TRIFLOXYSTROBIN (213)

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EXPLANATION

Trifloxystrobin (methyl (*E*)-methoxyimino-{(*E*)-α-[1-(α,α,α-trifluoro-meta-tolyl)ethylidene-aminooxy]-ortho-tolyl}acetate) was first evaluated for toxicology and residues by the JMPR in 2004. The Meeting derived an ADI of 0–0.04 mg/kg bw, decided that an ARfD is unnecessary and concluded that the residue definition for plant commodities for compliance with MRL values was trifloxystrobin *per se* and for animal commodities compliance with MRL values as well as for consumer risk assessment was the sum of trifloxystrobin and (*E,E*)-methoxyimino-{2-[1-(3-trifluoromethyl-phenyl) ethylidene-aminooxymethyl]-phenyl}acetic acid = CGA321113) (expressed as trifloxystrobin equivalents). The residue is fat soluble.

Trifloxystrobin was scheduled at the 48th session of the CCPR for the evaluation of additional MRLs for head cabbage, cauliflower, broccoli, spinach, cotton and ginseng by the 2017 JMPR.

RESIDUE ANALYSIS

Analytical methods

The method used for the analysis of head cabbage, cauliflower, broccoli, spinach and cotton samples was method 200177, which was evaluated by the 2004, and 2015 JMPR.

The Meeting received summarized information on an analytical method for trifloxystrobin and CGA321113 residues in ginseng. The analytical method used to analyse for trifloxystrobin involves homogenisation of a fresh ginseng and acetone mixture (with dry ice), filtering to remove solids. In the case of dried ginseng and red dried ginseng samples, distilled water was added to wet the samples prior to addition of acetonitrile, homogenisation and filtering to remove solids. Acetonitrile:distilled water:saturated lead acetate (5:2:1 v/v/v) are added to an aliquot of the dried ginseng and red ginseng extracts, the mixture shaken for 30 minutes prior to filtration to remove solids. The filter cake is washed with acetonitrile and the rinsate and filtrate combined.

The extracts obtained for fresh, dried and red ginseng are partitioned with distilled water:saturated NaCl:dichloromethane (6:2:1 v/v/v). The organic layer is separated and dried over anhydrous sodium sulphate and the solvent removed (35 °C, rotary evaporator). The residue is dissolved in n-hexane for clean-up on a Florsil column pre-washed with n-hexane followed by *n*-hexane:dichloromethane:acetonitrile (49.65:50:0.35 v/v/v) and the trifloxystrobin eluted with *n*-hexane:dichloromethane:acetonitrile (45:50:5 v/v/v) and the collected fraction concentrated to dryness (35 °C rotary evaporator) with the resulting residue dissolved in acetone for quantitation by GC-NPD.

Analysis of samples for CGA321113 involves homogenisation of a fresh ginseng, 0.1% H_3PO_4, and acetone mixture, filtering to remove solids. In the case of dried ginseng and red dried ginseng samples, distilled water was added to wet the samples and the mixture allowed to sit for one hour prior to addition of 0.1% H_3PO_4 and acetone, homogenisation and filtering to remove solids.

The extracts obtained for fresh, dried and red ginseng are partitioned with distilled water:saturated NaCl:*n*-hexane (3:1:1 v/v/v). The organic layer is discarded and the aqueous layer shaken with dichloromethane, which is separated, dried over anhydrous sodium sulphate and evaporated to dryness (35 °C, rotary evaporator). The residue is dissolved in n-hexane for clean-up on a Florsil column pre-washed with *n*-hexane followed by *n*-hexane:ethyl acetate (80:20 v/v) and the CGA321113 eluted with acetonitrile:formic acid (100:0.5 v/v) and the collected fraction concentrated to just dryness (35 °C rotary evaporator) with the resulting residue dissolved in acetone for quantitation by GC-NPD.

The detector response was linear for fresh ginseng, dried and red ginseng and water extract of dried ginseng over the concentration range 0.1-6 mg/kg for trifloxystrobin ($r^2 \geq 0.9987$, n=7) and 0.05–5 mg/kg for CGA321113 ($r^2 \geq 0.9991$, n=7). No interferences were reported.

Mean recoveries were within the range 70–120% for all commodities investigated and supported LOQs of 0.03 to 0.06 mg/kg for ginseng and 0.07 mg/kg for ginseng processed commodities. The method is suitable for the analysis of residues in ginseng and ginseng processed commodities.

Table 1 Summary of concurrent recovery data from trials conducted in 2013 and 2014 (Kyung 2014)

Commodity	trifloxystrobin Spike (mg/kg)	N	Mean recovery (%)	RSD (%)	CGA321113 Spike (mg/kg)	N	Mean recovery (%)	RSD (%)
Fresh ginseng	0.03	5	87	5.9	0.03	5	76	5.7
	0.06	5	88	3.9	0.1	5	75	3.7
	0.1	5	94	2.7	0.3	5	75	4.0
	0.3	5	86	3.0				
	0.6	5	92	2.2				
Dried ginseng	0.07	5	76	5.6	0.07	5	84	9.6
	0.13	5	107	6.7	0.2	5	76	4.7
	0.2	5	90	8.1	0.7	5	79	5.1
	0.7	5	88	4.0				
	1.3	5	85	6.4				
Red ginseng	0.07	5	86	5.5	0.07	5	82	6.1
	0.13	5	82	1.5	0.2	5	81	7.6
	0.2	5	80	5.6	0.7	5		
	0.7	5	88	4.1				
	1.3	5	90	1.8			81	8.0
Water extract of dried ginseng	0.07	5	100	15	0.07	5	88	5.4
	0.2	5	92	12	0.2	5	85	7.7
	0.7	5	90	17	0.7	5	73	5.5
Water extract of red ginseng	0.07	5	90	7.5	0.07	5	93	7.9
	0.2	5	83	6.8	0.2	5	75	4.1
	0.7	5	81	6.5	0.7	5	75	5.2

Stability of residues in stored analytical samples

The stability of residues in samples on frozen storage was evaluated by the 2004 JMPR for a range of commodities. Residues of trifloxystrobin and CGA321113 were stable under freezer storage conditions for at least 24 months in the case of grapefruit, cucumber, potato and wheat commodities (grain, straw and whole plant) or 18 months for apple (fruit, wet pomace), peanut (nutmeat, oil) and grape juice. The stability data covered commodities that are representative of high water content (apple, cucumber, grape juice), high acid content (grapefruit), high oil content (peanut) and high protein/starch content (potato, wheat) and can be extrapolated to the commodities considered at the current Meeting.

Maximum storage to analysis intervals were: 11.6 months for broccoli, 11.2 months for cauliflower, 10.8 months for cabbage, 11.3 months for spinach, 15 months for cottonseed, 10 months for gin by-products and 9.9 months for ginseng. The periods of demonstrated stability cover the frozen storage intervals used in the residue studies on crops.

Additionally information on spiked samples stored at for the same intervals as samples from residue trials was made available for ginseng. Samples of control ginseng were fortified with trifloxystrobin or CGA321113 at 0.03 to 1.3 mg/kg prior to freezing of the homogenised fresh, dried, red and water extracts. Residues in stored samples were 70–91% (five replicates per sample) of the nominal fortification level at the time of analysis, which occurred after 21 to 296 days of frozen storage.

USE PATTERN

The information available to the 2017 JMPR on registered uses of trifloxystrobin relevant to the residue trial data made available is summarised in Table 2.

Table 2 Use of trifloxystrobin

Crop	Country	No (int)	Growth stage	Application rate (g ai/ha)	Spray concentration (g ai/hL)	Spray volume (L/ha)	PHI (days)
Brassica leafy greens	USA	2-3 (5-14)		92-139 g ai/ha, max 281 g ai/ha/yr		- >18.7✈	0
Brassica, head and stem	USA	2-3 (7-14)		92-139 g ai/ha, max 281 g ai/ha/yr		- >18.7✈	0
Cotton (foliar)	USA	3 (14)		Foliar: 137 g ai/ha, max 411 g ai/ha/y		>93.5 grd >18.7✈	30
Cotton (in-furrow and banded)	USA	1		In-furrow or banded at planting: 137 g ai/ha or 0.835 g ai/100 m if row spacing 0.91 m		>93.5 grd	(30)
Ginseng	Republic of Korea	3 (10)	Flowering, Fruiting, Mature	(67)	3.35	2000 grd	21
Ginseng	USA	2 (14)		139 g ai/ha, max 281 g ai/ha/y		- >18.7✈	7
Leaf petioles	USA	2-4 (14)		73-106 g ai/ha, max 281 or 420 g ai/ha/y for all trifloxystrobin sources g ai/ha/year		- >18.7✈	7
Leafy greens	USA	2 (5-14)		139 g ai/ha, max 281 g ai/ha/y or 420 g ai/ha/y for all trifloxystrobin sources		- >18.7✈	0 broadcast Lettuce 0 broadcast, 20 d band

grd = application using ground based equipment

✈ = application by aircraft

RESIDUES STUDIES

The Meeting received information on supervised field trials on the following crops or crop groups:

Commodity	Table
Brassicas (broccoli, cauliflower, cabbage)	3
Spinach	4
Cotton	5
Ginseng	6

Residue values from the trials conducted according to maximum GAP have been used for the estimation of maximum residue levels. Those results included in the evaluation are underlined.

The sum of trifloxystrobin and CGA321113 was calculated and expressed as trifloxystrobin on the basis of the relative molecular masses. A conversion factor of 1.036 is required to express CGA321113 as trifloxystrobin. As CGA321113 does not generally constitute a significant proportion of the residue in crops, when the levels of trifloxystrobin or CGA321113 were below the LOQ, their sum was calculated as in the examples provided by the 2004 JMPR and copied below.

Trifloxystrobin (mg/kg)	CGA321113 (mg/kg)	Total (expressed as trifloxystrobin) (mg/kg)
< 0.02	< 0.02	< 0.02
< 0.02	0.03	0.05
0.10	< 0.02	0.10
0.92	0.16	1.1

Brassica vegetables

Table 3 Residues of trifloxystrobin and CGA321113 (mg/kg) in brassicas (Fischer and Harbin 2008 M-307503-01-1) (mean of duplicate samples) following applications of trifloxystrobin in an SC-formulation

Location, year, variety BRASSICAS	N (int)	Rate (g ai/ha)	Spray volume (L/ha)	GS BBCH	DALA	Sample	Trifloxystrobin (mg/kg)	CGA321113 (mg/kg)	Total (mg/kg)
Broccoli									
Raymondville TX USA 2007 Green Magic	2 (5)	148 143	186 180	49 49	0	Heads+stalks	0.68	0.02	0.70
Guadalupe CA USA 2007 Heritage	2 (5)	134 141	122 137	48 49	0	Heads+stalks	0.67	< 0.01	0.67
Sanger CA USA 2007 Marathon	2 (5)	141 143	136 163	49 49	0 1 3 7 10	Heads+stalks Heads+stalks Heads+stalks Heads+stalks Heads+stalks	0.44 0.37 0.30 0.19 0.16	0.02 0.03 0.03 0.03 0.04	0.46 0.40 0.33 0.22 0.20
Cauliflower									
King City CA USA 2007 Solvang	2 (6)	140 141	140 141	47 49	0	Heads+stalks	< 0.01	< 0.01	< 0.01
Sanger CA USA 2007 Shasta	2 (5)	142 150	136 170	46 47	0	Heads+stalks	0.32	0.03	0.36
Corvallis CA USA 2007 Minuteman [a]	2 (5)	147 140	148 141	48 49	0	Heads+stalks	0.01	< 0.01	0.01
Cabbage									
Germansville PA 2007 USA Blue Lagoon [b]	2 (5)	142 146	182 185	46 49	0 0 1 3 7 10	H+WL H-WL H+WL H+WL H+WL H+WL	0.37 0.01 0.33 0.16 0.05 0.10	0.02 < 0.01 0.02 0.02 0.02 0.02	0.39 0.01 0.34 0.18 0.07 0.12
Athens GA USA 2007 Bonnies Best hybrid	2 (5)	142 139	98 92	48 49	0	H+WL H-WL	0.47 0.01	0.01 < 0.01	0.48 0.01
Belle Glade FL 2007 USA Emblem	2 (5)	142 141	177 188	45 49	0	H+WL H-WL	0.58 0.03	0.01 < 0.01	0.60 0.03
Richland IA USA 2007 Stonehead [c]	2 (5)	142 142	155 155	49 49	0	H+WL H-WL	0.47 < 0.01	0.01 < 0.01	0.49 < 0.01
Uvalde TX USA 2007 Blue Thunder [d]	2 (5)	140 140	184 180	49 49	0	H+WL H-WL	0.11 < 0.01	< 0.01 < 0.01	0.11 < 0.01
Corning CA USA 2007 Copenhagen	2 (5)	139 142	187 187	48 49	0	H+WL H-WL	0.03 0.01	< 0.01 < 0.01	0.03 0.01

H+WL = heads with wrapper leaves

H-WL = heads without wrapper leaves

[a] Corvallis cauliflower: 18 mm rain within 24 hours of spray two

[b] Germansville cabbage: 21 mm rain within 24 hours of spray one

[c] Richland cabbage: 33 mm rain within 24 hours of spray one

[d] Uvalde cabbage: 0.3 mm rain within 24 hours of spray two

Spinach

Table 4 Residues of trifloxystrobin and CGA321113 (mg/kg) in spinach (Fischer and Harbin 2010 M-307507-02-1) (mean of duplicate samples) following applications of trifloxystrobin in an SC-formulation

Location, year, variety SPINACH	N (int)	Rate (g ai/ha)	Spray volume (L/ha)	GS BBCH	DALA	Sample	Trifloxystrobin (mg/kg)	CGA321113 (mg/kg)	Total (mg/kg)
Germansville PA USA 2007 Tyee	2 (5)	142 147	183 183	45 47	0	Leaves	7.6	0.06	7.6
Suffolk VA USA 2007 Tyee	2 (5)	142 142	123 120	45 45	0	Leaves	10	0.05	10
Hinton OK USA 2007 Bloomsdale	2 (7)	145 143	124 121	31 37	0	Leaves	9.5	0.05	9.6
Jerome ID USA 2007 Unipack 151	2 (6)	142 139	174 170	45 47	0	Leaves	4.8	0.02	4.8
King City CA USA 2007 Interceptor/Avenger	2 (5)	140 141	140 135	45 45	0 1 3 7 10	Leaves	5.3 4.8 4.2 1.3 0.60	0.03 0.03 0.02 0.02 < 0.01	5.4 4.8 4.3 1.3 0.60
Fresno CA USA 2007 Shasta	2 (5)	139 139	117 117	43 45	0	Leaves	7.6	0.04	7.6

Oilseed

Cotton seed

Table 5 Residues of trifloxystrobin and CGA321113 (mg/kg) in undelinted cotton seed (Netzband and Jerkins 2015 M-524431-01-1) (mean of duplicate samples) following applications of trifloxystrobin in an SC-formulation

Location, year, variety COTTON SEED	N (int)	Rate (g ai/ha)	Spray volume (L/ha)	GS BBCH	DALA	Sample [a]	Trifloxystrobin (mg/kg)	CGA321113 (mg/kg)	Total (mg/kg)
Talasee AL USA 2013 DPL 1050	3 (14 14)	142 140 139	188 278 183	69 71 80	29	Undelinted seed	< 0.01	< 0.01	< 0.01
Greenville MS USA 2013 Phytogen 499	3 (13 15)	142 140 138	133 140 134	74 76 80	31	Undelinted seed	< 0.01	< 0.01	< 0.01
Proctor AR USA 2013 FM1944 GLB2 10/9 [b]	3 (14 14)	137 138 138	103 96 96	75 75 83	30	Undelinted seed	0.01	< 0.01	0.01
Proctor AR USA 2013 DynaGro 2570 B2RF 15/9 [b]	3 (14 14)	139 139 140	106 97 97	75 75 82	25 29 35 40 44	Undelinted seed	< 0.01 < 0.01 < 0.01 0.03 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 0.03 < 0.01
Uvalde TX USA 2013 DPL 0912 B2RF [c]	3 (14 14)	139 140 139	112 113 112	65 80 84	23 29 34 38 44	Undelinted seed	0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.01 < 0.01 < 0.01 < 0.01 < 0.01

Location, year, variety COTTON SEED	N (int)	Rate (g ai/ha)	Spray volume (L/ha)	GS BBCH	DALA	Sample [a]	Trifloxystrobin (mg/kg)	CGA321113 (mg/kg)	Total (mg/kg)
Tahoka TX USA 2013 DP0912 B2RF	3 (14 12)	145 144 146	142 141 143	85	28	Undelinted seed	0.11	< 0.01	0.11
Levelland TX USA 2013 FM1944 GLB2	3 (14 13)	144 141 143	141 139 140	69 80 85	28	Undelinted seed	0.10 c0.03	< 0.01	0.10
Hinton OK USA 2013 FM1960 B2F [d]	3 (13 14)	139 140 140	150 186 194	73 79 84	26 30 36 40 43	Undelinted seed	0.04 0.05 c0.03 0.04 0.03 0.08	< 0.01 < 0.01 < 0.01 < 0.01 < 0.02	0.04 0.05 0.04 0.03 0.08
Wall TX USA 2013 FM1944 GLB2	3 (14 14)	141 139 141	138 132 135	77 78 87	27	Undelinted seed	0.03	< 0.01	0.03
Paso Robles CA USA 2013 DP 399	3 (14 14)	146 140 139	289 278 275	83 85 88	28	Undelinted seed	0.26	< 0.01	0.26
Sanger CA USA 2013 FM1944 GLB2	3 (14 14)	143 145 140	243 268 235	80 85 87	29	Undelinted seed	0.02	< 0.01	0.02
Porterville CA USA 2013 Phytogen 499	3 (14 13)	138 139 138	234 282 277	80 84 89	25 30	Undelinted seed	0.08 0.20	0.03 < 0.01	0.10 0.20

All applications were made using ground-based equipment. The adjuvant INDUCE PH (0.1% v/v) was used in all applications.

[a] samples were collected by hand except for three trials, Uvalde, Hinton and Wall where cotton seed was harvested using stripper equipment. Prior to analysis, seed cotton was sent for processing (ginning) into the derived commodity of undelinted cotton seed; in the three trials using stripper cotton, cotton gin by-products (gin trash), were also generated.

[b] the trials were conducted at the same location and with final application within a 5 day interval. These trials are not considered independent for the purposes of estimating maximum residue limits.

[c] Uvalde 15 mm rain within 24 hours of the third spray

[d] Hinton 5 mm rain within 24 hours of spray two

Trifloxystrobin is considered a meso-systemic fungicide, that is residues penetrate the leaf surface but are not significantly translocated to other plant parts. Residues in cotton seed generally increased with increase in growth stage reflecting the increasing proportion of bolls open (BBCH 80 = 1st open bolls, BBCH 81 = about 10% bolls open, BBCH 82 = about 20% bolls open, BBCH 83 = about 30% bolls open, BBCH 89 = about 90% bolls open), and therefore seeds potentially exposed to contact with spray liquid (Figure 1).

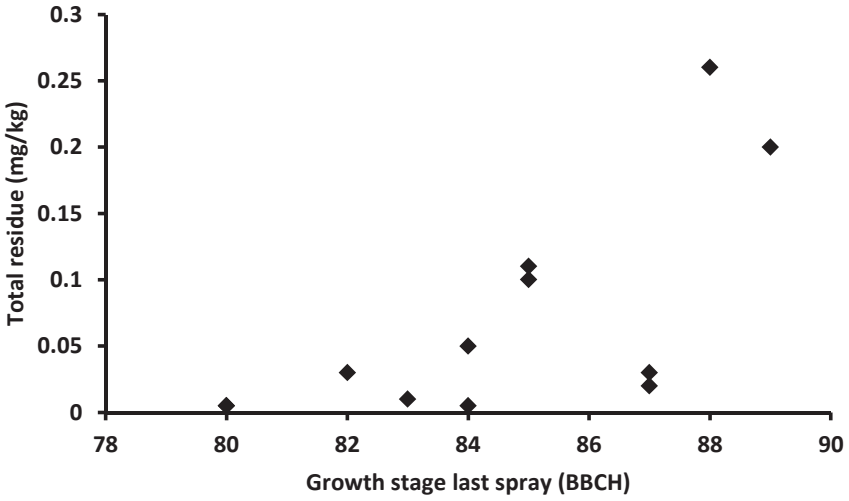

Figure 1 Residues in cotton seed versus BBCH growth stage for the timing of the last application

Ginseng

Table 6 Residues of trifloxystrobin and CGA321113 (mg/kg, mean of nine replicates) in ginseng (Kyung 2014) following applications of trifloxystrobin in an SC-formulation

Location, year, variety GINSENG	N	Rate (g ai/ha)	Spray Concentration (g ai/hL)	Spray volume (L/ha)	GS	DALA	Trifloxystrobin (mg/kg)	CGA321113 (mg/kg)	Total (mg/kg)
Yeongju-si Gyeongsangbuk-do, Korea 2013 7/9	3	65 67 65	3.33 3.35 3.33	1950 2000 1950	mature	21	< 0.06	< 0.03	< 0.06
2014 26/8 same plot as treated 2013 [a]	3	64 67 65	3.32 3.35 3.33	1925 2000 1950	mature	21	< 0.03	< 0.03	< 0.03
2014 26/8 new plot [a]	3	64 67 65	3.32 3.35 3.33	1925 2000 1950	mature	21	< 0.03	< 0.03	< 0.03
Geumsan-gun, Chungcheongnam-do Korea 2013 8/9	3	63 65 65	3.32 3.33 3.33	1900 1950 1950	mature	21	< 0.06	< 0.03	< 0.06
2014 27/8 same plot as treated 2013 [a]	3	65 67 63	3.33 3.35 3.32	1950 2000 1900	mature	21	< 0.03	< 0.03	< 0.03
2014 different plot [a]	3	65 67 63	3.33 3.35 3.32	1950 2000 1900	mature	21	< 0.03	< 0.03	< 0.03
Jeungpyeong-gun, Chungcheongbuk-do Korea 2013	3	65 64 67	3.33 3.32 3.35	1950 1925 2000	Mature	21	< 0.06	< 0.03	< 0.06
2014 same plot as treated 2013 [A]	3	65 65 67	3.33 3.33 3.35	1950 1950 2000	mature	21	< 0.03	< 0.03	< 0.03
2014 different plot [a]	3	65 65 67	3.33 3.33 3.35	1950 1950 2000	mature	21	< 0.03	< 0.03	< 0.03

[a] There is no carry-over of residues from one year to the next. Trials conducted at the same location with the same treatment dates are not considered independent for the purposes of estimating maximum residue limits

Animal feeds

Table 7 Residues of trifloxystrobin and CGA321113 (mg/kg) in cotton gin by-products (Netzband and Jerkins 2015 M-524431-01-1) (mean of duplicate samples) following applications of trifloxystrobin in an SC-formulation

Location, year, variety Gin by-products	N (int)	Rate (g ai/ha)	Spray volume (L/ha)	GS	DALA	Sample	Trifloxystrobin (mg/kg)	CGA321113 (mg/kg)	Total (mg/kg)
Uvalde TX USA 2013 DPL 0912 B2RF [a]	3 (14 14)	139 140 139	112 113 112	65 80 84	23 29 34 38 44	Gin byp82 88 89%DM 82 87	0.36 0.24 0.23 0.20 0.18	0.12 0.09 0.11 0.10 0.13	0.48 0.33 0.34 0.31 0.31
Hinton USA 2013 FM1960 B2F [b]	3 (13 14)	139 140 140	150 186 194	73 79 84	26 30 36 40 43	Gin byp 79 86 83%DM 85 87	1.3 0.93 c0.04 0.76 0.93 0.67	0.06 0.06 0.06 0.07 0.05	1.4 1.0 0.82 1.0 0.72
Wall TX USA 2013 FM1944 GLB2	3 (14 14)	141 139 141	138 132 135	77 78 87	27	Gin byp	0.71 c0.02	0.18	0.89

[a] Uvalde 15 mm rain within 24 hours of the third spray

[b] Hinton 5 mm rain within 24 hours of spray two

Fate of residues in processing

Broccoli

The effect on residues of the household preparation steps washing and cooking of broccoli was made available to the meeting (Fischer and Harbin 2008 M-307503-01-1).

Washed: Broccoli head and stems were rinsed under a stream of tap water for 30 sec, excess water shaken off and the samples placed on paper towels to dry for 2 minutes.

Cooked: Washed broccoli was cooked in boiling water for 8 minutes, drained and cooled.

Table 8 Residues of trifloxystrobin and CGA321113 (mg/kg) in broccoli after processing (Fischer and Harbin 2008 M-307503-01-1) (mean of duplicate samples)

Location, year, variety BROCCOLI	N (int)	Rate (g ai/ha)	DALA	Sample	Trifloxystrobin (mg/kg)	CGA321113 (mg/kg)	Total (mg/kg)	PFtri	PFtot
Sanger CA USA 2007 Marathon	2 (5)	141 143	0	Heads+stalks	0.41	0.03	0.44		
				washed	0.32	0.03	0.35	0.78	0.80
				Cooked	0.36	0.01	0.37	0.88	0.84

PFtri = processing factor for trifloxystrobin

PFtot = processing factor for the sum of trifloxystrobin and CGA321113

Spinach

The effect on residues of the household preparation steps washing and cooking of spinach was also investigated (Fischer and Harbin 2010 M-307507-02-1).

Washed: Spinach was washed by agitation in a sink of water for 30 seconds. The water was drained and the process repeated three times. After the last wash, excess water shaken off and the samples placed on paper towels to dry for 2 minutes.

Cooked: Washed spinach was cooked in boiling water for 2-3 minutes, drained and cooled.

Table 9 Residues of trifloxystrobin and CGA321113 (mg/kg) in spinach after processing (Fischer and Harbin 2010 M-307507-02-1) (mean of duplicate samples)

Location, year, variety SPINACH	N (int)	Rate (g ai/ha)	DALA	Sample	Trifloxystrobin (mg/kg)	CGA321113 (mg/kg)	Total (mg/kg)	PFtri	PFtot
King City CA USA 2007 Interceptor/Avenger	2 (5)	140 141	0	Fresh	5.14	0.04	5.19		
				Washed	5.22	0.04	5.26	1.0	1.0
				Cooked	1.83	0.02	1.85	0.36	0.36

PFtri = processing factor for trifloxystrobin

PFtot = processing factor for the sum of trifloxystrobin and CGA321113

Cotton

A study of the effect of processing cottonseed to oil on residues was made available to the meeting (Veal 2015 M-524279-01-1). Three applications of an SC formulation of trifloxystrobin were made to cotton at an application rate of approximately 700 g ai/ha with harvest about30 days after the last application.

Undelinted seed was processed into hulls, cottonseed meal, and refined oil. Samples were processed to simulate commercial practice as closely as possible. Prior to ginning, the seed cotton was cleaned with an attached stick extractor to remove gin trash (gin by-products). Seed was saw-ginned to remove most of the lint (ginned cottonseed = undelinted cottonseed). With approximately 11–15% remaining lint, the undelinted cottonseed was saw delinted to produce delinted cottonseed (ca. 3% lint remaining). The delinted seed was mechanically cracked on a roller mill followed by screening to separate the hulls from the kernel. The kernel material was adjusted to 13.5% moisture and processed into meal and crude oil by heating kernels to 79–91 °C for 28–32 minutes, after which the kernel material was flaked (roll gap 0.02–0.03 cm) and the flaked material fed into a continuous processor (extruder). As material moved through the extruder, steam was injected directly on the product. The maximum temperature of the exiting collets was 93–121 °C. Collets were dried in an oven at 66–82 °C for 30–40 minutes and then solvent extracted in batches (hexane 49–60 °C). After 30 minutes the hexane was drained, fresh hexane added and the extraction process repeated, three times in total. After final draining, the spent collets (meal) were heated to 99–104 °C in a steam heated paddle mixer to remove residual hexane and toasted in an electric cooker and the moisture adjusted to 15% and screened. The miscella (crude oil + hexane) was passed through a vacuum evaporator (91–96 °C) to remove the hexane and filtered prior to refining. The crude oil was heated to 70–76 °C, water added (to 3% weight of oil), stirred for 30–60 min and centrifuged to remove gums. The de-gummed oil was treated with 85% phosphoric acid equal to 0.03% the weight of oil and stirred for 29–31 minutes at 40–44 °C, centrifuged and filtered to remove slime. The resulting pre-clarified oil was mixed with NaOH at 20–24 °C for 15 minutes, the temperature increased to 63–67 °C for a further 12 minutes. The neutralised oil was centrifuged to remove the solids (soapstock) and the refined oil decanted and vacuum filtered. Refined oil was heated to 40–50 °C and activated bleaching earth added (1% by weight), the temperature increased to 85–100 °C and held at this temperature for 10–15 minutes, cooled and filtered to produce bleached oil. The bleached oil was held at 220–230 °C under vacuum for 28–32 minutes. After this period the oil was cooled during which time 0.5% citric acid was added and the resulting refined bleached deodorised oil collected (=oil, refined).

The processing factors range from 0.02 to 0.17, indicating that no concentration occurred in processed fractions.

Table 10 Residues of trifloxystrobin and CGA321113 (mg/kg) in cotton seed processed commodities (means of three individual analyses) (Veal 2015 M-524279-01-1)

Location, year, variety COTTON SEED	N	Rate (g ai/ha)	DALA	Sample	Trifloxystrobin (mg/kg)	CGA321113 (mg/kg) [a]	Total (mg/kg)	PFtri	PFtot
Cheneyville LA USA 2013 Phytogen 499 WRF 72.7 kg batch	3	730 711 728	29	Undelinted seed	0.178	0.021	0.181		
				Meal	< 0.01	< 0.01	< 0.01	< 0.06	< 0.05
				Hulls	0.020	0.013	0.033	0.11	0.17
				Crude oil, preclarified	0.014	< 0.01	0.014	0.08	0.12
				Oil, solvent extracted	0.014	< 0.01	0.014	0.08	0.12
				Crude oil, neutralised	< 0.01	< 0.01	< 0.01	< 0.06	< 0.05
				Oil, refined	< 0.01	< 0.01	< 0.01	< 0.06	< 0.05
Levelland TX USA 2013 DP0912 B2RF 80.5 kg batch	3	716 699 703	28	Undelinted seed	3.122	< 0.01	3.122		
				Meal	< 0.01	< 0.01	< 0.01	< 0.003	< 0.003
				Hulls	0.159	< 0.01	0.159	0.05	0.05
				Crude oil, preclarified	0.135	< 0.01	0.135	0.04	0.04
				Oil, solvent extracted	0.131	< 0.01	0.131	0.04	0.04
				Crude oil, neutralised	0.116	< 0.01	0.116	0.04	0.04
				Oil, refined	0.060	< 0.01	0.060	0.02	0.02

[a] CGA321113 determined as CGA321113 and calculated as trifloxystrobin

PFtri = processing factor for trifloxystrobin

PFtot = processing factor for the sum of trifloxystrobin and CGA321113

Ginseng

The residue trials on ginseng also contained information on processing (Kyung 2014). Processed products were processed by four different methods (dried ginseng, red ginseng, water extracts of dried ginseng and water extracts of red ginseng). Samples of processed ginseng products were transferred into polyethylene bags and stored at -20 °C until analysis.

Fresh ginseng were harvested and washed with tap water to remove soil particle from the roots. The washed fresh ginseng were blended, packed individually in polyethylene bags and stored at -20 °C until analysis.

Dried ginseng: Fresh washed ginseng were dried in a hot air drying machine set to 60 °C to water content < 14%. The dried ginseng were blended, packed individually in polyethylene bags and stored at -20 °C until analysis.

Red ginseng: Fresh washed ginseng were washed and steamed for 3 hours at 98 °C. The steamed ginsengs were dried in hot air drying machine set to 65 °C to reach a water content of about 50~55%. Small roots were removed and further drying processes were carried out under sunlight to reach a final water content < 14%. The dried ginsengs were blended, packed individually in polyethylene bags and stored at -20 °C until analysis.

Water extracts of dried ginseng and water extracts of red ginseng: Dried ginseng or red ginseng was cut into about 1 cm in size and extracted three times in refluxing extractor with water at 85 °C for about 18 hours. The extracts were evaporated with a vacuum rotary evaporator to reach the 72° Brix. The extracts were packed in polyethylene bottles individually and stored at -20 °C until analysis.

No residues were detected in fresh ginseng or in ginseng processed products. It was not possible to derive processing factors for ginseng.

In summary, trifloxystrobin did not concentrate in any of the processed commodities considered by the current meeting (Table 10).

Table 10 Summary of trifloxystrobin processing factors

Commodity	Processing fraction	PFtri	PFtot
Broccoli	Washed broccoli	0.78	0.80
	Cooked broccoli	0.88	0.84
Spinach	Washed spinach	1.0	1.0
	Cooked spinach	0.36	0.36
Cotton seed	Meal	< 0.003 < 0.06	< 0.003 < 0.05
	Hulls	0.05 0.11	0.05 0.17
	Crude oil	0.04 < 0.06	0.04 < 0.05
	Refined oil	0.02 < 0.06	0.02 < 0.05

PFtri = processing factor for trifloxystrobin

PFtot = processing factor for the sum of trifloxystrobin and CGA321113

APPRAISAL

Trifloxystrobin was first evaluated for toxicology and residues by the JMPR in 2004. The Meeting derived an ADI of 0–0.04 mg/kg bw, decided that an ARfD is unnecessary and concluded that the residue definition for plant commodities for compliance with MRL values was trifloxystrobin. For animal commodities for compliance with MRL values as well as for dietary risk assessment for both plant and animal commodities, the residue definition was the sum of trifloxystrobin and (*E,E*)-methoxyimino-{2-[1-(3-trifluoromethyl-phenyl) ethylidene-aminooxymethyl]-phenyl}acetic acid = CGA321113) (expressed as trifloxystrobin equivalents). The residue is fat soluble.

Trifloxystrobin was scheduled at the 48th session of the CCPR for the evaluation of additional MRLs for head cabbage, cauliflower, broccoli, spinach, cotton and ginseng by the 2017 JMPR.

Methods of analysis

The method used for the analysis of head cabbage, cauliflower, broccoli, spinach and cotton samples was method 200177 which was evaluated by the 2004 and 2015 JMPR. The Meeting received summarized information on an analytical method for trifloxystrobin and CGA321113 residues in ginseng. Mean recoveries were within the range 70–120% for all commodities investigated and supported LOQs of 0.03 mg/kg (2014 trials) to 0.06 mg/kg (2013 trials) for ginseng and 0.07 mg/kg for ginseng processed commodities. The method is suitable for the analysis of residues in ginseng and ginseng processed commodities.

Stability of residues in stored analytical samples

The stability of residues in samples on frozen storage was evaluated by the 2004 JMPR for a range of commodities. Residues of trifloxystrobin and CGA321113 were stable under freezer storage conditions for at least 24 months in the case of grapefruit, cucumber, potato and wheat commodities (grain, straw and whole plant) or 18 months for apple (fruit, wet pomace), peanut (nutmeat, oil) and grape juice. The stability data covered commodities that are representative of high water content (apple, cucumber, grape juice), high acid content (grapefruit), high oil content (peanut) and high protein/starch content (potato, wheat) and can be extrapolated to the commodities considered at the current Meeting. Samples in the trials considered by the current Meeting were stored frozen for periods less than the period of stability demonstrated in studies supplied to the 2004 JMPR and are therefore considered unlikely to have been adversely affected by storage.

Results of supervised residue trials on crops

The Meeting received information on supervised field trials on brassicas (broccoli, cauliflower and cabbage), spinach, cotton and ginseng.

The sum of trifloxystrobin and CGA321113 was calculated and expressed as trifloxystrobin on the basis of the relative molecular masses. A conversion factor of 1.036 is required to express CGA321113 as trifloxystrobin. As CGA321113 does not generally constitute a significant proportion of the residue in crops, when the levels of trifloxystrobin or CGA321113 were below the LOQ, their sum was calculated as in the examples provided by the 2004 JMPR and copied below.

Trifloxystrobin (mg/kg)	CGA321113 (mg/kg)	Total (expressed as trifloxystrobin) (mg/kg)
< 0.02	< 0.02	< 0.02
< 0.02	0.03	0.05
0.10	< 0.02	0.10
0.92	0.16	1.1

Brassicas

The critical GAP in the USA for brassica, head and stem vegetables is for two to three applications at a maximum rate of 139 g ai/ha, at 7–14 day intervals and a PHI of 0 days with a maximum seasonal rate of 281 g ai/ha/yr.

Three trials in broccoli (trifloxystrobin: 0.44, 0.67, 0.68 mg/kg) three trials in cauliflower (< 0.01, 0.01, 0.32 mg/kg) and six trials in head cabbage (with wrapper leaves) (0.03, 0.11, 0.37, 0.47, 0.48, 0.58 mg/kg) matched cGAP in the USA.

Corresponding total residues were:

Broccoli (0.46, 0.67, 0.70 mg/kg)

Cauliflower (< 0.01, 0.01, 0.36 mg/kg)

Head cabbage (without wrapper leaves) (< 0.01, < 0.01, 0.01, 0.01, 0.01, 0.03 mg/kg)

The number of trials on broccoli and cauliflower are too few to estimate a maximum residue level for these crops. The Meeting explored the possibility of estimating a group maximum residue level for subgroup 010A, flowerhead brassicas based on the combined dataset however, the residue data for broccoli and cauliflower were from two different populations and could not be combined. The Meeting concluded there is insufficient data to estimate a new maximum residue levels for both broccoli and cauliflower.

For cabbages, utilising the residues from trials (n=6) approximating cGAP (139 ± 25% g ai/ha) in the USA, the Meeting recommended a maximum residue level of 1.5 mg/kg, and an STMR of 0.01 mg/kg for head cabbage to replace the previous recommendation of 0.5 mg/kg.

Spinach

In the USA the critical GAP for leafy greens is for two applications at a maximum rate of 139 g ai/ha, at an interval of 14 days with a PHI of 0 days.

Six trials in spinach approximated critical GAP (139 ± 25% g ai/ha) with trifloxystrobin residues (n=6): 4.8, 5.3, 7.6, 7.6, 9.5 and 10 mg/kg.

The Meeting estimated a maximum residue level of 20 mg/kg for spinach.

Corresponding total residues were: 4.8, 5.4, 7.6, 7.6, 9.6 and 10 mg/kg. The Meeting estimated an STMR of 7.6 mg/kg for spinach.

Cotton seed

The critical GAP for cotton is for 3 applications at a maximum rate of 137 g ai/ha, at 14 day intervals and a PHI of 30 days.

In eleven trials approximating critical GAP (137 ± 25% g ai/ha) residues of trifloxystrobin were (n=11): < 0.01, < 0.01, < 0.01, 0.02, 0.03, 0.03, 0.08, 0.10, 0.11, 0.20 and 0.26 mg/kg.

The Meeting estimated a maximum residue level of 0.4 mg/kg for cottonseed.

Residues of CGA311123 were all < 0.01 mg/kg and did not contribute to the total residue. The Meeting estimated an STMR of 0.03 mg/kg.

Ginseng

The critical GAP for ginseng in the Republic of Korea is for 3 applications at a maximum rate of 3.35 g ai/hL, at 10 day intervals and a PHI of 21 days.

In six trials from the Republic of Korea approximating critical GAP (3.35 ± 25% g ai/hL) residues were (n=6): < 0.03 < 0.03 < 0.03, < 0.06, < 0.06, < 0.06 mg/kg.

The Meeting estimated a maximum residue level of 0.03* mg/kg and STMR of 0.03 mg/kg for ginseng.

Animal feedstuffs

Cotton gin by-products

The critical GAP for cotton is for 3 applications at a maximum rate of 137 g ai/ha, at 14 day intervals and a PHI of 30 days. In three trials approximating critical GAP residues of trifloxystrobin were (n=3): 0.24, 0.71, 0.93 mg/kg. The Meeting estimated a median residue for cotton gin by-products of 0.71 mg/kg and a highest residue of 0.93 mg/kg.

Fate of residues in processing

No residues were detected in fresh ginseng or in ginseng processed products. Residues in processed ginseng products will be covered by the maximum residue level recommended for fresh ginseng.

Trifloxystrobin did not concentrate in any of the other processed commodities considered by the current meeting.

Summary of trifloxystrobin processing factors

Commodity	Processing fraction	PFtri	PFtot (best estimate)	$STMR_{RAC\ tot}$	STMR-P = $STMR_{RAC\ tot}$ × PFtot
Broccoli	Washed broccoli	0.78	0.80		
	Cooked broccoli	0.88	0.84		
Spinach	Washed spinach	1.0	1.0		
	Cooked spinach	0.36	0.36		
Cotton seed	Meal	< 0.003 < 0.06	< 0.003 < 0.05 (0.003)	0.03	0.00009
	Hulls	0.05 0.11	0.05 0.17 (0.11)		0.0033
	Crude oil	0.04 < 0.06	0.04 < 0.05 (0.04)		0.0012
	Refined oil	0.02 < 0.06	0.02 < 0.05 (0.02)		0.0006

PFtri = processing factor for trifloxystrobin

PFtot = processing factor for the sum of trifloxystrobin and CGA321113

Residues in animal commodities

Estimation of livestock dietary burdens

The only commodity used as a livestock feed and for which the JMPR has made recommendations are cabbage with wrapper leaves and cotton gin by-products (gin trash). The additional contribution to the dietary burden using the estimated median and highest residue levels is less than 10% of the total. Based on the minor change in livestock dietary burden, the Meeting did not recalculate residues in animal commodities or revise its recommendations for maximum residue levels.

RECOMMENDATIONS

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

Residue in plant commodities

for compliance with MRLs: *trifloxystrobin*

for estimation of dietary exposure: *sum of trifloxystrobin and [(E,E)-methoxyimino-{2-[1-(3-trifluoromethylphenyl)ethylideneaminooxymethyl]phenyl}acetic acid] (CGA 321113), expressed as trifloxystrobin.*

Residue in animal commodities

For compliance with MRLs and estimation of dietary exposure: *sum of trifloxystrobin and [(E,E)-methoxyimino-{2-[1-(3-trifluoromethylphenyl)ethylideneaminooxymethyl]phenyl}acetic acid] (CGA 321113), expressed as trifloxystrobin.*

The residue is fat soluble.

Table of recommendations

Commodity		Recommended MRL (mg/kg)		STMR or STMR-P (mg/kg)	HR, HR-P, highest residue (mg/kg)
CCN	Name	New	Previous		
VB 0041	Cabbages, Head	1.5	0.5	0.01	
SO 0691	Cotton seed	0.4		0.03	
VR 0604	Ginseng	0.03*		0.03	
VL 0502	Spinach	20		7.6	

Table of additional STMR/median and HR/highest residue values for use in dietary intake and livestock dietary burden estimation.

Commodity		Recommended MRL (mg/kg)		STMR or STMR-P, median residue (mg/kg)	HR, HR-P, highest residue (mg/kg)
CCN	Name	New	Previous		
	Cotton seed meal			0.00009	
	Cotton seed hulls			0.0033	
OR 0691	Cotton seed refined oil, edible			0.0006	
	Cotton gin by-products			0.71	0.93

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The International Estimated Daily Intakes (IEDIs) of trifloxystrobin were calculated for the 17 GEMS/Food cluster diets using STMRs and STMR-Ps estimated by the JMPR in 2004, 2015 and current meeting. The results are shown in Annex 3 to the 2017 Report.

The ADI is 0–0.04 mg/kg bw and the calculated IEDIs were 1–7% of the maximum ADI. The Meeting concluded that the long-term dietary exposure to residues of trifloxystrobin from the uses considered by the JMPR is unlikely to present a public health concern

Short-term dietary exposure

The 2004 JMPR decided that an ARfD for trifloxystrobin was unnecessary. The Meeting therefore concluded that the short-term dietary exposure to residues of trifloxystrobin resulting from uses that have been considered by the JMPR is unlikely to present a public health concern.

REFERENCES

Author(s)	Year	Title
de Haan, R. A.	2002	Analytical method for the determination of residues of trifloxystrobin (Flint) and trifloxystrobin acid in/on tomatoes and peppers by LC-MS/MS. Bayer Corporation, Stilwell, KS, USA. Bayer CropScience AG, Report No.: 200177, Edition Number: M-070236-01-1. Unpublished
Fischer, R. D. and Harbin, A. M.	2008	AE C656948 500 SC and trifloxystrobin 500 SC - Magnitude of the residue in/on head and stem brassica (crop subgroup 5A). Bayer CropScience LP, Stilwell, KS, USA. Bayer CropScience AG, Report No. RAGMP076, Edition Number: M-307503-01-1. Unpublished.
Fischer, R. D. and Harbin, A. M.	2010	AE C656948 500 SC and trifloxystrobin 500 SC - Magnitude of the residue in/on leafy vegetables (crop subgroup 4). Bayer CropScience LP, Stilwell, KS, USA. Bayer CropScience AG, Report No. RAGMP085-1, Edition Number: M-307507-02-1. Unpublished.
Kyung K.S.	2014	Magnitude of trifloxystrobin and its metabolite CGA321113 residues in ginseng and its processed products in Korea. MFDS 2013-17889. Unpublished.
Netzband, D. and Jerkins, E.	2015	Proline 480 SC and Gem 500 SC - Magnitude of the residue in/on cotton. Bayer CropScience LP, RTP, NC, USA. Bayer CropScience AG, Report No. RAJAN029, Edition Number: M-524431-01-1. Unpublished.
Veal, M.	2015	Proline 480 SC and Gem 500 SC - Magnitude of the residue in/on cotton processed commodities. Bayer CropScience LP, RTP, NC, USA. Bayer CropScience AG, Report No. RAJAN030, Edition Number: M-524279-01-1. Unpublished.