BUPROFEZIN (173)

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EXPLANATION

Buprofezin, insecticide, was evaluated by JMPR in 1991 for the first time and then in 1995 and 1999. It was also reviewed under the Periodic Re-evaluation programme in 2008 for toxicity and residues. The 2008 JMPR allocated an ADI of 0–0.009 mg/kg bw and ARfD of 0.5 mg/kg bw. It concluded that the residue definition for compliance with the MRL and for estimation of dietary intake, both for animal and plant commodities, should be buprofezin and recommended eight maximum residue levels while withdrawing one previous recommendation.

The 2009 JMPR evaluated supervised trial data and recommended 18 MRLs for pome fruits, stone fruits, grapes (fresh and dried), olives, cucurbits, peppers, almonds (nuts and hulls) and foods of mammalian origin.

The current Meeting received information on supervised trials on banana, coffee and tea. The Meeting also received information on method validation and storage stability studies additional to those submitted to the 2008 and 2009 JMPR.

RESIDUE ANALYSIS

Analytical Methods

The 2008 JMPR evaluated a number of analytical methods for enforcement/monitoring and for supervised trials as well as storage stability on residue of buprofezin in foods of plant or animal origin. These are GC-NPD, GC-MS, HPLC-UV, or HPLC-MS-MS methods and can be used for the determination of buprofezin in plant commodities with high water content, high fat content, high acid or low water content, and animal commodities.

The current Meeting received information on the analytical methods used in the supervised residue trial studies on banana, coffee and tea.

Bananas

In the supervised residue trials conducted in the USA (Singer, 1997, R-1072; and Stewart, 2004, R-1160), buprofezin banana samples were analysed by using either the GC/NPD method NOR-AM BF/06/94 or NOR-AM BF/05/94 with minor modifications. These methods were reviewed by the 2008 JMPR to be suitable for the determination of buprofezin, the reverse Schiff base (BF9) and isopropylphenylurea (BF12) in grapes, cucumbers, tomatoes and its processed products, and lettuce.

Residues in homogenised banana samples were extracted with acetone. After rotary evaporation, the extracts were acidified with 1M HCl and partitioned with hexane. The hexane phase containing BF9 was concentrated and then cleaned up using Florisil column. The aqueous phase containing buprofezin and BF12 were neutralized to pH 7 and extracted with 50% (v/v) ethyl acetate/hexane. The organic phase was dried, combined with the Florisil eluate containing BF9, evaporated and re-dissolved in toluene. Buprofezin, BF9 and BF12 were quantified by GC-NPD. Validation of the method for the determination of buprofezin, BF9 and BF12 in banana was conducted at 0.01, 0.10 and 1.0 mg/kg. The reported LOQ was 0.01 mg/kg for each analyte.

Table 1 Recoveries by GC-NPD method NOR-AM BF/06/94 or NOR-AM BF/05/94 for the determination of buprofezin, BF9 and BF12 in banana (R1072, R-1160)

Matrix	Analyte	Fortification level (mg/kg)	Recovery rate (%)		RSD (%)	n
			Individual results	Mean		
Banana, pulp	Buprofezin	0.01	84, 91	88		2
		0.10	103, 113	108		2
		1.0	102, 111	107		2
		0.01–1.0	84–113	101	11	6
	BF9	0.01	77, 79	78		2
		0.10	103, 108	106		2
		1.0	100, 100	100		2
		0.01–1.0	77–108	95	14	6
	BF12	0.01	80, 85	83		2
		0.10	96, 101	99		2
		1.0	92, 96	94		2
		0.01–1.0	80–101	92	8.5	6
Banana, unpeeled	Buprofezin	0.01	81, 92	87		2
		0.10	74, 90	77		2
		1.0	77, 89	83		2
		0.01–1.0	74–92	84	9.0	6
	BF9	0.01	83, 99	91		2
		0.10	76, 89	83		2
		1.0	80, 92	86		2
		0.01–1.0	74–99	87	9.8	6
	BF12	0.01	71, 77	74		2
		0.10	70, 78	74		2
		1.0	75, 82	79		2
		0.01–1.0	70–82	76	6.0	6

In the supervised residue trials conducted in Spain (Chadwick, 2011, R-1398; and Sutherland, 2011, R-1429), buprofezin in banana samples were analysed using Huntingdon Life Sciences Method study number LMS0022 "Validation of methodology for the determination of buprofezin in tomato, whole oranges and olives".

Residues in banana samples were extracted and hydrolysed with a mixture of dioxane and 11 N HCl (5:2, v/v) and cleaned up by liquid-liquid partition followed by ENVI-Carb solid phase extraction (SPE) cartridges. Quantitation was performed using HPLC with tandem mass spectrometric detection (HPLC-MS-MS)(m/z 306 > 201 for buprofezin, m/z 251/106 for BF9 and m/z 179 > 94 for BF12). The method was previously validated at 0.01 and 0.1 mg/kg for buprofezin, BF9 and BF12 in tomatoes, whole oranges and olives. Validation of the method for the determination of buprofezin, BF9 and BF12 in banana pulp and peel was conducted at a number of concentrations in two different studies. The reported LOQ was 0.01 mg/kg for each analyte.

Table 2 Recoveries by HPLC-MS-MS method LMS0022 for the determination of buprofezin, BF9 and BF12 in bananas (R-1398 and R-1429)

Matrix	Analyte	Fortification level (mg/kg)	Recovery rate (%)		RSD (%)	n
			Individual results	Mean		
Banana, pulp	Buprofezin	0.01	73 [a], 82 [b]	78		2
		0.10	63 [a], 73 [b]	68		2
		0.01–0.10	63–82	73	11	4
	BF9	0.01	77 [b], 86 [a]	82		2
		0.10	70 [b], 80 [a]	75		2
		0.01–0.10	70–86	78	8.5	4
	BF12	0.01	71 [b] 81 [a]	76		2
		0.10	58 [b], 81 [a]	70		2
		0.01–0.10	58–81	73	15	4
Banana, Peel	Buprofezin	0.01	77 [b], 87 [b], 89 [a]	84		3
		0.10	66 [b] 91 [a], 99 [b]	85		3
		0.40	74 [b], 77 [b]	76		2

Matrix	Analyte	Fortification level (mg/kg)	Recovery rate (%)		RSD (%)	n
			Individual results	Mean		
		0.75	76 [a], 80 [a]	78		2
		0.01–0.75	66–99	82	12	10
	BF9	0.01	74 [b], 81 [a]	78		2
		0.10	80 [b], 96 [a]	88		2
		0.75	99 [a], 103 [a]	101		2
		0.01–0.75	74–103	89	13	6
	BF12	0.01	74 [b], 81 [a]	78		2
		0.10	76 [b], 93 [a]	85		2
		0.75	86 [a], 98 [a]	92		2
		0.01–0.75	74–98	85	11	6

a R-1398

b R-1429

Coffee

In the supervised residue trials conducted in the USA (Samoil, 2008, R-1219), buprofezin in coffee samples were analysed by the GC-NPD method NOR-AM BF/10/97 with minor modifications.

Residues in ground coffee samples were extracted with acetone. After evaporation, the extract was acidified with 1N HCl and cleaned up by partitioning with hexane. The acidic aqueous phase containing buprofezin was partitioned with methylene chloride. The methylene chloride layer was dried, evaporated to dryness and re-dissolved in hexane. The hexane solution was then cleaned up using Florisil column. Following clean-up, buprofezin was quantified by GC-NPD. The method was validated at 0.05, 0.50 and 5.0 mg/kg fortified to green coffee beans, roasted coffee bean and freeze-dried coffee. The calculated LOQ for buprofezin was 0.033, 0.065 and 0.039 mg/kg in green coffee beans, roasted coffee beans and freeze-dried coffee, respectively.

Table 3 Recoveries by GC-NPD method NOR-AM BF/10/97 for the determination of buprofezin in coffee bean and its processed products (R-1219)

Matrix	Fortification level (mg/kg)	Recovery rate (%)		RSD (%)	n
		Individual results	Mean		
Green coffee bean	0.05	92, 94, 95, 96, 98	95	2.1	5
	0.50	88, 89, 91, 92, 92	90	1.8	5
	5.0	88, 89, 90, 92, 93	90	2.1	5
	0.05–5.0	88–98	92	3.2	15
Roasted coffee bean	0.05	74, 76, 80, 104, 108	88	18	5
	0.50	95, 96, 97, 97, 98	97	1.2	5
	5.0	86, 87, 91, 92, 92	90	3.2	5
	0.05–5.0	74–108	92	11	15
Freeze-dried coffee	0.05	86, 96, 94, 100	94	6.3	4
	0.50	90, 98, 98, 99	96	4.4	4
	5.0	90, 91, 92, 94	92	1.9	4
	0.05–5.0	86–100	94	4.6	12

In the supervised residue trials conducted in Brazil (Carringer, 2011), buprofezin in coffee samples were analysed using a HPLC-MS-MS method, Morse Labs Analytical Method No. Meth-194, originally validated for the determination of buprofezin, BF-09 and BF-12 in wheat raw agricultural commodities.

Residues in ground coffee samples were extracted with acetone. The filtered extracts were combined and an aliquot of the combined extract was purified by means of an ENVI-Carb/LC-NH2 solid phase extraction (SPE) clean-up. The purified SPE eluate was concentrated. Determination and quantification of buprofezin were conducted using HPLC-MS-MS. The method was validated at 0.01 and 0.50 mg/kg fortification. The reported LOQ was 0.01 mg/kg.

Table 4 Recoveries by HPLC-MS-MS method, Morse Labs Analytical Method No. Meth-194 for the determination of buprofezin in coffee beans (R-1430)

Matrix	Fortification level (mg/kg)	Recovery rate (%)		RSD (%)	n
		Individual results	Mean		
Green coffee bean	0.05	72, 77, 78	76	4.2	3
	0.50	79, 82, 82	81	2.1	3
	0.05–0.50	72–82	78	4.8	6

Tea

In the supervised residue trials conducted in Japan in 1981 (Gotoh, 1981, R-1259), buprofezin in crude green tea was quantified by a GC-NPD method as follows.

The residues in pulverized crude green tea samples were extracted with a mixture of distilled water and acetone (1:3, v/v). The concentrated extract was acidified with 0.3 N hydrochloric acid and cleaned up by partitioning with isooctane. After neutralization with 1 N NaOH solution, buprofezin was extracted with isooctane from the aqueous phase. The concentrated isooctane extract was purified by silica gel column chromatography. Buprofezin was quantified by GC-NPD. The method was validated at 0.5 mg/kg in crude green tea. The reported LOD was 0.05 mg/kg.

Table 5 Recoveries by GC-NPD method for the determination of buprofezin in green tea (R-1259)

Matrix	Fortification level (mg/kg)	Recovery rate (%)		RSD (%)	n
		Individual results	Mean		
Crude green tea	0.5	88, 95	92		2

In the supervised residue trials conducted in Japan in 1996 (Komatsu and Yabusaki, 1996a and b), buprofezin in crude green tea was quantified according to the Official Test Guideline for Determination of Buprofezin published by the Japanese Ministry of the Environment (notification date: December 20, 1984) with some modification.

The residues in pulverized crude green tea were extracted with a mixture of distilled water and acetone (1:5, v/v) and cleaned up by macroporous diatomaceous earth cartridge column. Hexane elute from the column was further cleaned up by hexane-acetonitrile partitioning and Florisil column chromatography. Buprofezin was quantified by GC-NPD. The method was validated in two separate studies at 2.0 mg/kg fortification. The reported LOD was 0.05 mg/kg.

Table 6 Recoveries by GC-NPD method (Ministry of the Environment, Japan) for the determination of buprofezin in green tea (R-1325 and R-1338)

Matrix	Fortification level (mg/kg)	Recovery rate (%)		RSD (%)	n
		Individual results	Mean		
Crude green tea	2.0 [a]	81, 87, 89, 96	88	7.0	4
	2.0 [b]	89, 97, 103, 110	100	8.9	4
	2.0	81–110	94	10	8

a R-1325)

b R-1338)

Stability of pesticide residues in stored analytical samples

The current Meeting received information on the storage stability studies on bananas, coffee and tea to determine the stability of buprofezin following frozen storage.

Banana

The storage stability study (Reed, 2006, R-1189) on buprofezin, BF9 and BF12 fortified at 0.01 mg/kg to whole banana was already reviewed and summarized by the 2009 JMPR. These substances were stable up to the longest storing period of 70 days at -20 ± 5 °C.

Coffee

Samples of green coffee beans, roasted coffee beans and freeze-dried coffee were fortified with buprofezin at 0.50 mg/kg (Samoil, 2008, R-1219). At the end of storage at around -20 °C, the stored samples were analysed for buprofezin using GC-NPD method BF/10/97 which was reviewed by the 2008 JMPR.

Buprofezin is shown stable, when frozen at -20 °C, for at least 656 days in green coffee beans, 658 days in roasted coffee beans, and 680 days in freeze-dried coffee.

Table 7 Storage stability of buprofezin in coffee beans or coffee stored at -20 °C (R-1219)

Commodity	Fortification mg/kg	Storage days	% Remaining		Mean concurrent recovery, %
			Individual results	Mean	
Green coffee bean	0.5	0 656	 75, 77, 79	 77	87 84
Roasted coffee bean	0.5	0 658	 70, 73, 74	 72	91 94
Freeze-dried coffee	0.5	0 680	 90, 92, 93	 92	94 90.

Tea

Samples of crude green tea were fortified at 2 mg/kg and stored at -20 °C (Komatsu and Yabusaki, 1996a and 1996b). Samples were stored up to75 days and analysed using the official GC-NPD method of the Japanese Ministry of the Environment with some modification (see above). Buprofezin is shown stable, when frozen at -20 °C, for at least 75 days in crude green tea.

Table 8 Storage stability of buprofezin in crude green tea stored at -20 °C (R-1325, R-1338)

Commodity	Fortification mg/kg	Storage days	% Remaining		Mean concurrent recovery, %
			Individual results	Mean	
Crude green tea [a] (Mie)	2	0 56	 76, 77	 76	88 88
Crude green tea [a] (Kagoshima)	2	0 75	 75, 76	 76	88 88
Crude green tea [b] (Ibaraki)	2	0 50	 94, 95	 94	100 100
Crude green tea [b] (Kouchi)	2	0 75	 85, 93	 89	100 100

a R-1325

b R-1338

USE PATTERN

The Meeting received information on use pattern in Japan, Spain and the USA. Table 9 shows use pattern related to those crops on which supervised trials were conducted and provided to the current Meeting.

Table 9 Registered uses of buprofezin in the USA related to supervised residue trials submitted

Crop	Country	Form g ai/L or kg	Application					PHI days
			Method	Rate kg ai/ha	Rate kg ai/hL	Number	Min, interval days	
Banana	USA	700 WG	Foliar	0.34	0.24	4	14	1
Banana	Spain	250 WP	Foliar		0.01–0.02	–	–	7
Coffee	USA	700 WG	Foliar	1.12	0.24	4	14	0
Tea	Japan	250 WP	Foliar	–	0.025	2	–	14
Tea	Japan	200 SC	Foliar	2.0 [a]	0.020	2	–	14

a Calculated from the approved maximum concentration of 0.020 kg ai/hL and volume of 100 hL/ha.

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

The Meeting received data on supervised field trials of foliar application of for the following crops:

- Table 10 Assorted tropical and sub-tropical fruit-inedible peel—Banana
- Table 11 Seed for beverages and sweets—Coffee
- Table 12 Teas—Tea

All supervised trials were conducted outdoor with foliar applications.

Application rates were reported as buprofezin. Residue concentrations were reported for buprofezin and in some cases for two metabolites: reverse Schiff base (BF9) and isopropylphenylurea (BF12). Unquantifiable residues are shown as < LOQ. Residues below 1 mg/kg, application rates below 1 kg ai/ha and spray concentrations have been rounded to two significant figures. Residue concentrations are recorded unadjusted for recoveries or for residue values in control samples. Where multiple samples were taken from a single plot, individual results are reported, and the calculated average concentration is used for estimation of maximum residue level. Where trials were conducted in the same location, with the same varieties, same or similar formulations, and same equipment, and at the same or similar timing, they are not regarded as independent and only one result from these trials was chosen for the estimation of a maximum residue level.

Residues from the trials conducted according to maximum GAP have been used for the estimation of maximum residue levels and they are underlined.

Banana

The current Meeting received information on supervised field trials on banana conducted in the USA and Canary Islands, Spain as summarized in Table 10.

Four field trials were conducted in Hawaii, USA in 1996 (Singer, 1997) and one in Florida, USA in 2003 (Stewart, 2004) on banana. In three trials in Hawaii and one in Florida, each treated plot received four foliar applications at 0.034 or 0.035 kg ai/ha. In other one trial in Hawaii, the treated plot received first application at 0.84 kg ai/ha followed by three applications at a rate of 0.34 to 0.36 kg ai/ha. The interval of applications was 14 days. In those five trials, banana in one half of the trial site was protected with plastic bags and the other half was not. Samples were collected from both bagged and unbagged bananas. For analysis, one half of each sample was peeled so that residues could be determined in peeled and unpeeled bananas. In Table 10, U-U stands for unpeeled unbagged banana, U-P pulp of unbagged banana, B-U unpeeled bagged banana, and B-P pulp of bagged banana. No detectable residues were found in any of bagged banana samples or in banana pulp samples (whether bagged or unbagged). From each treated lot duplicate samples were taken and analysed. All

the samples were stored frozen less than 37 days. In one trial conducted on banana in Florida, USA in 2003, samples were stored frozen for 70 days.

Two field trials were conducted on banana in Spain in 2009 (Chadwick, 2011) and other two in 2010 (Sutherland, 2011). In these trials, each treated plot received one application at a spray concentration of 0.025–0.04 kg ai/hL (GAP in Spain specifies 0.01–0.02 kg ai/hL) resulting in 0.45–0.63 kg ai/ha. Residues on banana peels and pulps were determined, separately. A weight ratio of peel versus whole fruit was used to calculate the potential concentrations in whole banana. The dissipation of buprofezin residues on foliage or fruit is rapid, as demonstrated by the data from the USA where four applications at 14 day intervals were used were not so much different from data from Spain where only one application was used. It indicates that the residue level for buprofezin in crop commodities essentially depends on the dose rate of the last application, and previous applications made with a 7 or 14 day interval have little or no impact.

The LOQ for buprofezin in banana was 0.01 mg/kg. Buprofezin analysis of control samples all resulted in < 0.01 mg/kg.

In Table 10 the residue concentrations used for estimating a maximum level are underlined and those used for estimating an STMR are double underlined.

Table 10 Residues of buprofezin in banana from supervised trials conducted in the USA and Spain.

BANANA	Application					PHI, days	Portion analysed	Residues, mg/kg			Ref.
location year (variety)	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.			Buprofezin	BF9	BF12	(Trial no)
Trials in the USA											
US GAP	WG	0.34	0.24		4	1					
Corozal	WP	0.84	0.50	167	4	1	Whole banana				R-1072
Puerto Rico		0.34	0.17	200			U-U	< 0.01	< 0.01	< 0.01	
1996		0.36	0.20	176				0.020	< 0.01	< 0.01	(AA960316.01)
(Cavendish)		0.35	0.23	152				(0.02) [a]			
							U-P	< 0.01	< 0.01	< 0.01	
								≤ 0.01	< 0.01	< 0.01	
							B-U	< 0.01	< 0.01	< 0.01	
								< 0.01	< 0.01	< 0.01	
							B-P	< 0.01	< 0.01	< 0.01	
								< 0.01	< 0.01	< 0.01	
Kea'au [b]	WP	0.35	0.57	61	4	1	Whole banana				R-1072
Hawaii, HI		0.35	0.19	182			U-U	0.038	< 0.01	< 0.01	(AA960316.02)
USA		0.35	0.19	182				0.044	< 0.01	< 0.01	
1996		0.35	0.19	182				(0.04)			
(Cavendish)							U-P	< 0.01	< 0.01	< 0.01	
								≤ 0.01	< 0.01	< 0.01	
							B-U	< 0.01	< 0.01	< 0.01	
								< 0.01	< 0.01	< 0.01	
							B-P	< 0.01	< 0.01	< 0.01	
								< 0.01	< 0.01	< 0.01	
Kea'au [b]	WP	0.35	0.57	61	4	1	Whole banana				R-1072
Hawaii, HI		0.35	0.19	182			U-U	0.055	< 0.01	< 0.01	(AA960316.03)
1996		0.35	0.19	182				0.077	< 0.01	< 0.01	
(Cavendish)		0.35	0.19	182				(0.07)			
							U-P	< 0.01	< 0.01	< 0.01	
								≤ 0.01	< 0.01	< 0.01	
							B-U	< 0.01	< 0.01	< 0.01	
								< 0.01	< 0.01	< 0.01	
							B-P	< 0.01	< 0.01	< 0.01	
								< 0.01	< 0.01	< 0.01	
						3	U-U	0.03	< 0.01	< 0.01	
								0.03	< 0.01	< 0.01	
						7	U-U	0.02	< 0.01	< 0.01	
								0.12	< 0.01	< 0.01	
						14	U-U	0.03	< 0.01	< 0.01	
								0.04	< 0.01	< 0.01	

BANANA location year (variety)	Application Form	kg ai/ha	kg ai/hL	Water, L/ha	no.	PHI, days	Portion analysed	Residues, mg/kg Buprofezin	BF9	BF12	Ref. (Trial no)
Mt. View Hawaii, HI 1996 (Cavendish)	WP	0.35 0.35 0.35 0.35	0.57 0.19 0.19 0.19	61 182 182 182	4	1	Whole banana U-U U-P B-U B-P	 0.054 0.063 (0.06) < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	R-1072 (AA960316.04)
Kurtistown Hawaii, HI 1996 (Cavendish)	WP	0.35 0.35 0.35 0.35	0.60 0.20 0.20 0.20	58 174 174 174	4	1	Whole banana U-U U-P B-U B-P	 0.059 0.038 (0.05) < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	R-1072 (AA960316.05)
Sarasota, FL 2003 (Namwa)	WP	0.34 0.34 0.33 0.33	0.04 0.04 0.04 0.04	891 884 878 869	4	1	Whole banana (unbagged)	0.172 0.177 (0.18)	< 0.01 < 0.01	< 0.01 < 0.01	R-1160
Trials in Spain											
GAP in Spain	WP	–	0.01– 0.02			7					
San Andres y Sauces 2009 (Del Pais)	SC	0.525	0.03	1662	1	0 7	Peel Pulp Peel Pulp Whole banana [c]	0.33 < 0.01 0.22 < 0.01 0.10	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	R-1398
Villa de Mazo, Canarias 2009 (Pequeña enana)	SC	0.543	0.04	1375	1	0 1 3 5 7	Peel Pulp Peel Pulp Whole banana [c] Peel Pulp Peel Pulp Peel Pulp Whole banana [c]	0.34 < 0.01 0.34 < 0.01 0.15 0.19 < 0.01 0.17 < 0.01 0.24 < 0.01 0.10	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	R-1398
Puntallana, Tenerife 2010 (Pequeña enana)	SC	0.450	0.025	1800	1	0 7	Peel Pulp Peel Pulp Whole banana [c]	0.58 < 0.01 0.76 < 0.01 0.32	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	R-1429

BANANA	Application					PHI, days	Portion analysed	Residues, mg/kg			Ref.
location year (variety)	Form	kg ai/ha	kg ai/hL	Water, L/ha	no.			Buprofezin	BF9	BF12	(Trial no)
Villa de Mazo, Canarias 2010 (Del Pais)	SC	0.631	0.033	1893	1	0	Peel	0.64	< 0.01	< 0.01	R-1429
							Pulp	< 0.01	< 0.01	< 0.01	
						1	Peel	0.75	< 0.01	< 0.01	
							Pulp	< 0.01	< 0.01	< 0.01	
							Whole banana [c]	0.31			
						3		0.56	< 0.01	< 0.01	
							Peel	< 0.01	< 0.01	< 0.01	
							Pulp				
						5		0.57	< 0.01	< 0.01	
							Peel	< 0.01	< 0.01	< 0.01	
							Pulp				
						7		0.43	< 0.01	< 0.01	
							Peel	< 0.01	< 0.01	< 0.01	
							Pulp	0.18			
							Whole banana [c]				

a Average of analytical results of duplicate samples in parentheses.

b These trials were conducted in different sites.

c Calculated residue levels of whole fruit using the weight of peel and pulp (on average 41% and 56% respectively of whole fruit from 2009 trials). In the Spanish trials banana fruits were not bagged.

Coffee

The Meeting received information on supervised residue trials conducted in the USA and Brazil on coffee as summarized in Table 11.

A total of four independent and valid supervised field trials on coffee were conducted in the USA in 2004 (Samoil, 2008). Each treated plot received four foliar applications of the WP formulation at a rate of 1.12–1.23 kg ai/ha. The treatment interval was 14 days. The time from sampling to analysis was up to 656 days. Analyses were performed using a method similar to AgroEvo Method No. BF/06/94 (GC-NPD method). The LOQ of the method was 0.01 mg/kg for green coffee beans. The recovery percentage ranged from 70 to 90%. The residues in control plots were all below the LOQ.

Three additional trials on coffee were conducted in Brazil in 2011 (Carringer, 2011). In these trials, each treated plot received four foliar applications of the WP formulation at a rate of 1.12–1.15 kg ai/ha similar to the maximum US GAP rate. Buprofezin was analysed using a HPLC-MS-MS method with an LOQ of 0.01 mg/kg. The residues in control plots were all below the LOQ.

Table 11 Buprofezin residues in coffee from supervised trials in the USA and Brazil

COFFEE	Form	Application				PHI, days	Residues, mg/kg	Reference
Location, year (variety)		kg ai/ha	kg ai/hL	Water, L/ha	No.		Buprofezin	Trial no
Trials in the USA								
US GAP (max)		1.12			4	0		
Eleele, Kauai, HI, 2004 (Yellow Caturra)	WP	1.17 1.17 1.13 1.13	0.12 0.12 0.12 0.12	973 963 945 935	4	0	0.24	IR-4 08828 HI-04
		1.14	0.40	281	4	0	0.14, 0.08	HI-05
Kealakekua, Hawaii, HI, 2004 (Guatemalan Kona typical) [a]	WP	1.12 1.14 1.18 1.14 1.13	0.24 0.24 0.24 0.24 0.24	468 477 486 477 468	5 [b]	0	0.06, 0.10 (0.08) [c]	IR-4 08828 HI-06
Kealakekua,	WP	1.23	0.10	1235	4	0	0.12, 0.12	IR-4 08828

COFFEE Location, year (variety)	Form	Application kg ai/ha	kg ai/hL	Water, L/ha	No.	PHI, days	Residues, mg/kg Buprofezin	Reference Trial no
Hawaii, HI, 2004 (Guatemalan Kona typical) [a]		1.12 1.13 1.14	0.10 0.10 0.10	1122 1132 1150			(0.12)	HI-07
Lahaina, Maui, HI, 2004 (Red Catuai)	WP	1.13 1.12 1.13 1.13	0.080 0.079 0.080 0.080	1422 1412 1412 1422	4	0	0.16, 0.15 (0.155)	IR-4 08828 HI-08
Trials in Brazil								
Brazil (Arceburgo, MG) 2011 (Coffee/ Catuai Amarelo)	WP	1.13 1.14 1.15 1.12	0.25 0.24 0.24 0.25	454 481 473 452	4	0	0.06, 0.05 (0.055)	R-1430
Brazil (Dois Córregos, SP) 2011 (Coffee/ Catuai Vermelho)	WP	1.13 1.13 1.14 1.12	0.25 0.25 0.26 0.25	456 445 449 453	4	0	0.07, 0.08 (0.075)	R-1430
Brazil (Rolândia, PR) 2011 (Coffee/ Mundo Novo)	WP	1.13 1.12 1.14 1.12	0.13 0.12 0.13 0.13	906 905 896 898	4	0	0.09, 0.06 (0.075)	R-1430

a These trials were conducted in two different farms.

b Fifth application was made 8 days after the fourth application.

c Average of analytical results of duplicate samples in parentheses.

Tea

Six field trials were conducted on tea in Japan during 1981 (Gotoh, 1981) and 1996 (Komatsu and Yabusaki, 1996a and 1996b) following Japanese GAP. In these trials, each treated plot received two foliar applications at a spray concentration of 0.025 kg ai/hL (WP) or 0.02 kg ai/hL (SC). Only the dilution rate of formulated product is determined in Japanese GAP (spray volume depends on the tree height) so that the application in the trial at Mie was regarded by the Japanese Government as appropriate.

Table12 Residues of buprofezin in crude green tea from supervised trials in Japan

Location year (variety)	Form	Application kg ai/ha	kg ai/hL	Water L/ha	no.	PHI, days	Residues, mg/kg Buprofezin	Ref.
GAP, Japan	250 WP		0.025		2	14		
GAP, Japan	200 SC		0.020		2	14		
Kagoshima 1981 (Sayamamidori)	WP	2.5	0.025	10000	2	0 7 14 21	0.15 0.15 48.1 51.1 10.2 9.47 (9.84) [a] 2.08 1.93	R-1259

Location year (variety)	Form	Application				PHI, days	Residues, mg/kg	Ref.
		kg ai/ha	kg ai/hL	Water L/ha	no.		Buprofezin	
Nagasaki 1981 (Yabukita)	WP	2.5	0.025	10000	2	0 7 14 21	0.08 0.07 51.8 49.1 7.31 6.95 (7.13) 0.83 0.74	R-1259
Mie 1996 (Okumidori)	WP	0.5	0.025	2000	2	0 7 14 21 28	< 0.05 < 0.05 73.6 67.9 12.4 12.3 (12.4) 3.02 2.91 0.77 0.76	R-1325
Kagoshima 1996 (Yabukita)	WP	2.5	0.025	10000	2	0 7 14 21 28	< 0.05 < 0.05 46.6 44.7 8.56 7.91 (8.24) 3.13 2.82 0.95 0.85	R-1325
Ibaraki 1996 (Yabukita)	SC	1.0	0.02	5000	2	0 7 14 21 28	0.10 0.09 55.4 54.5 7.04 6.76 (6.90) 2.53 2.26 0.95 0.85	R-1338

Location year (variety)	Form	Application				PHI, days	Residues, mg/kg	Ref.
		kg ai/ha	kg ai/hL	Water L/ha	no.		Buprofezin	
Kouchi 1996 (Yabukita)	WP	2.0	0.02	10000	2	0 7 14 21 28	< 0.05 < 0.05 46.2 44.7 11.0 10.5 (10.8) 1.36 1.24 0.45 0.40	R-1338

a Average of analytical results of duplicate samples in parentheses.

FATE OF RESIDUES IN STORAGE AND PROCESSING

In processing

Information on the processing of green coffee beans to roasted coffee and freeze-dried coffee was received and reviewed by the 2009 JMPR. Calculated processing factors are transcribed below.

Table 13 Summary of calculated processing factors

Commodity	Calculated processing factor	Processing factor (median or best estimate)
Roasted coffee	0.32	0.32
Freeze-dried coffee	< 0.2	< 0.2

Residues in animal commodities

As none of banana, coffee, tea or their by-products is included in the OECD animal feed table, the Meeting concluded that there is no need to calculate animal dietary burden for this evaluation.

APPRAISAL

Buprofezin, insecticide, was evaluated by JMPR in 1991 for the first time and then in 1995 and 1999. It was also reviewed under the Periodic Re-evaluation Programme in 2008 for toxicity and residues. The 2008 JMPR allocated an ADI of 0–0.009 mg/kg bw and ARfD of 0.5 mg/kg bw. It concluded that the residue definition for compliance with the MRL and for estimation of dietary intake, both for animal and plant commodities, should be buprofezin. Buprofezin was further evaluated for additional maximum residue levels in 2009.

At the Forty-third Session, the CCPR included buprofezin in the Priority List for review by the current JMPR for additional MRLs.

The current Meeting received information on supervised trials on banana, coffee and tea. The Meeting also received information on method validation and storage stability studies additional to those submitted to the 2008 and 2009 JMPR.

Methods of analysis

The Meeting received information on validation of analytical methods used in the supervised field trial studies for determination of buprofezin in banana, coffee bean or crude green tea.

A number of CG-NPD methods and HPLC-MS-MS methods were validated for determination of buprofezin in banana (pulp and peel), coffee or green tea. Mean recoveries were within the acceptable range of 70–110% with RSDs less than 20%. The reported LOQ was 0.01 mg/kg for the methods used for determination of buprofezin in banana pulp and peel and coffee. For the two GC-NPD methods for determining buprofezin in green tea, reported limit of detection was 0.01–0.05 mg/kg.

Stability of residues in stored analytical samples

The current Meeting received information on the storage stability studies on banana, coffee and tea to determine the stability of buprofezin following frozen storage.

Buprofezin was demonstrated to be stable when stored frozen at -20 °C for at least the longest storing periods in studies: 70 days in whole banana (0.01 mg/kg), 656 days in green coffee beans (0.50 mg/kg), 658 days in roasted coffee beans (0.50 mg/kg) and 680 days in freeze-dried coffee (0.50 mg/kg) and 75 days in crude green tea (2 mg/kg). The storage duration of samples in supervised trials was within the above mentioned period for each commodity.

Results of supervised residue trials on crops

The Meeting received supervised trial data for buprofezin on banana, coffee and tea.

The OECD MRL calculator was used as a tool to assist in the estimation of maximum residue levels from the selected residue data set obtained from the supervised residue trials. As a first step, the Meeting reviewed trial conditions and other relevant factors related to each data set to arrive at a best estimate of the maximum residue level using expert judgement. Then, the OECD calculator was employed. If the statistical calculation spreadsheet suggested a different value, a brief explanation of the derivation was supplied.

Banana

Six supervised trials were conducted in the USA: one in Puerto Rico and four in Hawaii in 1996 and one in Florida in 2003. Four applications were made at around 0.34 kg ai/ha with an exception that in the trial in Puerto Rico the first application rate was 0.84 kg ai/ha. The registered use on banana in the USA allows the maximum of 4 foliar spray applications at the maximum rate of 0.34 kg ai/ha with PHI of 1 day. The maximum spray concentration is 0.24 kg ai/hL.

The Meeting decided that as about three times higher rate applied at the first application (42 days before the last application) in one trial did not seem to contribute significantly to residues in fruits at harvest, it was appropriate to use the residue data from this trial.

In five trials banana fruit was either bagged or unbagged and one half of each sample of bagged or unbagged banana was peeled and analysed with the rest unpeeled and analysed. No residues were found in pulp portion of both bagged and unbagged banana, or in bagged whole banana fruit. Therefore, the Meeting decided to use data set from unbagged banana. Residues in unbagged whole banana from trials conducted in USA following US GAP were in rank order: 0.02, 0.04, 0.05, 0.06, 0.07, and 0.18 mg/kg.

Corresponding residues in pulp were in rank order: < 0.01 mg/kg (5). In the trial in Florida, only whole fruits were analysed.

Additionally four trials were conducted in Spain: two in 2009 and two other in 2010. The registered use on banana in Spain allows the maximum spray concentration of 0.01–0.02 kg ai/hL with PHI of 7 days. The spray concentrations in trials were 0.025–0.04 kg ai/hL.

Residues in unbagged whole banana from trials conducted in Spain following Spanish GAP were: 0.32 mg/kg.

The GAP of the USA and that of Spain are significantly different and the data from trials in Spain were not sufficient for estimating a maximum residue level, the Meeting decided to use the results of S trials as a basis of estimating a maximum residue level, STMR and HR.

The Meeting estimated a maximum residue level of 0.3 mg/kg, an STMR of 0.01 mg/kg and HR of 0.01 mg/kg on a basis of US trials.

Coffee bean

Supervised trials were conducted on coffee in Hawaii in the USA in 2004 with four applications at 1.12–1.23 kg ai/ha. The 2009 JMPR reviewed these data and concluded that data were insufficient to recommend a maximum residue level.

Residues of buprofezin in green coffee beans from trials in the USA conducted following US GAP for coffee (1.12 kg ai/ha × 4, PHI 0 day) were re-evaluated by the current Meeting. These were in rank order: 0.08, 0.12, 0.16 and 0.24 mg/kg.

Additionally three trials were conducted in Brazil in 2011 with four applications at 1.12–1.14 kg ai/ha. These trials were in accordance with US GAP.

As the Meeting does not have sufficient information on normal agricultural practices in coffee cultivation in Brazil or the USA to determine their similarity, it concluded that it was not possible to estimate a maximum residue level for coffee bean.

Green tea

Supervised trials were conducted on tea in five Prefectures in Japan in 1981 and 1996 with two foliar applications at the spray concentration rate of 0.02 (SC) or 0.025(WP) kg ai/hL. The registered use in Japan allows maximum of two applications at the maximum spray concentration of 0.020 (in case of SC) or 0.025 (in case of WP) kg ai/hL. No maximum rate per ha is specified.

Residues of buprofezin in crude (unblended) green tea from trials in accordance with GAP in Japan were in rank order: 6.9, 7.1, 8.2, 9.8, 11 and 12 mg/kg.

The Meeting estimated a maximum residue level of 30 mg/kg and STMR of 9.0 mg/kg for tea, green.

As the processing of green tea is significantly different from that of black tea, the Meeting concluded that the estimated maximum residue level should be applicable only to green tea.

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.

Plant commodities and animal commodities:

Definition of the residue for compliance with MRLs and for estimation of dietary intake: *buprofezin.*

Commodity		Recommended MRL, mg/kg		STMR/STMR-P mg/kg	HR/HR-P mg/kg
CCN	Name	New	Previous		
FI 0327	Banana	0.3	-	0.01	0.01
DT	Tea, Green	30	-	9.0	-

DIETARY RISK ASSESSMENT

Long-term intake

The International Estimated Daily Intakes (IEDIs) of buprofezin were calculated for the 13 GEMS/Food cluster diets using STMRs and STMRPs estimated by the 2004, 2006, 2010 and current Meetings (Annex 3 of the 2012 JMPR Report). The ADI is 0–0.009 mg/kg bw and the calculated

IEDIs were 2–50% of the maximum ADI. The Meeting concluded that the long-term intake of residues of buprofezin resulting from the uses considered by the 2008, 2009 and current JMPR is unlikely to present a public health concern.

Short-term intake

The International Estimated Short-Term Intakes (IESTI) of buprofezin were calculated for food commodities and their processed commodities using HRs/HR-Ps or STMRs/STMR-Ps estimated by the current Meeting (see Annex 4 of the 2012 JMPR Report). The ARfD is 0.5 mg/kg and the calculated IESTIs were 0–7 % of the ARfD. The Meeting concluded that the short-term intake of residues of buprofezin, when used in ways that have been considered by the JMPR, is unlikely to present a public health concern.

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