FLUPYRADIFURONE (285)

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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.

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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

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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