CYPERMETHRINS (INCLUDING ALPHA- AND ZETA-CYPERMETHRIN) (118)

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EXPLANATION

Cypermethrin was evaluated by JMPR 1979 (T,R), 1981 (T,R), 1982 (R), 1983 (R), 1984 (R), 1985 (R), 1986 (R), 1987 (corr. to 1986 evaluation), 1988 (R), 1990 (R), 2006 (T), 2008 (R), 2009 (R). The last periodic review for toxicology was in 2006 and for residues in 2008 and included cypermethrin, alpha-cypermethrin and zeta-cypermethrin. The 2006 Meeting estimated the acceptable daily intake (ADI) for humans as 0–0.02 mg/kg bw and estimated the acute reference dose (ARfD) as 0.04 mg/kg bw. The 2008 Meeting defined the residue (for compliance with the MRL and for estimation of dietary intake) for plant and animal commodities as cypermethrin (sum of isomers). The residue is fat soluble. In 2009 adjacent information on the use of cypermethrin was submitted and evaluated. Cypermethrin was listed by the 42nd Session of the CCPR (ALINORM 10/33/24) for the evaluation of 2011 JMPR for additional MRLs.

The Meeting received information on zeta-cypermethrin from the manufacturer on storage stability, residue analysis, use pattern, residues resulting from supervised trials on citrus fruits and tree nuts, and fate of residue during processing. In addition, the Meeting received information on cypermethrin on residue analysis, use pattern, and residues resulting from supervised trials on asparagus and pomelo from Thailand. China and India submitted information on cypermethrin about storage stability, residue analysis, use pattern, residues resulting from supervised trials on teas, and on fate of residue during processing (China). Furthermore, the Meeting received information from Japan on cypermethrin on use pattern.

IDENTITY

A short summary about cypermethrins is provided below for clarity.

Table 1 Comparison between cypermethrin, alpha-cypermethrin and zeta-cypermethrin.

ISOMER	ACTIVITY RATIO	CYPER-METHRIN %	ALPHA-CYPER-METHRIN %	ZETA-CYPER-METHRIN %
1.1R-cis-R	0.60	14	–	3
2.1S-cis-S	0.03	14	–	22
3.1R-cis-S	13.5	11	50	22
4.1S-cis-R	0.04	11	50	3
5.1R-trans-R	0.40	14	–	3
6.1S-trans-S	0.03	14	–	22
7.1R-trans-S	3.20	11	–	22
8.1S-trans-R	0.01	11	–	3

Cypermethrin, alpha-cypermethrin and zeta-cypermethrin are similar pyrethroid insecticides with the same basic chemical formula and molecular weight.

Cypermethrin is a racemic mixture of eight isomers that only differ in the structural orientation of their chemical bonds (see Table 1). These eight isomers consist of two groups: those with a cis orientation across the cyclopropyl of the dichlorovinyl and ester groups and those with a trans-orientation. The ratio of cis to trans isomers ranges from 40:60 to 55:45 in cypermethrin. The two most active isomers are 1R-cis-S and 1R-trans-S.

The components of zeta-cypermethrin have the same chemical structure as those of cypermethrin.

RESIDUE ANALYSIS

Analytical methods

The Meeting received information on a residue enforcement method for foodstuff with high water and high acid content for the determination of zeta-cypermethrin in plant material. Furthermore, information on analytical methods for the determination of (zeta-)cypermethrin in supervised residue trials, processing studies and storage stability studies have been submitted to the meeting. Although analytical methods may discriminate between the different isomers of cypermethrin (depending on GC column used), residues are reported as cypermethrin (sum of isomers) in all cases.

Extended revision of Multi-residue DFG enforcement method S19 [Class, 2002, P 656 G]

The extended revision lists cypermethrin with average recoveries ranging from 80–113% and RSDs ranging from 3–30% for various plant matrix types for which the method can be considered validated. Additionally, the method was validated for use in watery (tomato) and acidic (orange) crop types by fortification with zeta-cypermethrin. The acidic plant material (orange) was neutralized with sodium hydrogen carbonate to adjust the pH to approximately 7. Then zeta-cypermethrin residues were extracted from tomato and orange with water/acetone (1:2 v/v). After partitioning into the organic phase by adding sodium chloride and ethyl acetate (EtOAc)/cyclohexane (1:1 v/v), aliquots of the upper organic phase were filtered through filter paper covered with sodium sulphate. The filtrate was evaporated to a watery remainder and resolved in EtOAc, where after sodium sulphate/sodium chloride (1:1 w/w) and cyclohexane were added. The specimen extract was cleaned up using gel permeation chromatography (GPC), and fractionated by column chromatographic fractionation on silica gel. Zeta-cypermethrin was predominantly present in fraction F2 (toluene). A portion of the final extract was then used for GC-ECD or GC-MS (electron impact ionisation, quantification m/z 181, confirmation m/z 127 and m/z 163) analysis for confirmation. In case the residues present in the specimens exceed 10× LOQ and most likely also the dynamic range of the GC-ECD detection, the final extracts were diluted by a factor 5. Recoveries, repeatability and calibration are presented in Table 2. The reported LOQ was 0.01 mg/kg.

Table 2 Validation results for the determination of zeta-cypermethrin using multi-residue DFG enforcement method S19

Commodity	Reported LOQ mg/kg	Spike level mg/kg	n	% Recovery Mean range	RSD_r	Control samples mg/kg (n)	Calibration	Reference, method
tomato	0.01	0.010 0.10	5 5	97 [a] 87–102 91 [a] 82–103	6% 9%	< 0.3× LOQ	1.0–200 ng/mL, 8 levels, Quadratic [b] R^2 > 0.99	P 656 G, GC-ECD
tomato	0.01	0.010 0.10	1 1	82 – 95 –	– –	< 0.3× LOQ	10–1000 ng/mL, 7 levels linear R^2 > 0.99	P 656 G, GC-MS
orange	0.01	0.010 0.10	5 5	109 103–120 87 78–95	6% 9%	< 0.3× LOQ	Quadratic [b], 8 levels 1.0–200 ng/mL Quadratic [b] R^2 > 0.99	P 656 G, GC-ECD
orange	0.01	0.010 0.10	1 1	85 – 85 –	– –	< 0.3× LOQ	10–1000 ng/mL, 7 levels linear R^2 > 0.99	P 656 G, GC-MS

[a] A background signal of 0.0019 mg/kg was subtracted.

[b] Non-linear GC-ECD response is observed frequently for analytes with intense ECD response. The use of linear calibration functions with shorter range may be possible, but would limit the dynamic range and require preliminary screening of final extracts followed possibly by dilution as to adjust the final concentration to the more limited linear quantification range.

Methods used in supervised residue trials

GC-ECD method A [Dow, 2004, P-3710; Zenide and Françon, 1994, A-17-94-02; Zenide, 1994, A-17-94-03; Markle, 1994, RAN-0147]

GC-ECD method A was described by Zenide and Françon (1994) to determine residues of zeta-cypermethrin on oranges and lemons. The homogenized samples were extracted with hexane:acetone (1:1, v/v). The extract was partitioned against water and the aqueous solution was extracted twice with hexane. From the combined organic phase, an aliquot was cleaned-up on Florisil column. The column eluate was evaporated and the residue redissolved in hexane. Analysis was performed by GC-ECD. Calibration was done by the external standard method with standard test solutions of 0.05–0.5 µg/mL. Reported LOQ was 0.05 mg/kg.

Dow (2004) used the same method for the determination of zeta-cypermethrin residues on almonds and pecans with some modifications. The acetone/hexane (50:50 v/v) extract was concentrated, reconstituted in hexane and cleaned-up with a silica gel solid phase extraction (SPE) cartridge. After concentrating, the specimen was analysed with GC-ECD and confirmed with GC-MS. Residues of zeta-cypermethrin were quantitated using a single point external standard calibration method at 0.0125–0.2 ng/µL. The reported LOQ was 0.05 mg/kg.

A comparable method was used in a storage stability study on lettuce, tomatoes, apples and soybeans (Markle, 1994). Crop samples were extracted with hexane/acetone (1:1 v/v). The extract was filtered and partitioned against water. The aqueous phase was extracted with hexane in the presence of sodium chloride, and the hexane fractions were passed through anhydrous sodium sulphate. The concentrated samples were then cleaned up by GPC (soybeans only) followed by Florisil. All samples were analysed with GC-ECD. Quantitation of residues was performed by external standard calibrations. The reported LOQ was 0.05 mg/kg. The validation results of the GC-ECD method are provided in Table 3.

Table 3 Validation results for GC-ECD method A for the determination of zeta-cypermethrin using GC-ECD.

Commodity	Reported LOQ mg/kg	Spike level mg/kg	n	% Recovery mean	range	RSD_r	Control samples mg/kg (n)	Calibration	Reference, method
almond hulls	0.05	0.05 2 10	1 1 1	90 113 88	– – –	– – –	< LOQ	0.0125–0.2 ng/µL, linearity not shown	P-3710, GC-ECD
almond nutmeat	0.05	0.05 0.1	2 1	107 116	97–117 –	– –	< LOQ	0.0125–0.2 ng/µL, linearity not shown	P-3710, GC-ECD
pecan nutmeat	0.05	0.05	2	84	75–94	–	< LOQ	0.0125–0.2 ng/µL, linearity not shown	P-3710, GC-ECD
lemon (whole fruit)	0.05	0.05 0.10	1 2	104 97	– 87–106	– –	< LOQ (4)	0.05–0.5 µg/mL, 4 duplicate points, linear by graph	A-17-94-02, GC-ECD
orange (whole fruit)	0.05	0.05 0.10 1	3 2 1	97 98 94	88–104 91–104 –	8.3% – –	< LOQ (8)	0.05–0.5 µg/mL, 4 duplicate points, linear by graph	A-17-94-03, GC-ECD

GC-ECD method B [Culligan, 2005, P-3753]

To determine zeta-cypermethrin in whole citrus fruit, pulp only (wet), dried pulp and orange juice, samples were extracted with acetone/water (70:30 v/v) and partitioned into hexane. Orange oil was extracted with hexane and partitioned into acetonitrile (ACN). All citrus fruit extracts were cleaned-up using a silica gel SPE cartridge and analysed with GC-ECD. Residues of zeta-cypermethrin were quantitated using a single point external standard calibration method within the concentration range

12.5 to 500 pg/µL. The reported LOQ was 0.05 mg/kg. The validation results of the GC-ECD method are provided in Table 4.

Table 4 Validation results for GC-ECD method B for the determination of zeta-cypermethrin using GC-ECD.

Commodity	Reported LOQ mg/kg	Spike level mg/kg	n	% Recovery mean	range	RSD_r	Control samples mg/kg (n)	Calibration	Reference, method
lemon (whole fruit)	0.05	0.05 0.1 0.2	1 1 1	126 115 72	– – –	– – –	< LOQ	12.5–500 pg/µL linearity not shown	P-3753, GC-ECD
grapefruit (whole fruit)	0.05	1.0	1	90	–	–	< LOQ	12.5–500 pg/µL linearity not shown	P-3753, GC-ECD
grapefruit (wet pulp)	0.05	0.5	1	65	–	–	< LOQ	12.5–500 pg/µL linearity not shown	P-3753, GC-ECD
orange (whole fruit)	0.05	0.05 0.2 0.5	1 2 2	77 87 89	– 81–92 86–92	– – –	< LOQ	12.5–500 pg/µL linearity not shown	P-3753, GC-ECD
orange (wet pulp)	0.05	0.05	1	117	–	–	< LOQ	12.5–500 pg/µL linearity not shown	P-3753, GC-ECD
orange (dried pulp)	0.05	0.10	1	106	–	–	< LOQ	12.5–500 pg/µL linearity not shown	P-3753, GC-ECD
orange oil	0.05	0.05 0.5 5.0	1 1 1	122 92 89	– – –	– – –	< LOQ	12.5–500 pg/µL linearity not shown	P-3753, GC-ECD
orange juice	0.05	1.0	1	74	–	–	< LOQ	12.5–500 pg/µL linearity not shown	P-3753, GC-ECD

GC-ECD method C [Chen et al., 2011]

For the determination of cypermethrin, dry made tea samples were extracted with acetone. The extract was filtered, evaporated to dryness and redissolved in hexane. In case of tea infusion, the samples were extracted with n-hexane-acetone (9:1 v/v) and extracts were dried with $NaSO_4$, evaporated to dryness and concentrated. The extracts from made tea or tea infusion were cleaned-up with Florisil chromatographic column. Quantification was done with GC-ECD. Validation results are presented in Table 5. Tea infusion was not separately validated.

Table 5 Validation results for GC-ECD method C for the determination of cypermethrin using GC-ECD.

Commodity	Reported LOQ mg/kg	Spike level mg/kg	n	% Recovery mean	range	RSD_r	Control samples mg/kg (n)	Calibration	Reference, method
made tea	0.025	0.1 1 10 30	5 5 5 5	93 95 102 99	89–96 92–98 98–106 96–101	2.8% 2.3% 3.0% 1.9%	< 0.3 LOQ (5)	linearity not shown	Chen *et al.*, 2011 GC-ECD

GC-ECD method D [Barooah and Selvan, 2009]

For the determination of cypermethrin, dry made tea samples were re-hydrated with distilled water and mixed with acetone and n-hexane. An aliquot of the upper layer was transferred into a sodium chloride solution (2% w/v) and residues were partitioned into hexane. The hexane phase was concentrated followed by clean-up on an alumina column. The eluate was concentrated and quantification was done by GC-ECD using single point calibration. Validation results are presented in Table 6. The reported LOQ was 0.05 mg/kg.

For the determination of cypermethrin in tea infusion, the brew was allowed to cool and residues were partitioned into n-hexane and residues were determined directly by GC-ECD. No separate validation was performed on tea brew.

Table 6 Validation results for GC-ECD method D for the determination of cypermethrin using GC-ECD

Commodity	Reported LOQ mg/kg	Spike level mg/kg	n	% Recovery mean	range	RSD_r	Control samples mg/kg (n)	Calibration	Reference, method
black tea (made tea)	0.05	0.05 0.1 0.5	5 5 5	83 90 96	80–86 88–93 93-98	4.0 2.3 1.9	< 0.3 LOQ (8)	0.05–0.5 mg/mL [a], linearity not shown	Barooah and Selvan, 2011 GC-ECD

[a] Additional information provided by e-mail (Rai, 2011)

GC-ECD method E [Barooah and Selvan, 2009]

For the determination of cypermethrin, black tea samples were extracted with ACN:water (2:1 v/v) and filtered. Thereafter, 4% NaCl and hexane were added, and after partitioning the hexane layer was passed through an anhydrous sodium sulphate layer. The extract was evaporated to dryness and re-dissolved in hexane. ACN-saturated with hexane was added and the ACN-layer was drained into anhydrous sodium sulphate. The concentrated residue was cleaned up by adsorption column chromatography using 5% Florisil. The eluate was concentrated to dryness, redissolved in hexane and quantitated by GC-ECD using single point calibration. Validation results are presented in Table 7. The reported LOQ was 0.1 mg/kg.

For the determination of cypermethrin in tea infusion, the brew was allowed to cool and residues were partioned into n-hexane and residues were determined directly by GC-ECD. No separate validation was performed on tea brew.

Table 7 Validation results for GC-ECD method E for the determination of cypermethrin using GC-ECD.

Commodity	Reported LOQ mg/kg	Spike level mg/kg	n	% Recovery mean	range	RSDr	Control samples mg/kg (n)	Calibration	Reference, method
black tea (made tea)	0.1	0.1 1.0 2.0	3 3 3	97 93 93	96–98 92–95 91–95	0.71 1.7 1.8	< 0.3 LOQ (8)	0.14–0.5 mg/mL a, linearity not shown	Barooah and Selvan, 2011 GC-ECD

[a] Additional information provided by e-mail (Rai, 2011)

GC-ECD method F [Steinwandter, 1985]

GC-ECD method F was used for the determination of cypermethrin residues in pomelo and asparagus. Samples were extracted with acetone and dichloromethane in the presence of NaCl and the organic phase was dried with $NaSO_4$ and evaporated to dryness and redissolved in ethylacetate. The extracts were then cleaned up with silica gel SPE column and the eluate was evaporated to dryness and dissolved in hexane before analysis with GC-ECD. The reported LOQ was 0.01 mg/kg.

Method validation on pomelo and chilli pepper [Pongsapitch, 2011] is summarized in Table 8.

Table 8 Validation results for GC-ECD method F for the determination of cypermethrin using GC-ECD

Commodity	Reported LOQ mg/kg	Spike level mg/kg	n	% Recovery mean	range	RSD_r	Control samples mg/kg (n)	Calibration	Reference method
pomelo	0.01	0.01 0.1 1.0	10 10 10	79 88 93	73–84 81–98 83–105	11% 5.6% 7.7%	< 0.3LOQ (3)	0.01–2.0 mg/kg 10 single points in solvent linear, r^2> 0.99	Pongsapitch, 2011, GC-ECD
chilli peppers	0.01	0.01 0.05 0.4 4.0	10 10 10 10	78 97 108 103	70-90 86–110 101–120 96–110	8.1% 8.3% 5.2% 4.7%	–	0.01–1.0 mg/kg 8 single points in solvent linear, r^2> 0.999	Pongsapitch, 2011, GC-ECD

GC-ECD method G [US FDA, 1996]

GC-ECD method G is the modified PAM version 1 multi-residue method as used in the USA (FDA, 1996). This method was used for the determination of cypermethrin residues in asparagus. Samples were extracted in ACN, where after the ACN extract was mixed with water, saturated NaCl and petroleum ether. The extracts were cleaned up on Florisil column and analysed with GC-ECD. The reported LOQ was 0.01 mg/kg. The individual recoveries varied between 90–96%; further details were not available. No further details were provided on calibration.

Stability of pesticide residues in stored analytical samples

Stability cypermethrin in various crops

Untreated crop samples were fortified with 0.5 mg/kg cypermethrin before storage at –18 °C for 18 months [Markle, 1994, RAN-0147]. Cypermethrin (sum of isomers) were analysed with GC-ECD (method A) at five sampling intervals. Duplicate fortifications were run for each crop at each sampling interval. The reported LOQ was 0.05 mg/kg. The results of the storage stability study are provided in Table 9.

Table 9 Stability of cypermethrin in lettuce, tomatoes, apples and soybeans after storage at -18 °C

Commodity	Fortification level (m g/kg)	Storage time (months)	% Remaining [a], n = 3 mean	range	RSD_r	Concurrent recovery mean, range, n = 2		Reference, method
lettuce	0.500	0	106	104–108	1.9	91	89–93	RAN-0147, GC-ECD
		3	99	96–102	3.1	92	92–92	
		6	99	98–100	1.2	98	95–100	
		12	97	92–102	5.2	102	100–104	
		18	99	96–104	4.2	92	90–94	
tomatoes	0.500	0	97	94–100	3.2	100	96–104	RAN-0147, GC-ECD
		3				94	92–95	
		6	99	98–100	1.2	97	97–97	
		12	99	94–102	4.2	96	91–100	
		18	105	102–110	4.4	98	97–99	
			93	90–96	3.3			
apples	0.500	0	104	102–106	1.9	89	85–93	RAN-0147, GC-ECD
		3	93	90–94	2.5	98	97–98	

Commodity	Fortification level (m g/kg)	Storage time (months)	% Remaining [a], n = 3 mean range RSD$_r$			Concurrent recovery mean, range, n = 2		Reference, method
		6	107	100–112	6.0	90	88–93	
		12	98	96–100	2.0	92	91–94	
		18	104	98–108	5.1	82	77–87	
soya beans	0.500	0	96	92–102	5.5	98	94–101	RAN-0147, GC-ECD
		3	103	102–106	2.2	825	80–83	
		6	91	88–94	3.4	87	86–88	
		12	103	98–114	8.9	98	95–101	
		18	103	98–110	5.9	78	76–81	

[a] corrected for average recovery values at each sampling interval, uncorrected data not available

USE PATTERN

Cypermethrin is registered for pest control in several countries (see also JMPR, 2008). For this evaluation, additional use patterns were submitted for citrus fruits (pomelo, grapefruit, lemon, oranges), tree nuts (hazelnut, macadamia), teas and asparagus. Only use patterns for which the original labels were provided by the manufacturer and for which the dose rates could be verified by the Meeting are listed in Table 10. In addition, the Agricultural Toxic Substances Group of the Department of Agriculture in Thailand, the Tea Research Institute of the Chinese Academy of Agricultural Sciences in China, the Ministry of Health & Family Welfare in India and Food and Agricultural Materials Inspection Centre of Japan provided information on use patterns of cypermethrin.

Table 10 Registered pre-harvest uses of cypermethrin and zeta-cypermethrin.

Crop	Country	Form	Application				PHI, days
			Method	Rate g ai/ha	Spray conc, g ai/hL	Number	
Citrus fruits							
Pomelo	Thailand	EC 62.5 g/L cypermethrin (+ EC 225 g/L phosalone)	foliar spraying	0.94–1.2 g/tree [d]	9.4–12	4 (interval 7 days)	14
Citrus fruits [b]	USA	EC 180 g/L zeta-cypermethrin	foliar spray, ground equipment foliar spray; aerial	56 (max 224 g ai/ha per season)	conc spray 30 g ai/hL dilute spray 6.0 g ai/hL aerial spray 60 g ai/hL	ns, interval at least 14 days	1
Orange	Italy	EC 15 g/L zeta-cypermethrin	foliar spray		2.3–2.6	–	14
Lemon	Italy	EC 15 g/L zeta-cypermethrin	foliar spray		2.3–2.6	–	14
Citrus fruits	Japan	WP 60 g/kg cypermethrin	high volume foliar spray		3	1–5	7
Citrus fruits	Japan	EC 60 g/L cypermethrin	high volume foliar spray		3–6	1–5	7
Stem vegetables							
Asparagus	Thailand	EC 250 g/L cypermethrin	foliar spraying	188 [d]	25	when infested	3
Tree nuts							
Tree nuts [c]	USA	EC 180 g/L zeta-cypermethrin	foliar spray, ground equipment	56 (max 280 g ai/ha	ground 60 g ai/hL aerial 300 g ai/hL	ns, interval at least 7 days	7

Crop	Country	Form	Application				PHI, days
			Method	Rate g ai/ha	Spray conc, g ai/hL	Number	
			foliar spray; aerial	per season)			
Tree nuts [c]	USA	EC 96 g ai/L zeta-cypermethrin	foliar spray, ground equipment foliar spray; aerial	22–28 (max 140 g ai/ha per season)	ground 24–30 g ai/hL aerial 120–150 g ai/hL	ns, interval at least 7 days	7
Hazelnut	Turkey	EW 100 g/L zeta-cypermethrin	ground	20	–	1	7
Macadamia	South Africa	EW 100 g/L zeta-cypermethrin	foliar spray, ground or air equipment	–	2	2–3	30
Chestnuts	Japan	WP 60 g/kg cypermethrin	high volume foliar spray		2–6	1–5	7
Walnuts	Japan	WP 60 g/kg cypermethrin			3–6	1–3	1
Teas							
Tea	China	EC 100 g ai/L cypermethrin	foliar spray	30–45	3.3–5.0	1 [a]	7
Tea	India	EC 100–250 g/L cypermethrin	foliar spray	10–63	2.5–16	1	7
Tea (green, black)	Japan	WP 60 g/kg cypermethrin	high volume foliar spray		3–6	1	14

[a] Application at growth stage: tea shoots with one bud and two leaves stage

[b] Includes: calamondin, citrus citron, chironja, tangelo, tangor, grapefruit, kumquat, lemon, lime, mandarin (tangerine), orange (sweet and sour), pomelo, satsuma mandarin.

[c] Includes: almond, beech nut, Brazil nut, butternut, cashew, chestnut, chinquapin, hazelnut, hickory nut, macadamia nut, pecan, walnut (black and English).

[d] Application rate (g ai/tree for citrus or g ai/ha for asparagus) were calculated by Thailand from spray concentration (approved label) and spray volume (additional recommendation from Department of Agriculture, Thailand)

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

The meeting received information on supervised residue trials of foliar treatments for the following crops:

Group	Commodity	Table No
Citrus fruits	Oranges	11
	Lemons	12
	Shaddocks or pomelos	13
Stem vegetables	Asparagus	14
Tree nuts	Almonds	15
	Pecans	16
Miscellaneous fodder and forage crops	Almond hulls	17
Teas	Tea (green)	18
	Tea (black)	19

Application rates were reported as zeta-cypermethrin (oranges, lemons, grapefruit, almonds and pecans) or cypermethrin (pomelo, asparagus and tea). Unquantifiable residues are shown as below the reported LOQ (e.g. < 0.05 mg/kg). Residues, application rates and spray concentrations have been rounded to two figures. Residue data are recorded unadjusted for percentage recoveries or for residue values in control samples unless stated otherwise. Where multiple samples were taken from a single plot individual values are reported unless stated otherwise. Where multiple analyses were conducted on a single sample, the average value is reported. Where results from separate plots with distinguishing characteristics such as different formulations, varieties or treatment schedules were reported, results are listed for each plot.

Residues from the trials conducted according to critical GAP have been used for the estimation of maximum residue levels (MRLs), STMR and HR values. Those results are underlined.

Citrus fruits

Information about residues of cypermethrin after foliar spraying (pre-harvest treatment) of zeta-cypermethrin was received for oranges (USA, Italy), lemons (USA, Italy), and grapefruit (USA), and after foliar spraying of cypermethrin for pomelo (Thailand).

Oranges

For oranges, supervised residue trials executed in the USA (2004) and Italy (1993) were submitted. Results are presented in Table 11. Samples from Trial 01, 02, 09, 11 were split into two equal portions of which one portion was analysed as RAC and from the other portion the peel was removed before freezing (analysed as pulp only, see Table 24). Oranges from Trial 01 were also further processed into orange juice, dried pulp and orange oil (see fate of residues in processing and Table 20).

Table 11 Residues of cypermethrin on whole oranges after pre-harvest treatment with zeta-cypermethrin

ORANGE Location, year, (variety)	Form	No	Interval (days)	g ai/ha	g ai/hL	method, timing last application	soil type	DAT	Cypermethrin (sum of isomers), mg/kg	Reference
Sebring, FL, USA 2004 (Valencia)	EC 180 g/L	4	13 14 14	56	8.3 8.2 8.1 8.2	foliar spray; 28 Jun; mature fruit	sand	1	< 0.05 , < 0.05 mean < 0.05	Trial 01 [b d]
Lake Placid, FL, USA 2004 (Hamlin)	EW 180 g/L	4	13 13 14	56	6.4 6.2 6.2 6.2	foliar spray; 22 Nov; mature fruit	sand	1	< 0.05, < 0.05 mean < 0.05	Trial 02 [b d]
Lake Placid, FL, USA 2004 (Valencia)	EW 180 g/L	4	13 14 14	56	2.6 3.3 3.4 3.3	foliar spray; 28 Jun; mature fruit	sand	1	0.14, 0.16 mean <u>0.15</u>	Trial 08 [b d]
Mount Dora, FL, USA 2004 (Hamlin)	EC 180 g/L	4	14 14 13	56	2.7 2.8 2.6 2.6	foliar spray; 16 Nov; mature fruit	sand	1	0.16, 0.16 mean <u>0.16</u>	Trial 03 [b d]
Mount Dora, FL, USA 2004 (Valencia)	EC 180 g/L	4	14 14 14	56	8.8 8.7 8.3 8.3	foliar spray; 28 Jun; mature fruit	sand	1	< 0.05, < 0.05 mean < 0.05	Trial 05 [a b d]
DeLeon Springs, FL, USA 2004 (Valencia)	EW 180 g/L	4	14 14 14	56	3.3 2.9 2.8 2.9	foliar spray; 28 Jun; mature fruit	fine sand	1	0.13, 0.14 mean <u>0.14</u>	Trial 04 [b d]
DeLeon	EW	4	14	56	8.7	foliar	fine	1	0.12, 0.13	Trial 06

ORANGE Location, year, (variety)	Form	No	Interval (days)	g ai/ha	g ai/hL	method, timing last application	soil type	DAT	Cypermethrin (sum of isomers), mg/kg	Reference
Springs, FL, USA 2004 (Navel)	180 g/L		15 12		8.5 8.0 7.9	spray; 16 Nov; mature fruit	sand	 8 15 22	mean 0.12 0.11, 0.15 mean 0.13 0.15, 0.12 mean 0.14 0.11, 0.13 mean 0.12	[b d]
Vero Beach, FL, USA 2004 (Hamlin)	EC 180 g/L	4	13 13 14	56	3.3 3.2 3.2 3.2	foliar spray; 22 Nov; mature fruit	fine sand	1	0.11, 0.16 mean 0.14	Trial 07 [b d]
Alamo, TX, USA 2004 (N-33)	EC 180 g/L	4	14 14 13	56	8.0 8.1 8.0 7.9	foliar spray; 8 Nov; 6.4 to 8.9 cm diameter	sandy clay loam	1	< 0.05, < 0.05 mean < 0.05	Trial 09 [b d]
Richgrove, CA, USA 2004 (Valencia Olinda)	EW 180 g/L	4	14 14 14	56	7.2 7.3 7.3 7.2	foliar spray; 14 Jul; BBCH89	sandy loam	1	< 0.05, < 0.05 mean < 0.05	Trial 10 [b d]
Visalia, CA, USA 2004 (Valencia Olinda)	EC 180 g/L	4	14 14 14	56	3.8 3.8 3.8 3.8	foliar spray; 08 Dec; mature fruit	sandy loam	1	0.15, 0.14 mean 0.14	Trial 11 [b d]
Reedley, CA, USA 2004 (Valencia)	EW 180 g/L	4	14 14 14	56	2.3 2.3 2.3 2.3	foliar spray; 30 Aug; 6.4 to 7.6 cm fruit	loamy sand	1	0.13, 0.13 mean 0.13	Trial 12 [b d]
Catania, Italy, 1993 (Tarocco)	EW 50 g/L	1	–	50	0.3	foliar spray; 12 Oct; mature fruit	loamy sand	0 3 7 14	0.09, 0.08 0.08, 0.08 0.07, 0.07 < 0.05, < 0.05	A-17-94-03 [c]
Catania, Italy, 1993 (Tarocco)	EW 50 g/L	1	–	100	0.5	foliar spray; 12 Oct; mature fruit	loamy sand	0 3 7 14	0.15, 0.16 0.10, 0.15 0.10, 0.07 < 0.05, < 0.05	A-17-94-03 [c]

[a] Rainfall within 6 hours after last application.

[b] Replicate field samples, the mean may be selected for MRL derivation if according to critical GAP.

[c] Based on the field data sheets provided by the manufacturer, it is assumed by that these trials were replicate field trials. Therefore, the maximum value may be selected for MRL derivations if according to cGAP

[d] Formulation EC1.5 and EW1.5 contained 1.5 lb ai/gal as answered by manufacturer to questions, 25-05-2011. In deviation of the answer of the manufacturer, it is assumed that there is no difference in active ingredient between EC and EW, so both formulations contained 180 g ai/L.

[Culligan, 2005 Trial 01 to Trial 12] No unusual weather conditions, except rainfall within 8 hours after application 3 (Trial 01, 08), within 3 and 5 hours after application 1 and 3 (Trial 04), within 3 and 6 hours after application 3 and 4 (Trial 05). Crop age between 9–25 years old. Plot size 4–6 trees per plot. Spray equipment ARA Airblast spraying (Trial 01-08), Orchard Airblast sprayer (DP 50 Air, Trial 09), Tractor-mounted Airblast sprayer (Trial 10), or Handgun sprayer (Trial 11, 12) with spray volumes 636–897 L/ha (low volume) for Trials 01, 02, 05, 06, 09, 10 and 1477–2467 L/ha (diluted) for Trials 03, 04, 07, 08, 11, 12. Each sample (mature fruit) consisted of minimum of 24 fruit from several places on at least four individual trees from all four quadrants of the tree, both inside and outside the foliage, to total at least 2 kg. In addition, bulk samples were taken from Trial 01 for processing of approximately 272 kg. Total storage at –18 °C before analysis 3–8 months. Sample analysis with GC-ECD method B [Culligan, 2005, P-3753]. LOQ = 0.05 mg/kg. Results not corrected for individual recoveries (77–92%) and controls (< 0.05 mg/kg).

[Zenide, 1994 A-17-94-03] No unusual weather conditions not stated. Plot size 10 trees/plot, 2 repeats. Back mechanic sprayer; spray volume 2000 L/ha. Sample size 20 fruits. Frozen storage for 357 days until analysis. Analysis with GC-

ECD method A [Zenide, 1994, A-17-94-03]. Individual recoveries ranged between 88 and 104%. Controls were below LOQ (< 0.05 mg/kg).

Lemon

The meeting received supervised residue trials executed in the USA (2004) and Italy (1993) for lemons. Results are presented in Table 12. Samples from Trial 14 and 15 were also split into two equal portions of which one portion was analysed as RAC and from the other portion the peel was removed before freezing (analysed as pulp only, see fate of residues in processing).

Table 12 Residues of cypermethrin on whole lemons after pre-harvest treatment with zeta-cypermethrin

LEMON Location, year, (variety)	Form	No	Interval (days)	g ai/ha	g ai/hL	Method, timing last application	Soil type	DAT	Cypermethrin (sum of isomers), mg/kg	Reference
Vera Beach, FL, USA 2004 (Bearrs)	EC 180 g/L	4	14 14 14	56	9.8 11 11 11	foliar spray; 19 Aug; mature fruit	sand	1	0.08, 0.08 mean 0.08	Trial 13 [a c]
Yuma, AZ, USA 2004 (Lisbon)	EW 180 g/L	4	6 7 7	56	11 23 12 12	foliar spray; 22 Nov; mature fruit	sand	1	0.07, 0.09 mean 0.08	Trial 14 [a,c]
Porterville, CA, USA 2004/2005 (Pryor)	EC 180 g/L	4	13 14 15	56	2.6 2.5 2.5 2.5	foliar spray; 12 Jan 2005; mature fruit	clay	1	0.08, 0.06 mean 0.07	Trial 15 [a,c]
Visalia, CA, USA 2004 (Lisbon)	EW 180 g/L	4	14 14 15	56	3.0 3.0 3.0 3.0	foliar spray; 8 Dec; mature fruit	sandy loam	1	0.05, 0.07 mean 0.06	Trial 16 [a,c]
Reedley, CA, USA 2004 (Lisbon)	EC 180 g/L	4	14 14 15	56	9.1 9.1 9.1 9.1	foliar spray; 8 Dec; mature fruit	loamy sand	1 7 14 21	0.06, 0.06 mean 0.06 0.06, 0.05 mean 0.06 0.08, 0.09 mean 0.08 0.08, 0.09 mean 0.08	Trial 17 [a,c]
Catania, Italy, 1993 (Femminello)	EW 50 g/L	1	–	50	0.3	foliar spray; 12 Oct; mature fruit	loamy sand	7 14	0.12, 0.08 0.09, 0.09	A-17-94-02 [b]
Catania, Italy, 1993 (Femminello)	EW 50 g/L	1	–	100	0.5	foliar spray; 12 Oct; mature fruit	loamy sand	7 14	0.26, 0.23 0.14, 0.10	A-17-94-02 [b]

[a] Replicate field samples, the mean may be selected for MRL derivation if according to critical GAP.

[b] Based on the field data sheets provided by the manufacturer, it is assumed by that these trials were replicate field trials. Therefore, the maximum value may be selected for MRL derivations if according to cGAP

[c] Formulation EC1.5 and EW1.5 contained 1.5 lb ai/gal as answered by manufacturer to questions, 25-05-2011. In deviation of the answer of the manufacturer, it is assumed that there is no difference in active ingredient between EC and EW, so both formulations contained 180 g ai/L.

[Culligan, 2005 Trial 13 to Trial 17] No unusual weather conditions, except rainfall within 6 and 8 hours after application 3 (Trial 15). Crop age between 8–39 years old. Plot size 4–6 trees per plot. Spray equipment ARA Airblast spraying (Trial 13, 14), Tractor-mounted Airblast sprayer (Trial 15), or Handgun sprayer (Trial 16, 17) with spray volumes 481–618 L/ha (low volume) for Trials 13, 14, 17 and 1841–2262 L/ha (diluted) for Trials 15, 16. Each sample (mature fruit) consisted of minimum of 24 fruit from several places on at least 4 individual trees from all four quadrants of the tree, both inside and outside the foliage, to total at least 2 kg. Total storage at –18 °C before analysis 2–6 months. Sample analysis with GC-ECD method B [Culligan, 2005, P-3753]. LOQ = 0.05 mg/kg. Results not corrected for individual recoveries (72–126%) and controls (< 0.05 mg/kg).

[Zenide and Françon, 1994 A-17-94-02] No unusual weather conditions. Plot size 10 plants/plot, 2 repeats. Back mechanic sprayer; spray volume 2000 L/ha Sample size 20 fruits. Frozen storage for 349 days until analysis. Analysis with GC-ECD method A [Zenide and Françon, 1994 A-17-94-02]. Individual recoveries ranged between 87 and 106%. Controls were below LOQ (< 0.05 mg/kg).

Shaddocks or pomelos

Supervised residue trials were furthermore submitted for grapefruits, for which trials were executed in the USA (2004). Samples from Trial 20 and 22 were also split into two equal portions of which one portion was analysed as RAC and from the other portion the peel was removed before freezing (analysed as pulp only, see fate of residues in processing). Supervised residue trials for pomelo were received from Thailand (2008, 2009). Results of the field trials for grapefruit and pomelo are presented in Table 13 for zeta-cypermethrin treatment and Table 14 for cypermethrin treatment. .

Table 13 Residues of cypermethrin on whole grapefruit after pre-harvest treatment with zeta-cypermethrin

GRAPEFRUIT Location, year, (variety)	Form	No	Interval (days)	g ai/ha	g ai/hL	Method, timing last application	Soil type	DAT	Cypermethrin (sum of isomers), m g/kg	Reference
DeLeon Springs, FL, USA 2004 (Grapefruit/Texas Star Ruby)	EC 180 g /L	4	14 15 12	56	3.0 3.0 2.9 2.8	foliar spray; 16 Nov; mature fruit	fine sand	1 8 15 22	0.12, 0.12 mean 0.12 0.11, 0.11 mean 0.11 0.09, 0.09 mean 0.09 0.11, 0.12 mean 0.12	Trial 18 [a,b]
Ft. Pierce, FL, USA 2004 (Grapefruit/White)	EW 180 g /L	4	13 13 14	56	6.6 6.5 6.5 6.4	foliar spray; 22 Nov; BBCH 74	sand	1	0.16, 0.15 mean 0.16	Trial 19 [a,b]
Vero Beach, FL, USA 2004 (Grapefruit/Reds Ruby)	EC 180 g /L	4	13 13 14	56	6.6 6.5 6.5 6.3	foliar spray; 22 Nov; mature fruit	fine sand	1	0.20, 0.20 mean 0.20	Trial 20 [a,b]
Alamo, TX, USA 2004 (Grapefruit/Rio Red)	EW 180 g /L	4	14 14 13	56	2.3 2.4 2.4 2.3	foliar spray; 8 Nov; 8.9 to 11 cm diameter	sandy clay loam	1	0.06, 0.07 mean 0.06	Trial 21 [a,b]
Porterville, CA, USA 2004 (Grapefruit/Mello Gold)	EC 180 g /L	4	12 14 14	56	2.5 2.6 2.6 2.6	foliar spray; 22 Nov; mature fruit	loam	1	0.05, 0.05 mean 0.05	Trial 22 [a,b]
Visalia, CA, USA 2004 (Grapefruit/Marsh)	EW 180 g /L	4	14 14 15	56	11 11 11 11	foliar spray; 8 Dec; mature fruit	sandy loam	1	0.11, 0.14 mean 0.12	Trial 23 [a,b]

[a] Replicate field samples, the mean may be selected for MRL derivation if according to critical GAP.

[b] Formulation EC1.5 and EW1.5 contained 1.5 lb ai/gal as answered by manufacturer to questions, 25-05-2011. In deviation of the answer of the manufacturer, it is assumed that there is no difference in active ingredient between EC and EW, so both formulations contained 180 g ai/L.

[Culligan, 2005 Trial 18 to Trial 23] No unusual weather conditions, except that during Trial 19 the region experienced severe weather from tropical systems and one hurricane resulting in 60% fruit loss, however with addition of 2 extra trees per plot enough for RAC trial. Crop age between 8–20 years old (unknown Trial 22). Plot size 4–6 trees per plot. Spray equipment ARA Airblast spraying (Trial 18–20), Orchard Airblast sprayer (DP 50 Air, Trial 21), Tractor-mounted Airblast sprayer (Trial 22), or Handgun sprayer (Trial 23) with spray volumes 496–884 L/ha (low volume) for Trials 19, 20, 23 and 1879–2402 L/ha (diluted) for Trials 18, 21, 22. Each sample (mature fruit) consisted of minimum of 24 fruit from several places on at least 4 individual trees from all four quadrants of the tree, both inside and outside the foliage, to total at least 2 kg. Total storage at –18 °C before analysis 2–4 months. Sample analysis with GC-ECD method B

[Culligan, 2005, P-3753]. LOQ = 0.05 mg/kg. Results not corrected for individual recoveries (90%) and controls (< 0.05 mg/kg).

Table 14 Residues of cypermethrin on whole pomelo after pre-harvest treatment with cypermethrin

POMELO Location, year, (variety)	Form	No	Interval (days)	g ai/ha	g ai/hL	Method, timing last application	Soil type	DAT	Cypermethrin (sum of isomers), mg/kg	Reference
Samut Songkhram, Thailand 2008 (Pummelo–Khao Yai)	EC 62.5 g/L	4	7 7 7	0.94 g a i/tree	9.4	foliar spray; at harvesting stage; 28 May; mature fruits	sandy loam	0 1 3 5 7 10 14 21	0.29, 0.24, 0.33 0.22, 0.19, 0.24 0.15, 0.14, 0.21 0.13, 0.12, 0.18 0.13, 0.12, 0.13 0.12, 0.10, 0.12 0.10, 0.08, 0.11 0.07, 0.08, 0.08	CY-PO-01 [a,b]
Samut Songkhram, Thailand 2008/9 (Pummelo–Khao Yai)	EC 62.5 g/L	4	7 7 7	0.94 g a i/tree	9.4	foliar spray; at harvesting stage; 13 Nov; mature fruits	sandy loam	0 1 3 5 7 10 14 21	0.71, 0.66, 0.60 0.71, 0.62, 0.58 0.37, 0.30, 0.33 0.26, 0.22, 0.25 0.22, 0.21, 0.19 0.19, 0.18, 0.15 0.16, 0.13, 0.16 0.14, 0.11, 0.12	CY-PO-04 [a,b]
Samut Songkhram, Thailand 2009 (Pummelo–Khao Yai)	EC 62.5 g/L	4	7 7 7	0.94 g a i/tree	9.4	foliar spray; at harvesting stage; 31 Jul; mature fruits	sandy loam	0 1 3 5 7 10 14 21	0.24, 0.28, 0.25 0.22, 0.24, 0.22 0.20, 0.19, 0.21 0.17, 0.16, 0.20 0.13, 0.15, 0.17 0.11, 0.11, 0.13 0.09, 0.08, 0.11 0.05, 0.05, 0.06	CY-PO-06 [a,b]
Nakorn Pathom, Thailand 2008 (Pummelo–Khao Num Pung)	EC 62.5 g/L	4	7 7 7	0.94 g a i/tree	9.4	foliar spray; at harvesting stage; 28 Jun; mature fruits	loam	0 1 3 5 7 10 14 21	0.35, 0.39, 0.42 0.26, 0.28, 0.33 0.21, 0.27, 0.28 0.17, 0.25, 0.25 0.15, 0.19, 0.23 0.11,0.17, 0.20 0.08, 0.09, 0.14 0.04, 0.06, 0.09	CY-PO-02 [a,b]
Nakorn Nayok, Thailand 2009 (Pummelo–Thong Dee)	EC 62.5 g/L	4	7 7 7	0.94 g a i/tree	9.4	foliar spray; at harvesting stage; 13 Aug; mature In table 14 fruits	loam	0 1 3 5 7 10 14 21	0.39, 0.40, 0.42 0.32, 0.36, 0.39 0.27, 0.30, 0.32 0.23, 0.25, 0.27 0.21, 0.23, 0.24 0.20, 0.21, 0.21 0.18, 0.16, 0.17 0.13, 0.11, 0.13	CY-PO-05 [a,b]
Prachinburi, Thailand 2008 (Pummelo–Thong Dee)	EC 62.5 g/L	4	7 7 7	0.94 g a i/tree	9.4	foliar spray; at harvesting stage; 5 Nov; mature fruits	loam	0 1 3 5 7 10 14 21	0.43, 0.52, 0.41 0.53, 0.60, 0.47 0.45, 0.48, 0.45 0.32, 0.41, 0.37 0.25, 0.37, 0.31 0.23, 0.29, 0.27 0.23, 0.25, 0.22 0.16, 0.18, 0.14	CY-PO-03 [a,b]

[a] Formulation with phosalone 225 g/L.

[b] Results came from 4 replicate field trials, but only 3 replicates were reported; the maximum value may be selected for MRL derivations if according to cGAP.

[Pimpan, 2008 CY-PO-01 to CY-PO-03] No unusual weather conditions. 4 plots with 1 tree (10 years of age) per plot. High pressure pump spraying, spray to run off with spray volume 10 L/tree. Whole fruit sampled with 5 fruits per sample (from each tree or plot, total 7.5–12.5 kg/tree). Samples were analysed as whole fruit. No storage. Analysis at same day as sampling with GC-ECD method F [Steinwandter, 1985]. LOQ = 0.01 mg/kg. Results were not corrected for recovery (83–108%) or control (< 0.01 mg/kg). Additional data on replicates was provided by Pongsapitch (2011).

[Pimpan, 2009 CY-PO-06] No unusual weather conditions. 4 plots with 1 tree (10 years of age) per plot. High pressure pump spraying, spray to run off with spray volume 10 L/tree. Whole fruit sampled with 5 fruits per sample (from each tree or plot, total 7.5–12.5 kg/tree). Samples were analysed as whole fruit. No storage. Analysis at same day as sampling with GC-ECD method F [Steinwandter, 1985]. LOQ = 0.01 mg/kg. Results were not corrected for recovery (87–105%) or control (< 0.01 mg/kg). Additional data on replicates was provided by Pongsapitch (2011).

[Phaikaew, 2008 CY-PO-04] No unusual weather conditions. 4 plots with 1 tree (10 years of age) per plot. High pressure pump spraying, spray to run off with spray volume 10 L/tree. Whole fruit sampled with 5 fruits per sample (from each tree or plot, total 7.5–12.5 kg/tree). Samples were analysed as whole fruit. No storage. Analysis at same day as sampling with GC-ECD method F [Steinwandter, 1985]. LOQ = 0.01 mg/kg. Results were not corrected for recovery (88–108%) or control (< 0.01 mg/kg). Additional data on replicates was provided by Pongsapitch (2011).

[Phaikaew, 2009 CY-PO-05] No unusual weather conditions. 4 plots with 1 tree (10 years of age) per plot. High pressure pump spraying, spray to run off with spray volume 10 L/tree. Whole fruit sampled with 5 fruits per sample (from each tree or plot, total 7.5–12.5 kg/tree). Samples were analysed as whole fruit. No storage. Analysis at same day as sampling with GC-ECD method F [Steinwandter, 1985]. LOQ = 0.01 mg/kg. Results were not corrected for recovery (87–105%) or control (< 0.01 mg/kg). Additional data on replicates was provided by Pongsapitch (2011).

Stem vegetables

The meeting received data on cypermethrin residues for asparagus (Thailand).

Asparagus

Supervised residue trials on green asparagus (pre-harvest treatment) were submitted by Thailand (2003, 2010). The results are provided in Table 15.

Table 15 Residues of cypermethrin on whole asparagus after pre-harvest treatment with cypermethrin

ASPARAGUS Location, year, (variety)	Form	No	Interval (days)	g ai/ha	g ai/hL	Method, timing last application	Soil type	DAT	Cypermethrin (sum of isomers), mg/kg	Reference
Nakornpratom, Thailand 2003 (Green asparagus)	EC 250 g/L	4	7 7 7	189	25	foliar spray; 8 Dec; shooting	sandy loam	0 1 3 5 7 10	0.52, 0.61, 0.60, 0.53 0.43, 0.44, 0.57, 0.43 0.14, 0.20, 0.20, 0.16 < 0.01, < 0.01, < 0.01, < 0.01 < 0.01, < 0.01, < 0.01, < 0.01 < 0.01, < 0.01, < 0.01, < 0.01	CY-AS-01 [a]
Nakornpatom, Thailand 2010 (Green asparagus)	EC 250 g/L	3	7 7	188	25	foliar spray; 11 Mar; shooting	loam	0 1 2 3 4 5 7	0.87, 0.51, 0.76 0.54, 0.35, 0.39 0.17, 0.13, 0.16 0.09, 0.04, 0.07 0.02, 0.02, 0.02 0.02, 0.01, 0.02 < 0.01, 0.01, < 0.01	CY-AS-03 [a]
Nakornpatom, Thailand 2010 (Green asparagus)	EC 250 g/L	4	7 7 7	188	25	foliar spray; 18 Apr; shooting	sandy clay loam	0 1 3 5 7 9	0.25, 0.25, 0.25 0.06, 0.05, 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	CY-AS-05 [a]
Nakornpatom, Thailand 2010 (Green asparagus)	EC 250 g/L	4	7 7 7	188	25	foliar spray; 5 Jun; shooting	loam	0 1 2	0.57, 0.59, 0.51 0.18, 0.12, 0.17 0.09, 0.06, 0.07	CY-AS-04 [a]

ASPARAGUS Location, year, (variety)	Form	No	Interval (days)	g ai/ ha	g ai/h L	Method, timing last application	Soil type	DAT	Cypermethrin (sum of isomers), mg/kg	Reference
								3	0.03, 0.02, 0.03	
								4	< 0.01, 0.01, < 0.01	
								5	0.01, 0.01, 0.01	
								7	0.01, 0.01, 0.01	
Karnjanaburi, Thailand 2003 (Green asparagus)	EC 250 g/L	4	6 7 7	19 4	25	foliar spray; 17 Dec; shooting	sandy loam	0	0.75, 0.71, 0.83, 0.79	CY-AS-02 [a]
								1	0.45, 0.40, 0.44, 0.54	
								3	0.09, 0.04, 0.06, 0.06	
								5	< 0.01, < 0.01, < 0.01, < 0.01	
								7	< 0.01, < 0.01, < 0.01, < 0.01	
								10	< 0.01, < 0.01, < 0.01, < 0.01	

[a] Results came from 4 replicate field trials, but in some cases only 3 replicates were reported. The maximum value may be selected for MRL derivations if according to cGAP.

[Dachanuraknukul, 2003 CY-AS-01 to CY-AS-02]: No unusual weather conditions. 4 plots with 360–380 plants per plot. Knapsack Sprayer, spray to run-off. Shoot (spear) sampled with 2 samples per plot, 40–50 spears per sample (1–2 kg). No storage. Analysis at same day as sampling with GC-ECD method F [Steinwandter, 1985]. LOQ = 0.01 mg/kg. Results were not corrected for recovery (104–107%) or control (< 0.01 mg/kg). Additional data on replicates was provided by Pongsapitch (2011).

[Poomongkutchai, 2010 CY-AS-03]: No unusual weather conditions, except rainfall during harvesting period trial CY-AS-05. 3 plots with 330 plants per plot (only given for CY-AS-03) in 3 rows. Knapsack Sprayer, spray to run-off. Shoot (spear) sampled with 2 samples per plot, > 12 spears per sample (at least 2 kg). No storage. Analysis at same day as sampling with GC-ECD method F [Steinwandter, 1985]. LOQ = 0.01 mg/kg. Results were not corrected for recovery (83%) or control (< 0.01 mg/kg). Additional data on replicates was provided by Pongsapitch (2011).

[Akcaboot and Chawengsri, 2010 CY-AS-04]: No unusual weather conditions, except rainfall during harvesting period trial CY-AS-05. 3 plots with 330 plants per plot (only given for CY-AS-03) in 3 rows. Knapsack Sprayer, spray to run-off. Shoot (spear) sampled with 2 samples per plot, > 12 spears per sample (at least 2 kg). No storage. Analysis at same day as sampling with GC-ECD method F [Steinwandter, 1985]. LOQ = 0.01 mg/kg. Results were not corrected for recovery (88%) or control (< 0.01 mg/kg). Additional data on replicates was provided by Pongsapitch (2011).

[Santaweesook, 2010 CY-AS-05]: No unusual weather conditions, except rainfall during harvesting period trial CY-AS-05. 3 plots with 330 plants per plot (only given for CY-AS-03) in 3 rows. Knapsack Sprayer, spray to run-off. Shoot (spear) sampled with 2 samples per plot, > 12 spears per sample (at least 2 kg). No storage. Analysis at same day as sampling with GC-ECD method G [US FDA, 1996]. LOQ = 0.01 mg/kg. Results were not corrected for recovery (90–96%) or control (< 0.01 mg/kg). Additional data on replicates was provided by Pongsapitch (2011).

Tree nuts

The meeting received data about cypermethrin residues on almonds and pecans. The supervised residue trials were conducted in the USA (2003) with foliar spraying of zeta-cypermethrin as pre-harvest treatment.

Almonds

Supervised residue trials performed in the USA were submitted for almonds (2003). The residues of cypermethrin found on almond nutmeat are represented in Table 16.

Table 16 Residues of cypermethrin on almond nutmeat after pre-harvest treatment with zeta-cypermethrin

ALMOND Location, year, (variety)	Form	No	Interval (days)	g ai/ha	g ai/hL	Method, timing last application	Soil type	DAT	Cypermethrin (sum of isomers), mg/kg	Reference
Farmersville, CA, USA 2003	EC 180 g/L	5	7 7	56	8.0 8.0	foliar spray; 21 Aug; 95% hull	fine sandy	1	< 0.05, < 0.05 mean < 0.05	Trial 01 [a,b]

ALMOND Location, year, (variety)	Form	No	Interval (days)	g ai/ha	g ai/hL	Method, timing last application	Soil type	DAT	Cypermethrin (sum of isomers), mg/kg	Reference
(Carmel)			7 7		8.0 8.0 8.0	split	loam	3 7 14	< 0.05, < 0.05 mean < 0.05 < 0.05, < 0.05 mean < 0.05 < 0.05, < 0.05 mean < 0.05	
Reedley, CA, USA 2003 (Mono)	EW 180 g/L	5	7 7 7 7	56	8.0 8.0 8.1 8.0 8.0	foliar spray; 21 Aug; 100% hull split	loamy sand	7	< 0.05, < 0.05 mean < 0.05	Trial 02 [a,b]
Poplar, CA, USA 2003 (Non-pareil)	EC 180 g/L	5	7 7 7 7	56	14 14 13 14 13	foliar spray; 5 Aug; hull split	loamy fine sand	7	< 0.05, < 0.05 mean < 0.05	Trial 03 [a,b]
Porterville, CA, USA 2003 (Padre)	EW 180 g/L	5	7 7 7 7	56	3.4 3.4 3.2 3.3 3.2	foliar spray, 2 Sep; 100% hull split	sandy loam	7	< 0.05, < 0.05 mean < 0.05	Trial 04 [a,b]
Yuba City, CA, USA 2003 (Non-pareil)	EC 180 g/L	5	7 7 7 7	56	3.7 3.7 3.7 3.7 3.7	foliar spray; 6 Aug; 100% hull split	clay loam	7	< 0.05, < 0.05 mean < 0.05	Trial 05 [a,b]

[a] Replicate field samples, the mean may be selected for MRL derivation if according to critical GAP.

[b] Formulation EC1.5 and EW1.5 contained 1.5 lb ai/gal as answered by manufacturer to questions, 25-05-2011. In deviation of the answer of the manufacturer, it is assumed that there is no difference in active ingredient between EC and EW, so both formulations contained 180 g ai/L.

[Dow, 2004 Trial 01 to Trial 05] No unusual weather conditions. Crop age between 3–27 years old. Plot size 4–6 trees per plot. Spray equipment Handgun sprayer (Trial 01, 02), Orchard Airblast sprayer (Trial 03, 04), or with Rears Pak Blast sprayer with spray volumes 409–702 L/ha (concentrated) for Trials 01–03 and 1495–1766 L/ha (diluted) for Trials 04, 05. Each sample collected at normal harvest for each commodity, shaken from trees and collected from all four quarters surrounding the trees at least 4.5 kg, almond hulls separated and collected from nutmeat. Nutmeat sample size 0.9–1.4 kg. Total storage at −18 °C before analysis 6–8 months. Sample analysis with GC-ECD method A [Dow, 2004, P-3710]. LOQ = 0.05 mg/kg. Results not corrected for individual recoveries (97–117%) and controls (< 0.05 mg/kg).

Pecans

For pecans, the Meeting received supervised residue trials conducted in the USA (2003). The residues of cypermethrin found on pecan nutmeat are represented in Table 17.

Table 17 Residues of cypermethrin on pecan nutmeat after pre-harvest treatment with zeta-cypermethrin

PECAN Location, year, (variety)	Form	No	Interval (d)	g ai/ha	g ai/hL	Method, timing last application	Soil type	DAT	Cypermethrin (sum of isomers), mg/kg	Reference
Chula, GA, USA 2003 (Sumner Sly)	EW 180 g/L	5	7 7 7 7	56	11 11 11 11	foliar spray; 14 Nov; 100% hull split	loamy sand	7	< 0.05, < 0.05 mean < 0.05	Trial 06 [a,b,c]

PECAN Location, year, (variety)	Form	No	Interval (d)	g ai/ha	g ai/hL	Method, timing last application	Soil type	DAT	Cypermethrin (sum of isomers), mg/kg	Reference
					11					
Sycamore, GA, USA 2003 (Stewart)	EC 180 g/L	5	7 7 7 7	56	3.5 3.5 3.4 3.4 3.4	foliar spray; 21 Nov; 100% mature	loamy sand	7	< 0.05, < 0.05 mean < 0.05	Trial 07 [b,c]
Greenville, MS, USA 2003 (Desirable)	EW 180 g/L	5	7 7 7 7	56	14 14 14 14 14	foliar spray; 5 Nov; 7 DBH	fine sandy loam	7	< 0.05, < 0.05 mean < 0.05	Trial 08 [b,c]
Iago, TX, USA 2003 (Oconee)	EC 180 g/L	5	6 6 8 6	56	2.9 2.9 2.9 2.9 2.9	foliar spray; 20 Oct; shuck split	silt loam	7	< 0.05, < 0.05 mean < 0.05	Trial 09 [b,c]
Rincon, NM, USA 2003 (Western Schley)	EW 180 g/L	5	7 8 6 7	56	12 11 13 12 12	foliar spray; 2 Dec; shuck split	fine sandy loam	7	< 0.05, < 0.05 mean < 0.05	Trial 10 [b,c]

[a] Rainfall within 32 hours after last application.

[b] Replicate field samples, the mean may be selected for MRL derivation if according to critical GAP.

[c] Formulation EC1.5 and EW1.5 contained 1.5 lb ai/gal as answered by manufacturer to questions, 25-05-2011. In deviation of the answer of the manufacturer, it is assumed that there is no difference in active ingredient between EC and EW, so both formulations contained 180 g ai/L.

[Dow, 2004 Trial 06 to Trial 10] No unusual weather conditions, except rainfall within 32 hours after application 5 (Trial 06), within 1 day after application 2 (Trial 10), and for Trial 09 cooler and wetter than normal however no adverse effects expected due to location on high ground and well drainage. Crop age between 6–25 years old. Plot size 4–6 trees per plot. Spray equipment ARA Airblast sprayer (Trial 06, 07), Swanson Airblast sprayer (Trial 08), Tractor/Airblast sprayer (Trial 09), or with Airblast sprayer (Trial 10) with spray volumes 398–522 L/ha (concentrated) for Trials 06, 08, 10 and 1594–1944 L/ha (diluted) for Trials 04, 05. Each sample collected at normal harvest for each commodity, shaken from trees and collected from all four quarters surrounding the trees at least 0.9 kg. Total storage at –18 °C before analysis 4–5 months. Sample analysis with GC-ECD method A [Dow, 2004, P-3710]. LOQ = 0.05 mg/kg. Results not corrected for individual recoveries (75–94%) and controls (< 0.05 mg/kg).

Miscellaneous fodder and forage crops

Residues of cypermethrin on almond hulls were analysed after pre-harvest treatment with foliar spraying of zeta-cypermethrin on almonds in the USA (2003). The results are presented in Table 18.

Table 18 Residues of cypermethrin on almond hulls after pre-harvest treatment with zeta-cypermethrin

ALMOND HULLS Location, year, (variety)	Form	No	Interval (days)	g ai/ha	g ai/hL	Method, timing last application	Soil type	DAT	Cypermethrin (sum of isomers), mg/kg	Reference
Farmersville, CA, USA 2003 (Carmel)	EC 180 g/L	5	7 7 7 7	56	8.0 8.0 8.0 8.0 8.0	foliar spray; 21 Aug; 95% hull split	fine sandy loam	1 3 7 14	3.8, 5.6 mean 4.7 2.9, 3.1 mean 3.0 2.1, 3.3 mean 2.7 2.2, 3.0 mean 2.6	Trial 01 [a,b]
Reedley, CA, USA 2003	EW 180 g/L	5	7 7	56	8.0 8.0	foliar spray; 21 Aug; 100% hull	loamy sand	7	2.8, 2.0 mean 2.4	Trial 02 [a,b]

ALMOND HULLS Location, year, (variety)	Form	No	Interval (days)	g ai/ha	g ai/hL	Method, timing last application	Soil type	DAT	Cypermethrin (sum of isomers), mg/kg	Reference
(Mono)			7 7		8.1 8.0 8.0	split				
Poplar, CA, USA 2003 (Non-pareil)	EC 180 g/L	5	7 7 7 7	56	14 14 13 14 13	foliar spray; 5 Aug; hull split	loamy fine sand	7	0.80, 1.1 mean 0.95	Trial 03 [a,b]
Porterville, CA, USA 2003 (Padre)	EW 180 g/L	5	7 7 7 7	56	3.4 3.4 3.2 3.3 3.2	foliar spray, 2 Sep; 100% hull split	sandy loam	7	2.3, 2.3 mean 2.3	Trial 04 [a,b]
Yuba City, CA, USA 2003 (Non-pareil)	EC 180 g/L	5	7 7 7 7	56	3.7 3.7 3.7 3.7 3.7	foliar spray; 6 Aug; 100% hull split	clay loam	7	0.85, 0.95 mean 0.90	Trial 05 [a,b]

[a] Replicate field samples, the mean may be selected for MRL derivation if according to critical GAP.
[b] Formulation EC1.5 and EW1.5 contained 1.5 lb ai/gal as answered by manufacturer to questions, 25-05-2011. In deviation of the answer of the manufacturer, it is assumed that there is no difference in active ingredient between EC and EW, so both formulations contained 180 g ai/L.

[Dow, 2004 Trial 01 to Trial 05] No unusual weather conditions. Crop age between 3–27 years old. Plot size 4–6 trees per plot. Spray equipment Handgun sprayer (Trial 01, 02), Orchard Airblast sprayer (Trial 03, 04), or with Rears Pak Blast sprayer with spray volumes 409–702 L/ha (concentrated) for Trials 01–03 and 1495–1766 L/ha (diluted) for Trials 04, 05. Each sample collected at normal harvest for each commodity, shaken from trees and collected from all four quarters surrounding the trees at least 4.5 kg, almond hulls separated and collected from nutmeat. Almond hulls sample size 0.9–3.2 kg. Total storage at –18 °C before analysis 6–8 months. Sample analysis with GC-ECD method A [Dow, 2004, P-3710]. LOQ = 0.05 mg/kg. Results not corrected for individual recoveries (88–113%) and controls (< 0.05 mg/kg).

Tea

The Meeting received data on cypermethrin residues in made tea (dried tea leaves as traded). The definition of made tea for China is: directly after picking the tea leaves are transported to an indoor cool place and then processed (a process of cooling and drying) within one day to the end product 'made tea'. For India, made tea is manufactured from fresh tea leaves by withering (reduction of moisture in plucked shoots), rolling, oxidation (fermentation) and drying (95–115 °C).

The results of the supervised residue trials with cypermethrin in China (2008, 2009) on made green tea and made black tea and in India (2005, 2006) on made black tea are presented in Table 19 (green tea) and Table 20 (black tea).

Table 19 Residues of cypermethrin after pre-harvest treatment with cypermethrin on made green tea (dried tea leaves)

TEA Location, year, (variety)	Form	No	Interval (days)	g ai/ha	g ai/hL	Method, timing	Soil type	DAT	Cypermethrin (sum of isomers), mg/kg	Reference
Hangzhou City, Zhejiang, China, 2008 (Longjing 43)	EC 100 g/L	1	–	30	3.3	foliar spray, 21 Apr, shoots one bud and two leaves	loamy clay	0 1 4 7 10 14	7.5, 7.5, 8.4 7.5, 7.6, 8.3 3.5, 3.4, 4.2 1.8, 1.4, 2.2 0.76, 0.45, 0.77 0.56, 0.62, 0.65	2008/A-01 [a]
Hangzhou City,	EC 100 g/	1	–	45	5.0	foliar spray, 21 Apr, shoots	loamy clay	0 1	24, 25, 26 23, 23, 21	2008/B-01 [a]

TEA Location, year, (variety)	Form	No	Interval (days)	g ai/ha	g ai/hL	Method, timing	Soil type	DAT	Cypermethrin (sum of isomers), mg/kg	Reference
Zhejiang, China, 2008 (Longjing 43)	L					one bud and two leaves		4 7 10 14	9.6, 8.6, 8.2 3.4, 3.6, 3.9 2.9, 1.7, 0.72 0.65, 0.58, 0.78	
Hangzhou City, Zhejiang, China, 2009 (Longjing 43)	EC 100 g/L	1	–	30	3.3	foliar spray, 26 Jun, shoots one bud and two leaves	loamy clay	0 1 4 7 10 14	9.2, 7.9, 9.0 7.7, 6.4, 6.8 3.3, 2.2, 2.1 2.0, 1.8, 1.7 0.43, 0.32, 0.21 0.20, 0.14, 0.08	2009/A-01 [a]
Hangzhou City, Zhejiang, China, 2009 (Longjing 43)	EC 100 g/L	1	–	45	5.0	foliar spray, 26 Jun, shoots one bud and two leaves	loamy clay	0 1 4 7 10 14	23, 23, 23 22, 21, 21 6.7, 6.3, 6.2 5.6, 5.5, 5.5 2.7, 1.9, 2.8 0.42, 0.43, 0.50	2009/B-01 [a]
Nanping City, Fujian, China, 2008 (Maoxie)	EC 100 g/L	1	–	30	3.3	foliar spray, 17 Jul, shoots one bud and two leaves	loamy clay	0 1 4 7 10 14	7.6, 8.3, 8.4 6.8, 7.0, 5.1 4.4, 4.8, 4.0 2.5, 1.6, 1.5 0.58, 0.47, 0.31 0.11, 0.05, 0.05	2008/A-03 [a]
Nanping City, Fujian, China, 2008 (Maoxie)	EC 100 g/L	1	–	45	5.0	foliar spray, 17 Jul, shoots one bud and two leaves	loamy clay	0 1 4 7 10 14	30, 26, 30 22, 19, 23 9.3, 8.4, 9.7 4.8, 4.9, 4.9 2.3, 2.2, 2.1 0.92, 0.88, 0.93	2008/B-03 [a]
Nanping City, Fujian, China, 2009 (Maoxie)	EC 100 g/L	1	–	30	3.3	foliar spray, 10 Sep, shoots one bud and two leaves	loamy clay	0 1 4 7 10 14	9.9, 11, 11 8.6, 8.7, 9.6 8.6, 7.9, 8.4 1.2, 1.1, 1.1 0.32, 0.22, 0.24 < 0.025, < 0.025, < 0.025	2009/A-03 [a]
Nanping City, Fujian, China, 2009 (Maoxie)	EC 100 g/L	1	–	45	5.0	foliar spray, 10 Sep, shoots one bud and two leaves	loamy clay	0 1 4 7 10 14	20, 22, 20 2.9, 13, 13 6.2, 5.7, 6.0 1.6, 1.6, 1.4 0.94, 0.83, 0.81 0.10, 0.15, 0.11	2009/B-03 [a]

[a] Results came from 3 replicate field trials, the maximum value may be selected for MRL derivation if according to critical GAP.

[Chen et al., 2011, trials 2008/A1, A3, B1, B3] No unusual weather conditions. 3 plots with 180 plants per plot in 10 rows. Knapsack sprayer, spray volume 900 L/ha. Fresh tea shoots containing one bud and two leaves (2 kg) collected, 1 sample per plot. Sample storage for 1 to 4 months at –20 ºC. Analysis with GC-ECD method C. Results were not corrected for recovery (88–102%) or control (< 0.025 mg/kg).

[Chen et al, 2011, trials 2009/A1, A3, B1, B3] No unusual weather conditions. 3 plots with 180 plants per plot in 10 rows. Knapsack sprayer, spray volume 900 L/ha. Fresh tea shoots containing one bud and two leaves (2 kg) collected, 1 sample per plot. Sample storage for 1 to 4 months at –20 ºC. Analysis with GC-ECD method C. Results were not corrected for recovery (88–102%) or control (< 0.025 mg/kg).

Table 20 Residues of cypermethrin after pre-harvest treatment with cypermethrin on made black tea (dried tea leaves)

TEA Location, year, (variety)	Form	No	g ai/ha	g ai/hL	Method, timing	Soil type	DAT	Cypermethrin (sum of isomers), mg/kg	Reference
Changsha City, Hunan, China, 2008 (Xiangbolù)	EC 100 g/L	1	30	3.3	foliar spray, 8 May, shoots one bud and two leaves	loamy clay	0 1 4 7 10 14	14, 15, 16 9.6, 9.4, 9.1 2.8, 3.2, 3.7 0.53, 0.65, 0.74 0.42, 0.56, 0.71 0.18, 0.13, 0.29	2008/A-02 [a]
Changsha City, Hunan, China, 2008 (Xiangbolù)	EC 100 g/L	1	45	5.0	foliar spray, 8 May, shoots one bud and two leaves	loamy clay	0 1 4 7 10 14	15, 16, 16 7.9, 8.0, 8.3 2.4, 2.4, 2.1 1.2, 1.6, 1.2 0.65, 0.53, 0.57 0.34, 0.51, 0.47	2008/B-02 [a]
Changsha City, Hunan, China, 2009 (Xiangbolù)	EC 100 g/L	1	30	3.3	foliar spray, 8 Aug, shoots one bud and two leaves	loamy clay	0 1 4 7 10 14	11, 12, 15 9.6, 11, 12 5.7, 4.7, 4.4 2.4, 2.3, 2.3 1.2, 1.4, 1.3 0.57, 0.56, 0.64	2009/A-02 [a]
Changsha City, Hunan, China, 2009 (Xiangbolù)	EC 100 g/L	1	45	5.0	foliar spray, 8 Aug, shoots one bud and two leaves	loamy clay	0 1 4 7 10 14	28, 26, 27 19, 22, 20 7.8, 7.0, 12 3.3, 3.5, 3.6 1.9, 1.7, 2.4 1.3, 1.2, 1.2	2009/B-02 [a]
Tocklai, Assam, India, 2005 (mixture of cultivars)	EC 100 g/L	1	10	2.5	foliar spray, 31 Oct, active vegetative growth (dry season)	sandy loam	0 7 14	2.3, 2.4, 2.5 0.47, 0.32, 0.40 < 0.05, < 0.05, 0.05	Barooah and Selvan, 2011 [a]
Tocklai, Assam, India, 2005 (mixture of cultivars)	EC 100 g/L	1	10	2.5	foliar spray, 22 Aug, active vegetative growth (wet season)	sandy loam	0 7 14	1.3, 0.98, 1.0 0.16, 0.14, 0.19 < 0.05, < 0.05, < 0.05	Barooah and Selvan, 2011 [a]
Tocklai, Assam, India, 2005 (mixture of cultivars)	EC 100 g/L	1	20	5.0	foliar spray, 31 Oct, active vegetative growth (dry season)	sandy loam	0 7 14 21	3.7, 4.0, 4.0 1.0, 0.93, 0.97 0.23, 0.18, 0.17 < 0.05, < 0.05, 0.05	Barooah and Selvan, 2011[a]
Tocklai, Assam, India, 2005 (mixture of cultivars)	EC 100 g/L	1	20	5.0	foliar spray, 22 Aug, active vegetative growth (wet season)	sandy loam	0 7 14 21	2.8, 2.6, 2.6 0.67, 0.54, 0.51 0.09, 0.08, 0.06 < 0.05, < 0.05, < 0.05	Barooah and Selvan, 2011 [a]
Valparai, Tamil Nadu, India, 2006 (mixed clones)	EC 250 g/L	1	63	16	foliar spray, 23 Jun, active vegetative growth (wet season)	sandy loam	0 7 10 14	12 2.0 0.83 < 0.1	Barooah and Selvan, 2011
Valparai, Tamil Nadu, India, 2006 (mixed clones)	EC 250 g/L	1	63	16	foliar spray, 22 Sep, active vegetative growth (dry season)	sandy loam	0 7 10 14	13 1.1 0.36 < 0.1	Barooah and Selvan, 2011
Valparai, Tamil Nadu, India, 2006 (mixed clones)	EC 250 g/L	1	126	32	foliar spray, 23 Jun, active vegetative growth (wet season)	sandy loam	0 7 10 14	22 3.3 2.0 < 0.1	Barooah and Selvan, 2011
Valparai, Tamil Nadu, India, 2006 (mixed clones)	EC 250 g/L	1	126	32	foliar spray, 22 Sep, active vegetative growth (dry season)	sandy loam	0 7 10 14	22 1.7 0.83 0.63	Barooah and Selvan, 2011

[a] Results came from replicate field trials; the maximum value may be selected for MRL derivation if according to critical GAP.

[Chen et al, 2011, trials 2008/A2, B2] No unusual weather conditions. 3 plots with 180 plants per plot in 10 rows. Knapsack sprayer, spray volume 900 L/ha. Fresh tea shoots (2 kg) collected, 1 sample per plot. Sample storage for 1 to 4 months at
–20 ºC. Analysis with GC-ECD method C. Results were not corrected for recovery (88–102%) or control (< 0.025 mg/kg).

[Chen et al., 2011, trials 2009/A2, B2] No unusual weather conditions. 3 plots with 180 plants per plot in 10 rows. Knapsack sprayer, spray volume 900 L/ha. Fresh tea shoots (2 kg) collected, 1 sample per plot. Sample storage for 1 to 4 months at –20 ºC. Analysis with GC-ECD method C. Results were not corrected for recovery (88–102%) or control (< 0.025 mg/kg).

[Barooah and Selvan, 2009] Dry or wet season. Plot size 77–100 m^2 with 72–100 plants/plot. Backpack sprayer, high volume spray 400 L/ha. Green leaf samples (minimum of 70% two leaves and a bud) from each of treated plots of about 1-2 kg was collected from fresh harvest. Tea leaves manufactured to normal black tea. For residue estimation, 0.2 kg was used. Samples immediately extracted upon receipt in laboratory, no extensive storage prior to analysis. Samples from Tocklai were analysed by GC-ECD method D and samples from Valparai were analysed by GC-ECD method E. Results were not corrected for concurrent recoveries (90–94%) or control (< 0.05 mg/kg).

FATE OF RESIDUES IN STORAGE AND PROCESSING

In storage

The Meeting received data on the fate of residues during storage of made tea samples (dried tea leaves as traded) at both room temperature (25 °C) and cold storage (4 °C).

During the tea growing stage, tea plants were treated with cypermethrin in an EC formulation (100 g ai/L) assumed at 3.3 g ai/hL (sample 1) and 5.0 g ai/hL (sample 2), respectively [Chen *et al.,* 2011]. Tea leaves were collected after 0 days (sample 1) or 4 days (sample 2) and manufactured to made tea with initial cypermethrin contents of 3.6 mg/kg and 13 mg/kg, respectively. The samples were stored at both room temperature (25 °C) and cold storage (4 °C). The results and degradation rates are presented in Table 21.

Table 21 Decrease of cypermethrin residues in made tea (dried tea leaves) during storage conditions

	Content of cypermethrin in made tea (mg/kg)	Storage conditions	Content of cypermethrin residue in made tea (mg/kg)				Degradation rate over 4 months
			Aug.20 (one month after storage)	Sep.20 (two months after storage)	Oct.20 (three months after storage)	Nov.20 (four months after storage)	
Tea Sample No. 1	3.6	Room temperature (25 °C)	3.6	3.5	3.4	3.4	5.6%
		Cold storage (4 °C)	3.6	3.6	3.5	3.5	3.5%
Tea Sample No. 2	13	Room temperature (25 °C)	13	13	13	12	3.3%
		Cold storage (4 °C)	13	13	13	13	2.1%

IN PROCESSING

The Meeting received data on the on the magnitude of residues for processing of oranges and made tea.

Processing study on oranges

A processing study was conducted on oranges as part of the field trials in the USA [Culligan, 2005, P-3753]. Orange trees were treated four times with an EC formulation (182 g/L) at a rate of 56 g ai/ha. Further details can be found in Table 11 (trial 1). Mature fruits were collected 1 day after treatment and stored for less than 1 month at 2–7 °C until processing. Orange samples were processed into dried pulp, orange oil and pasteurized orange juice according to Figure 1 simulating commercial practices.

Cleaned fruit was put into an extractor for production of three product streams: raw juice with some solids, oil-in-water emulsion with some solids and solid components.

Preparation of Orange juice

Solids in raw juice were removed and added to the solid fraction. After Brix and pH adjustment, the fresh juice was pasteurised by heating to 95 °C.

Preparation of orange oil

Oil-in-water emulsion was sieved to remove the solids which were added to the solid fraction. The emulsion was then centrifuged to reduce water content and provide clarified peel oil.

Preparation of orange dried pulp

The insoluble solids were dewatered and milled to produce a consistently sized product. Thereafter, the solids were dried at 99 °C to 107 °C.

Information on storage of the processed products was not available. The results of the extraction of zeta-cypermethrin from the processed products of oranges analysed with GC-ECD method B are presented in Table 22. Processing factors could not be determined as the zeta-cypermethrin residue was below the LOQ for the whole fruit.

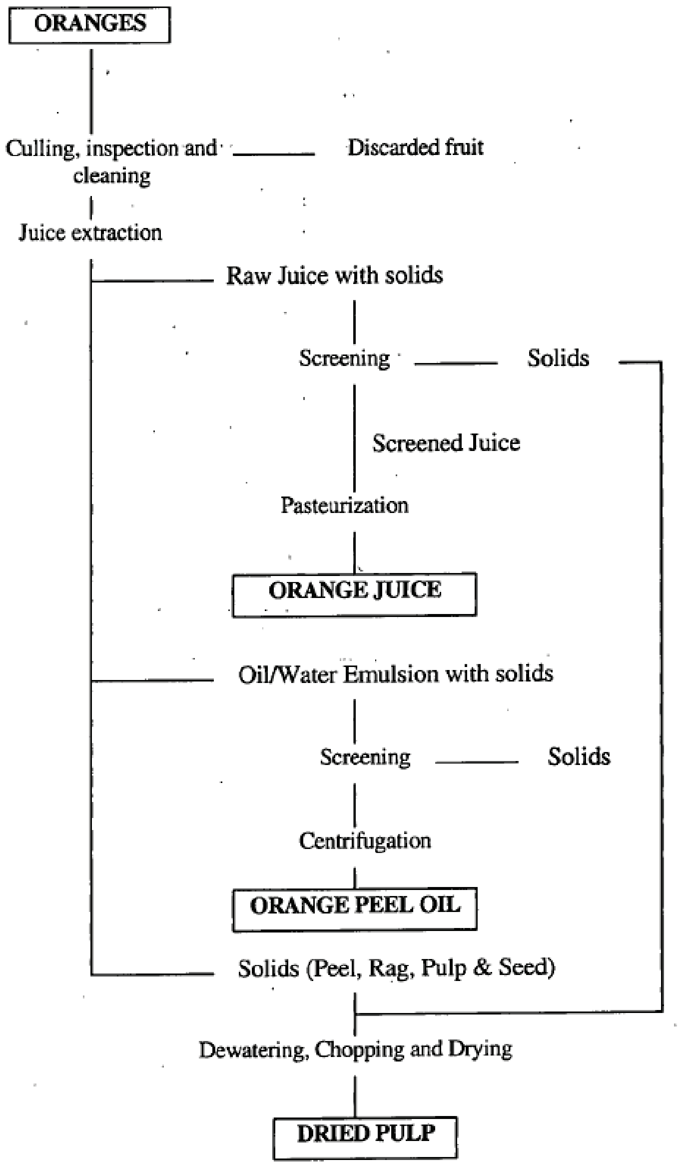


Figure 1 Processing of oranges into orange juice, orange oil and dried pulp.

Table 22 Residues of cypermethrin after processing of oranges

Location, year, (variety)	Treatment	DAT	Processed products	Cypermethrin (sum of isomers), mg/kg	PF	Reference
Sebring, FL, USA 2004 (Orange/Valencia)	foliar spraying, EC 180 g/L, 4× 56 g ai/ha	1	whole fruit (RAC) dried pomace orange oil pasteurized juice	< 0.05, < 0.05 0.32, 0.22 0.50, 0.63 < 0.05, < 0.05	–	Trial 01 [a]

[a] Results came from two replicate field samples. Since each field sample was processed independently, results are considered as coming from two independently processed samples.

[Culligan, 2005 Trial 01] LOQ< 0.05 mg/kg. Results were not corrected for individual recoveries (77–92% for whole orange, 106% for dried pulp, 89–122% for orange oil, 74% for orange juice) or controls (< 0.05 mg/kg).

Processing study on made tea

Made tea leaves (dried tea leaves as traded) are not consumed raw. They are used for preparations of infusions in hot water and only the liquor (called brew) is used for consumption. Data on cypermethrin residues after processing of made tea to tea brew was submitted by China and India.

For China [Chen *et al.*, 2011], tea plants were pre-treated as described before (see supervised trials in tea) and taken at day 0 (samples 1–4 from Fujian province) and day 1 (samples 5–8 from Hunan province) and prepared into green and black made tea samples. The tea infusion was made by blending 3.0 g crushed made tea in 150 mL bathing water for 20 minutes, where after this was repeated twice (3.0 g tea in 450 mL water).

For India [Barooah and Selvan, 2009] the tea infusion was made by brewing 3 g made tea (7 day PHI) sample in 150 ml of boiling hot water for 5–6 mins as per standard of the International Organisation for Standardization (ISO) for organoleptic testing of teas for preparation of infusion. Samples were analysed using GC-ECD method D (LOQ 0.05 mg/kg) for Tocklay and GC-ECD method E (LOQ 0.1 mg/k) for Valparai samples.

The results of the processing studies together with the processing factors are presented in Table 23 (green tea) and Table 24 (black tea).

Table 23 Residues of cypermethrin after processing of made tea (dried green tea leaves)

Location, year, (variety)	Treatment	DAT	Processed products	Cypermethrin (sum of isomers), mg/kg	PF	Reference
Green tea No. 1	foliar spraying 3.3 g ai/hL	0	made tea (RAC) tea infusion	11 0.10	0.0088	[Chen *et al,* 2011] [a]
Green tea No. 2	foliar spraying 3.3 g ai/hL	0	made tea (RAC) tea infusion	11 0.12	0.011	[Chen *et al*, 2011] [a]
Green tea No. 3	foliar spraying 3.3 g ai/hL	0	made tea (RAC) tea infusion	11 0.090	0.0083	[Chen *et al,* 2011] [a]
Green tea No. 4	foliar spraying 3.3 g ai/hL	0	made tea (RAC) tea infusion	11 0.090	0.0085	[Chen *et al,* 2011] [a]

[a] For calculation of processing factors (PF), cypermethrin residues were not rounded.

Table 24 Residues of cypermethrin after processing of made tea (dried black tea leaves)

Location, year, (variety)	Treatment	DAT	processed products	cypermethrin (sum of isomers), mg/kg	PF	reference
Black tea No. 5	foliar spraying 5.0 g ai/hL	1	made tea (RAC) tea infusion	8.4 0.084	– 0.010	[Chen *et al,* 2011] [a]
Black tea No. 6	foliar spraying 5.0 g ai/hL	1	made tea (RAC) tea infusion	7.9 0.068	– 0.0086	[Chen *et al,* 2011] [a]
Black tea No. 7	foliar spraying 5.0 g ai/hL	1	made tea (RAC) tea infusion	8.0 0.11	– 0.013	[Chen *et al,* 2011] [a]
Black tea No. 8	foliar spraying 5.0 g ai/hL	1	made tea (RAC) tea infusion	8.1 0.086	– 0.011	[Chen *et al,* 2011] [a]
Black tea, Tocklai dry season	foliar spraying 10 g ai/ha	7	made tea (RAC) tea infusion	0.39 < 0.05	– < 0.13	[Barooah and Selvan, 2009] [a]
Black tea, Valparai wet season	foliar spraying 10 g ai/ha	7	made tea (RAC) tea infusion	1.1 < 0.1	– < 0.089	[Barooah and Selvan, 2009] [a]

[a] For calculation of processing factors (PF), cypermethrin residues were not rounded.

Processing studies summary

An overview of calculated processing factors for black tea and green tea is given in Table 25. Since the datasets with processing factors are similar, the average value of all the processing factors is taken forward for calculations.

Table 25 Overview of calculated processing factors

Commodity	Processed fraction	Processing factors (n = 4)	Best estimate
Tea (black)	Tea infusion (3 g/450 ml, 20 min)	0.0083, 0.0085, 0.0088, 0.011	0.0099
Tea (green)	Tea infusion (3 g/450 ml, 20 min)	0.0086, 0.010, 0.011, 0.013	
Tea (black)	Tea infusion (3 g/150 ml, 5 min)	< 0.089, < 0.13	

Residues in the edible portion of food commodities

The meeting received data on the fate of zeta-cypermethrin residues in citrus fruits (oranges, lemons and grapefruit) analysed as pulp only [Culligan, 2005 P-3753]. Samples from eight trials (four oranges, two lemons and two grapefruit) in the study were shipped ambient to remove the peel before freezing the sample. For more details on the study see comments to Table 11, Table 12 and Table 13. The zeta-cypermethrin residues found in oranges, lemons and grapefruit pulp are provided in Table 26.

Table 26 Residues of cypermethrin on orange, lemon or grapefruit pulp after pre-harvest treatment with zeta-cypermethrin.

Location, year, (variety)	Form	No	Interval (days)	g ai/ha	g ai/hL	Method, timing	Soil type	DAT	Cypermethrin (sum of isomers), mg/kg	Reference
Sebring, FL, USA 2004 (Orange/Valencia)	EC 180 g/L	4	13 14 14	56	8.3 8.2 8.1 8.2	foliar spray; 18 May; mature fruit	sand	1	< 0.05, < 0.05 mean < 0.05	Trial 01 [a,b]
Lake Placid, FL, USA 2004 (Orange/Hamlin)	EW 180 g/L	4	13 13 14	56	6.4 6.2 6.2 6.2	foliar spray; 13 Oct; mature fruit	sand	1	< 0.05, < 0.05 mean < 0.05	Trial 02 [a,b]
Alamo, TX, USA 2004 (Orange/N-33)	EC 180 g/L	4	14 14 13	56	8.0 8.1 8.0 7.9	foliar spray; 28 Sep; mature fruit	sandy clay loam	1	< 0.05, < 0.05 mean < 0.05	Trial 09 [a,b]
Visalia, CA, USA 2004 (Orange/Valencia Olinda)	EC 180 g/L	4	14 14 14	56	3.8 3.8 3.8 3.8	foliar spray; 26 Oct; mature fruit	sandy loam	1	< 0.05, < 0.05 mean < 0.05	Trial 11 [a,b]
Yuma, AZ, USA 2004 (Lemon/Lisbon)	EW 180 g/L	4	6 7 7	56	11 23 12 12	foliar spray; 2 Nov; mature fruit	sand	1	< 0.05, < 0.05 mean < 0.05	Trial 14 [a,b]
Porterville, CA, USA 2004/2005 (Lemon/Pryor)	EC 180 g/L	4	13 14 15	56	2.6 2.5 2.5 2.5	foliar spray; 1 Dec; mature fruit	clay	1	< 0.05, < 0.05 mean < 0.05	Trial 15 [a,b]
Vero Beach, FL, USA 2004 (Grapefruit/Reds Ruby)	EC 180 g/L	4	13 13 14	56	6.6 6.5 6.5 6.3	foliar spray; 13 Oct; mature fruit	fine sand	1	< 0.05, < 0.05 mean < 0.05	Trial 20 [a,b]
Porterville, CA, USA 2004 (Grapefruit/Mello Gold)	EC 180 g/L	4	12 14 14	56	2.5 2.6 2.6 2.6	foliar spray; 13 Oct; mature	loam	1	< 0.05, < 0.05 mean < 0.05	Trial 22 [a,b]

Location, year, (variety)	Form	No	Interval (days)	g ai/ha	g ai/hL	Method, timing	Soil type	DAT	Cypermethrin (sum of isomers), mg/kg	Reference
						fruit				

[a] Replicate field samples, the mean may be selected for MRL derivation if according to critical GAP.

[b] Formulation EC1.5 and EW1.5 contained 1.5 lb ai/gal as answered by manufacturer to questions, 25-05-2011. In deviation of the answer of the manufacturer, it is assumed that there is no difference in active ingredient between EC and EW, so both formulations contained 180 g ai/L.

NATIONAL RESIDUE DEFINITIONS

The residue definition for the United States is zeta-cypermethrin. The Thailand residue definition is total cypermethrin. Also the residue definition for China and India is total cypermethrin.

APPRAISAL

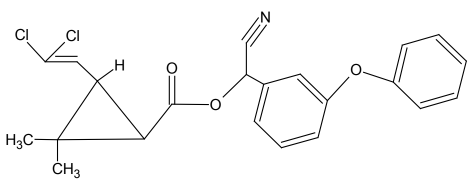
Cypermethrins was evaluated by JMPR 1979 (T, R), 1981 (T, R), 1982 (R), 1983 (R), 1984 (R), 1985 (R), 1986 (R), 1987 (corr. to 1986 evaluation), 1988 (R), 1990 (R), 2006 (T), 2008 (R) and 2009 (R). The last periodic review for toxicology was in 2006 and for residues in 2008 and included cypermethrin, alpha-cypermethrin and zeta-cypermethrin. The 2006 Meeting estimated the acceptable daily intake (ADI) as 0–0.02 mg/kg bw and estimated the acute reference dose (ARfD) as 0.04 mg/kg bw. The 2008 Meeting defined the residue (for compliance with the MRL and for estimation of dietary intake) for plant and animal commodities as cypermethrin (sum of isomers). The residue is fat soluble. In 2009 additional information on the use of cypermethrin was submitted and evaluated. Cypermethrin was listed by the Forty-second Session of the CCPR[2] for the evaluation of 2011 JMPR for additional maximum residue levels.

The Meeting received information on zeta-cypermethrin from the manufacturer on storage stability, residue analysis, use patterns, residues resulting from supervised trials on citrus fruits and tree nuts, and fates of residue during processing. In addition, the Meeting received information on cypermethrin on residue analysis, use patterns, and residues resulting from supervised trials on asparagus and pomelo from Thailand. China and India submitted information on cypermethrin about storage stability, residue analysis, use patterns, residues resulting from supervised trials on teas, and on fates of residue during processing (China). Furthermore, the Meeting received information from Japan on use patterns of cypermethrin.

Chemical name

(Zeta)-Cypermethrin or (*RS*)-α-cyano-3-phenoxybenzyl-(1*RS*,3*RS*;1*RS*,3*RS*)-3-(2,2-dichlorovinyl)-2,2-dimethylcyclopropanecarboxylate or (*RS*)-α-cyano-3-phenoxybenzyl-(1*RS*)-cis-trans-3-(2,2-dichlorovinyl)-2,2-dimethylcyclopropanecarboxylate

Structural formula:



Cypermethrin and zeta-cypermethrin are similar pyrethroid insecticides with the same basic chemical formula and molecular weight. Both products are mixtures of eight individual isomers that only differ in the structural orientation of their chemical bonds.

[2] ALINORM 10/33/24

Analytical methods

The Meeting received description and validation data for analytical methods of cypermethrin and zeta-cypermethrin.

The extended revision of GC-ECD/GC-MS method DFG S19 was submitted to the Meeting as multiresidue enforcement method. This method was already shown to be valid for the determination of cypermethrin in various plant matrices. The method is now shown to be valid for the determination of zeta-cypermethrin in plant material with high water content and/or high acid content (LOQ = 0.01 mg/kg).

In addition, seven GC-ECD analytical methods were received for use in the supervised residue trials, processing studies and storage stability studies for the determination of cypermethrin or zeta-cypermethrin in plant material (LOQ varied between 0.01, 0.025 and 0.05 mg/kg). The Meeting noted that for two of the methods recoveries were acceptable, but there were only a limited number of samples (1–2 per concentration level).

Stability of pesticide residues in stored analytical samples

The Meeting received information on the stability of cypermethrin in plant commodities stored frozen. The 2008 Meeting considered storage stability studies of cypermethrin and zeta-cypermethrin mutually supportive.

In additional studies provided for the present Meeting, cypermethrin was shown to be stable when stored at -18 °C for at least 18 months in crops with high water content (apple, lettuce and tomatoes) and high oil content (soya beans). Storage at +25 and +4 °C showed no degradation of cypermethrin residues in tea samples (dried tea leaves as traded) for a period of at least 4 months. Since the cypermethrins do not dissociate in water, storage stability on crops with high water content can be extrapolated to crops with high acid content.

Samples from supervised residue trials on citrus fruits, asparagus and tea were either analysed directly after harvest or were analysed within the storage stability periods indicated above.

Results of supervised trials on crops

The Meeting received supervised trials data for zeta-cypermethrin on oranges, lemons, grapefruit, almonds and pecans, and for cypermethrin on pomelo, asparagus and tea.

In 2008 trials were available for alpha-cypermethrin on citrus fruits, almonds and tea but no suitable GAP was available to evaluate them. Since no suitable GAP is available in 2011, these trials cannot be used here and they will not be mentioned in the text below.

The recommendations proposed by the Meeting were verified using the OECD MRL calculator. For all trials the outcome of the OECD MRL calculator agreed with the recommendation made by the Meeting.

Citrus fruits

Field trials for zeta-cypermethrin or cypermethrin treatment on shaddocks and pomelos were conducted in the Thailand (pomelo) and the USA (grapefruit).

Critical GAP for cypermethrin on pomelos in Thailand is for four foliar spray applications at 12 g ai/hl at a 7-day interval and PHI 14 days. In trials from Thailand (4 × 9.4 g hL, PHI 14 days) matching this GAP cypermethrin (sum of isomers) residues in pomelo whole fruit were 0.11, 0.11, 0.14, 0.16, 0.18 and 0.25 mg/kg (n = 6).

Critical GAP for zeta-cypermethrin on citrus fruits in the USA is for an unspecified number of ground or aerial applications to foliage at 56 g ai/ha (max 224 g ai/ha per season) with an interval of at least 14 days and PHI 1 day. In trials from the USA (4 × 56 g ai/ha, PHI 1 day, interval 14 days) matching this GAP cypermethrin (sum of isomers) residues in grapefruit whole fruit were 0.05, 0.06, 0.12, 0.12, 0.16 and 0.20 mg/kg (n = 6). In two of the trials residues were also measured in the pulp and showed that cypermethrin (sum of isomers) residues were < 0.05 (2) mg/kg.

Since the GAPs for Thailand and the USA are different, the data on grapefruit and pomelo cannot be combined. Since the highest residue is found in the Thai dataset, the Meeting agreed to use the Thai dataset for pomelo. The Meeting estimated a maximum residue level of 0.5 mg/kg for shaddocks and pomelos and an STMR of 0.05 mg/kg and an HR of 0.05 mg/kg based on the residues in the pulp from trials in the USA.

Field trials with zeta-cypermethrin treatment on oranges were conducted in the USA and Italy.

Critical GAP for zeta-cypermethrin on citrus fruits in the USA is for an unspecified number of ground or aerial applications to foliage at 56 g ai/ha (maximum of 224 g ai/ha per season) with an interval of at least 14 days and PHI 1 day. In trials from the USA (4 × 56 g ai/ha, PHI 1 day, interval 14 days) matching this GAP cypermethrin (sum of isomers) residues in orange whole fruit were < 0.05, < 0.05, < 0.05, < 0.05, < 0.05, 0.13, 0.14, 0.14, 0.14, 0.14, 0.15 and 0.16 mg/kg (n = 12). Where residues were higher at longer PHIs these were selected instead. In four of the trials residues were also measured in the pulp and showed that cypermethrin (sum of isomers) residues were < 0.05 (4) mg/kg.

No GAP matched the field trial conducted in Italy.

Field trials with zeta-cypermethrin treatment on lemons were conducted in the USA and Italy.

Critical GAP for zeta-cypermethrin on citrus fruits in the USA is for an unspecified number of ground or aerial applications to foliage at 56 g ai/ha (maximum 224 g ai/ha per season) with an interval of at least 14 days and PHI 1 day. In trials from the USA (4 × 56 g ai/ha, PHI 1 day, interval 14 days) matching this GAP cypermethrin (sum or isomers) residues in lemon whole fruit were 0.06, 0.07, 0.08, 0.08 and 0.08 mg/kg (n = 5). In two of the trials residues were also measured in the pulp and showed that cypermethrin (sum of isomers) residues were < 0.05 (2) mg/kg.

No GAP matched the field trial conducted in Italy.

The Meeting noted that the datasets for oranges and lemons matching USA GAP for citrus fruit were from similar populations (Mann-Whitney U test). Since residue behaviour within the citrus fruit group is expected to be similar, the Meeting agreed that the datasets for oranges and lemons could be combined to estimate a maximum residue level for citrus fruits, except shaddocks and pomelos. Cypermethrin residues in oranges and lemons (whole fruit) were: < 0.05, < 0.05, < 0.05, < 0.05, < 0.05, 0.06, 0.07, 0.08, 0.08, 0.08, 0.13, 0.14, 0.14, 0.14, 0.14, 0.15 and 0.16 mg/kg (n = 17).

Based on the dataset matching the GAP of the USA, the Meeting estimated a maximum residue level of 0.3 mg/kg for citrus fruits, except shaddocks and pomelos. The Meeting estimated an STMR of 0.05 mg/kg and an HR of 0.05 mg/kg based on residues in the pulp from trials in the USA.

Stem vegetables

The JMPR 2008 Meeting estimated an STMR value and an HR value of 0.01 and 0.01 mg/kg, respectively, for cypermethrin residues in asparagus. The estimated maximum residue level was 0.01* mg/kg for asparagus. These estimations were based on seven alpha-cypermethrin trials on asparagus in France with conditions in line with German GAP for asparagus (0.0125 kg ai/ha) and no specified PHI). In addition, Thailand provided data in 2008 on two asparagus trials with cypermethrin treatments. However, these trials were considered insufficient for estimating a maximum residue level.

Additional field trials for cypermethrin treatments to asparagus were conducted in Thailand with the same GAP as the trials provided to the 2008 JMPR Meeting.

Critical GAP for asparagus in Thailand is for an unspecified number of foliar spray applications at 25 g ai/hL and a PHI of 3 days. In trials from Thailand (2–3 × 25 g ai/hL, PHI 3 days) matching this GAP from both the 2008 and the 2011 datasets cypermethrin (sum of isomers) residues in green asparagus were: 0.01, 0.03, 0.06, 0.09, 0.09, 0.18 and 0.20 mg/kg (n = 7).

Since the GAP from Thailand resulted in higher residues than the GAP of Germany, the Meeting decided to withdraw the previous maximum residue level recommendation. Based on the dataset matching the Thai GAP, the Meeting estimated a maximum residue level of 0.4 mg/kg for asparagus to replace the previous recommendation of 0.01* mg/kg. The Meeting estimated an STMR of 0.09 mg/kg and an HR of 0.20 mg/kg.

Tree nuts

Field trials for zeta-cypermethrin treatment on almonds were conducted in the USA.

Critical GAP for zeta-cypermethrin on tree nuts in the USA is for an unspecified number of ground or aerial foliar applications at 56 g ai/ha (maximum of 280 g ai/ha per season) with a treatment interval of 7 days and a PHI of 7 days. In trials from the USA (5 × 56 g ai/ha, PHI 7 days, interval 7 days) matching this GAP cypermethrin (sum of isomers) residues in almond nutmeat were: < 0.05, < 0.05, < 0.05, < 0.05 and < 0.05 mg/kg (n = 5).

Field trials for zeta-cypermethrin treatment on pecans were conducted in the USA.

Critical GAP for zeta-cypermethrin on tree nuts in the USA is for an unspecified number of ground or aerial foliar applications at 56 g ai/ha (maximum of 280 g ai/ha per season) with an interval of 7 days and a PHI of 7 days. In trials from the USA (5 × 56 g ai/ha, PHI 7 days, interval 7 days) matching this GAP cypermethrin (sum of isomers) residues in pecan nutmeat were: < 0.05, < 0.05, < 0.05, < 0.05 and < 0.05 mg/kg (n = 5).

The Meeting agreed that the USA data sets for almonds and pecans are similar and could be combined to estimate a maximum residue level for tree nuts. The combined dataset resulted in the following residues: < 0.05 (10) mg/kg.

Based on the dataset for almonds and pecans. matching USA GAP, the Meeting estimated a maximum residue level of 0.05* mg/kg for tree nuts and an STMR value of 0.05 mg/kg and an HR value of 0.05 mg/kg for tree nuts.

Miscellaneous fodder and forage crops

Field trials for zeta-cypermethrin treatment on almond hulls were conducted in the USA.

Critical GAP for zeta-cypermethrin on tree nuts in the USA is for an unspecified number of ground or aerial foliar applications at 56 g ai/ha (maximum of 280 g ai/ha per season) with an interval of 7 days and PHI 7 days. In trials from the USA (5 × 56 g ai/ha, PHI 7 days, interval 7 days) matching this GAP cypermethrin (sum of isomers) residues in almond hulls were: 0.90, 0.95, 2.3, 2.4 and 2.7 mg/kg, as received (n = 5).

Based on these data, the Meeting estimated a median value of 2.3 mg/kg.

Teas

Field trials for cypermethrin treatment on tea were conducted in China and India. Directly after picking the fresh tea leaves, the tea was processed into dried tea leaves as traded. Residues listed here are for the processed tea.

Critical GAP for cypermethrin on tea in China is for one spray application at 45 g ai/ha and a PHI of 7 days. In trials from China (45 g ai/ha, PHI 7 days) matching this GAP cypermethrin (sum of isomers) residues in green processed tea were: 1.6, 3.9, 4.9 and 5.6 mg/kg (n = 4). In trials from China matching this GAP, cypermethrin (sum of isomers) residues in black processed tea were 1.6 and 3.6 mg/kg (n = 2). The Meeting agreed that the data sets for green and black processed tea are similar and could be combined to form the following dataset for green and black processed tea: 1.6, 1.6, 3.6, 3.9, 4.9 and 5.6 mg/kg (n = 6).

Critical GAP for cypermethrin on tea in India is for one foliar spray application at 63 g ai/ha and a PHI of 7 days. In trials from India (63 g ai/ha, PHI 7 days) matching this GAP cypermethrin residues in black processed tea were 1.1 and 2.0 mg/kg (n = 2).

Since the highest residue is found in the Chinese dataset, the Meeting decided to use the tea data corresponding to the GAP of China. Residues for green and black processed tea were: 1.6, 1.6, 3.6, 3.9, 4.9 and 5.6 mg/kg (n = 6).

Based on the dataset for green and black processed tea matching Chinese GAP, the Meeting estimated a maximum residue level of 15 mg/kg for tea, green, black (black, fermented and dried) and an STMR value of 3.75 mg/kg.

Fate of residues in storage

The effect of storage on cypermethrin residues in processed tea (dried tea leaves as traded) at +4 °C and +25 °C on cypermethrin residues was investigated by determination of degradation rates. After 4 months of storage the degradation rates were 3.3–5.6% for storage at +25 °C and 2.1–3.5% for storage at +4 °C.

The Meeting, therefore, concluded that storage for 4 months at room temperature or under cold storage did not influence the fate of cypermethrin residues in processed tea.

Fate of residues during processing

In the 2008 Report of the JMPR it was shown that alpha-cypermethrin and cypermethrin residues were stable during hydrolysis conditions simulating pasteurization, baking, brewing and boiling. The 2008 JMPR Meeting calculated processing factors for a number of food processes. Additional processing studies with (zeta-) cypermethrin were submitted for oranges and tea in the 2011 Meeting.

Cypermethrin (sum of isomers) residue levels in dried orange pomace (0.22 and 0.32 mg/kg) and orange oil (0.50 and 0.63 mg/kg) were higher than in the corresponding RAC (< 0.05 mg/kg). Processing factors could not be derived for the processing of oranges in orange juice, orange oil or dried pulp (pomace) as cypermethrin (sum of isomers) residues in the RAC were below the LOQ (< 0.05 mg/kg).

For the processing of tea (dried tea leaves as traded) into tea infusion, processing factors could be derived. In the table below relevant processing factors for tea infusion are summarized. The STMR-P is calculated as $STMR_{RAC}$ × processing factor.

Commodity	Processed fraction	Processing factors (n = 4)	Best estimate
Tea (black)	Tea infusion (3 g/450 mL, 20 min)	0.0083, 0.0085, 0.0088, 0.011	0.0099
Tea (green)	Tea infusion (3 g/450 mL, 20 min)	0.0086, 0.010, 0.011, 0.013	
Tea (black)	Tea infusion (3 g/150 mL, 5 min)	< 0.089, < 0.13	

Livestock dietary burden

For estimating the livestock dietary burden, relevant trials were received by the present Meeting involving almond hulls. These feed commodities are additional to the feed commodities already taken into account at the 2008 and 2009 JMPR Meetings. The present Meeting estimated the dietary burden of cypermethrin residues on the basis of the 2009 livestock diets as listed in the FAO manual appendix IX (OECD feedstuff table). Calculation from highest residue, STMR (some bulk commodities) and STMR-P values provides the levels in feed suitable for estimating maximum residue levels, while calculation from STMR and STMR-P values from feed is suitable for estimating STMR values for animal commodities.

All plant commodities used in the dietary burden calculation are listed below. Dietary burden for livestock might be underestimated, since residue data are not available for several feedstuff derived from crops treated with cypermethrin.

Codex Group	CROP	FEED STUFF	Highest residue	STMR	DM (%)
AL	Alfalfa	forage	11	3.65	35
AL	Alfalfa	hay	20	11.5	100
AF/AS	Barley	forage	1.4	0.39	30
AF/AS	Barley	straw	6.9	3.6	100
AL	Bean	vines	2.1	0.71	35
AM/AV	Beet, sugar	tops	8.3	1.5	100
AM/AV	Cabbage	heads, leaves	0.65	0.02	15
AF/AS	Corn, field	forage/silage	0.1	0.05	40
AF/AS	Corn, field	stover	6.9	3.6	100
AF/AS	Corn, pop	stover	6.9	3.6	100
AF/AS	Corn, sweet	forage	0.1	0.05	48
AF/AS	Corn, sweet	stover	6.9	3.6	100
AL	Cow pea	forage	0.86	0.45	30
AL	Cow pea	hay	1.1	0.42	100
AM/AV	Kale	leaves	0.52	0.07	15
AF/AS	Millet	straw	6.9	3.6	100
AF/AS	Oat	straw	6.9	3.6	100
AL	Pea	vines	0.86	0.45	25
AL	Pea	hay	1.1	0.42	100
AM/AV	Rape	forage	0.24	0.05	30
AF/AS	Rice	straw	6.9	3.6	100
AF/AS	Rye	straw	6.9	3.6	100
AF/AS	Sorghum, grain	stover	6.9	3.6	100
AF/AS	Triticale	straw	6.9	3.6	100
AF/AS	Wheat	forage	1.4	0.38	25
AF/AS	Wheat	straw	6.9	3.6	100
VR	Carrot	culls	0.01	0.01	12
VR	Cassava/tapioca	roots	0.01	0.01	37
VR	Potato	culls	0.01	0.01	20
VR	Swede	roots	0.01	0.01	10
VR	Turnip	roots	0.01	0.01	15
GC	Barley	grain	–	1.38	88
VD	Bean	seed	–	0.05	88
GC	Corn, field	grain	–	0.035	88
GC	Corn, pop	grain	–	0.035	88
VD	Cowpea	seed	–	0.05	88
VD	Lupin	seed	–	0.05	88
GC	Millet	grain	–	0.035	88
GC	Oat	grain	–	1.38	89
VD	Pea	seed	–	0.05	90
GC	Rice	grain	–	0.57	88
GC	Rye	grain	–	1.38	88
GC	Sorghum, grain	grain	–	0.035	86
VD	Soya bean	seed	–	0.05	89
GC	Triticale	grain	–	0035	89
VD	Vetch	seed	–	0.05	89
GC	Wheat	grain	–	1.38	89
AM/AV	Almond	hulls	–	2.3	90
SO	Cotton	undelinted seed		0.05	88
AM/AV	Cotton	gin by-products	0.55	0.36	90
AB	Grape	pomace, wet	–	0.032	15
CM/CF	Wheat	milled by-products	–	3.45	88

Dietary burden calculations for beef cattle, dairy cattle, broilers and laying poultry are provided in Annex 6. A mean and maximum dietary burden for livestock, based on cypermethrin use, is shown in the table below.

Animal dietary burden for cypermethrin, expressed as ppm of dry matter diet

	US	EU	AU	JPN	Overall	
	max	max	max	max	max	
beef cattle	6.07	24.4	31.4	4.71	31.4 (AU)	a
dairy cattle	9.35	17.1	21.6	7.23	21.6 (AU)	b
poultry broiler	2.74	1.89	2.03	1.96	2.74 (US)	
poultry layer	2.74	3.10	2.03	1.20	3.10 (EU)	c,d
	mean	mean	mean	mean	mean	
beef cattle	4.30	8.48	11.3	3.86	11.3 (AU)	a
dairy cattle	4.66	6.86	8.47	5.11	8.47 (AU)	b
poultry broiler	2.74	1.89	2.03	0.91	2.74 (US)	
poultry layer	2.74	2.26	2.03	1.20	2.74 (US)	c,d

[a] Highest mean and maximum beef or dairy cattle dietary burden suitable for maximum residue level and STMR estimates for mammalian meat.

[b] Highest mean and maximum dairy cattle dietary burden suitable for maximum residue level and STMR estimates for milk.

[c] Highest mean and maximum poultry broiler or poultry layer dietary burden suitable for maximum residue level and STMR estimates for poultry meat.

[d] Highest mean and maximum poultry layer suitable for maximum residue level and STMR estimates for eggs.

Livestock feeding studies

Livestock feeding studies with alpha-cypermethrin, zeta-cypermethrin and cypermethrin for cattle and poultry have been submitted and evaluated by JMPR 2008.

Residues in animal commodities

Cattle

The estimated mean and maximum dietary burden for beef and dairy cattle remained the same compared with estimates from JMPR 2009, so there is no change in estimated maximum residue levels, STMRs and HRs for mammalian meat, fat, offal and milk.

Poultry

The estimated mean dietary burden for broiler and layer poultry (2.74 ppm) remained the same compared with estimates from JMPR 2009, so there is no change in estimated STMR values for poultry meat, fats, edible offal and eggs.

The estimated maximum dietary burden for broiler and laying poultry (3.10 ppm) was lower than estimated at JMPR 2009 (3.89 ppm). This lower value was due only to the 2009 change in livestock diets. For maximum residue level estimation, the high residues in eggs, muscle, liver and fat were calculated by interpolating the maximum dietary burden (3.10 ppm) between the relevant feeding levels (0, 1.6 and 7.2 ppm) from the alpha-cypermethrin laying hen feeding study and using the highest meat and egg concentrations from those feeding groups (see table below).

				Residues (mg/kg) in:		
	Feed level (ppm) for egg residues	Residues (ng/g) in eggs	Feed level (ppm) for tissue residues	Muscle	Liver	Fat
Maximum residue level - layer and broiler poultry						
Feeding study [a,b]	0 7.2	0 0.011	0 7.2	0 < 0.05	0 < 0.05	0 0.088
Dietary burden and residue estimate	3.10	0.0047	3.10	0.022	0.022	0.038

[a] highest residues for tissues and eggs

[b] residues were interpolated between 0 and 7.2 ppm feed, because the 1.6 ppm level resulted in residues below LOQ for all matrices.

The Meeting estimated an HR value of 0.022 mg/kg in muscle and liver and an HR of 0.038 mg/kg for fat.

The Meeting estimated a maximum residue level of 0.1 mg/kg for poultry meat (fat) and poultry fats and 0.05* mg/kg for edible offal of poultry. These recommendations take into account that cypermethrin is fat-soluble, and that higher residues could be expected in the fat of broilers in line with the decision taken in JMPR 2008.

The Meeting estimated an HR value of 0.0047 mg/kg for eggs and a maximum residue level of 0.01* mg/kg for eggs.

RECOMMENDATIONS

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

Definition of the residue for compliance with the MRL and for estimation of dietary intake for plant and animal commodities: cypermethrin (sum of isomers). The residue is considered fat-soluble.

CCN	Commodity	Recommended MRL mg/kg		STMR or STMR-P mg/kg	HR or HR-P mg/kg	Source
		new	previous			
AM 0660	Almond hulls			2.3		B
VS 0621	Asparagus	0.4	0.01*	0.09	0.20	a, C
FC 0001	Citrus fruits (except shaddocks or pomelos)	0.3	2	0.05	0.05	a, B
PE 0112	Eggs	0.01*	0.01*	0.0042	0.0047	–
PF 0111	Poultry, Edible offal of	0.05*	0.05 *	0.002	0.022	–
PM 0110	Poultry meat	0.1 (fat)	0.1 (fat)	0.002 (muscle) 0.034 (fat)	0.022 (muscle) 0.048 (fat)	–
PF 0111	Poultry fats	0.1		0.034	0.038	–
FC 0005	Shaddocks or pomelos	0.5		0.05	0.05	a, b , C
DT 1114	Tea, green, black (black, fermented and dried)	15	20	3.75		C
TN 0085	Tree nuts	0.05*		0.05	0.05	a, B

Source of data supporting the proposed MRL:

[a] = alpha-cypermethrin;

[b] = zeta-cypermethrin;

[c] = cypermethrin.

Capital letters shows the primary source of data responsible for the maximum residue level estimate. Small letters show the sources of other data for that commodity.

DIETARY RISK ASSESSMENT

Long-term intake

Based on the evaluation of cypermethrin, alpha-cypermethrin and zeta-cypermethrin, maximum residue levels, HRs and STMRs were estimated for raw and processed commodities in JMPR 2008, 2009 and at the present Meeting. When data on consumption were available for the listed food commodities, dietary intakes were calculated for the GEMS/Food Consumption Cluster Diets. The results are shown in Annex 3 of 2011 JMPR Report.

The IEDI of in the 13 GEMS/Food cluster diets, based on the estimated STMRs were in the range 7–30% of the maximum ADI of 0.02 mg/kg bw. The Meeting concluded that the long-term intake of residues of cypermethrins from uses considered by the Meeting is unlikely to present a public health concern.

Short-term intake

The International Estimated Short Term Intake (IESTI) for cypermethrin, alpha-cypermethrin and zeta-cypermethrin was calculated for the food commodities (and their processing factors) for which maximum residue levels, STMRs and HRs were estimated at the present Meeting. The results are shown in Annex 4 of the 2011 JMPR Report.

The IESTI varied from 0–8% of the ARfD (0.04 mg/kg bw). The Meeting concluded that the short-term intake of residues of cypermethrins from uses considered by the Meeting is unlikely to present a public health concern.

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