FLUENSULFONE (265)

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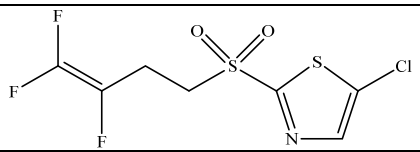
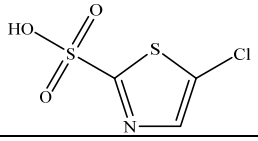
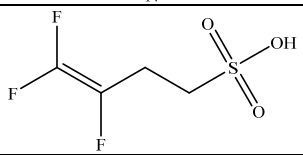
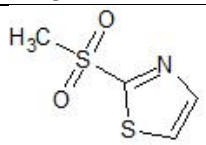
EXPLANATION

Fluensulfone is the International Organisation for Standardisation (ISO)-provisionally approved name for 5-chloro-1,3-thiazol-2-yl 3,4,4-trifluorobut-3-en-1-yl sulfone. Fluensulfone is a non-fumigant nematicide containing sulfone, thiazolyl and fluoroalkenyl functional groups. It was evaluated by JMPR in 2013 for toxicology and in 2014 and 2016 for residues. The 2014 Meeting established an ADI of 0-0.01 mg/kg bw/day and an ARfD of 0.3 mg/kg bw. The 2016 Meeting concluded that for plant commodities, the definition of the residue for enforcement is the sum of fluensulfone and 3,4,4-trifluorobut-3-ene-1-sulfonic acid (BSA), expressed as fluensulfone equivalents. The definition of the residue for risk assessment for plant commodities, and for enforcement and risk assessment in animal commodities is fluensulfone. The 2016 Meeting further concluded that the residue is fat soluble.

Fluensulfone was listed by the 48th Session of the CCPR for evaluation of additional MRLs. The 2017 Meeting received residue data reflecting use of fluensulfone on citrus, soya bean, sugarcane, coffee, and black pepper. Additional data were provided relating to analytical methods, storage stability, and processing.

Common name/abbreviation	Chemical name	Structure	Molecular weight
Fluensulfone, MCW-2	5-Chloro-2-[(3,4,4-trifluorobut-3-en-1-yl) sulfonyl]thiazole		291.7
Thiazole sulfonic acid, TSA, M-3625	5-Chloro-thiazole-2-sulfonic acid		199.6
Butene sulfonic acid, BSA, M-3627	3,4,4-Trifluorobut-3-ene-1-sulfonic acid		190.1
MeS, M-3626	2-Methylsulfonylthiazole		131.2

ANALYTICAL METHODS

Except for samples of dried orange pulp, orange juice, orange oil, refined sugar, and blackstrap molasses generated from trials conducted in the US, analysis of fluensulfone and its metabolites BSA, TSA, and MeS in the residue and storage stability studies provided to the current Meeting was by an LC-MS/MS method comparable to the method evaluated at the 2014 and 2016 Meetings (method number 1977W). Suitable validation data were generated concurrently with each residue study.

For citrus, soya bean, sugarcane, peanut, coffee, and black pepper, recoveries from spiked samples ranged from 70 to 120%, with relative standard deviations ranging from 2 to 18% across all three analytes and across all raw commodities and processed commodities included in the submission. Limits of quantification (LOQ) are based on the lowest limit of method validation and are reported in the supervised trial section, below.

For the citrus matrices noted above, analysis was for residues of fluensulfone and BSA only (Report R-31461). Samples of citrus dried pulp were hydrated with water, and residues were extracted with methanol. Analysis of BSA was accomplished by LC-MS/MS without further sample clean-up. For analysis of fluensulfone, the extract was cleaned-up using C18 solid-phase extraction prior to determining residues by LC-MS/MS. Orange juice was analysed in the same manner as the dried pulp, but without the hydration step. For orange oil, samples were simultaneously extracted with acetonitrile:H_2O (1:1, v/v) and partitioned against hexane. The acetonitrile/water phase was retained, and an aliquot was concentrated by evaporation under nitrogen and subsequently extracted with ethyl acetate. The aqueous layer was collected, filtered, and analysed for residues of BSA using LC-MS/MS, and the organic phase was collected, evaporated to near dryness under nitrogen, brought to volume in acetonitrile, filtered, and analysed for residues of fluensulfone by LC-MS/MS. Matrix-matched standards were used for quantitation of fluensulfone residues in orange oil; residues in other matrices were quantified using standards in HPLC mobile phase.

For sugarcane blackstrap molasses, residues of fluensulfone and BSA were extracted with acetonitrile:H_2O (1:1, v/v). An aliquot of the extract was centrifuged, filtered, and diluted with acetonitrile:H_2O (1:3, v/v) by at least 5-fold for fluensulfone analysis and by at least 50-fold for BSA analysis. Analysis was by LC-MS/MS. For refined sugar, the sugar sample was dissolved in water, and methanol was added to the sugar solution. For analysis of BSA, the methanol:H_2O extract was filtered and analysed by LC-MS/MS. For analysis of fluensulfone, the methanol:H_2O extract was cleaned-up using C18 solid-phase extraction prior to LC-MS/MS analysis. Calibration standards were prepared in HPLC mobile phase.

Table 1 Method validation recoveries for orange (R-31461) and sugarcane (R-35574) matrices

MATRIX	FORTIFICATION LEVEL, PPM	RECOVERIES, %	MEAN ± STD. DEV.%
Fluensulfone			
Oranges	0.01	87, 82, 78	82 ± 4.3
	0.1	82, 78, 77	79 ± 2.3
Orange dried pulp (concurrent recovery)	0.01	76	76
	0.1	70	70
Orange juice	0.01	72, 82, 73	94 ± 11
	0.1	97, 89, 93	93 ± 4.0
Orange oil	0.02	86, 96, 96	93 ± 5.6
	0.1	75, 82, 75	78 ± 4.2
Sugarcane	0.01	92, 90, 74	86 ± 10
	0.1	94	94
	0.2	90, 90	90 ± 0
Blackstrap molasses	0.01	78, 75, 88	81 ± 6.7
	0.1	72, 72, 74	73 ± 1.2
Refined sugar	0.01	87, 78, 84	83 ± 4.7
	0.1	97, 96, 96	96 ± 0.23
BSA			
Oranges	0.01	72, 82, 73	76 ± 5.4
	0.1	72, 72, 70	72 ± 1.1
Orange dried pulp (concurrent recovery)	0.01	77	77
	0.1	82	82
	0.4	92, 86	89 ± 4.2
Orange juice	0.01	85, 90, 76	83 ± 7.1
	0.1	89, 92, 97	93 ± 4.2
Orange oil	0.02	99, 101, 101	100 ± 1.2
	0.1	100, 98, 100	99 ± 0.93
Sugarcane	0.01	95, 103, 84	94 ± 9.4
	0.1	92	92
	0.2	94, 101	98 ± 4.6
Blackstrap molasses	0.01	82, 72, 77	77 ± 4.8
	0.1	92, 95, 91	93 ± 2.0
Refined sugar	0.01	96, 86, 106	96 ± 10
	0.1	93, 94, 89	92 ± 2.8

STABILITY OF PESTICIDE RESIDUES IN STORED ANALYTICAL SAMPLES

Plant matrices

Peanuts

As part of a field trial and processing study conducted with peanuts, samples of shelled peanuts, peanut hay, peanut meal, and refined peanut oil were fortified with fluensulfone and BSA at approximately 0.1 mg/kg. Samples were placed into frozen storage and analysed after ca. 6 months and 12 months; peanut hay included a 0-time sample. Residues were analysed using Method 1977W.

Table 2 Storage stability of fluensulfone and its metabolites in peanut matrices

Matrix	Storage interval, days	Nominal fortification, mg/kg	Residues remaining, mg/kg	% remaining [mean]
Fluensulfone				
Shelled peanuts	180	0.100, 0.0998	0.0892, 0.0878	89, 88 [88]
	378	0.0998, 0.0994	0.0958, 0.0803	96, 81 [88]
Peanut hay	0	0.0996, 0.100	0.0841, 0.0860	84, 86 [85]
	180	0.100, 0.0998	0.0784, 0.0739	78, 74 [76]
	378	0.0998, 0.100	0.0786, 0.0732	79, 73 [76]
Peanut meal	180	0.100, 0.100	0.0892, 0.0824	89, 82 [86]
	378	0.100, 0.0996	0.0916, 0.0924	92, 93 [92]
Peanut oil	180	0.100, 0.0998	0.101, 0.107	101, 107 [104]
	378	0.0998, 0.0996	0.104, 0.103	104, 103 [104]
BSA				
Shelled peanuts	180	0.104, 0.104	0.0776, 0.0743	75, 71 [73]
	378	0.104, 0.103	0.105, 0.0934	101, 91 [96]
Peanut hay	0	0.104, 0.104	0.0926, 0.0924	89, 89 [89]
	180	0.104, 0.104	0.0880, 0.0852	85, 82 [83]
	378	0.104, 0.104	0.0924, 0.0910	89, 88 [88]
Peanut meal	217	0.104, 0.104	0.0933, 0.0950	90, 91 [90]
	378	0.104, 0.104	0.0988, 0.101	95, 97 [96]
Peanut oil	180	0.104, 0.104	0.123, 0.123	118, 118 [118]
	378	0.104, 0.104	0.121, 0.120	116, 115 [116]

Residues of fluensulfone and its BSA metabolite were stable in peanut matrices during frozen storage for at least 378 days.

Sugar cane

Concurrent storage stability samples were prepared and analysed along with samples of sugar cane matrices in an Australian sugar can field trial (Report ASA-13-254). Control samples of sugar cane billets, tops, and trash were fortified with fluensulfone, BSA, TSA, and MeS at 0.1 mg/kg each on Day 0 and Day 35 and placed into frozen storage. Samples were analysed 20 and 55 days after fortification using a modified version of the method PTRL West 2061 W which was evaluated by the 2014 JMPR.

Table 3 Storage stability of fluensulfone and its metabolites in sugar cane matrices

Matrix	Storage interval, days	Nominal fortification, mg/kg	Residues remaining, mg/kg	% remaining [mean]
Fluensulfone				
Sugar cane billets	20	0.1	0.093, 0.092	93, 92 [92]
	55	0.1	0.088, 0.087	88, 87 [88]
Sugar cane tops	20	0.1	0.093, 0.094	93, 94 [94]
	55	0.1	0.089, 0.088	89, 88 [88]
Sugar cane trash	20	0.1	0.094, 0.094	94, 94 [94]

Matrix	Storage interval, days	Nominal fortification, mg/kg	Residues remaining, mg/kg	% remaining [mean]
	55	0.1	0.090, 00.090	90, 90 [90]
BSA				
Sugar cane billets	20	0.1	0.093, 0.093	93, 93 [93]
	55	0.1	0.096, 0.094	96, 94 [95]
Sugar cane tops	20	0.1	0.089, 0.090	89, 90 [90]
	55	0.1	0.083, 0.095	83, 95 [89]
Sugar cane trash	20	0.1	0.093, 0.091	93, 91 [92]
	55	0.1	0.094, 0.093	94, 93 [94]
TSA				
Sugar cane billets	20	0.1	0.095, 0.096	95, 96 [96]
	55	0.1	0.089, 0.088	89, 88 [88]
Sugar cane tops	20	0.1	0.092, 0.093	92, 93 [92]
	55	0.1	0.088, 0.088	88, 88 [88]
Sugar cane trash	20	0.1	0.093, 0.093	93, 93 [93]
	55	0.1	0.092, 0.090	92, 90 [91]
MeS				
Sugar cane billets	20	0.1	0.090, 0.092	90, 92 [91]
	55	0.1	0.093, 0.094	93, 94 [94]
Sugar cane tops	20	0.1	0.093, 0.092	93, 92 [92]
	55	0.1	0.084, 0.096	84, 96 [90]
Sugar cane trash	20	0.1	0.090, 0.090	90, 90 [90]
	55	0.1	0.088, 0.087	88, 87 [88]

Residues of fluensulfone and its metabolites were stable in cane billets, tops, and trash during frozen storage for at least 55 days.

USE PATTERN

Labels demonstrating registrations for the uses considered by the Meeting were not provided.

RESIDUES RESULTING FROM SUPERVISED TRIALS

The Meeting received supervised residue trial data for citrus (oranges only), soya bean, sugar cane, coffee, and black pepper. All studies were in field-grown crops and used the analytical method (modified PTRL 1977W) described above. No residues above the LOQ were found in any of the untreated control samples. For determining the sum of fluensulfone and the BSA metabolite (residue definition for enforcement), the concentration of BSA in each sample was multiplied by 1.53 [the ratio of the molecular weights of fluensulfone (291.7 amu) and BSA (190.1 amu)] and the resulting product added to the concentration of fluensulfone. For purposes of calculating total residues, residues reported as less than the LOQ were assumed to bear residues at the LOQ and residues reported as less than the LOD were assumed to be zero. Trials that are not considered to be independent have their location information surrounded by a heavy border (Table 5, for example).

Group	Commodity	Countries	Table
Citrus	Orange, lemon, grapefruit	Brazil	5
Pulses	Soya bean	Brazil	6
Grasses for sugar or syrup production	Sugar cane	Brazil, Australia	7
Seed for beverages and sweets	Coffee	Brazil	8
Spices	Black pepper	Brazil	9

Citrus fruits

In total, eight supervised residues trials were conducted in oranges in Brazil during 2012 (four locations; Report LRN02611CT_2013i) and 2014 (four locations; Report LRN02716CT_2015 + addendum). All trials were conducted with a single treatment of fluensulfone applied as a 0.1-m-wide band. The trials in 2012 were conducted with a nominal treatment rate of 1.92 kg ai/ha, and those in 2014 were conducted with a nominal treatment rate of 0.96 kg ai/ha. Samples were harvested 90, 120, 150, 180, 210, and 240 (2 locations only) days after treatment and placed into frozen storage prior to shipment to the analytical facility. Samples were stored frozen for no more than 269 days prior to analysis. For all three analytes, the LOQ is 0.08 mg/kg in the Brazilian trials and 0.01 mg/kg in the USA trials.

Residue trials in oranges, lemons, and grapefruit were conducted in the US during 2014 and 2015 (Kelly, Report R-31461). For all trials, fluensulfone was applied once by drip line, micro-emitter, or micro-sprinkler at either 4 kg ai/ha or 20 kg ai/ha and watered-in by irrigation or natural rainfall. Fruits were harvested at commercial maturity (*ca.* 60 days after treatment). Additional samples were collected 1 week and 2 weeks, each before and after commercial harvest, to investigate residue decline. Harvested samples were immediately frozen and maintained frozen during shipment to the analytical facility. Samples designated for processing were maintained at ambient temperature at the analytical facility prior to processing; all other samples were maintained frozen. Whole citrus fruits were stored up to 490 days prior to analysis.

Table 5 Residues of fluensulfone in citrus

Location, Year (variety)	Application					Crop	Residues, mg/kg [mean]			
	No.	Timing, type	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days		Fluen-sulfone	BSA	TSA	Total[a]
GAP Brazil	1	Beginning of rainy season, banded	0.96	1000-2000	n.s.	Citrus	--	--	--	--
2012 (Report LRN02611CT_2013i)										
Andirá, PR, BR 2012 (Pera Rio)	1	BBCH 71, banded	1.92	500	90	Orange	< 0.08	< 0.08	< 0.08	< 0.2
					120	Orange	< 0.08	< 0.08	< 0.08	< 0.2
					150	Orange	< 0.08	< 0.08	< 0.08	< 0.2
					180	Orange	< 0.08	< 0.08	< 0.08	< 0.2
					210	Orange	< 0.08	< 0.08	< 0.08	< 0.2
Cornélio Procópio, PR, BR 2012 (Folha murcha)	1	BBCH 71, banded	1.92	500	90	Orange	< 0.08	< 0.08	< 0.08	< 0.2
					120	Orange	< 0.08	< 0.08	< 0.08	< 0.2
					150	Orange	< 0.08	< 0.08	< 0.08	< 0.2
					180	Orange	< 0.08	< 0.08	< 0.08	< 0.2
					210	Orange	< 0.08	< 0.08	< 0.08	< 0.2
Iracemápolis, SP, BR 2012 (Valência)	1	BBCH 68, banded	1.92	500	90	Orange	< 0.08	< 0.08	< 0.08	< 0.2
					120	Orange	1.04	0.21	< 0.08	1.4

Location, Year (variety)	Application					Crop	Residues, mg/kg [mean]			
	No.	Timing, type	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days		Fluen-sulfone	BSA	TSA	Total[a]
					150	Orange	0.74	0.32	< 0.08	1.2
					180	Orange	0.61	0.28	< 0.08	1.0
					210	Orange	0.38	0.44	< 0.08	1.1
					240	Orange	0.40	0.43	< 0.08	1.1
Leme, SP, BR 2012 (Pera Coroa)	1	BBCH 62, banded	1.92	500	90	Orange	< 0.08	< 0.08	< 0.08	< 0.2
					120	Orange	< 0.08	< 0.08	< 0.08	< 0.2
					150	Orange	< 0.08	< 0.08	< 0.08	< 0.2
					180	Orange	< 0.08	< 0.08	< 0.08	< 0.2
					210	Orange	< 0.08	< 0.08	< 0.08	< 0.2
					240	Orange	< 0.08	< 0.08	< 0.08	< 0.2
2014 (Report LRN02716CT_2015)										
Santa Mariana, PR, BR 2014 (Pera Rio)	1	BBCH 72, banded	0.96	500	90	Orange	< 0.08	< 0.08	< 0.08	< 0.2
					120	Orange	< 0.08	< 0.08	< 0.08	< 0.2
					150	Orange	< 0.08	< 0.08	< 0.08	< 0.2
					180	Orange	< 0.08	< 0.08	< 0.08	< 0.2
					210	Orange	< 0.08	< 0.08	< 0.08	< 0.2
					240	Orange	< 0.08	< 0.08	< 0.08	< 0.2
Uraí, PR, BR 2014 (Pera Rio)	1	BBCH 75, banded	0.96	500	90	Orange	< 0.08	< 0.08	< 0.08	< 0.2
					120	Orange	< 0.08	< 0.08	< 0.08	< 0.2
					150	Orange	< 0.08	< 0.08	< 0.08	< 0.2
					180	Orange	< 0.08	< 0.08	< 0.08	< 0.2
					210	Orange	< 0.08	< 0.08	< 0.08	< 0.2
					240	Orange	< 0.08	< 0.08	< 0.08	< 0.2
Andirá, PR, BR 2014 (Pera Rio)	1	BBCH 73, banded	0.96	500	90	Orange	< 0.08	< 0.08	< 0.08	< 0.2
					120	Orange	< 0.08	< 0.08	< 0.08	< 0.2
					150	Orange	< 0.08	< 0.08	< 0.08	< 0.2
					180	Orange	< 0.08	< 0.08	< 0.08	< 0.2
					210	Orange	< 0.08	< 0.08	< 0.08	< 0.2

Location, Year (variety)	Application					Crop	Residues, mg/kg [mean]			
	No.	Timing, type	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days		Fluen-sulfone	BSA	TSA	Total[a]
					240	Orange	< 0.08	< 0.08	< 0.08	< 0.2
Iracemápolis, SP, BR 2014 (Valência)	1	BBCH 79, banded	0.96	500	90	Orange	< 0.08	< 0.08	< 0.08	< 0.2
					120	Orange	< 0.08	< 0.08	< 0.08	< 0.2
					150	Orange	< 0.08	< 0.08	< 0.08	< 0.2
					180	Orange	< 0.08	< 0.08	< 0.08	< 0.2
					210	Orange	< 0.08	< 0.08	< 0.08	< 0.2
					240	Orange	< 0.08	< 0.08	< 0.08	< 0.2
2014-2015 (Report R-31461)										
Oviedo, FL, US 2014-2015 (Navel) OR FL 1	1	Microjet emitter	4.00	18800	60	Orange	0.014, 0.015 [0.014]	0.018, 0.024 [0.021]	--	0.040, 0.052 [0.046]
Oviedo, FL, US 2014-2015 (Hamlin) OR FL 3	1	Microjet emitter	4.00	18800	59	Orange	0.024, < 0.01 [0.017]	0.019, 0.018 [0.018]	--	0.053, 0.038 [0.045]
Oak Hill, FL, US 2014-2015 (Hamlin) OR FL 2	1	Microjet emitter	4.00	18800	58	Orange	< 0.01, < 0.01 [< 0.01]	0.021, 0.020 [0.021]	--	0.042, 0.041 [0.042]
Mims, FL, US 2014-2015 (Navel)OR FL 4	1	Microjet emitter	4.00	18800	58	Orange	< 0.01, < 0.01 [< 0.01]	0.012, 0.011 [0.011]	--	0.028, 0.026 [0.027]
Oviedo, FL, US 2014-2015 (Murcott and Minneola) OR FL 5	1	Microjet emitter	4.00	18800	60	Mandarin	< 0.01, < 0.01 [< 0.01]	0.037, 0.045 [0.041]	--	0.066, 0.079 [0.072]
Mims, FL, US 2014-2015 (Minneola) OR FL 6	1	Microjet emitter	4.00	18800	58	Mandarin	< 0.01, < 0.01 [< 0.01]	0.029, 0.033 [0.031]	--	0.055, 0.061 [0.058]
Oviedo, FL, US 2014-2015 (Valencia) OR FL 7	1	Microjet emitter	3.99	18800	46	Orange	< 0.01, < 0.01 [< 0.01]	< 0.01, < 0.01 [0.01]	--	< 0.025, < 0.025 [0.025]
					53	Orange	< 0.01, < 0.01 [< 0.01]	0.01, 0.01 [0.01]	--	0.025, 0.025 [0.025]
					60	Orange	< 0.01, < 0.01 [< 0.01]	< 0.01, < 0.01 [< 0.01]	--	< 0.025, < 0.025 [< 0.025]
					68	Orange	< 0.01, < 0.01 [< 0.01]	< 0.01, < 0.01 [< 0.01]	--	< 0.025, < 0.025 [< 0.025]
					75	Orange	< 0.01, < 0.01 [< 0.01]	0.011, 0.011 [0.011]	--	0.026, 0.027 [0.027]
Orlando, FL, US 2014-2015 (Valencia) OR-FL-8	1	Microjet emitter	3.99	18800	60	Orange	< 0.01, < 0.01 [< 0.01]	< 0.01, < 0.01 [< 0.01]	--	< 0.025, < 0.025 [< 0.025]
			20	18900	60	Orange	< 0.01, < 0.01	0.034, 0.033	--	0.062, 0.060,

Location, Year (variety)	Application					Crop	Residues, mg/kg [mean]			
	No.	Timing, type	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days		Fluen-sulfone	BSA	TSA	Total[a]
							[< 0.01]	[0.034]		[0.061]
Raymondville, TX, US 2014-2015 (N-33 Navel) OR-TX	1	Post-directed spray	4.01	19100	60	Orange	< 0.01, < 0.01 [< 0.01]	< 0.01, < 0.01 [< 0.01]	--	< 0.025, < 0.025 [< 0.025]
Porterville, CA, US 2014-2015 (Valencia) OR-CA-1	1	Micro-Sprinkler	3.99	18900	59	Orange	< 0.01, < 0.01 [< 0.01]	< 0.01, 0.016 [< 0.013]	--	< 0.025, 0.034 [0.030]
Porterville, CA, US 2014-2015 (Murcott) OR-CA-2	1	Micro-Sprinkler	3.99	18900	60	Mandarin	< 0.01, < 0.01 [< 0.01]	0.013, 0.015 [0.014]	--	0.031, 0.033 [0.032]
Richgrove, CA, US 2014-2015 (Valencia) OR-CA-3	1	Micro-Sprinkler	3.99	18900	60	Orange	< 0.01, < 0.01 [< 0.01]	0.016, 0.023 [0.020]	--	0.035, 0.046 [0.040]
			20	18900	60	Orange	< 0.01, < 0.01 [< 0.01]	0.065, 0.035 [0.050]	--	0.11, 0.064 [0.087]
Clermont, FL, US 2014-2015 (Bearrs) LE-FL	1	Microjet emitter	4.00	18900	60	Lemon	< 0.01, < 0.01 [< 0.01]	0.047, 0.054 [0.050]	--	0.081, 0.092 [0.087]
Porterville, CA, US 2014-2015 (Pryor) LE-CA-1	1	Micro-Sprinkler	4.00	18900	61	Lemon	< 0.01, < 0.01 [< 0.01]	< 0.01, < 0.01 [< 0.01]	--	< 0.025, < 0.025 [< 0.025]
Porterville, CA, US 2014-2015 (Lison) LA-CA-2	1	Micro-Sprinkler	4.00	18900	60	Lemon	< 0.01, < 0.01 [< 0.01]	< 0.01, < 0.01 [< 0.01]	--	< 0.025, < 0.025 [< 0.025]
Somis, CA, US 2014-2015 (Eureka)LA-CA-3	1	Micro-Sprinkler	4.00	18900	60	Lemon	0.063, 0.035 [0.049]	0.063, 0.046 [0.054]	--	0.16, 0.11 [0.13]
Richgrove, CA, US 2014-2015 (Lisbon) LA-CA-4	1	Micro-Sprinkler	4.00	18900	60	Lemon	< 0.01, < 0.01 [< 0.01]	< 0.01, < 0.01 [< 0.01]	--	< 0.025, < 0.025 [< 0.025]
Oak Hill, FL, US 2014-2015 (Pink) GF-FL-1	1	Microjet emitter	4.00	18800	58	Grape-fruit	< 0.01, < 0.01 [< 0.01]	< 0.01, < 0.01 [< 0.01]	--	< 0.025, < 0.025 [< 0.025]
Oviedo, FL, US 2014-2015 (White/Flame) GF-FL-2	1	Microjet emitter	4.00	18900	60	Grape-fruit	< 0.01, 0.018 [< 0.014]	0.042, 0.040 [0.041]	--	0.074, 0.080 [0.077]
Mims, FL, US 2014-2015 (Ray Red) GF-FL-3	1	Microjet emitter	4.00	18800	58	Grape-fruit	< 0.01, < 0.01 [< 0.01]	0.011, 0.01 [0.01]	--	0.026, 0.025 [0.026]
Raymondville, TX, US 2014-2015 (Rio Red) GF-TX	1	Post-directed spray	4.01	19100	60	Grape-fruit	< 0.01, < 0.01 [< 0.01]	< 0.01, < 0.01 [< 0.01]	--	< 0.025, < 0.025 [< 0.025]
Porterville, CA, US 2014-2015 (Star Ruby) GF-CA-1	1	Micro-Sprinkler	4.00	18900	58	Grape-fruit	< 0.01, < 0.01 [< 0.01]	< 0.01, < 0.01 [< 0.01]	--	< 0.025, < 0.025 [< 0.025]
Porterville, CA, US 2014-2015 (Mellogold) GF-CA-2	1	Micro-Sprinkler	3.98	18900	46	Grape-fruit	< 0.01, < 0.01 [< 0.01]	< 0.01, < 0.01 [< 0.01]	--	< 0.025, < 0.025 [< 0.025]
					53		< 0.01,	< 0.01,	--	< 0.025,

Location, Year (variety)	Application					Crop	Residues, mg/kg [mean]			
	No.	Timing, type	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days		Fluen-sulfone	BSA	TSA	Total[a]
							< 0.01 [< 0.01]	< 0.01 [< 0.01]		< 0.025 [< 0.025]
					60		< 0.01, < 0.01 [< 0.01]	< 0.01, < 0.01 [< 0.01]	--	< 0.025, < 0.025 [< 0.025]
					67		< 0.01, < 0.01 [< 0.01]	< 0.01, < 0.01 [< 0.01]	--	< 0.025, < 0.025 [< 0.025]
					73		< 0.01, < 0.01 [< 0.01]	< 0.01, < 0.01 [< 0.01]	--	< 0.025, < 0.025 [< 0.025]

[a] Sum of fluensulfone and BSA, expressed as fluensulfone.

Pulses

Soya bean

Four supervised residues trials were conducted in soya beans in Brazil during 2012 (Report LRN02609SJ_2013i + addendum). All trials were conducted with a single treatment of fluensulfone applied as a 0.1-m-wide band immediately prior to planting. The trials were conducted with a nominal treatment rate of 0.24 kg ai/ha. Soya bean seeds were harvested 110, 115, 120, and 125 days after treatment and placed into frozen storage prior to shipment to the analytical facility. Samples were stored frozen for no more than 270 days prior to analysis of fluensulfone and MeS, and no more than 480 days prior to analysis of BSA and TSA. For all three analytes, the LOQ is 0.08 mg/kg.

Table 6 Residues of fluensulfone in soya bean

Location, Year (variety)	Application					Residues, mg/kg			
	No.	Timing, type	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days	Fluensulfone	BSA	TSA	Total[a]
GAP BR	1	Banded just prior to planting	0.24	500	n.s.	--	--	--	--
Sorocabal, SP, BR 2012 (BMX Potência RR)	1	Banded, pre-plant	0.24	40	110	< 0.08	< 0.08	< 0.08	< 0.2
					115	< 0.08	< 0.08	< 0.08	< 0.2
					120	< 0.08	< 0.08	< 0.08	< 0.2
					125	< 0.08	< 0.08	< 0.08	< 0.2
Iracemápolis, SP, BR 2012 (BMX Potência RR)	1	Banded, pre-plant	0.24	40	110	< 0.08	< 0.08	< 0.08	< 0.2
					115	< 0.08	< 0.08	< 0.08	< 0.2
					120	< 0.08	< 0.08	< 0.08	< 0.2
					125	< 0.08	< 0.08	< 0.08	< 0.2
Santa Mariana, PR, BR 2012 (BMX Potência RR)	1	Banded, pre-plant	0.24	40	110	< 0.08	< 0.08	< 0.08	< 0.2

Location, Year (variety)	Application					Residues, mg/kg			
	No.	Timing, type	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days	Fluensulfone	BSA	TSA	Total[a]
					115	< 0.08	< 0.08	< 0.08	< 0.2
					120	< 0.08	< 0.08	< 0.08	< 0.2
					125	< 0.08	< 0.08	< 0.08	< 0.2
Bandeiranles, PR, BR 2012 (BR 369 km 54)	1	Banded, pre-plant	0.24	40	110	< 0.08	< 0.08	< 0.08	< 0.2
					115	< 0.08	< 0.08	< 0.08	< 0.2
					120	< 0.08	< 0.08	< 0.08	< 0.2
					125	< 0.08	< 0.08	< 0.08	< 0.2

[a] Sum of fluensulfone and BSA, expressed as fluensulfone.

Grasses for sugar or syrup production

Sugar cane

Supervised residues trials were conducted in sugar cane in Brazil during 2012 (four trials; Report LRN02607CN_2013i) and 2014 (four trials; Report LRN02724CN_2015), in Australia during 2015 (four trials; Report ASA-13-254), and in the USA during 2015 (eight trials; Report R-34474). All trials were conducted with a single treatment of fluensulfone applied as a 0.5-m-wide band to the furrow (with billets). The trials in Brazil were conducted with a nominal treatment rate of 0.96 kg ai/ha, and sugar cane stalks were harvested 11, 12, 13, and 14 months after treatment. The trials in Australia were conducted at rates of 1.92 and 3.84 kg ai/ha, and sugar cane billets, tops, and trash were harvested 10 or 12 months after treatment. The trials in the USA were conducted at rates of either 4 kg ai/ha or 20 kg ai/ha. Samples from all trials were placed into frozen storage immediately after harvest and shipped frozen to the analytical facility. Samples were stored frozen for no more than 49 days (Australia), 173 days (USA), or 198 days (Brazil) prior to analysis. For all three analytes, the LOQ is 0.08 mg/kg in the Brazilian trials and 0.01 mg/kg in the Australian and USA trials (USA trials analysed only for fluensulfone and BSA).

Table 7 Residues of fluensulfone in sugar cane

Location, Year (variety)	Application					Matrix[a]	Residues, mg/kg [mean]			
	No.	Timing, type	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days		Fluensulfone	BSA	TSA	Total[b]
GAP BR	1	Banded, at planting or banded after harvest (ratoon)	0.96	1000-2000	n.s.	--	--	--	--	--
Brazil 2012 (Report 002 075 11 B)										
Rio Claro, SP, BR 2012 (IAC 110)	1	Banded, at planting	0.96	200	335	Cane	< 0.08	< 0.08	< 0.08	< 0.2
					364	Cane	< 0.08	< 0.08	< 0.08	< 0.2
					395	Cane	< 0.08	< 0.08	< 0.08	< 0.2
					425	Cane	< 0.08	< 0.08	< 0.08	< 0.2
Piracicaba, SP, BR 2012 (IAC 110)	1	Banded, at planting	0.96	200	335	Cane	< 0.08	< 0.08	< 0.08	< 0.2
					364	Cane	< 0.08	< 0.08	< 0.08	< 0.2

Location, Year (variety)	Application					Matrix [a]	Residues, mg/kg [mean]			
	No.	Timing, type	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days		Fluensulfone	BSA	TSA	Total [b]
					395	Cane	< 0.08	< 0.08	< 0.08	< 0.2
					425	Cane	< 0.08	< 0.08	< 0.08	< 0.2
Andirá, PR, BR 2012 (BR 89)	1	Banded, at planting	0.96	200	334	Cane	< 0.08	< 0.08	< 0.08	< 0.2
					365	Cane	< 0.08	< 0.08	< 0.08	< 0.2
					395	Cane	< 0.08	< 0.08	< 0.08	< 0.2
					426	Cane	< 0.08	< 0.08	< 0.08	< 0.2
Abatiá, PR, BR 2012 (BR 89)	1	Banded, at planting	0.96	200	334	Cane	< 0.08	< 0.08	< 0.08	< 0.2
					365	Cane	< 0.08	< 0.08	< 0.08	< 0.2
					395	Cane	< 0.08	< 0.08	< 0.08	< 0.2
					426	Cane	< 0.08	< 0.08	< 0.08	< 0.2
Brazil 2014 (Report RA 002 004 14 B)										
Iracemápolis, SP, BR 2014 (RB 88)	1	Banded, at planting	0.96	200	331	Cane	< 0.08	< 0.08	< 0.08	< 0.2
					360	Cane	< 0.08	< 0.08	< 0.08	< 0.2
					390	Cane	< 0.08	< 0.08	< 0.08	< 0.2
					420	Cane	< 0.08	< 0.08	< 0.08	< 0.2
Santa Mariana, PR, BR 2014 (RB 88)	1	Banded, at planting	0.96	200	330	Cane	< 0.08	< 0.08	< 0.08	< 0.2
					360	Cane	< 0.08	< 0.08	< 0.08	< 0.2
					390	Cane	< 0.08	< 0.08	< 0.08	< 0.2
					420	Cane	< 0.08	< 0.08	< 0.08	< 0.2
Cabarám, PR, BR 2014 (RB 88)	1	Banded, at planting	0.96	200	330	Cane	< 0.08	< 0.08	< 0.08	< 0.2
					360	Cane	< 0.08	< 0.08	< 0.08	< 0.2
					390	Cane	< 0.08	< 0.08	< 0.08	< 0.2
					420	Cane	< 0.08	< 0.08	< 0.08	< 0.2
Anápolis, GO, BR 2014 (RB 855453)	1	Banded, at planting	0.96	200	330	Cane	< 0.08	< 0.08	< 0.08	< 0.2
					360	Cane	< 0.08	< 0.08	< 0.08	< 0.2
					390	Cane	< 0.08	< 0.08	< 0.08	< 0.2
					420	Cane	< 0.08	< 0.08	< 0.08	< 0.2
Australia 2015 (Report ASA-13-254)										
Bellenden Ker, QLD, AU 2015 (Q208)	1	Banded, at planting	1.99	440	307	Billets	< 0.01	< 0.01	0.20	< 0.025
						Tops	< 0.01	< 0.01	0.75	< 0.025
						Trash	< 0.01	< 0.01	1.61	< 0.025
	1	Banded, at planting	3.98	440	307	Billets	< 0.01	< 0.01	0.52	< 0.025
						Tops	< 0.01	< 0.01	3.38	< 0.025
						Trash	< 0.01	< 0.01	2.70	< 0.025
Miriwinni, QLD, AU 2015 (Q220)	1	Banded, at planting	1.99	440	307	Billets	< 0.01	< 0.01	0.34	< 0.025

Location, Year (variety)	Application					Matrix [a]	Residues, mg/kg [mean]			
	No.	Timing, type	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days		Fluensulfone	BSA	TSA	Total [b]
						Tops	< 0.01	< 0.01	1.23	< 0.025
						Trash	< 0.01	< 0.01	0.96	< 0.025
	1	Banded, at planting	3.98	440	307	Billets	< 0.01	< 0.01	0.76	< 0.025
						Tops	< 0.01	< 0.01	4.38	< 0.025
						Trash	< 0.01	< 0.01	3.46	< 0.025
Stapylton, QLD, AU 2015 (Q183)	1	Banded, at planting	2.03	408	372	Billets	< 0.01	< 0.01	0.13	< 0.025
						Tops	< 0.01	< 0.01	0.56	< 0.025
						Trash	< 0.01	< 0.01	0.41	< 0.025
	1	Banded, at planting	4.06	408	372	Billets	< 0.01	< 0.01	0.34	< 0.025
						Tops	< 0.01	< 0.01	1.55	< 0.025
						Trash	< 0.01	< 0.01	1.11	< 0.025
Stotts Creek, NSW, AU 2015 (Q244)	1	Banded, at planting	2.03	408	379	Billets	< 0.01	< 0.01	0.23	< 0.025
						Tops	< 0.01	< 0.01	0.84	< 0.025
						Trash	< 0.01	< 0.01	0.77	< 0.025
	1	Banded, at planting	4.06	408	379	Billets	< 0.01	< 0.01	0.67	< 0.025
						Tops	< 0.01	< 0.01	2.11	< 0.025
						Trash	< 0.01	< 0.01	1.81	< 0.025
USA 2015 (Report R-35574)										
Oviedo, Florida, US 2015 (2143)	1	Banded, at planting	3.88	280	267	Cane	< 0.0033, < 0.0033 [< 0.01]	0.022, 0.024 [0.023]	--	0.034, 0.037 [0.035]
Moore Haven, Florida, US 2015 (CP1252)	1	Banded, at planting	3.93	290	274	Cane	< 0.0033, < 0.0033 [< 0.01]	< 0.0033, 0.012 [< 0.01]	--	< 0.005, 0.018 [0.018]
Jupiter, Florida, US 2015 (1743)	1	Banded, at planting	4.02	290	235	Cane	< 0.0033, < 0.0033 [< 0.01]	< 0.01, < 0.01 [< 0.01]	--	< 0.015, < 0.015 [< 0.015]
Washington, Louisiana, US 2015 (540)	1	Banded, at planting	4.02	240	274	Cane	< 0.0033, < 0.0033 [< 0.01]	< 0.0033, < 0.0033 [< 0.01]	--	< 0.005, < 0.005 [< 0.015]
Washington, Louisiana, US 2015 (540)	1	Banded, at planting	4.09	250	266	Cane	< 0.0033, < 0.0033 [< 0.01]	< 0.01, < 0.01 [< 0.01]	--	< 0.015, < 0.015 [< 0.015]
					270	Cane	< 0.0033, < 0.0033 [< 0.01]	< 0.01, < 0.0033 [< 0.01]	--	< 0.015, < 0.005 [< 0.015]
					274	Cane	< 0.0033, < 0.0033 [< 0.01]	< 0.0033, < 0.0033 [< 0.01]	--	< 0.005, < 0.005 [< 0.015]
					278	Cane	< 0.0033, < 0.0033 [< 0.01]	< 0.01, < 0.0033 [< 0.01]	--	< 0.015, < 0.005 [< 0.015]
					282	Cane	< 0.0033, < 0.0033 [< 0.01]	< 0.01, < 0.0033 [< 0.01]	--	< 0.015, < 0.005 [< 0.015]
Church Point, Louisiana, US 2015 (540)	1	Banded, at planting	4.09	250	274	Cane	< 0.0033, < 0.0033 [< 0.01]	< 0.0033, < 0.0033 [< 0.01]	--	< 0.005, < 0.005 [< 0.015]
	1	Banded, at	20	250	280	Cane	0.075, 0.042	0.122,	--	0.30,

Location, Year (variety)	Application					Matrix [a]	Residues, mg/kg [mean]			
	No.	Timing, type	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days		Fluensulfone	BSA	TSA	Total [b]
		planting					[0.058]	0.104 [0.113]		0.22 [0.26]
Raymondville, Texas, US 2015 (1210)	1	Banded, at planting	4.12	290	314	Cane	< 0.0033, < 0.0033 [< 0.01]	< 0.0033, < 0.0033 [< 0.01]	--	< 0.005, < 0.005 [< 0.015]
Kunia, Hawaii, US 2015 (65-7052)	1	Banded, at planting	4.18	340	365	Cane	< 0.0033, < 0.0033 [< 0.01]	< 0.0033, < 0.0033 [< 0.01]	--	< 0.005, < 0.005 [< 0.015]

[a] Cane and billet residue data are presented on a fresh-weight basis; tops and trash are presented on a dry-weight basis.

[b] Sum of fluensulfone and BSA, expressed as fluensulfone.

Seed for beverages and sweets

Coffee

Supervised residues trials were conducted in coffee in Brazil during 2012 (four trials; Report LRN02612CF_2013i + addendum) and 2014 (four trials; Report RA 002 003 14 B). All trials were conducted with a single treatment of fluensulfone applied as a 0.5-m-wide band. The trials were conducted with a nominal treatment rate of 1.92 kg ai/ha (2012) or 0.96 kg ai/ha (2014). Coffee beans were harvested 150, 165, 180, 195, and 210 days after treatment and sun-dried prior to being processed to remove the pulp. After pulp removal, the beans were placed into frozen storage. Samples were stored frozen for no more than 373 days prior to analysis of fluensulfone and MeS, and no more than 178 days prior to analysis of BSA and TSA. For all three analytes, the LOQ is 0.08 mg/kg.

Table 8 Residues of fluensulfone in coffee

Location, Year (variety)	Application					Residues, mg/kg			
	No.	Timing, type	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days	Fluen-sulfone	BSA	TSA	Total[a]
GAP BR	1	Beginning of the rainy season, banded.	0.96	1000-2000	n.s.	--	--	--	--
2012 (LRN02612CF_2013i)									
Ribeirão do Pinhal, PR, BR 2012 (Obatá)	1	BBCH 71, banded	1.92	200	150	< 0.08	< 0.08	< 0.08	< 0.2
					165	< 0.08	< 0.08	< 0.08	< 0.2
					180	< 0.08	< 0.08	0.15	< 0.2
					195	< 0.08	< 0.08	< 0.08	< 0.2
					210	< 0.08	< 0.08	< 0.08	< 0.2
Bandeirantes, PR, BR 2012 (Obatá)	1	BBCH 72, banded	1.92	200	150	< 0.08	< 0.08	< 0.08	< 0.2
					165	< 0.08	< 0.08	0.08	< 0.2
					180	< 0.08	< 0.08	0.12	< 0.2
					195	< 0.08	< 0.08	0.13	< 0.2
					210	< 0.08	< 0.08	< 0.08	< 0.2
Laranjal Paulista, SP, BR 2012 (ICATU)	1	BBCH 70, banded	1.92	200	150	< 0.08	< 0.08	< 0.08	< 0.2
					165	< 0.08	< 0.08	< 0.08	< 0.2

Location, Year (variety)	Application					Residues, mg/kg			
	No.	Timing, type	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days	Fluen-sulfone	BSA	TSA	Total[a]
					180	< 0.08	< 0.08	< 0.08	< 0.2
					195	< 0.08	< 0.08	< 0.08	< 0.2
					210	< 0.08	< 0.08	< 0.08	< 0.2
Iracemápolis, SP, BR 2012 (ICATU)	1	BBCH 59, banded	1.92	200	150	< 0.08	< 0.08	< 0.08	< 0.2
					165	< 0.08	< 0.08	< 0.08	< 0.2
					180	< 0.08	< 0.08	0.12	< 0.2
					195	< 0.08	< 0.08	0.32	< 0.2
					210	< 0.08	< 0.08	0.12	< 0.2
2014 (Report RA 002 003 14 B)									
Santa Mariana, PR, BR 2014 (Caturra Amerelo)	1		0.96	200	150	< 0.08	< 0.08	< 0.08	< 0.2
					165	< 0.08	< 0.08	< 0.08	< 0.2
					180	< 0.08	< 0.08	< 0.08	< 0.2
					195	< 0.08	< 0.08	< 0.08	< 0.2
					210	< 0.08	< 0.08	< 0.08	< 0.2
Bandeirantes, PR, BR 2014 (Catuai Vermelho)	1		0.96	200	150	< 0.08	< 0.08	< 0.08	< 0.2
					165	< 0.08	< 0.08	< 0.08	< 0.2
					180	< 0.08	< 0.08	< 0.08	< 0.2
					195	< 0.08	< 0.08	< 0.08	< 0.2
					210	< 0.08	< 0.08	< 0.08	< 0.2
Procópio, PR, BR 2014 (Catuai Vermelho)	1		0.96	200	150	< 0.08	< 0.08	< 0.08	< 0.2
					165	< 0.08	< 0.08	< 0.08	< 0.2
					180	< 0.08	< 0.08	< 0.08	< 0.2
					195	< 0.08	< 0.08	< 0.08	< 0.2
					210	< 0.08	< 0.08	< 0.08	< 0.2
Iracemápolis, SP, BR 2014 (Catuai Vermelho)	1		0.96	200	150	< 0.08	< 0.08	< 0.08	< 0.2
					165	< 0.08	< 0.08	< 0.08	< 0.2
					180	< 0.08	< 0.08	< 0.08	< 0.2
					195	< 0.08	< 0.08	< 0.08	< 0.2
					210	< 0.08	< 0.08	< 0.08	< 0.2

[a] Sum of fluensulfone and BSA, expressed as fluensulfone.

Spices

Black pepper

Four supervised residues trials were conducted in black pepper in Brazil during 2013 (Report LRN02616PR_2013i + addendum). All trials were conducted with a single treatment of fluensulfone applied as a 0.5-m-wide band. The trials were conducted with a nominal treatment rate of 0.96 kg ai/ha. Fruits were harvested 55, 60, 65, and 70 days after treatment. Fruits were dried to form

peppercorns and placed into frozen storage prior to shipment to the analytical facility. Samples were stored frozen for no more than 95 days prior to analysis. For all three analytes, the LOQ is 0.08 mg/kg.

Table 9 Residues of fluensulfone in black pepper

Location, Year (variety)	Application					Residues, mg/kg			
	No.	Timing, type	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days	Fluensulfone	BSA	TSA	Total [a]
GAP BR	1	Beginning of the rainy season, banded.	0.96	1000-2000	n.s.	--	--	--	--
Jaguaré, ES, BR 2013 (Bragantina)	1	BBCH 71, banded	0.96	100	55	< 0.08	< 0.08	< 0.08	< 0.2
					60	< 0.08	< 0.08	< 0.08	< 0.2
					65	< 0.08	< 0.08	< 0.08	< 0.2
					70	< 0.08	< 0.08	< 0.08	< 0.2
Linhares, ES, BR 2013 (Bragantina)	1	BBCH 71, banded	0.96	100	55	< 0.08	< 0.08	< 0.08	< 0.2
					60	< 0.08	< 0.08	< 0.08	< 0.2
					65	< 0.08	< 0.08	< 0.08	< 0.2
					70	< 0.08	< 0.08	< 0.08	< 0.2
Aracruz, ES, BR 2013 (Bragantina)	1	BBCH 72, banded	0.96	100	55	< 0.08	< 0.08	< 0.08	< 0.2
					60	< 0.08	< 0.08	< 0.08	< 0.2
					65	< 0.08	< 0.08	< 0.08	< 0.2
					70	< 0.08	< 0.08	< 0.08	< 0.2
Mucuri, BA, BR 2013 (Bragantina)	1	BBCH 71, banded	0.96	100	55	< 0.08	< 0.08	< 0.08	< 0.2
					60	< 0.08	< 0.08	< 0.08	< 0.2
					65	< 0.08	< 0.08	< 0.08	< 0.2
					70	< 0.08	< 0.08	< 0.08	< 0.2

[a] Sum of fluensulfone and BSA, expressed as fluensulfone.

FATE OF RESIDUES IN PROCESSING

Citrus

Samples of oranges treated at the exaggerated rate of 8.1 kg ai/ha from the US field trials described above (R-31461) were processed into juice, wet pomace, dried pulp, oil, and marmalade using simulated commercial practices. A subsample of washed fruits was retained for processing into marmalade and a separate subsample was put into an abrasive peeler for scarifying. The oil-water emulsion from the scarifying process was separated into oil and water components and the oil retained for analysis. Abraded fruits were transferred to a juice extractor, and the resulting juice was screened to remove membranes, seeds, and peel fragments.

The subsample of oranges for marmalade were peeled, and the rind was chipped in a food processor. The rind chips and orange pulp were cooked, separately, and then combined. Lemon juice and sugar were added to the combined rind and pulp, and the mixture was boiled for 3 minutes. Pectin was added and the mixture was boiled for an additional 2 minutes, after which the finished product

was transferred to sterile jars and cooled. Samples of marmalade were then transferred to frozen storage prior to analysis of residues.

Wet orange pomace was produced by combining the peel from the juice extractor, the scarified flavedo from the oil production, and the screenings (membranes, seeds, peel fragments) from the juice production. Lime was added to the wet pulp and mixed, and the resulting limed pulp was pressed. A subsample of the pressed wet pulp was placed into frozen storage prior to analysis of residues and a separate subsample was air dried to <10% moisture. The dried pulp was processed in a hammer mill and then transferred to frozen storage prior to analysis of residues.

Residues of fluensulfone and BSA were determined using the methods described above, with an LOQ of 0.01 mg/kg for both analytes.

Table 10 Residues of fluensulfone in processed citrus commodities (R-31461)

Matrix	Analyte [a]	Residues, mg/kg	Processing factor [median/best estimate]
Whole fruit	Fluensulfone	< 0.01, < 0.01	--
	BSA	0.033, 0.050	--
	Total	0.060, 0.086	--
Juice	Fluensulfone	< 0.01, < 0.01	Unknown [1]
	BSA	0.006, 0.020	1.9, 0.61
	Total	0.019, 0.041	0.32, 0.67 [0.49]
Marmalade	Fluensulfone	< 0.01, < 0.01	Unknown [1]
	BSA	< 0.01, < 0.01	<0.30, < 0.20
	Total	< 0.025, < 0.025	>0.42, < 0.29 [0.35]
Oil	Fluensulfone	0.698, 0.047	>70, >4.7 [1000; per OECD Guidance Document No. 96]
	BSA	< 0.01, < 0.01	<0.30, < 0.20
	Total	0.71, 0.062	12, 0.72 [6.3]
Pomace (wet)	Fluensulfone	< 0.01, < 0.01	Unknown [1]
	BSA	0.047, 0.085	1.4, 1.7
	Total	0.082, 0.14	1.4, 1.6 [1.5]
Pulp (dry)	Fluensulfone	0.020, < 0.01	>2, unknown [2]
	BSA	0.187, 0.351	5.7, 7.0
	Total	0.31, 0.55	5.1, 6.3 [5.7]

[a] Total = sum of fluensulfone and BSA, expressed as fluensulfone.

Sugar cane

A sample of sugar cane from a US field trial (FL-1; as described above) was harvested at commercial maturity 267 days after application of fluensulfone and processed into molasses and refined sugar using simulated commercial practices (Report No. R-35574). Samples of cane were cleaned and pressed using a cane crusher to express the juice from the cane. The juice was retained and the fibres (bagasse) were weighed and discarded. The juice was screened, adjusted to pH 7.2–7.6, and heated to 97–100 °C. After centrifugation to separate solids (mud) from thin juice, the mud was weighed and discarded, and the juice was evaporated to form thick juice. Raw sugar was crystalized from the thick juice and separated from the molasses by centrifugation. Raw sugar was processed into refined sugar by dissolving in water, adjusting to acidic pH (5.0–5.8), bringing back to near neutral pH, heating, and filtering in the presence of activated charcoal. Refined sugar was crystalized during vacuum evaporation and cooling.

Residues of fluensulfone and BSA were determined using the methods described above, with an LOQ of 0.01 mg/kg for both analytes.

Table 11 Residues of fluensulfone in processed sugar cane commodities (Report R-35574)

Matrix	Analyte [a]	Residues, mg/kg	Processing factor [best estimate]
Whole cane	Fluensulfone	< 0.0033	--
	BSA	0.023	--
	Total	0.035	--

Matrix	Analyte [a]	Residues, mg/kg	Processing factor [best estimate]
Blackstrap molasses	Fluensulfone	< 0.0033	Unknown [1]
	BSA	0.17	7.4
	Total	0.26	7.4 [7.4]
Refined sugar	Fluensulfone	< 0.0033	Unknown [1]
	BSA	< 0.0033	<0.14
	Total	< 0.01	< 0.28 [0.28]

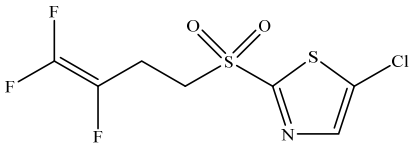
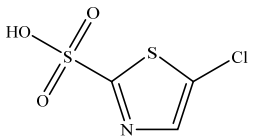
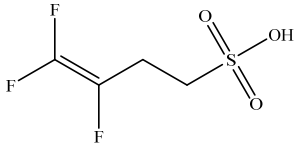
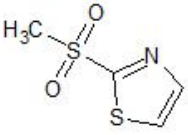
[a] Total = sum of fluensulfone and BSA, expressed as fluensulfone.

APPRAISAL

Fluensulfone was evaluated by JMPR for the first time for toxicology in 2013, when an ADI of 0–0.01 mg/kg bw/day and an ARfD of 0.3 mg/kg bw were established. Residues aspects of fluensulfone were evaluated by the 2014 and 2016 JMPR. The evaluation by the 2016 Meeting included a revision to the residue definitions for enforcement and dietary assessment as follows: The definition of the residue for enforcement for plant commodities is the sum of fluensulfone and 3,4,4-trifluorobut-3-ene-1-sulfonic acid (BSA), expressed as fluensulfone; the definition of the residue for dietary risk assessment for plant commodities is fluensulfone; and the definition of the residue for enforcement and dietary risk assessment for animal commodities is fluensulfone. The 2016 Meeting further determined that the residue is fat soluble.

The 48th Session of the CCPR, scheduled fluensulfone for evaluation of additional crop uses by the 2017 JMPR. The 2017 Meeting received residue data reflecting use of fluensulfone on citrus fruits, soya bean, sugarcane, coffee, and black pepper. Additional data were provided relating to analytical methods, storage stability, and processing.

The following residue components are discussed. Structures and chemical names are tabulated below.

Common name/abbreviation	Chemical name	Structure	Molecular weight
Fluensulfone, MCW-2	5-Chloro-2-[(3,4,4-trifluorobut-3-en-1-yl)sulfonyl]thiazole		291.7
Thiazole sulfonic acid, TSA, M-3625	5-Chloro-thiazole-2-sulfonic acid		199.6
Butene sulfonic acid, BSA, M-3627	3,4,4-Trifluorobut-3-ene-1-sulfonic acid		190.1
MeS, M-3626	2-Methylsulfonylthiazole		131.2

Methods of analysis

In the residue and storage stability studies provided to the current Meeting for crops except citrus pulp (dry), orange juice, and orange oil, residues of fluensulfone parent compound and its metabolites TSA and BSA were determined using an LC-MS/MS method (method number 1977W) that was evaluated and found acceptable by the 2014 JMPR. That method uses acetonitrile:H_2O extraction and C18 solid-phase extraction (SPE) for clean-up (BSA and TSA only). Modifications to that method were used for processed citrus and sugar cane commodities.

For orange juice, residues were extracted into methanol. For dried citrus pulp, the pulp was hydrated and then residues were extracted into methanol. For both matrices, the methanol extract was analysed directly for residues of BSA and after C18 SPE clean-up for residues of fluensulfone. For oil, residues were extracted into acetonitrile:H_2O and partitioned against hexane and then against ethyl acetate. The aqueous phase was analysed for residues of BSA and the organic phase was analysed for residues of fluensulfone; slightly different protocols were used depending on the matrix. For citrus pulp, orange juice, and orange oil, analyses were for fluensulfone and BSA only.

Blackstrap molasses was extracted with acetonitrile:H_2O, centrifuged, filtered, and diluted prior to analysis for both fluensulfone and BSA. Refined sugar was dissolved in water and then methanol was added. For analysis of BSA, the methanol:H_2O extract was analysed without further clean-up; for analysis of fluensulfone, the extract was cleaned-up by C-18 SPE prior to analysis.

For all matrices, analysis was by LC-MS/MS. Validation data generated concurrently with each residue study demonstrated adequate method performance (recoveries 70–100%, maximum 11% RSD).

Stability of residues in stored analytical samples

The stability of fluensulfone residues in various matrices during frozen storage was evaluated by the 2014 and 2016 Meetings. Data submitted to the 2014 Meeting demonstrated stability in raw tomatoes for at least 15 months, processed tomato commodities for at least 6 months, in capsicum, cucumber and melon for at least 16 months. The 2016 Meeting received data for residues of fluensulfone, including the metabolites BSA and TSA, demonstrating stability during frozen storage (≤ -20 °C) in oranges for at least 18 months, in carrots for at least 17.5 months, in potato tubers for at least 23 months, and in dried potatoes and wet peel for at least 25 months for each of the three analytes.

The 2017 Meeting received storage stability data for peanut matrices including shelled nutmeat, hay, meal, and oil; and sugar cane matrices including billets, tops, and trash. For both crops, storage stability studies were conducted concurrently with supervised residue trials and processing studies.

Residues of both fluensulfone and BSA were stable in peanut matrices for at least 378 days and in sugar cane matrices for at least 55 days. The Meeting noted that although zero-day data from spiked samples were not provided except for peanut hay, the data support the stability determination based on the similarity in residues between the two sample points taken (180 and 378 days for peanut, 20 and 55 days for sugar cane) and on the stability determinations made by the previous Meetings for other matrices.

Results of supervised residue trials on crops

The Meeting received supervised trial data for applications of fluensulfone to citrus, soya beans, sugar cane, coffee, and black pepper. No evidence of registration for use on any of the crops under consideration was provided to the current Meeting; therefore, the Meeting was unable to make estimates of maximum residue levels.

Residues in animal commodities

Of the uses under consideration by the Meeting, citrus, soya bean, and sugar cane have significant livestock feed items. Data were provided for citrus dried pulp, soya bean seed, sugar cane tops, and

sugarcane molasses; data were not provided for soya bean feed items (forage, hay, silage, or meal) or sugar cane bagasse.

As there were no GAPs available for the data considered by the Meeting, the Meeting did not re-evaluate the previous dietary burdens and recommendations from the 2016 Meeting, i.e. that '*maximum residue levels for mammalian commodities should be estimated at the LOQ* [0.01 mg/kg], *with dietary parameters at 0'* and that '*maximum residue levels for eggs, poultry meat and poultry offal should be estimated at the LOQ, with dietary parameters at 0*.'

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