW, an oil-in-water emulsion, was applied at a concentration of 0.4% product (0.1% active ingredient; 0.1 kg ai/hL). The homogeneity of the application solution in the whole area of the fruits was verified with filter paper evenly spaced between the crates. Fruits were stored in dark at 4 °C.

Samples were collected at 0 day and 3, 7 and 13 (14) days after the treatment. The maximum storage period of deep-frozen samples before analysis was up to 273 days.

Residues of tebuconazole were analysed using Methods 00462 and 00462/E001. The LOQ was 0.05 mg/kg for tebuconazole in fruit, peel and pulp. Procedural recoveries for tebuconazole in fruit, peel and pulp were performed at 3 spiking levels (0.05, 0.5 and 5 mg/kg). The average recoveries were 77–89%. The RSD was ≤13%. The full dataset on oranges and mandarins is presented in Table 3 for mandarins and in Table 4 for oranges.

Table 3 Results of residue trials conducted in 1996 in Germany with Tebuconazole 250 EW on mandarins after indoor post-harvest treatment

Crop Variety	Application			Residues			Reference
	No	kg/hL (ai)	GS	Portion analysed	DALT (days)	tebuconazole (mg/kg)	
Satsumas	1	0.1	85	fruit	0	<u>0.48</u>	RA-2076/96
					3	0.43	60272/8
					7	0.33	0272-96
					13	0.35	
				pulp	3	<0.05	
					13	<0.05	
				peel	3	1.4	
					13	1.3	
Mandarin (*Cleme-nules*)	1	0.1	83	fruit	0	<u>0.38</u>	RA-2076/96
					3	0.36	60578/6
					7	0.35	0578-96
					13	0.32	
				pulp	3	<0.05	
					13	<0.05	
				peel	3	1.1	
					13	1.5	

Crop Variety	Application			Residues			Reference
	No	kg/hL (ai)	GS	Portion analysed	DALT (days)	tebuconazole (mg/kg)	
Mandarin (*Clause-llina*)	1	0.1	89	fruit	0	0.46	RA-2076/96 60804/1 0804-96
					3	0.35	
					7	0.38	
					13	0.32	
				pulp	3	<0.05	
					13	<0.05	
				peel	3	1.3	
					13	1.4	
Mandarin (*Nova*)	1	0.1	79	fruit	0	0.40	RA-2076/96 60807/6 0807-96
					3	0.35	
					7	0.33	
					13	0.30	
				pulp	3	<0.05	
					13	<0.05	
				peel	3	1.1	
					13	1.2	

Table 4 Results of residue trials conducted in 1996 in Germany with Tebuconazole 250 EW on oranges after indoor post-harvest treatment

Crop Variety	Application			Residues			Reference
	No	kg/hL (ai.)	GS	Portion analysed	DALT (days)	tebuconazole (mg/kg)	
Orange (*Navel)*	1	0.1	85	fruit	0	0.22	RA-2076/96 60273/6 0273-96
					3	0.23	
					7	0.25	
					14	0.27	
				pulp	3	<0.05	
					14	<0.05	
				peel	3	0.82	
					14	0.83	
Orange (*Lanetate*)	1	0.1	82	fruit	0	0.28	RA-2076/96 60577/8 0577-96
					3	0.25	
					7	0.28	
					14	0.20	
				pulp	3	<0.05	
					14	<0.05	
				peel	3	1.2	
					14	1.2	
Orange (*Navelina*)	1	0.1	87	fruit	0	0.20	RA-2076/96 60806/8 0806-96
					3	0.23	
					7	0.25	
					14	0.20	
				pulp	3	<0.05	
					14	<0.05	
				peel	3	0.92	
					14	0.84	

Crop Variety	Application			Residues			Reference
	No	kg/hL (ai.)	GS	Portion analysed	DALT (days)	tebuconazole (mg/kg)	
Orange (*New Hall*)	1	0.1	89	fruit	0	0.23	RA-2076/96
					3	0.27	60808/4
					7	0.25	0808-96
					14	0.20	
				pulp	3	<0.05	
					14	<0.05	
				peel	3	0.91	
					14	0.90	

Fate of residues in processing

Oranges (marmalade, juice)

Two processing studies were carried out in Germany (RA-3076/96) in order to determine the residues of tebuconazole in the processed commodities juice and marmalade. Tebuconazole EW 250 (an oil-in-water emulsion) was applied at a spray concentration of 0.4% product (0.1 kg ai/hL) in 240 L/ha wax, i.e. 0.024 g tebuconazole/m^2) on harvested fruits in two different residue trials. Oranges for processing were collected 3 days after treatment. Fruit samples were processed into marmalade and juice, simulating both household and commercial practice. Processed samples were stored for up to 223–250 days before analysis. Residues of tebuconazole were determined according to Method Nos. 00462 and 00462/E001. The LOQ was 0.05 mg/kg. The processing procedures for peeled fruits, marmalade and juice are described below.

Preparation of peeled oranges: Orange samples were peeled with a knife. Peel and pulp were separated and homogenized in the presence of dry ice.

Preparation of Orange Marmalade: Orange fruits were washed and peeled with a knife. Subsequently the peel was cut into small strips and the fruit pulp was minced with a mixer and subsequently passed through a strainer to separate pulp waste and fruit puree. Sugar, gelling agent and the peel strips were added to the fruit puree. The orange marmalade was heated to 98–100 °C for about 3 minutes. After cooking, the marmalade samples were taken and stored at -18 °C.

Preparation of orange juice (pasteurized): Orange fruits were washed and peeled with a knife. The peeled oranges were pressed into pulp waste and raw juice. After pressing the raw orange juice was pasteurized at temperatures up to 85 °C. After pasteurization, juice samples were taken and were stored at -18 °C.

The results of the trials are summarized in Table 5. No residues of tebuconazole above the LOQ were found in the control sample.

Table 5 Results of marmalade and juice (pasteurized) processing trials conducted in 1996 in Germany with Tebuconazole 250 EW on oranges

Crop Variety	Residues		Processing factor	References
	Portion analysed	Tebuconazole (mg/kg)		
Orange (*Navelina*)	Fruit (RAC)	0.23		RA-2076/96
	juice	<0.05	*not calculated**	60806/8
	marmalade	<0.05	<0.22	0806-96
Orange (*New Hall*)	Fruit (RAC)	0.27		RA-2076/96
	juice	<0.05	*not calculated**	60808/4
	marmalade	0.17	0.63	0808-96

*PF was not calculated since fruits were peeled before processing.

Citrus (pomace, oil, juice)

One processing study was conducted in Southern California (RAHWN001) in order to determine the residues of tebuconazole in the processed commodities dried pulp (pomace), pasteurized juice and oil. Tebuconazole 250 g/L EW was applied at a spray concentration of 0.4% product (0.1 kg ai/hL) on harvested fruits. Following treatment, the treated fruit was placed into cold storage for two to three days to simulate commercial post-harvest storage practices. Following cold storage, the fruit (RAC) samples were removed and triplicate subsamples were taken. The remaining oranges were processed to generate the processed commodities of dried pulp (pomace), oil, and juice (pasteurized). Samples were stored up to 103 days before analysis. Residues of tebuconazole were determined according to method No. HW-002-P09-01. The LOQ was 0.01 mg/kg. Mean procedural recoveries were 88–114% with RSD below 14% for all citrus matrices. The processing procedures for dried pulp (pomace), oil, and juice (pasteurized) are described below:

Preparation of oil: Before processing fruits were cleaned with a rotating brush and washed with warm water. From oil extraction, fruits were passed through a scarifier to scarify the flavedo (epicarp), and the collected flavedo and oil-water emulsion were passed through a mesh to separate the water emulsion from the flavedo fragments. The water emulsion was passed through a separator to separate the oil.

Preparation of juice (pasteurized): An aliquot of the scarified oranges was transferred to a juicer to separate the juice from the peel. The collected juice was screened and pasteurized (89 °C, 15sec.)

Preparation of dried pulp (pomace): The collected peel from the juicing process along with the scarified flavedo was combined to generate the wet peel/pulp. Calcium oxide was added and the limed peel was pressed and then dried to below 10% moisture.

The results of the trial are summarized in Table 6. In orange fruit, average residues of tebuconazole were at 5.6 mg/kg (3 days after treatment). After processing, residues were 40.2 mg/kg in dried pulp (pomace), 137 mg/kg in oil and 0.036 mg/kg in juice. No residues of tebuconazole above the LOQ were found in the control samples, except for oil (0.067 mg/kg).

Table 6 Results of dried pulp (pomace), oil, and juice (pasteurized) processing trials conducted in USA in 2012 with tebuconazole 250 EW in/on oranges

Crop Variety	Residues			References
	Portion analysed	Tebuconazole (mg/kg)	Processing factor	
Orange (*Valencia*)	Fruit (RAC) juice oil pomace, dried	5.6 0.036 137/0.067** 40.2	 *not calculated** 24.5 7.2	RAHWN001 HWN001-12PA

* PF was not calculated since fruits were peeled before processing

** residues in control

The Meeting received two processing studies for oranges. An overview of the available processing factors derived in the current evaluation is presented in Table 7 below.

Table 7 Overall summaries of the available Processing Factors in oranges

RAC	Processed Commodity	Processing Factors [best estimate]
oranges	marmalade	<0.22, 0.63 [0.63]
	oil	24.5
	pomace, dried	7.2

APPRAISAL

Tebuconazole is a triazole fungicide in the DMI (demethylation inhibitor) class. Tebuconazole was first evaluated by JMPR in 1994 (T, R). The latest residue evaluation was conducted in 2017 (R).

The 2010 JMPR review of tebuconazole reaffirmed an ADI of 0–0.03 mg/kg bw and established an ARfD of 0.3 mg/kg bw. The residue definition for compliance with the MRL and for estimation of dietary exposure for plant and animal commodities is parent tebuconazole. The residue is not fat soluble.

It was scheduled at the Fiftieth Session of the CCPR for the evaluation of additional uses by the 2019 Extra JMPR. The Meeting received additional residue studies to support the additional uses in citrus fruits.

Methods of analysis

One new analytical method (HW-002-P09-01) was submitted that was used in the processing studies and is considered suitable for the determination of tebuconazole residues in orange fruits, dried pulp, oil and juice. The method is based on a simple extraction with 3:1 v/v acetone:water followed by determination with LC-MS/MS. The LOQ of the method is set at 0.01 mg/kg.

Stability of residues in stored analytical samples

Storage stability studies were not provided to the current Meeting. The 2011 Meeting concluded that residues of tebuconazole are stable in high-acid commodities for at least 30 months in frozen storage. Samples considered by the current meeting were stored for up to 273 days (ca. 9 months).

Results of supervised residue trials on crops

In Spain, tebuconazole is registered for post-harvest use on citrus fruits as a drench spray with a concentration of 0.1 kg ai/hL; no withholding period is specified. Four trials each for mandarins and oranges were conducted approximating the Spanish GAP. For post-harvest treatment the variability is expected to be significantly less than that of field trials thus four trials can be considered sufficient.

Mandarins (Subgroup of)

In mandarins (whole fruit), residues of tebuconazole were (n=4): 0.38, 0.40, 0.46 and 0.48 mg/kg. Residues in pulp were < 0.05 (4) mg/kg.

The Meeting estimated a maximum residue level of 0.7 mg/kg (mean + 4SD) in mandarin subgroup. Based on residues in pulp, the Meeting estimated a STMR of 0.05 mg/kg and HR of 0.05 mg/kg in mandarin subgroup.

Oranges, Sweet, Sour (subgroup of)

In oranges (whole fruit), residues of tebuconazole were (n=4): 0.25, 0.27 (2), and 0.28 mg/kg. Residues in pulp were < 0.05 (4) mg/kg, and residues in peel were (n=4): 0.83, 0.91, 0.92, and 1.2 mg/kg.

The Meeting estimated a maximum residue level of 0.4 mg/kg (mean + 4SD) in orange, sweet, sour (subgroup). Based on residues in pulp and peel, the Meeting estimated a STMR of 0.05 mg/kg and HR of 0.05 mg/kg in orange, sweet, sour (subgroup) pulp and a STMR of 0.915 mg/kg and HR of 1.2 mg/kg in orange peel.

Fate of residues during processing

The Meeting received processing studied for oranges. In one study (RA-3076/96), fruits were peeled prior to processing, which is not reflective of commercial processing, where whole fruits are pressed to obtain juice. Since residues of tebuconazole are on the surface of the fruits, peeling removed a significant amount of the residue that otherwise could have been transferred to the juice. In a second study (RAHWN001), oranges were scarified prior to juicing. This also removed a significant amount of the surface residue that could otherwise be transferred to juice. Therefore, the Meeting decided not to use either study to estimate a processing factor for citrus juice. The processing factors derived from

the processing studies and the resulting recommendations for STMR-Ps, HR-Ps, and/or maximum residue levels are summarized in the table below.

Processing (Transfer) Factors from the Processing of Raw Agricultural Commodities (RACs) with Field-Incurred Residues from Foliar Treatment with tebuconazole

RAC	Processed Commodity	Processing Factors [best estimate]	RAC MRL	Processed Commodity MRL	RAC STMR	Processed Commodity STMR-P
oranges	marmalade	<0.22, 0.63 [0.63]	0.4	--	0.27	0.17
	oil	24.5	0.4	10	0.27	6.6
	pomace, dried	7.2	0.4	3	0.27	1.9

Estimated maximum and mean dietary burdens of farm animals

The Meeting estimated the contribution from citrus pulp (dry) to the livestock dietary burden and based on the small increase by 0.8 ppm of dry matter diet, in relation to the maximum dietary burden estimate from the 2011 JMPR (54 ppm of dry matter diet), no change to the residue situation in animal commodities is expected. The Meeting 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 are suitable for establishing maximum residue limits and for IEDI and IESTI assessments.

The residue definition for compliance with the MRL and for dietary risk assessment for plant and animal commodities is parent tebuconazole.

The residue is not fat soluble.

CCN	Commodity, subgroups	Recommended MRL (mg/kg)		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
FC 0003	Mandarins (subgroup of)	0.7 (Po)	-	0.05	0.05
FC 0004	Oranges, Sweet, Sour (subgroup of)	0.4 (Po)	-	0.05	0.05
AB 0001	Citrus pulp, Dry	3 (dw)	-	1.9 (median residue)	-
OR 0001	Orange oil, edible	10	-	6.6	-
	Orange peel	-	-	0.915	1.2
	Orange marmalade			0.17	

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for tebuconazole is 0–0.03 mg/kg bw. The International Estimated Daily Intakes (IEDI) for tebuconazole was 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 2019 Extra JMPR Report.

The IEDIs ranged from 1–5% 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 2019 Extra JMPR Report.

The IESTIs were 0–1% (children) and 0% (general population) of the ARfD. 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

Report number	Author(s)	Year	Title, Source, Company name, Report No., Date, GLP status published or not
HW-002-P09-01	Stoughton, S. M.	2009	Analytical method for the determination of residues of tebuconazole in plant matrices using HPLC/MS/MS. Bayer CropScience LP, Stilwell, KS, USA. Bayer AG, Crop Science Division, Method No.: HW-002-P09-01, Edition Number: M-367751-01-1. No GLP, Unpublished
RA-2076/96	Allmendinger, H.	1998	Quantitation of residues of tebuconazole on mandarin and orange after post-harvest treatment of Folicur (250 EW). Bayer AG, Leverkusen, Germany. Bayer AG, Crop Science Division, Edition Number: M-023612-01-2. Unpublished.
RA-3076/96	Allmendinger, H. and Walz-Tylla, B.	1998	Quantitation of residues of tebuconazole in processed commodities of orange after post-harvest treatment of Folicur (250 EW). Bayer AG, Leverkusen, Germany. Bayer AG, Division Crop Science, Edition Number: M-008702-01-1. Unpublished
RAHWN001	Lenz, C. and Freeseman, P.	2012	Tebuconazole 250 EW - Magnitude of the residue in/on orange processed commodities following post-harvest treatment. Bayer CropScience LP, RTP, NC, USA. Bayer AG, Crop Science Division, Edition Number: M-440417-01-1. Unpublished.

THIABENDAZOLE (065)

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

EXPLANATION

Thiabendazole is a benzimidazole compound used as a systemic fungicide in agriculture, and also as a broad-spectrum anthelmintic in various animal species. It was first evaluated by JMPR in 1970, and the latest evaluation was conducted in 2006 (T, R).

The residue definitions for thiabendazole are for compliance with MRL and for dietary risk assessment for plant commodities: *thiabendazole.*

For compliance with MRL for animal commodities: *sum of thiabendazole and 5-hydroxythiabendazole*

And for dietary risk assessment for animal commodities: *sum of thiabendazole, 5-hydroxythiabendazole and its sulfate conjugate*.

The compound was scheduled at the Fiftieth Session of the CCPR for the evaluation of additional uses by the 2019 Extra JMPR. Plant metabolism studies, analytical methods and residue trials on mango, beans, peas and sweet potato, and processing studies were submitted to the Meeting.

Plant metabolism

Two plant metabolism studies not previously evaluated by the Meeting were submitted, one study investigated the metabolism of thiabendazole in oranges treated post-harvest and one in maize when used as a seed treatment.

Orange, post-harvest

In the study conducted by Piskorski (2012), [Phenyl-U-^{14}C]-thiabendazole was applied to orange fruits in a single dose at 0.2 kg ai/hL prior to storage in the dark at approximately 5 °C and at a relative humidity of about 85%, and oranges sampled and analysed directly after application and at intervals of 8 and 16 weeks. At each timepoint, radioactive residues were extracted from the fruit surface with acetonitrile and the washed oranges were separated into peel and flesh, which were milled and then homogenized under liquid nitrogen prior to combustion/LSC analysis. Radioactivity of orange flesh was always < 0.01 mg eq/kg, and only the homogenised, washed peel were sequentially shaken with 1N NaOH/phosphate buffer pH 8, extracted with ethyl acetate, phosphate buffer pH 8 again and then with acetonitrile. The liquid and solid phases were separated by centrifugation and the supernatant was filtered, the radioactivity in each extract quantified by LSC, and further concentrated for TLC and HPLC analysis for residues identification and characterization. Aliquots of the post extraction debris were analysed by combustion/LSC. Analysis of samples was completed within six months of each sampling date and therefore no storage stability analysis was required. The results are shown in Tables 1 and 2.

Table 1 Summary of total radioactive residues and extractability in mature oranges treated post-harvest with [^{14}C]thiabendazole

Period of storage	Initial Fruit Wash		Washed Fruit Peel				Flesh		TRR[a]
			Extracted Radioactivity		Non-extracted Radioactivity				
	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	mg eq/kg
0 days	94.35	4.982	4.37	0.231	1.24	0.066	0.04	0.002	5.280
8 weeks	80.93	4.379	14.02	0.759	4.92	0.266	0.14	0.007	5.411
16 weeks	72.59	3.001	22.51	0.931	4.78	0.198	0.11	0.005	4.134

[a] The total radioactive residue (mg/kg) is calculated by the summation of the radioactivity present in the initial fruit surface wash, extract and debris generated from analysis of the washed peel and the direct combustion of orange flesh.

Table 2 Summary of identification and characterization of residues found in orange peel washes

		Day 0		8 weeks of storage		16 weeks of storage	
TRR by summation (mg/kg)		5.280[a]		5.411[a]		4.134[a]	
TRR by direct quantification (mg eq/kg)		5.333[b]		5.556[b]		4.207[b]	
Percentage of TRR for chromatography (%)on a conveyor belt		98.72		94.94		95.11	
Origin of component	Component	% TRR	Residue (mg eq/kg)	% TRR	Residue (mg eq/kg)	% TRR	Residue (mg eq/kg)
Chromato-graphed	Thiabendazole	98.56	5.204	92.30	4.994	89.91	3.717
	5-hydroxythiabendazole	N/D	N/D	0.34	0.018	0.39	0.016
	Benzimidazole	N/D	N/D	0.04	0.002	ND	ND
	Benzimidazole-COOH	N/D	N/D	0.30	0.016	0.30	0.012
	Unassigned [c]	0.02	0.001	1.65	0.089	2.28	0.094
	Baseline	0.20	0.011	0.31	0.017	2.22	0.092
	Unextracted (peel)	1.24	0.066	4.92	0.266	4.78	0.198
	Unextracted (flesh)	0.04	0.002	0.14	0.007	0.11	0.005
	Total	100	5.28	100	5.41	100	4.13

ND: Not detected;

[a] TRR determined by summation of radioactivity present in the surface wash, solvent extracts of washed peel, debris following solvent extraction of washed peel and combusted flesh;

[b] The radioactive residue determined by summation of radioactivity present in the surface wash and direct quantification from washed fruit (separately peel and flesh) employing combustion/LSC;

[c] Unassigned radiocomponents which chromatographed away from the origin in 2D-TLC SSA. For the day 0 samples this consists of 2 components, none > 0.01%TRR (<0.001 mg/kg); for week 8 samples this consists of 9 components, none > 0.53% TRR (0.029 mg/kg); for week 16 samples, this consists of 7 components, none > 1.13%TRR (0.047 mg/kg);

Maize, seed treatment

[Phenyl-U-^{14}C]-thiabendazole, formulated as a suspension concentrate, was applied as a single seed dressing to maize seeds at the maximum rate of 0.09 mg/seed (MacKinnon, 2005). Treated maize was grown under glasshouse conditions with plants harvested at growth stages representing commercial forage, sweet corn and maturity. Samples of forage were taken at the growth stage of V12 or BBCH 19, 81 days after sowing. Samples of forage, cobs and kernels were taken at the sweet corn stage of R4 or BBCH 83–85, 101 days after sowing and at maturity growth stage R6 or BBCH 89, 116 days after sowing. All samples were stored frozen at ≤ -20 °C.

No residues were detected in the cobs or kernels from the sweet corn or at maturity growth stages. TRR of foliage from the sweet corn stage and at maturity were < 0.01 mg eq/kg (0.005 and 0.002 mg eq/kg, respectively, and were not analysed. Foliage from the forage stage sample had TRR of 0.014 mg eq/kg and was extracted with acetonitrile:water (8:2), dried and reconstituted with acetone:water (3:2). After filtration, the solution was reduced to dryness and reconstituted with acetone:water (1:1), resulting in a bi-phasic system of organic and aqueous fractions. 42.5% of the TRR (0.006 mg/kg) were extracted and 55.5% remained unextracted (0.008 mg/kg). Aliquots of the organic and aqueous fractions submitted to normal and reversed-phase TLC. TRR were characterised to be composed of multiple minor metabolites without the presence of thiabendazole. No further attempt was made to extract and characterise the post extraction solid residue.

RESIDUE ANALYSIS

Analytical methods

In Method GRM040.01A, samples are extracted with aqueous phosphate buffer (pH 6) and partitioned against ethyl acetate (McLean and Nelson, 2008). Any conjugates of thiabendazole or benzimidazole were extracted with ethyl acetate following the addition of a glucosidase enzyme to the aqueous phase. The ethyl acetate phase was analysed by LC-MS/MS, with a LOQ of 0.01 mg/kg for each analyte. Table 3 shows the recovery data of this procedure.

Table 3 Recoveries of thiabendazole from crops using method GRM040.01A

Commodity	Compound	Fortification level, mg/kg	No.	Mean recovery (%)	RSD (%)	Mean recovery (%)	RSD (%)
				Primary transition		Confirmatory transistion	
Spinach (leaves)	Thiabendazole (m/z = 202 → 131)	0.01	5	101	2	102	2
		0.1	5	97	2	98	1
	Benzimidazole (m/z = 119 → 92)	0.01	5	87	2	87	1
		0.10	5	88	3	87	3
Wheat (grain)	Thiabendazole (m/z = 202 → 131)	0.01	5	94	2	94	2
		0.1	5	94	2	93	2
	Benzimidazole (m/z = 119 → 92)	0.01	5	92	2	96	2
		0.10	5	92	2	94	2
Wheat (straw)	Thiabendazole (m/z = 202 → 131)	0.01	5	95	3	95	2
		0.1	5	94	2	93	1
	Benzimidazole (m/z = 119 → 92)	0.01	5	87	2	90	2
		0.10	5	89	1	88	2
Carrot (roots)	Thiabendazole (m/z = 202 → 131)	0.01	5	105	1	105	2
		0.1	5	96	1	96	2
	Benzimidazole (m/z = 119 → 92)	0.01	5	93	2	94	1
		0.10	5	89	1	89	2
Bean (with pods)	Thiabendazole (m/z = 202 → 131)	0.01	3	98	2.4		
		1.0	3	96	6.2		
	Benzimidazole (m/z = 119 → 92)	0.01	3	95	1.4		
		1.0	3	85	8.8		
Pea (vines)	Thiabendazole (m/z = 202 → 131)	0.01	3	89	1.4		
		1.0	3	92	1.9		
	Benzimidazole (m/z = 119 → 92)	0.01	3	75	1.8		
		1.0	3	76	4.3		
Sweet potato (roots)	Thiabendazole (m/z = 202 → 131)	0.01	3	85	4.1		
		0.1	3	84	14		
		10	3	75	2.0		
	Benzimidazole (m/z = 119 → 92)	0.01	3	90	7.1		
		0.10	3	88	12		
		10	3	73	2.2		
Sweet potato (chips)	Thiabendazole (m/z = 202 → 131)	0.01	3	86	3.6		
		1.0	3	82	1.9		
		10	3	67	3.1		
	Benzimidazole (m/z = 119 → 92)	0.01	3	83	2.6		
		1.0	3	81	0.1		
		10	3	68	6.8		
Sweet potato (flakes)	Thiabendazole (m/z = 202 → 131)	0.01	3	89	4.5		
		1.0	3	91	9.5		
		10	3	73	17		
	Benzimidazole (m/z = 119 → 92)	0.01	3	80	7.9		
		1.0	3	72	6.4		
		10	3	65	32		

In Method GRM046.04A, samples were extracted twice with ethyl acetate, centrifuged and purified using a cation exchange SPE cartridge (Crook, 2012). The purified extracts were quantified by LC-MS/MS, monitoring for the primary transition (m/z = 202→175) and the confirmatory transition (m/z = 202→131). The response of the LC-MS/MS was shown to be linear for both primary and confirmatory transitions for thiabendazole over a concentration range of between 30% LOQ and 20% above the upper fortification level, with correlation coefficients > 0.99. Insignificant enhancement or suppression (< 20%) of detector response was observed for both analytes and all the matrices. The recovery data are shown in Table 4.

Table 4 Recoveries of thiabendazole from crops using method GRM046.04A

Commodity	Fortification level (mg/kg)	No.	Mean recovery (%)	RSD (%)	Mean recovery (%)	RSD (%)
			m/z = 202→175		*m/z* = 202→131	
Lettuce	0.01	5	90	5.0	92	3.4
	0.1	5	81	13.5	82	14.2
Tomato	0.01	5	88	7.7	88	7.7
	0.1	5	82	11.4	83	11.7
Orange	0.01	5	80	7.7	86	9.7
	0.10	5	81	2.7	79	7.2
Potato	0.01	5	90	4.9	90	2.7
	0.1	5	83	8.6	83	8.3
Avocado	0.01	5	86	4.4	85	4.7
	0.10	5	87	5.0	88	4.7
Maize (whole cob)	0.01	5	93	1.9	94	1.2
	0.10	5	78	11.8	79	12.0
Mango (peel)	0.01	5	87	6.4	-	-
	0.1	5	88	7.7	-	-
	1.0	5	92	4.0	-	-
	20	5	86	4.1	-	-
Mango (pulp)	0.01	5	79	5.6	-	-
	0.01	5	84	4.6	-	-
	1.0	5	103	5.8	-	-

The extraction efficiency of the methods used in the trials was shown by analysing oranges treated with [Phenyl-U-^{14}C]-thiabendazole from the metabolism study (Piskorski, 2012). The results are shown in Table 5.

Table 5 Comparison of solvent extractabilities of thiabendazole residues from whole fruit orange from metabolism and residue analytical methods

Method	Radioactivity extracted		Relative efficiency (%)[1]	Thiabendazole		Relative thiabendazole recovery (%)[a]
	%TRR	mg eq/kg		%TRR	mg/kg	
Metabolism extraction[b]	95.1	3.931	N/A	89.9	3.717	N/A
M-046[c]	81.8	1.849	86.0	80.8	1.827	89.9
M-049[c]	68.1	1.538	71.6	67.2	1.519	74.7
Modified M-049[c]	70.1	1.585	73.7	66.7	1.508	74.2

N/A - Not applicable;

[a] (Residue method %TRR extracted/metabolism method %TRR extracted) × 100%;

[b] Results from oranges stored for 16 weeks; sum of washed and extracted radioactivity from the orange peel. The flesh was not extracted as the radioactive residue present was only 0.005 mg/kg;

[c] Mean values of 2 replicates.

A QuEChERS method (EN 15662:2009-02 or L-00.00-115) was validated for thiabendazole in crop commodities (Class and Bacher, 2012). A sample aliquot (5 or 10 g) is extracted with acetonitrile:water. magnesium sulphate, sodium chloride, sodium citrate tribasic dihydrate and sodium citrate dibasic sesquihydrate are added, the mixture is shaken, centrifuged and transferred to a dispersive SPE (dSPE) clean-up tube. If necessary, the upper acetonitrile phase is stored frozen for approximately three hours to remove fat or waxes. Thiabendazole is determined by LC-MS/MS, at a LOQ of 0.01 mg/kg. The recovery results are shown in Table 6.

Table 6 Recovery of thiabendazole from crops using the QuEChERS method

Commodity	Fortification level (mg/kg)	No.	Mean recovery (%)	RSD (%)	Mean recovery (%)	RSD (%)
			m/z=202→175		m/z=202→131	
Lettuce	0.01	5	101	3	101	4
	0.1	5	103	2	102	2
Orange (whole fruit)	0.01	5	99	4	101	5
	0.1	5	97	5	99	4
Wheat (grain)	0.01	5	88	5	87	4
	0.1	5	89	4	89	3
Olives	0.01	5	80	8	84	9
	0.1	5	83	4	85	4

The LC-MS/MS response was shown to be linear over the range from ≤ 20% of the LOQ to 20 × LOQ for both primary and confirmatory transitions, with correlation coefficients (r) ≥ 0.99. Significant matrix effects (> 20%) were observed for orange, wheat grain and olive matrices, and matrix-matched standards were used for quantification. A method validation was conducted by an independent laboratory, and the data are presented in Table 7 (Amic, 2012).

Table 7 Recovery of thiabendazole from crops using the QuEChERS method

Commodity	Fortification level (mg/kg)	No.	Mean recovery (%)	RSD (%)	Mean recovery (%)	RSD (%)
			m/z=202→175		m/z=202→131	
Lettuce	0.01	5	94	2	92	2
	0.1	5	93	1	93	1
Olives	0.01	5	90	3	93	2
	0.1	5	92	2	83	5

The QuEChERS method was also validated for thiabendazole and 5-hydroxy-thiabendazole in animal matrices and the recovery results are shown in Table 8 (Class and Backer, 2012; Watson, 2014).

Table 8 Recovery of thiabendazole and 5-hydroxy-thiabendazole in animal commodities using the QuEChERS method

Commodity	Compound	Fortification level (mg/kg)	No.	Range of Recovery (%)	Mean recovery (%)	RSD (%)
Primary transition						
Muscle	Thiabendazole (m/z=202→175)	0.01	5	93-99	97	2.5
		0.1	5	90-96	92	2.5
	5-Hydroxy-thiabendazole (m/z=218→147)	0.01	5	70-79	75	5.9
		0.1	5	77-82	80	2.3
Fat	Thiabendazole (m/z=202→175)	0.01	5	95-105	101	3.9
		0.1	5	98-103	100	1.9
	5-Hydroxy-thiabendazole (m/z=218→147)	0.01	5	96-106	100	4.5
		0.1	5	90-99	94	3.8
Liver	Thiabendazole (m/z=202→175)	0.01	5	87-93	90	2.7
		0.1	5	86-88	87	0.9
	5-Hydroxy-thiabendazole (m/z=218→147)	0.01	5	70-80	75	5.3
		0.1	5	70-72	71	1.2
Milk	Thiabendazole (m/z=202→175)	0.01	5	93-98	96	1.9
		0.1	5	91-95	93	1.9
	5-Hydroxy-thiabendazole (m/z=218→147)	0.01	5	82-93	88	4.7
		0.1	5	82-89	85	3.4
Egg	Thiabendazole (m/z=202→175)	0.01	5	86-94	92	3.5
		0.1	5	98-103	101	2.3

Commodity	Compound	Fortification level (mg/kg)	No.	Range of Recovery (%)	Mean recovery (%)	RSD (%)
	5-Hydroxy-thiabendazole (m/z=218→147)	0.01	5	69-86	79	8.5
		0.1	5	91-98	95	2.9
Confirmatory transition						
Muscle	Thiabendazole (m/z=202→131)	0.01	5	92-100	94	3.4
		0.1	5	90-97	93	2.7
	5-Hydroxy-thiabendazole (m/z=218→191)	0.01	5	74-87	81	6.0
		0.1	5	75-79	77	1.9
Fat	Thiabendazole (m/z=202→131)	0.01	5	99-106	101	2.6
		0.1	5	99-106	102	2.9
	5-Hydroxy-thiabendazole (m/z=218→191)	0.01	5	95-103	99	3.8
		0.1	5	90-97	93	3.0
Liver	Thiabendazole (m/z=202→131)	0.01	5	87-91	89	1.9
		0.1	5	85-87	86	0.9
	5-Hydroxy-thiabendazole (m/z=218→191)	0.01	5	71-78	75	3.5
		0.1	5	71-74	72	1.8
Milk	Thiabendazole (m/z=202→131)	0.01	5	92-99	96	2.6
		0.1	5	94-97	95	1.2
	5-Hydroxy-thiabendazole (m/z=218→191)	0.01	5	79-91	85	5.1
		0.1	5	84-90	87	3.1
Egg	Thiabendazole (m/z=202→131)	0.01	5	84-95	91	4.9
		0.1	5	99-105	102	2.4
	5-Hydroxy-thiabendazole (m/z=218→191)	0.01	5	71-86	80	7.4
		0.1	5	91-97	95	2.2

The QuEChERS method for animal commodities was validated by an independent laboratory (Amic, 2012), and recovery data are shown in Table 9.

Table 9 Recovery of thiabendazole and 5-hydroxy-thiabendazole in animal commodities obtained by an independent laboratory

Commodity	Compound	Fortification level (mg/kg)	No.	Range of Recovery (%)	Mean recovery (%)	RSD (%)
Primary transition						
Muscle	Thiabendazole (m/z=202→175)	0.01	5	119-122	120	1
		0.1	5	96-102	99	2
	5-Hydroxy-thiabendazole (m/z=218→147)	0.01	5	92-103	99	4
		0.1	5	72-78	75	3
Fat	Thiabendazole (m/z=202→175)	0.01	5	103-110	107	2
		0.1	5	92-105	99	5
	5-Hydroxy-thiabendazole (m/z=218→147)	0.01	5	110-114	111	2
		0.1	5	94-107	100	5
Liver	Thiabendazole (m/z=202→175)	0.01	5	97-103	99	3
		0.1	5	89-98	94	4
	5-Hydroxy-thiabendazole (m/z=218→147)	0.01	5	71-80	75	4
		0.1	5	66-73	70	4
Milk	Thiabendazole (m/z=202→175)	0.01	5	92-103	96	5
		0.1	5	96-104	100	3
	5-Hydroxy-thiabendazole (m/z=218→147)	0.01	5	68-87	78	9
		0.1	5	84-93	89	4
Egg	Thiabendazole (m/z=202→175)	0.01	5	90-100	96	4
		0.1	5	96-101	98	2
		0.01	5	77-96	86	8

Commodity	Compound	Fortification level (mg/kg)	No.	Range of Recovery (%)	Mean recovery (%)	RSD (%)
	5-Hydroxy-thiabendazole (m/z=218→147)	0.1	5	80-95	87	7
Confirmatory transition						
Muscle	Thiabendazole (m/z=202→131)	0.01	5	115-121	119	2
		0.1	5	96-100	97	2
	5-Hydroxy-thiabendazole (m/z=218→191)	0.01	5	92-101	97	4
		0.1	5	71-80	75	5
Fat	Thiabendazole (m/z=202→131)	0.01	5	102-109	106	3
		0.1	5	90-103	97	5
	5-Hydroxy-thiabendazole (m/z=218→191)	0.01	5	106-114	110	3
		0.1	5	94-106	100	4
Liver	Thiabendazole (m/z=202→131)	0.01	5	93-102	97	3
		0.1	5	89-97	93	4
	5-Hydroxy-thiabendazole (m/z=218→191)	0.01	5	73-83	78	5
		0.1	5	69-73	71	3
Milk	Thiabendazole (m/z=202→131)	0.01	5	90-105	97	6
		0.1	5	96-104	100	3
	5-Hydroxy-thiabendazole (m/z=218→191)	0.01	5	65-81	74	8
		0.1	5	83-90	87	4
Egg	Thiabendazole (m/z=202→131)	0.01	5	88-98	93	4
		0.1	5	94-100	97	2
	5-Hydroxy-thiabendazole (m/z=218→191)	0.01	5	76-93	88	8
		0.1	5	79-93	87	7

Stability of pesticide residues in stored analytical samples

Homogenised samples aliquots were fortified with known amounts of either thiabendazole or benzimidazole in methanol at 0.1 mg/kg. The solvent was allowed to evaporate and the samples sealed and stored in a freezer at -20 ± 5 °C (Manson, 2014). The initial concentration was determined by analysis of two freshly-prepared samples fortified with both thiabendazole and benzimidazole using Method GRM040.01A, with acceptable procedural recoveries. The results are shown in Table 10.

Table 10 Storage stability data for thiabendazole and benzimidazole residues in frozen plant matrices

Matrix	Analyte	Interval (days/months)	Fortification level (mg/kg)	Residue level in freezer storage	
				mg/kg	% remaining (mean)
Navy beans (dry seed)	Thiabendazole	0 / 0	0.1	0.090, 0.091	90, 91 (91)
		132 / 4	0.1	0.083, 0.080	83, 80 (82)
		300 / 10	0.1	0.089, 0.092	89, 92 (91)
		377 / 12	0.1	0.094, 0.094	94, 94 (94)
		544 / 18	0.1	0.083, 0.080	83, 80 (82)
		743 / 24	0.1	0.093, 0.090	93, 90 (92)
	Benzimidazole	0 / 0	0.1	0.077, 0.081	77, 81 (79)
		132 / 4	0.1	0.066, 0.064	66, 64 (65)
		300 / 10	0.1	0.092, 0.094	92, 94 (93)
		377 / 12	0.1	0.084, 0.080	84, 80 (82)
		544 / 18	0.1	0.066, 0.072	66, 72 (69)
		743 / 24	0.1	0.087, 0.081	87, 81 (84)
Soya bean (Seed)	Thiabendazole	0 / 0	0.1	0.088, 0.088	88, 88 (88)
		100 / 3	0.1	0.085, 0.080	85, 80 (83)
		275 / 9	0.1	0.077, 0.074	77, 74 (76)
		379 / 12	0.1	0.082, 0.084	82, 84 (83)
		544 / 18	0.1	0.066, 0.075	66, 75 (71)

Matrix	Analyte	Interval (days/months)	Fortification level (mg/kg)	Residue level in freezer storage	
				mg/kg	% remaining (mean)
		730 / 24	0.1	0.083, 0.080	83, 80 (82)
	Benzimidazole	0 / 0	0.1	0.077, 0.073	77, 73 (75)
		100 / 3	0.1	0.073, 0.074	73, 74 (74)
		275 / 9	0.1	0.077, 0.076	77, 76 (77)
		379 / 12	0.1	0.064, 0.061	64, 61 (63)
		544 / 18	0.1	0.058, 0.054	58, 54 (56)
		730 / 24	0.1	0.072, 0.069	72, 69 (71)
Spinach (leaves)	Thiabendazole	0 / 0	0.1	0.090, 0.092	90, 92 (91)
		91 / 3	0.1	0.077, 0.080	77, 80 (79)
		271 / 9	0.1	0.070, 0.068	70, 68 (69)
		371 / 12	0.1	0.083, 0.085	83, 85 (84)
		544 / 18	0.1	0.079, 0.091	79, 91 (85)
		740 / 24	0.1	0.088, 0.087	88, 87 (88)
	Benzimidazole	0 / 0	0.1	0.081, 0.075	81, 75 (78)
		91 / 3	0.1	0.058, 0.056	58, 56 (57)
		271 / 9	0.1	0.056, 0.056	56, 56 (56)
		371 / 12	0.1	0.048, 0.047	48, 47 (48)
		544 / 18	0.1	0.042, 0.047	42, 47 (45)
		740 / 24	0.1	0.036, 0.035	36, 35 (36)
Barley (grain)	Thiabendazole	0 / 0	0.1	0.091, 0.096	91, 96 (94)
		91 / 3	0.1	0.081, 0.078	81, 78 (80)
		271 / 9	0.1	0.072, 0.075	72, 75 (74)
		371 / 12	0.1	0.088, 0.084	88, 84 (86)
		544 / 18	0.1	0.070, 0.068	70, 68 (69)
		747 / 25	0.1	0.085, 0.085	85, 85 (85)
	Benzimidazole	0 / 0	0.1	0.071, 0.076	71, 76 (74)
		91 / 3	0.1	0.070, 0.069	70, 69 (70)
		271 / 9	0.1	0.075, 0.074	75, 74 (75)
		371 / 12	0.1	0.076, 0.075	76, 75 (76)
		544 / 18	0.1	0.061, 0.064	61, 64 (63)
		747 / 25	0.1	0.079, 0.079	79, 79 (79)
Orange (whole fruit)	Thiabendazole	0 / 0	0.1	0.095, 0.098	95, 98 (97)
		100 / 3	0.1	0.093, 0.090	93, 90 (92)
		275 / 9	0.1	0.068, 0.074	68, 74 (71)
		392 / 13	0.1	0.098, 0.105	98, 105 (102)
		544 / 18	0.1	0.082, 0.084	82, 84 (83)
		730 / 24	0.1	0.092, 0.093	92, 93 (93)
	Benzimidazole	0 / 0	0.1	0.081, 0.070	81, 70 (76)
		100 / 3	0.1	0.070, 0.068	70, 68 (69)
		275 / 9	0.1	0.076, 0.074	76, 74 (75)
		392 / 13	0.1	0.079, 0.077	79, 77 (78)
		544 / 18	0.1	0.073, 0.082	73, 82 (78)
		730 / 24	0.1	0.084, 0.081	84, 81 (83)

USE PATTERNS

Table 11 shows the use patterns of thiabendazole for the crops and treatments relevant to this evaluation.

Table 11 Registered uses of thiabendazole using SC formulation for seed and post harvest treatments

Crop	Country	Application	kg ai/tonne	kg ai/hL	PHI (days)
Beans [a] (except soya bean)	USA	Seed treatment	0.55	-	-
Chickpea	USA	Seed treatment	0.65	-	-
Lentil	USA	Seed treatment	0.34	-	-

Crop	Country	Application	kg ai/tonne	kg ai/hL	PHI (days)
Peas [1)]	USA	Seed treatment	0.33	-	-
Mango	Belize, Costa Rica, Dominican Republic, Guatemala, Honduras, Panama and Nicaragua,	Dip (post-harvest)	-	0.24	0
	Brazil	Post-harvest	-	0.19	0
Soya bean	USA	Seed treatment	0.20	-	-
Sweet potato	USA	Dip (post-harvest)	-	0.16	0
		Spray (post-harvest), in a conveyor line	0.006		0

[a] According to USA. Crop Group 6: Legume Vegetables (Succulent or Dried);

RESULTS OF SUPERVISED RESIDUE TRIALS ON CROPS

Mango

Eight post-harvest trials were conducted on mango in Brazil in 2017, using either a dip or a spray application. Samples were maintained in frozen storage prior to extraction for periods of up to 60 days. The results are summarized in Table 12.

Table 12 Residues of thiabendazole on mango from post-harvest trials conducted in 2017 with dip or spray applications in Brazil (Report: LBS17004)

Location (variety)	Application Rate (kg ai/hL)	PHI (days)	Crop Part	Residue (mg/kg) Thiabendazole	Trial
Taquaritinga, São Paulo (Palmer)	0.25 dip	0	Peel Pulp Whole fruit [a]	8.3, 7.2 (7.7) 0.025, 0.030 (0.027) 2.4	LBS17004-01
	0.097 spray	0	Peel Pulp Whole fruit [a]	8.9, 7.6, 5.9, 5.6, 6.2 (6.8) <0.01, <0.01 (<0.01) 2.3	
Juazeiro, Bahia (Keit)	0.25 dip	0	Peel Pulp Whole fruit [a]	15, 16, 13, 14, 14 (14) 0.011, 0.013 (0.012) 4.5	LBS17004-02
	0.097 spray	0	Peel Pulp Whole fruit [a]	8.2, 7.3 (7.7) <0.01, <0.01 (<0.01) 2.5	
Casa-Nova, Bahia (Kent)	0.25 dip	0	Peel Pulp Whole fruit [a]	11, 11, 12, 12 (11) 0.010, <0.01 (0.01) 3.4	LBS17004-03
	0.097 spray	0	Peel Pulp Whole fruit [a]	10, 7.9 (9.0) <0.01, <0.01 (<0.01) 2.6	
Petrolina, Pernambuco (Tommy)	0.25 dip	0	Peel Pulp Whole fruit [a]	10, 8.1 (9.1) 0.021, 0.025 (0.023) 2.6	LBS17004-04
	0.097 spray	0	Peel Pulp Whole fruit [a]	8.0, 7.7 (7.8) <0.01, <0.01 (<0.01) 2.1	

[a] Calculated from residues in the pulp and in the peel

Twelve trials were conducted on fresh beans in the USA in 2013/14 with seed-treated bean seeds. The results are summarized in Table 13.

Table 13 Residues of thiabendazole on green beans with or without pods from seed treatment trials conducted in the USA in 2013 (Report: TK0180600)

Region (Variety)	Application Rate (kg ai/ton seed)	DAT (days)	Crop Part	Residues, mg/kg	Trial
Germansville, PA (Provider)	0.62	47	Beans with pods	<0.01, <0.01 (<u>≤0.01</u>)	TK0180600-01
Chula, GA (Provider)	0.62	61	Beans with pods	<0.01, <0.01 (<u>≤0.01</u>)	TK0180600-02
Athens, GA (Provider)	0.62	48	Beans with pods	<0.01, <0.01 (<u>≤0.01</u>)	TK0180600-03
Richland, IA (Provider)	0.62	60	Beans with pods	<0.01, <0.01 (<u>≤0.01</u>)	TK0180600-04
Geneva, MN (Provider)	0.62	67	Beans with pods	<0.01, <0.01 (<u>≤0.01</u>)	TK0180600-05
Paso Robles, CA (Provider)	0.62	84	Beans with pods	<0.01, <0.01 (<u>≤0.01</u>)	TK0180600-06
Athens, GA (Fordhook 242)	0.21	91	Beans green w/o pods	<0.01, <0.01 (<0.01)	TK0180600-07
Seven Springs (Fordhook 242)	0.21	142	Beans green w/o pods	<0.01, <0.01 (<0.01)	TK0180600-08
Lenexa, KS (Fordhook 242)	0.21	104	Beans green w/o pods	<0.01, <0.01 (<0.01)	TK0180600-10
Chico, CA (Fordhook 242)	0.21	135	Beans green w/o pods	<0.01, <0.01 (<0.01)	TK0180600-11
Parkdale, OR (Fordhook 242)	0.21	132	Beans green w/o pods	<0.01, <0.01 (<0.01)	TK0180600-12
Chula, GA (Jackson Wonder)	0.42	75	Beans green w/o pods	<0.01, <0.01 (<u>≤0.01</u>)	TK0180600-41

Nine supervised harvest trials were conducted on fresh peas in the U.S.A. in 2013/14 with thiabendazole treated pea seeds, three trials on peas with pods and six trials on peas without pods. The results are summarized in Table 14.

Table 14 Residues of thiabendazole on green peas with or without pods from seed treatment trials conducted in the USA in 2013-21014 (Report: TK0180600)

Country (Region)	Application rate (kg ai/ton seed)	DAT (days)	Crop Part	Residues (mg/kg)	Trial
Germansville, PA (Sugar Ann)	0.99	51	Peas with pods	<0.01, <0.01 (<0.01)	TK0180600-13
Athenas, GA (Sugar Ann)	1	53	Peas with pods	<0.01, <0.01 (<0.01)	TK0180600-14
Lenexa, KS (Sugar Ann)	0.99	53	Peas with pods	<0.01, <0.01 (<0.01)	TK0180600-15
Northwood, ND (Premium)	1.2	49	Peas green w/o pods	<0.01, <0.01 (<0.01)	TK0180600-17
Verona, WI (Premium)	1.2	51	Peas green w/o pods	<0.01, <0.01 (<0.01)	TK0180600-18
Parkdale, OR (Premium)	1.2	71	Peas green w/o pods	<0.01, <0.01 (<0.01)	TK0180600-19
Payette, ID (Premium)	1.2	54	Peas green w/o pods	<0.01, <0.01 (<0.01)	TK0180600-20
Hilsboro, OR (Premium)	1.2	61	Peas green w/o pods	<0.01, <0.01 (<0.01)	TK0180600-21

Country (Region)	Application rate (kg ai/ton seed)	DAT (days)	Crop Part	Residues (mg/kg)	Trial
Lenexa, KS (Premium)	1.2	38	Peas green w/o pods	<0.01, <0.01 (<0.01)	TK0180600-39

Nine supervised trials were conducted on dry beans in the U.S.A. in 2013 following thiadendazole treatment of bean seeds. The results are summarized in Table 15.

Table 15 Residues of thiabendazole on dry beans, following seed-treatment conducted in the USA in 2013 (Report: TK0180600)

Region (Variety)	Application rate (kg ai/ton seed)	DAT (days)	Residue (mg/kg)	Trial
Verona, WI (Lariat)	0.44	109	<0.01, <0.01 (<u><0.01</u>)	TK0180600-22
Royalton, MN (Lariat)	0.44	117	<0.01, <0.01 (<u><0.01</u>)	TK0180600-23
York, NE (Lariat)	0.44	102	<0.01, <0.01 (<u><0.01</u>)	TK0180600-24
Geneva, MN (Lariat)	0.44	104	<0.01, <0.01 (<u><0.01</u>)	TK0180600-25
Grand Island (Red Hawk)	0.41	107	<0.01, <0.01 (<u><0.01</u>)	TK0180600-26
San Angelo, TX (Red Hawk)	0.41	167	<0.01, <0.01 (<u><0.01</u>)	TK0180600-27
Jerome, ID (Red Hawk)	0.41	95	<0.01, <0.01 (<u><0.01</u>)	TK0180600-28
Chico, CA (Red Hawk)	0.41	94	<0.01, <0.01 (<u><0.01</u>)	TK0180600-29
American Falls (Red Hawk)	0.41	110	<0.01, <0.01 (<u><0.01</u>)	TK0180600-30

Ten supervised harvest trials were conducted on dry peas in the U.S.A. with thiabendazole treated pea seeds in 1996 or 2013. The results are summarized in Table 16.

Table 16 Residues of thiabendazole on dry peas after seed treatment from trials conducted in the USA in 1996 and 2013.

Region (Variety)	Application rate (kg ai/ton seed)	DAT (days)	Residue (mg/kg)	Report, Trial - Year
Prosser, WA (SS Alaska Dry Pea)	0.9	83	<0.05, <0.05 (<u><0.05</u>)	IR-4 06532, 96-WA85 - 1996
Prosser, WA (Umatilla Dry Pea)	0.9	83	<0.05, <0.05 (<u><0.05</u>)	IR-4 06532, 96-WA84 – 1996
Prosser, WA (Columbia Dry Pea)	0.9	83	<0.05, <0.05 (<u><0.05</u>)	IR-4 06532, 96-WA83 – 1996
Kimberly, ID (Umatilla Dry Pea)	0.9	87	<0.05, <0.05 (<u><0.05</u>)	IR-4 06532, 96-ID09 – 1996
Kimberly, ID (Columbia Dry Pea)	0.9	90	<0.05, <0.05 (<u><0.05</u>)	IR-4 06532, 96-ID10 -1996
Ephrata, WA (Montex 4153)	0.8	98	<0.01, <0.01 (<u><0.01</u>)	TK0180600 TK0180600-31 – 2013
Parkdale, OR (Montex 4153)	0.8	93	<0.01, <0.01 (<u><0.01</u>)	TK0180600 TK0180600-32 – 2013
American Falls (Montex 4153)	0.8	105	<0.01, <0.01 (<u><0.01</u>)	TK0180600 TK0180600-33 – 2013
Chubbuck, ID (Montex 4153)	0.8	105	<0.01, <0.01 (<u><0.01</u>)	TK0180600 TK0180600-34 - 2013
Payette, ID (Montex 4153)	0.8	81	<0.01, <0.01 (<u><0.01</u>))	TK0180600 TK0180600-35 - 2013

Sweet Potato

Eight residue trials relevant to the use of thiabendazole on sweet potato were conducted in the USA in 2016. The results are summarized in Table 17.

Table 17 Residues of thabendazole on sweet potato from post-harvest trials in USA in 2016 (IR-4 Project No. 11859)

Region (variety)	Application rate (kg ai/tone)	Application rate (kg ai/hL)	DAT (days)	Residue (mg/kg)	Trial
Kibler, AR	0.006 (spray)	-	0	0.382, 0.368 (0.38)	AR499
(Beauregard)	-	0.16 (dip)	0	4.68, 4.85 (4.8)	
Parlier, CA	0.006 (spray)	-	0	1.32, 1.08 (1.2)	CA498
(Covington)	-	0.16 (dip)	0	2.84, 2.60 (2.7)	
Parlier, CA (Covington)	-	0.16 (dip)	0	4.4	CA525
Tifton, GA	0.006 (spray)	-	0	0.26, 0.250 (0.26)	GA*503
(Beauregard)	-	0.16 (dip)	0	5.22, 5.85 (5.5)	
Tifton, GA	0.006 (spray)	-	0	0.201, 0.219 (0.21)	GA*504
(Covington)	-	0.16 (dip)		4.93, 5.89 (5.4)	
Clinton, NC	0.006 (spray)	-	0	0.452, 0.462 (0.46)	NC500
(Covington)	-	0.18 (dip)	0	6.97, 5.55 (6.3)	
Fremont, OH	0.006 (spray)	-	0	0.531, 0.491 (0.51)	OH*502
(Beauregard)	-	0.16 (dip)	0	4.29, 4.79 (4.5)	
Weslaco, TX	0.006 (spray)	-	0	0.549, 0.526 (0.54)	TX501
(Beauregard)	-	0.16 (dip)	0	4.62, 4.61 (4.6)	

Animal feedstuffs

Table 18 Residues of thiabendazole on dry peas vines and hay from seed treatment trials conducted in the USA in 2013 Report: TK0180600

Region (Variety)	Application rate (kg ai/ton seed)	DAT (days)	Crop Part	Residue (mg/kg)	Trial
Ephrata, WA (Montex 4153)	0.8	71 71	Vines Hay	0.010, 0.022 (0.02) 0.064, 0.089 (0.08)	TK0180600-31
Parkdale, OR (Montex 4153)	0.8	56 59	Vines Hay	0.015, 0.024 (0.02) 0.056, 0.051 (0.05)	TK0180600-32
American Falls (Montex 4153)	0.8	64 70	Vines Hay	<0.01, <0.01 (<0.01) 0.015, <0.01 (0.01)	TK0180600-33 2013
Chubbuck, ID (Montex 4153)	0.8	64 69	Vines Hay	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	TK0180600-34
Payette, ID (Montex 4153)	0.8	55 59	Vines Hay	0.013, <0.01 (0.01) 0.018, 0.056 (0.04)	TK0180600-35

Table 19 Residues of thiabendazole on cowpea (California Black Eyed #5) animal feed after seed treatment in the USA in 2013. Report: TK0180600

Region	Application rate (kg ai/ton seed)	DAT (days)	Crop Part	Residue (mg/kg)	Trial
Blackville, SC	0.8	46 50	Forage Hay	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	TK0180600-36
Blackville, SC	0.8	70 75	Forage Hay	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	TK0180600-37

Region	Application rate (kg ai/ton seed)	DAT (days)	Crop Part	Residue (mg/kg)	Trial
Hinton, OK	0.8	62 65	Forage Hay	<0.01, <0.01 (<0.01) <0.01, <0.01 (<0.01)	TK0180600-38

FATE OF RESIDUES DURING PROCESSING

Effects on the nature of the residues during processing

A study on the nature of residues in processed commodities was submitted to the Meeting (Adam, 1999). Individual aqueous solutions of [phenyl-U-^{14}C]-thiabendazole were prepared in sterile buffer solutions, and duplicate samples were incubated under different conditions and the recovered in Table 20. The actual concentrations of [phenyl-U-^{14}C]-thiabendazole was in average 5.3 mg/L. For each sample to be analysed the ^{14}C-activity was measured by LSC after the samples were taken, the pH of the solution measured at ambient temperature and thereafter, the samples neutralised to pH 7. Subsamples of the neutralised test solutions were directly analysed by HPLC and 2D-TLC for test substance and degradation products. HPLC and TLC analysis of the radioactivity in the neutralised, aqueous buffer solutions after incubation revealed only parent compound at all three pH-values tested. The results of the radioactivity recovered at each condition is also shown in Table 20.

Table 20 Hydrolytic conditions simulating processing and % of radioactive recovery for thiabendazole

Process represented	Temperature (°C)	Time (min)	pH	Radioactive recovery, thiabendazole (%)
Pasteurisation	90	20	4	103.5, 103.3 (103.4)
Baking, Brewing, Boiling	100	60	5	99.9, 102.5 (101.2)
Sterilisation	120	20	6	99.8, 98.9 (99.4)

Effects on the level of the residues during processing

Sweet potato

Samples from the field phase of post-harvest trial CA525 (dip application) were used for processing of sweet potato roots into flakes, chips, baked sweet potatoes and French fries (Jolly, 2018). Treated sweet potatoes were batch-tub washed for 5 minutes before submitted to processing.

Sweet potato flake: Sweet potatoes were batch steam-peeled for ~ 45 seconds, scrubbed and a sample of the steam-peeled sweet potatoes was analysed. The remaining were cut into slabs, which were spray-washed in water for ~ 30 seconds to remove free starch, pre-cooked at 70–77 °C for 20 minutes and cooled for about 20 minutes in water. The sweet potato slabs were steam-cooked at 94–100 °C for 30 minutes, mashed using a modified meat grinder, and the mash/puree analysed. An aliquot of the sweet potato mash was mixed with an emulsion of pre-weighed food additives and fed onto a drum dryer to dry into a thin sheet, which were fed into a hammer mill for uniform milling of the finished sweet potato flakes.

Sweet potato chip: Washed sweet potatoes were peeled, cut into thin slices (~ 0.16 cm) using a restaurant-style cutter/slicer and discharged into a tub of hot water to remove free starch. The slices were drained and fried in a restaurant-style deep fat fryer at about 165–191 °C for ~60 seconds. The fried sweet potato chips were drained, salted and analysed.

Baked sweet potato: Washed sweet potatoes were punctured with a fork or knife then placed in a preheated oven at 220 °C and baked for about 1 hour to reach an internal temperature of about 88–92 °C, allowed to cool and analysed.

Sweet potato fries: Fries were produced by first slicing washed, unpeeled sweet potatoes into strips (approx. 1 cm) using a French fry cutter, the strips were fried in a deep fat fryer for about 2.5 minutes at 180 °C, the fries were allowed to drain and cool and analysed.

Residues of thiabendazole in samples of sweet potato RAC roots prior to processing and after processing, and the processing factors are shown in Table 21.

Table 21 Processing study on sweet potato with thiabendazole

	Residues (mg/kg)	Processing factor
RAC roots before processing	3.76	-
Washed roots	0.989	0.26
Raw washed & peeled roots	0.124	0.03
Wet Peel	0.013	0.003
Baked washed with peel	1.06	0.28
Chips	0.092	0.02
Puree	0.060	0.02
Fries	0.437	0.12
Flakes	0.319	0.08

APPRAISAL

Thiabendazole, a benzimidazole fungicide, was first evaluated by JMPR in 1970, and the latest residue evaluation was conducted in 2006 (T, R).

The Joint FAO/WHO Expert Committee on Food Additives (JECFA) established an ADI of 0–0.1 mg/kg and the 2006 JMPR established an ARfD of 0.3 mg/kg bw for women of childbearing age and of 1 mg/kg bw for the general population.

The residue definitions for thiabendazole for compliance with the MRL and dietary risk assessment for plant commodities: *thiabendazole.*

The residue definitions for thiabendazole for compliance with the MRL for animal commodities: *sum of thiabendazole and 5-hydroxythiabendazole.*

The residue definitions for thiabendazole for dietary risk assessment for animal commodities: *sum of thiabendazole, 5-hydroxythiabendazole and its sulfate conjugate*.

The compound was scheduled at the Fiftieth Session of the CCPR for the evaluation of additional uses by the 2019 Extra JMPR. Plant metabolism studies on orange (post-harvest) and maize (seed treatment), analytical methods and residue studies on mango, beans, peas and sweet potato, and processing studies were submitted to the Meeting.

Plant metabolism

[Phenyl-U-^{14}C]-thiabendazole was applied post-harvest to orange fruits in a single dose at 0.2 kg ai/hL prior to storage in the dark at 5 °C, and samples were analysed just after application, 8 and 16 weeks later. Radioactivity was extracted from the fruit surface with acetonitrile, and oranges separated into peel and flesh. Radioactivity in orange flesh was < 0.01 mg eq/kg (0.002–0.007 mg/kg eq) and was not further investigated. From 94 (day 0) to 73% (16 weeks) of TRR were recovered from the fruit surface. About 98% TRR in the orange peel on day 0 was thiabendazole (5.2 mg/kg), with residues dropping to 90%TRR after 16 weeks (3.7 mg/kg). Only minor metabolites of thiabendazole were observed in orange peel, arising via hydroxylation of the phenyl ring to produce 5-hydroxy-thiabendazole (~0.02 mg eq/kg), and elimination of the thiazole ring to produce benzimidazole (0.002 mg/kg at 8 weeks) and carboxylated benzimidazole (0.02 mg eq/kg at 8 weeks).

[Phenyl-U-^{14}C]-thiabendazole was applied to maize seed at 0.09 mg/seed. Treated maize was grown under glasshouse and plants were harvested at stages representing commercial forage, sweet corn and maturity. No residues were found in cobs and kernels. The TRRs of foliage from the sweet corn stage and maturity were 0.005 and 0.002 mg eq/kg, respectively. Only the foliage from the forage stage had TRR > 0.01 mg eq/kg (0.014 mg eq/kg), from which 55.5% remained unextracted (0.008 mg

eq/kg). Extracted residues in forage were composed of multiple minor metabolites without the presence of thiabendazole. No further attempt was made to characterise the unextracted residue.

In summary, thiabendazole was the only relevant residue found in orange after post-harvest treatment and no thiabendazole related residues were found in maize commodities after seed treatment.

Methods of analysis

Additional methods of analysis and validation data for crop commodities were submitted to the Meeting. In general, samples are extracted with ethyl acetate, cleaned-up with cation exchange SPE and analysed by LC-MS/MS with a LOQ of 0.01 mg/kg. In another LC-MS/MS method, conjugates of thiabendazole or benzimidazole are extracted with ethyl acetate following addition of glucosidase enzyme to the aqueous phase (LOQ of 0.01 mg/kg). The efficiency of ethyl acetate extraction was confirmed with orange (whole fruit) treated post-harvest from the metabolism study. Additionaly, the QuEChERS method was validated for thiabendazole in crop commodities and for thiabendazole and 5-hydroxy thiabendazole in animal commodities, with a LOQ of 0.01 mg/kg in all cases.

Storage stability of residues under frozen conditions

Stability studies conducted with beans (dry seed), soya beans, spinach, barley and oranges showed that residues were stable under frozen conditions (-20 ºC) for at least 24 months.

Results of supervised residue trials on crops

Mango

Thiabendazole is registered for post-harvest use in a dip solution at a concentration of 0.24 kg ai/hL in Central American countries and 0.19 kg ai/hL in Brazil. In four trials conducted in Brazil according to central American GAP, residues were 2.4, 2.6, 3.4 and 4.5 mg/kg in the whole fruit and 0.01, 0.012, 0.023, and 0.027 (highest individual level of 0.030) mg/kg in the pulp.

The Meeting agreed that four trials were enough to make a recommendation for mango due to the lower variability of the residues in post-harvest treatment, using the mean + 4×SD approach.

The Meeting estimated a maximum residue level of 7 mg/kg (Po), a STMR of 0.0175 mg/kg and a HR of 0.030 mg/kg for thiabendazole in mango.

Succulent beans and peas subgroups

Thiabendazole is registered in the USA as a seed treatment in beans (succulent and dry, except soya bean) at 0.55 kg ai/tonne seed. The GAP for soya bean is 0.20 kg ai/tonne seed. In seven bean trials conducted in the USA approximating the GAP, residues in beans with pods were < 0.01 (6) mg/kg and residues in bean without the pods in one trial were < 0.01 mg/kg.

The GAP rate for peas (succulent and dry) in the USA is 0.33 kg ai/tonne seed. In nine trials conducted in peas at about 3–4 times the GAP rate, residues in the peas without the pods were < 0.01 (9) mg/kg.

As the trials conducted with beans at GAP and the trials conducted with peas at a rate higher than the GAP gave no quantified residues, and the GAP for soya bean is lower, the Meeting agreed that the residue data provided support a recommendation for the subgroups of succulent beans and peas.

The Meeting estimated a maximum residue level of 0.01(*) mg/kg, a STMR and HR of 0 mg/kg for thiabendazole for the subgroups of Beans with pods, Peas with pods, Succulent beans without pods and Succulent peas without pods

Dry beans and peas, subgroups

Thiabendazole is registered in the USA as a seed treatment in beans (succulent and dry, except soya bean) at 0.55 kg ai/tonne seed. The GAP for soya bean is 0.20 kg ai/tonne seed. In nine trials conducted approximating the GAP in the USA, residues in dry beans were < 0.01 (9) mg/kg.

The GAP rate for peas (succulent and dry) in the USA is 0.33 kg ai/tonne seed. In 10 trials conducted with peas using at least 2.4 times the GAP rate, residues in dry peas were < 0.01 (5) and < 0.05 (5) mg/kg.

As the trials conducted with beans at GAP and the trials conducted with peas at a higher rate than the GAP gave no quantified residues, and the GAP for soya bean is lower, the Meeting agreed that the residue data provided support a recommendation for the subgroups of dry beans and peas.

The Meeting estimated a maximum residue level of 0.01(*) mg/kg and a STMR of 0 mg/kg for thiabendazole for the subgroups of Dry beans and Dry peas.

Sweet potato

Thiabendazole is registered in the USA as post-harvest dip in a 0.16 kg ai/hL solution or spray (on a conveyor belt) at 0.006 kg ai/tonne.

In seven trials conducted according to the spray GAP, residues were 0.21, 0.26, 0.38, 0.46, 0.51, 0.54 and 1.2 mg/kg.

In eight trials conducted according to the dip GAP, residues were 2.7, 4.4, 4.5, 4.6, 4.8, 5.4, 5.5, and 6.3 (highest individual level of 6.97) mg/kg.

Based on the dip trials, which gives the highest residues, and on the mean + (4×SD) approach, the Meeting estimated a maximum residue level of 9 mg/kg (Po), a STMR of 4.7 mg/kg and a HR of 6.97 mg/kg for thiabendazole in sweet potato.

Animal feedstuffs

The GAP rate for peas (succulent and dry) in USA is 0.33 kg ai/tonne seed. In the trials conducted with pea in the USA at 2.4 times the GAP, residues ranged from < 0.01 to 0.02 mg/kg in the vines and from < 0.01 to 0.08 mg/kg in the hay. In three trials conducted with cowpea beans at 1.4 times the USA GAP for beans, residues in vines and hay were < 0.01 mg/kg.

As no trials were conducted according to GAP, no recommendations were made for thiabendazole in legume animal feeds.

Fate of residues during processing

In a study to simulate the hydrolysis of thiabendazole under different temperature/time and pH conditions, 99-103% of the applied radioactivity was recovered.

Sweet potatoes treated post-harvest with a dipping solution were processed to flake, chip, baked and fries. The processing factors and estimated STMRs for the processed commodities are shown below.

Crop	PF	STMR/STMR-P, mg/kg	HR/HR-P, mg/kg
Raw sweet potato	-	4.7	6.97
Baked washed with peel	0.28	1.3	1.95
Chips	0.02	0.094	0.139
Puree	0.02	0.094	0.139
Fries	0.12	0.564	0.836
Flakes	0.08	0.376	0.558

Residues in animal commodities

The estimations conducted by the present Meeting do not impact the previous calcutated dietary burden of thiabendazole and do not affect the recommendations made by the JMPR 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 and IESTI assessment.

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

Definition of the residue for compliance with the MRL for animal commodities: *sum of thiabendazole and 5-hydroxythiabendazole*

Definition of the residue for dietary risk assessment for animal commodities: *sum of thiabendazole, 5-hydroxythiabendazole and its sulfate conjugate*.

CCN	Commodity	Recommended Maximun residue level (mg/kg)		STMR or STMR-P (mg/kg)	HR or HR-P (mg/kg)
		New	Previous		
FI 0345	Mango	7 (Po)	5 (Po)	0.0175	0.030
VP 2060	Beans with pods	0.01*		0	0
VP 2061	Peas with pods	0.01*		0	0
VP 2062	Succulent beans without pods	0.01*		0	0
VP 2063	Succulent peas without pods	0.01*		0	0
VD 2065	Dry beans	0.01*		0	
VD 2066	Dry peas	0.01*		0	
VR 0508	Sweet potato	9 (Po)		4.7	6.97
	Sweet potato Baked washed with peel			1.3	1.95
	Sweet potato Chips			0.094	0.139
	Sweet potato Puree			0.094	0.139
	Sweet potato Fries			0.564	0.836
	Sweet potato Flakes			0.376	0.558

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for thiabendazole is 0–0.1 mg/kg bw. The International Estimated Daily Intakes (IEDIs) for thiabendazole were estimated for the 17 GEMS/Food Consumptiion Cluster diets using the STMR or STMR-P values estimated by the JMPR. The results are shown in Annex 3 of the 2019 Extra JMPR Report.

The IEDIs accounted for 2 to 10% of the maximum ADI. The Meeting concluded that the long-term dietary exposure to residues of thiabendazole from uses considered by the JMPR is unlikely to present a public health concern.

Acute dietary exposure

The ARfDs for thiabendazole is 1 mg/kg bw for the general population and 0.3 mg/kg bw for women of child-bearing age. The International Estimate of Short Term Intakes (IESTIs) for thiabendazole 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 2019 Extra JMPR Report.

The IESTIs were 0–20% (children) and 0–7% (general population) of the ARfD for the general population; and from 0–9% of the ARfD for women of child bearing age. The Meeting concluded that the acute dietary exposure to residues of thiabendazole from uses considered by the present Meeting is unlikely to present a public health concern.

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