Pyriproxyfen (200)

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EXPLANATION

Pyriproxyfen is classified as a juvenile hormone mimic that interferes with normal insect development and reproduction. Metamorphosis of immature life stages is affected but adults are not directly controlled, although production of viable eggs is affected by transovarial activity. Pyriproxyfen is absorbed through the insect cuticle but may also act by ingestion.

Pyriproxyfen was first evaluated by the JMPR in 1999 and then in 2000 and 2001. In the 1999 evaluation for toxicity and residues an ADI of 0–0.1 mg/kg bw was established. The Meeting also concluded that it was not necessary to establish an ARfD because of the low acute toxicity of pyriproxyfen.

The 1999 JMPR recommended the following residue definition for pyriproxyfen:

Definition of the residue for compliance with the MRL and dietary risk assessment for plant and animal commodities: *pyriproxyfen*

The residue is fat-soluble.

The current Meeting received new information on use patterns for pyriproxyfen in bananas, mangoes, papayas, pineapples, cucumbers, cantaloupe melons, peppers and tomatoes supported by additional analytical methods, storage stability data, supervised field trials and nature of residues studies simulating typical processing conditions.

RESIDUE ANALYSIS

Analytical methods

Pyriproxyfen was first evaluated by the JMPR in 1999 where the following analytical methods for plant commodities were assessed:

Table 1 Overview of analytical methods for pyriproxyfen submitted and evaluated by the 1999 JMPR

Method	Commodity	Analyte	LOQ	Reference
CHE 33383-02R	Citrus (peel & pulp)	Parent	0.01 mg/kg	Gardner, 1997 Benwell, 1997 (NNA-0068)
CHE 33383-02R	Citrus (whole fruit, peel & pulp)	Parent	0.01 mg/kg	Van Zyl, 1997 (NNR-0048)
Equivalent to: CHE 33383-02R	Tomato	Parent	0.01 mg/kg	Orpella *et al.*, 1997 (NNA-0069)
RM-33P-2-2	Cotton (seed)	Parent and PYPAC	0.02 mg/kg	Kruplak, 1996 (NNA-0058)
RM-33P-2-3	Cotton (seed and processed fractions)	Parent and PYPAC	0.02 mg/kg	
FDA multi-residue GLC method	Apple, cotton seed	Parent	0.05 mg/kg	Nandihalli, 1996 (NNA-0055)

PYPAC: (RS)-2-(2-pyridyloxy)propionic acid.

For the current Meeting new analytical methods, including multi-residues methods, for pyriproxyfen were submitted.

Table 2 Overview of analytical methods for pyriproxyfen and 4-OH-pyriproxyfen

Method	Matrix	Extraction	Clean-Up	Detection, LOQ
L-00.00-115 (modified "QuEChERS")	High water High acid	Acidic acetonitrile + "QuEChERS"-salts	None	GC-MS/MS Pyriproxyfen: m/z 136→96, 136→78 & 226→186, LOQ: 0.01 mg/kg or by HPLC-MS/MS: m/z 322→96 & 322→185 & 322→227, LOQ: 0.02 mg/kg
RM-33P-1-3 & RM-33P-1-3a	High water High acid	Acetonitrile/hexane	Silica gel SPE	GC-NPD (pyriproxyfen), LOQ: 0.02 mg/kg HPLC-FLD (4-OH-pyriproxyfen), LOQ: 0.02 mg/kg
L-00.00-34	High water	Acetone/water (2:1 v/v)	Gel permeation	GC-MS

Method	Matrix	Extraction	Clean-Up	Detection, LOQ
(formerly "DFG S19")		Ethyl acetate/cyclohexane (1:1 v/v)	chromatography	Pyriproxyfen: m/z 136→78 for quantification and 226 as qualifier, LOQ: 0.01 mg/kg

Plant materials

Method L-00.00-115 (European Committee for Standardization) "QuEChERS"

Banana peel, pulp and whole fruit (Borbón, 2017, PYRIP_001)

Pineapple peel, pulp and whole fruit (Candanedo, 2016, PYRIP_002)

A homogenized sample aliquot (15 g) is weighed into a plastic tube (50 mL) and acidified (1%) acetonitrile is added. The sample is shaken for 10 minutes in an automatic shaker at 2500 rpm. Magnesium sulfate (6 g) and ammonium acetate (1.5 g) are added and the sample is shaken for a further minute. After centrifugation, an aliquot (8 mL) of the clear supernatant is taken and magnesium sulfate (1.2 g) and PSA (0.4 g) are added. The solution is blended for one minute followed by centrifugation. An aliquot (940 µL) is transferred into a glass vial; an internal standard (parathion-ethyl-D10, 30 µL) and protectant solution (D-sorbitol and glucolactone, 30 µL) are added to get a final volume of 1 mL.

Residues of pyriproxyfen are determined by GC-MS/MS (mass transitions: m/z = 136 –96, 136 –78 and 226 –186)

Mango (Keong, 2017, PYRIP_003; Chuan Mun Choy, 2015, PYRIP_004 and Fauzan bin Yunus, 2017, PYRIP_005)

A homogenized sample aliquot (15 g) is weighed into a plastic tube (50 mL) and acidified (1%) acetonitrile (15 mL) is added. The sample is shaken for 1 minute. Magnesium sulfate (6 g) and sodium acetate (1.5 g) are added and the sample shaken for a further minute. After centrifugation, an aliquot (3 mL) of the clear supernatant is taken and magnesium sulfate (450 mg), C18 (150 mg) and PSA (150 mg) are added. The solution is vigorously shaken followed by centrifugation. An aliquot (1 mL) is transferred into a glass vial and diluted with 0.1% formic acid in water (1 mL).

Residues of pyriproxyfen are determined by HPLC-MS/MS (mass transitions: m/z = 322 –96, 322 –185 and 322 –227)

Table 3 Recovery data for method L-00.00-115 (European Committee for Standardization) "QuEChERS" in plant matrices

Matrix	Fortification level (mg/kg)	n	Recovery in % (mean)	RSD (%)	Method, Analyte, Mass transition, Reference
Banana, peel	0.01	9	109–118 (115)	2.9	Quechers (modified)
	0.11	3	76–81 (79)	3.9	pyriproxyfen
	2.1	8	83–96 (88)	4.2	m/z = 136 → 96
Banana, pulp	0.01	9	95–112 (105)	5.1	m/z = 136 → 78
	0.11	3	70–74 (71)	3.9	m/z = 226 → 186
	2.1	8	84–98 (91)	5.8	PYRIP_001
Banana, fruit	0.01	9	97–118 (112)	7.3	
	0.11	3	87–88 (87)	0.7	
	2.1	7	84–99 (91)	5.2	
Pineapple, peel	0.01	6	80–110 (101)	11	Quechers (modified)
	0.2	4	80–108 (99)	13	pyriproxyfen
	2.0	3	107–120 (113)	5.8	m/z = 136 → 96
Pineapple, pulp	0.01	6	102–120 (113)	8.2	m/z = 136 → 78
	0.2	4	92–110 (104)	8.6	m/z = 226 → 186
	2.0	3	106–113 (109)	3.2	PYRIP_002
Pineapple, fruit	0.01	10	77–106 (91)	9.5	
	0.2	4	2.7	1.5	
	2.0	3	105–111 (109)	3.0	
Mango, fruit	0.02	12	70–104 (87)	11	Quechers (modified),
	0.06	3	81–104 (91)	12	Pyriproxyfen, m/z = 322→96,
	0.2	6	77–105 (92)	13	322→185, 322→227
	2.0	9	80–108 (93)	11	PYRIP_003, PYRIP_004, PYRIP_005

Method RM-33P-1-3 (Green, 1996, PYRIP_006, Wood, 1997, PYRIP_007) and modified method RM-33P-1-3a (Green, 1998, PYRIP_008; Tejada, 2017, PYRIP_031)

A sample aliquot is blended with acetone for 5 minutes, three times. The combined extracts are evaporated to the aqueous remainder. The extract is acidified with hydrochloric acid (2 x 25 mL, 1M). Except for apples a first partitioning is performed with acetonitrile and hexane. The hexane phase is additionally washed twice with acetonitrile. The combined acetonitrile extracts were evaporated to dryness and the hexane phase is discarded. The second partitioning uses an aqueous sodium chloride solution which is extracted three times with dichloromethane. The combined dichloromethane phases were evaporated just to dryness. The residue is dissolved in hexane/ ethyl acetate (80:20; v/v; 5 mL).

The extract is split for column clean-up of pyriproxyfen (silica gel; washing/elution: hexane/ethyl acetate 80:20, v/v) and 4-OH-pyriproxyfen (silica gel; washing/elution: hexane/ethyl acetate 60:40, v/v).

Residues of pyriproxyfen are determined by gas chromatography with nitrogen/phosphorous detection (GC-NPD), while residues of 4-OH-pyriproxyfen are determined by high performance liquid chromatography with fluorescence detection (HPLC-FLD).

The modified method RM-33P-1-3a is nearly indentical, except for a slightly lower amount of sample required for the initial extraction due to omission of 4-OH-pyriproxyfen determination.

Table 4 Recovery data for method RM-33P-1-3 and its modification RM-33P-1-3a in plant matrices

Matrix	Fortification level (mg/kg)	n	Recovery, mean (%)	RSD (%)	Method, Analyte, Determination, Reference
Apple	0.02	2	79–80 (80)	-	RM-33P-1-3, pyriproxyfen
	0.2	2	78–82 (80)	-	GC-NPD, PYRIP_007
	0.02	2	80–100 (90)	-	RM-33P-1-3, 4-OH-pyriproxyfen
	0.2	2	71–85 (78)	-	HPLC-FLD, PYRIP_007
Orange	0.02	2	82–88 (86)	-	RM-33P-1-3, pyriproxyfen
	0.3	2	80–90 (85)	-	GC-NPD, PYRIP_007
	0.02	2	84–94 (89)	-	RM-33P-1-3, 4-OH-pyriproxyfen
	0.3	2	80–82 (81)	-	HPLC-FLD, PYRIP_007
Orange	0.02	4	83–96 (88)	6.7	RM-33P-1-3a, pyriproxyfen
	0.1	6	91–97 (94)	2.4	GC-NPD, PYRIP_008
Papaya	0.02	3	93–129 (108)	17	RM-33P-1-3a, pyriproxyfen
	0.2	3	97–113 (104)	8.0	GC-MS/MS, PYRIP_XXX
	2.0	3	89–127 (108)	17	
	0.02	30	68–96 (80)	9.8	RM-33P-1-3a, pyriproxyfen
	0.2	6	84–90 (87)	3.4	LC-MS/MS, PYRIP_XXX
	2.0	6	78–86 (83)	4.0	

Extraction efficiency of method RM-33P-1-3 (Pensyl, 1997, PYRIP_011)

The extraction efficiency of method RM-33P-1-3 was tested on apple pomace samples obtained from the plant metabolism study on apples evaluated by the 1999 JMPR. Apple pomace samples were exhaustively extracted within the plant metabolism study and the known amounts of pyriproxyfen and its metabolite 4- OH-pyriproxyfen were compared with the residues found when method RM-33P-1-3 was applied to the same sample. The following extraction efficiency of method RM-33P-1-3 was found for apple pomace:

Table 4 Overview of the extraction efficiency of method RM-33P1-3 in apple pomace

Commodity	Analyte	Residues in mg/kg		Extraction efficiency (%)
		Method RM-33P-1-3	Metabolism study	
Apple pomace	Pyriproxyfen	0.365	0.466	78
	4-OH-pyriproxyfen	0.068	0.078	87

Method L-00.00-34 (European Committee for Standardization) - formerly "DFG S19" (Weber, 2000, PYRIP_009; Kretschmer, 2001, PYRIP_010)

Samples were homogenized in acetone/water 2:1 (v/v, taking into account the water content of the sample).

After addition of sodium chloride the mixture was partitioned using ethyl acetate/cyclohexane 1:1 (v/v). The organic phase was partially evaporated and redissolved in ethyl acetate, after which sodium sulphate, sodium chloride and cyclohexane

were added. The organic phase was subjected to gel permeation chromatography on Bio Beads S-X3 polystyrene gel using a mixture of ethyl acetate/cyclohexane. The eluate was evaporated and redissolved in ethyl acetate.

Residues of pyriproxyfen were determined by GC-MS (mass transitions: m/z 136 → 78 for quantitation and 226 as qualifier).

Table 6 Recovery data for method L-00.00-34 (European Committee for Standardization) in plant matrices

Matrix	Fortification level (mg/kg)	n	Recovery, mean (%)	RSD (%)	Method, Analyte, Mass transition, Reference
Cucumber	0.01	5	90–105 (97)	7.2	L-00.00-34, pyriproxyfen
	0.1	5	82–97 (90)	6.1	m/z = 136→78, PYRIP_009
Cucumber	0.01	5	79–88 (83)	5.0	L-00.00-34, pyriproxyfen
	0.1	5	80–99 (90)	9.0	m/z = 136→78, PYRIP_010

Stability of pesticides in stored analytical samples

Pyriproxyfen was first evaluated by the JMPR in 1999 and the storage stability of residues in plant commodities was evaluated. The 1999 Meeting concluded that pyriproxyfen was stable in tomato homogenate for 12 months, cotton seed for 13 months and gin trash for 8 months. PYPAC [(RS)-2-(2-pyridyloxy)propionic acid] was stable in cotton seed for 13 months. New data on storage stability are available for pepper, papaya, mango and pineapple.

Plant matrices

Pepper (Pensyl, 1998, PYRIP_012)

The study presents storage stability data for pyriproxyfen and the metabolite PYPA [(RS)-2-(2-pyridyloxy)propyl alcohol] in homogenized pepper samples. Homogenized samples of pepper fruits (10 g) were fortified with pyriproxyfen at a level of 0.1 mg/kg and stored at -20 °C for up to 91 days.

Levels of pyriproxyfen in stored samples were determined using the residue analytical method RM-33P-8, a modified version of residue analytical method RM-33P-2, adapted to high water containing crops (tomato), with an LOQ of 0.02 mg/kg.

Table 7 Storage stability of pyriproxyfen in peppers

Matrix	Spike level (mg/kg)	Storage interval (days/months)	Recovered residues		
			Stored sample		Procedural recovery
			mg/kg	% nominal (mean)	%
Pepper (whole fruit)	0.1	0 / 0	0.105, 0.101	105, 101 (103)	103
		30 / 1	0.082, 0.082	82, 82 (82)	94
		63 / 2	0.078, 0.072	78, 72 (75)	103
		66 / 3	0.074, 0.071	71, 68 (70)	92
		91 / 3	0.084, 0.076	84, 76 (80)	97

Papaya (Samoil, 2011, PYRIP_013; Tejada, 2017, PYRIP_031)

Papaya fruit samples (20 g) were homogenised, fortified with pyriproxyfen at a level of 0.20 mg/kg and stored at approximately 20 °C for 574 days. Duplicate freshly fortified samples were used for procedural recoveries. Levels of pyriproxyfen in stored samples were determined using the residue analytical method RM-33P-1-3a, with an LOQ of 0.02 mg/kg for papaya.

Table 8 Storage stability of pyriproxyfen in papaya fruits

Matrix	Spike level (mg/kg)	Storage interval (days/months)	Recovered residues		
			Stored sample		Procedural recovery
			mg/kg	% nominal (mean)	%
Papaya (whole fruit)	0.2 [a]	574 / 19	0.176, 0.175, 0.178	88, 88, 89 (88)	107, 110 (109)
	0.2 [b]	559 / 18	0.198, 0.198, 0.199	96, 102, 101 (100)	115, 83 (99)
	0.2 [c]	634 / 21	0.212, 0.238, 0.210	106, 119, 105 (110)	63, 69 (66)

[a] PYRIP_013

[b] PYRIP_031: Food Development Center

[c] PYRIP_031: Central Laboratory Services

Mango (Keong, 2017, PYRIP_003; Chuan Mun Choy, 2015, PYRIP_004 and Fauzan bin Yunus, 2017, PYRIP_005)

Samples of mango whole fruit (15 g) were fortified with pyriproxyfen at a level of 0.20 mg/kg and stored at about 20 °C for 176 days. Levels of pyriproxyfen in stored samples were determined using a modified QuEChERS residue analytical method, with a validated LOQ of 0.02 mg/kg for mango whole fruit (see above).

Table 9 Storage stability of pyriproxyfen in mango fruits

Matrix	Spike level (mg/kg)	Storage interval (days/months)	Recovered residues		
			Stored sample		Procedural recovery
			mg/kg	% nominal (mean)	%
Papaya (whole fruit)	0.2 /[a]	174 / 6	0.164, 0.158, 0.158	82, 79, 79 (80)	88
	0.2 /[b]	176 / 6	0.211, 0.198, 0.208, 0.202, 0.182	106, 99, 104, 101, 91 (100)	89

[a] Veterinary Public Health Laboratory

[b] Central Laboratory Service

Pineapple (Candanedo, 2016, PYRIP_002)

Samples of pineapple peel, pulp and whole fruit (15 g) were fortified with pyriproxyfen at a level of 0.20 mg/kg and stored at about -20 °C for 497 days. Levels of pyriproxyfen in stored samples were determined using a modified version of the QuEChERS residue analytical method, with an LOQ of 0.01 mg/kg for pineapple peel, pulp and whole fruit (see above).

Table 10 Storage stability of pyriproxyfen in pineapples (peel, pulp and whole fruits)

Matrix	Spike level (mg/kg)	Storage interval (days/months)	Recovered residues		
			Stored sample		Procedural recovery
			mg/kg	% nominal (mean)	%
Pineapple (peel)	0.2	497 / 16	0.147, 0.153, 0.149	72, 73, 75 (74)	79
Pineapple (pulp)	0.2	497 / 16	0.204, 0.203, 0.203	100, 100, 100 (100)	92
Pineapple (whole fruit)	0.2	497 / 16	0.195, 0.184, 0.188	90, 92, 96 (93)	99

Animal matrices

No additional data submitted.

USE PATTERN

Pyriproxyfen is intended for post-emergence use as an insecticide in various crops such as banana, mango, papaya, pineapple, cucurbits, peppers, and tomatoes by a foliar spray application for the control of banded wing whitefly, greenhouse whitefly, silverleaf whitefly (Bemisia tabaci) and sweet potato whitefly.

Pyriproxyfen is registered in many countries all over the world. For the purposes of this document, emphasis is given to registered uses in countries where supervised trials have been conducted or in countries with GAPs similar to those where the supervised trials were carried out.

Table 11 List of uses of pyriproxyfen

Crops or crop groups	Country	Application detail					
		kg ai/ha	Growth stage at last treatment	Indoor/ Outdoor	No.	Interval in days	Pre harvest interval (PHI) in days
Group 006 - Assorted tropical and sub-tropical fruits - inedible peel							
Banana	Panama	0.1	At infestation	Outdoor	n.s.	n.s.	1
Banana	U.S.A	0.12	At infestation	Outdoor	3	14	14
Mango	Malaysia	0.05	At infestation	Outdoor	2	14	1
Papaya	Philippines	0.1		Outdoor	2	14	1
Pineapple	Panama	0.3	At infestation	Outdoor	3	n.s.	1
Pineapple	U.S.A.	0.06	At infestation	Outdoor	2	21	1
Group 011 - Fruiting vegetables, Cucurbits							
Cucumber, courgette	Italy	0.12	At infestation	Indoor	2	14	3
Cucurbits [a]	U.S.A.	0.075	At infestation	Outdoor	2	14	7

Crops or crop groups	Country	Application detail					
		kg ai/ha	Growth stage at last treatment	Indoor/ Outdoor	No.	Interval in days	Pre harvest interval (PHI) in days
Group 012 - Fruiting vegetables, other than Cucurbits							
Pepper, tomato, eggplant	Italy	0.12	At infestation	Indoor	2	14	3
Fruiting vegetables US Crop Group 8-10 [b]	U.S.A.	0.06	At infestation	Indoor/ Outdoor	2	14	1

n.s. not stated

[a] US Crop Group 9: Balsam apple, balsam pear, bitter melon, cantaloupe, chayote, Chinese cucumber, Chinese wax gourd, citron melon, cucumber, edible gourd, gherkin, Momordica spp., muskmelon, pumpkin, summer squash, watermelon, winter squash

[b] US Crop Group 8-10: African eggplant, bush tomato, bell pepper, cocona, currant tomato, eggplant, garden huckleberry, goji berry, ground cherry, Martynia, naranjilla, okra, pea eggplant, pepino, pepper bell, pepper non-bell, roselle, scarlet eggplant, sunberry, tomatillo, tomato, tree tomato, further cultivars and hybrids of these

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

Residue levels were reported as measured. Application rates were always reported as pyriproxifen equivalents. When residues were not detected they are shown as below the LOQ, e.g. <0.01 mg/kg. Application rates, spray concentrations and mean residue results have generally been rounded to the even with two significant figures. Residue values from the trials, conducted according to maximum GAP, that have been used for the estimation of maximum residue levels and STMRs are underlined.

Laboratory reports included method validation, including batch recoveries with spiking at residue levels similar to those occurring in samples from the supervised trials. Dates of analyses or duration of residue sample storage were also provided. Field reports provided data on the sprayers used and their calibration, plot size, residue sample size and sampling date. Although trials included control plots, no control data are recorded in the tables except where residues in control samples exceeded the LOQ. Residue data are recorded unadjusted for % recovery.

Pyriproxyfen - supervised residue trials

Commodity	Indoor/Outdoor	Treatment	Countries	Table
Banana	Outdoor	Foliar	Costa Rica, Guatemala	Table 12
Mango	Outdoor	Foliar	Malaysia	Table 13
Papaya	Outdoor	Foliar	Brunei, Malaysia, Philippines	Table 14
Pineapple	Outdoor	Foliar	Panama	Table 15
Cucumber	Indoor	Foliar	France, Greece, Italy, Spain	Table 16
Cantaloupe	Outdoor	Foliar	USA	Table 17
Peppers	Indoor	Foliar	France, Greece, Italy, Spain	Table 18
Tomatoes	Indoor	Foliar	France, Greece, Italy, Spain	Table 19

Table 12 Residues of pyriproxyfen following spray treatment on bananas

Location, Year (variety)	Application kg ai/ha	Inter-val	kg ai/hL	Growth stage	Residues, mg/kg Sample	DALA	Parent	Report/Trial No., Reference, analytical method, validation data, storage period
USA: 3×0.12 kg ai/ha, 14 d RTI, 14 d PHI								
Costa Rica, Sixaola 2016 (Cavendish)	0.14 0.14 0.14	- 14 d 14 d	0.56 0.56 0.56	Fruiting	Whole fruit	0	0.083	B11399/15-CR11, PYRIP_015 Method: L-00.00-115 Storage period: 5 months
Costa Rica, Tortuguero 2016 (Cavendish)	0.14 0.14 0.14	- 14 d 14 d	0.4 0.4 0.4	Fruiting	Whole fruit Pulp Peel	0 0 0	0.1 <0.01 0.37	B11399/15-CR12, PYRIP_015 Method: L-00.00-115 Storage period: 5 months
Costa Rica,	0.14	-	0.47	Fruiting	Whole fruit	0	0.043	B11399/15-CR13, PYRIP_015

Location, Year (variety)	Application kg ai/ha	Inter-val	kg ai/hL	Growth stage	Residues, mg/kg Sample	DALA	Parent	Report/Trial No., Reference, analytical method, validation data, storage period
San Clemente 2016 (Cavendish)	0.14 0.14	14 d	0.47 0.47					Method: L-00.00-115 Storage period: 5 months
Costa Rica, Martina 2016 (Cavendish)	0.14 0.14 0.14	- 14 d 14 d	0.47 0.47 0.47	Fruiting	Whole fruit	0	0.029	B11399/15-CR14, PYRIP_015 Method: L-00.00-115 Storage period: 5 months
Costa Rica, Puerto Viejo 2016 (Cavendish)	0.14 0.14 0.14	- 14 d 14 d	0.47 0.47 0.47	Fruiting	Whole fruit Pulp Peel	0 0 0	0.018 <0.01 0.039	B11399/15-CR15, PYRIP_015 Method: L-00.00-115 Storage period: 5 months
Costa Rica, Cariari 2016 (Cavendish)	0.14 0.14 0.14	- 14 d 14 d	0.35 0.35 0.35	Fruiting	Whole fruit Pulp Peel	0 3 7 14 21 0 14 0 14	0.080 0.20 0.13 0.023 <u>0.036</u> <0.01 <0.01 0.28 0.11	B11399/15-CR16, PYRIP_015 Method: L-00.00-115 Storage period: 5 months
Costa Rica, El Carmen 2016 (Cavendish)	0.14 0.14 0.14	- 14 d 14 d	0.35 0.35 0.35	Fruiting	Whole fruit	0	0.056	B11399/15-CR17, PYRIP_015 Method: L-00.00-115 Storage period: 5 months
Guatemala, Aldea Los Chatos 2016 (Cavendish)	0.14 0.14 0.14	- 14 d 14 d	0.56 0.56 0.56	Fruiting	Whole fruit Pulp Peel	0 0 0	0.012 <0.01 <0.01	B11399/15-GU42, PYRIP_015 Method: L-00.00-115 Storage period: 5 months

DALA: days after last treatment; d: days

Table 13 Residues of pyriproxyfen following spray treatment on mangos

Location, Year (variety)	Application kg ai/ha	Inter-val	kg ai/hL	Growth stage	Residues, mg/kg Sample	DALA	Parent	Report/Trial No., Reference, analytical method, validation data, storage period
cGAP : Malaysia, 2×0.05 kg ai/ha, 14 day interval, PHI : 14 days								
Malaysia, Pedang 2012 (Chok-Anan)	0.056 0.059	- 14 d	0.005 0.005	Fruiting	Whole fruit	14	<0.02, <0.02	IR-4 PR No. 10990/12-MY01, PYRIP_003, PYRIP_004, PYRIP_005 Method: L-00.00-115 Storage period: 1 months
Malaysia, Masmago 2012 (Chok-Anan)	0.057 0.058	- 13 d	0.005 0.005	Fruiting	Whole fruit	14	<0.02, <0.02	IR-4 PR No. 10990/12-MY02, PYRIP_003, PYRIP_004, PYRIP_005 Method: L-00.00-115 Storage period: 1.5 months
Malaysia, Dulan 2013	0.052 0.053	- 13 d	0.005 0.005	Fruiting	Whole fruit	14	<0.02, <0.02	IR-4 PR No. 10990/12-MY03, PYRIP_003, PYRIP_004, PYRIP_005

Location, Year (variety)	Application kg ai/ha	Inter-val	kg ai/hL	Growth stage	Residues, mg/kg Sample	DALA	Parent	Report/Trial No., Reference, analytical method, validation data, storage period
(Chok-Anan)								Method: L-00.00-115 Storage period: 1.5 months
Malaysia, Serdang 2013 (Chok-Anan)	0.05 0.056	- 13 d	0.005 0.005	Fruiting	Whole fruit	14	<0.02, <0.02	IR-4 PR No. 10990/12-MY04, PYRIP_003, PYRIP_004, PYRIP_005 Method: L-00.00-115 Storage period: 1 months
Malaysia, Rawang 2014 (Chok-Anan)	0.054 0.054	- 15 d	0.005 0.005	Fruiting	Whole fruit	14	<0.02, <0.02	IR-4 PR No. 10990/12-MY06, PYRIP_003, PYRIP_004, PYRIP_005 Method: L-00.00-115 Storage period: 1 months
Malaysia, Dulan 2014 (Chok-Anan)	0.055 0.055	- 14 d	0.005 0.005	Fruiting	Whole fruit	0 3 7 14 21	<0.02, <0.02 <0.02, <0.02 <0.02, <0.02 <0.02, <0.02 <0.02, <0.02	IR-4 PR No. 10990/12-MY07, PYRIP_003, PYRIP_004, PYRIP_005 Method: L-00.00-115 Storage period: 1 months

DALA: days after last treatment; d: days

Table 14 Residues of pyriproxyfen following spray treatment on papaya

Location, Year (variety)	Application kg ai/ha	Inter-val	kg ai/hL	Growth stage	Residues, mg/kg Sample	DALA	Parent	Report/Trial No., Reference, analytical method, validation data, storage period
cGAP : Philippines, 2×0.1 kg ai/ha, 14 day interval, PHI : 1 day								
Philippines, Balingasag, Misamis Oriental 2014 (SNX)	0.092 0.099	- 13 d	0.013 0.013	Fruiting	Whole fruit	0 1 3 7 14	0.10, 0.10 (0.10) 0.07, 0.07 (0.07) 0.06, 0.06 (0.06) 0.05, 0.05 (0.05) 0.03, 0.03 (<u>0.03</u>)	IR-4 PR No. 11251/14-PH01, PYRIP_031 Method: RM-33P-1-3A Storage period: 9.6 months
Philippines, Brgy. Caingin, San Rafael, Bulacan 2014 (Sinta)	0.050 0.050	- 13 d	0.006 0.006	Fruiting	Whole fruit	1 14	0.09, 0.09 (0.09) 0.02, 0.04 (0.03)	IR-4 PR No. 11251/14-PH02, PYRIP_031 Method: RM-33P-1-3A Storage period: 12 months
	0.116 0.115	- 13 d	0.013 0.013	Fruiting	Whole fruit	1 14	0.16, 0.20 (0.18) 0.10, 0.11 (<u>0.10</u>)	
Philippines, Perez Papaya Farm, Brgy. Canda, Sariaya, Quezon 2015 (Red Lady)	0.050 0.051	- 14 d	0.003 0.006	Fruiting	Whole fruit	1 15	0.07, 0.07 (0.07) 0.17, 0.03 (<u>0.10</u>)	IR-4 PR No. 11251/14-PH03, PYRIP_031 Method: RM-33P-1-3A Storage period: 12 months

Location, Year (variety)	Application				Residues, mg/kg			Report/Trial No., Reference, analytical method, validation data, storage period
	kg ai/ha	Inter-val	kg ai/hL	Growth stage	Sample	DALA	Parent	
	0.099 0.101	- 14 d	0.012 0.013	Fruiting	Whole fruit	1 15	0.08, 0.07 (0.07) 0.09, 0.03 (0.06)	
Malaysia, MARDI, Persiaran, Mardi-Upm, Serdang, 13400, Selangor 2015 (Eksotika)	0.102 0.100	- 15 d	0.013 0.013	Fruiting	Whole fruit	0 1 3 6 13	2x 0.092 (0.092) 0.083, 0.096 (0.090) 0.039, 0.060 (0.050) 0.029, 0.054 (0.042) 0.033, 0.026 (0.030)	IR-4 PR No. 11251/14-MY04, PYRIP_031 Method: RM-33P-1-3A Storage period: 21 months
Malaysia, Pauh, Perlis 2015 (Sekaki)	0.050 0.049	- 14 d	0.007 0.005	Fruiting	Whole fruit	1 13	0.064, 0.052 (0.058) 0.042, 0.045 (0.044)	IR-4 PR No. 11251/14- MY05, PYRIP_031 Method: RM-33P-1-3A Storage period: 20 months
	0.105 0.099	- 14 d	0.010 0.013	Fruiting	Whole fruit	1 13	0.148, 0.138 (0.143) 0.145, 0.150 (0.148)	
Malaysia, Pauh, Perlis 2016 (Eksotika)	0.051 0.052	- 14 d	0.006 0.007	Fruiting	Whole fruit	1 14	0.182, 0.180 (0.181) 0.035, 0.038 (0.037)	IR-4 PR No. 11251/14- MY06, PYRIP_031 Method: RM-33P-1-3A Storage period: 8.3 months
	0.107 0.106	- 14 d	0.012 0.012	Fruiting	Whole fruit	1 14	0.166, 0.200 (0.183) 0.045, 0.034 (0.040)	
Brunei, Brunei 2015 (--)	0.051 0.054	- 14 d	0.006 0.004	Fruiting	Whole fruit	1 14	0.169, 0.116 (0.143) 0.083, 0.073 (0.078)	IR-4 PR No. 11251/14-BN07, PYRIP_031 Method: RM-33P-1-3A Storage period: 12 months
	0.192 0.196	- 14 d	0.013 0.025	Fruiting	Whole fruit	1 14	0.164, 0.251 (0.208) 0.094; 0.062 (0.078)	

DALA: days after last treatment; d: days

Table 15 Residues of pyriproxyfen following spray treatment on pineapples

Location, Year (variety)	Application kg ai/ha	Inter-val	kg ai/hL	Growth stage	Residues, mg/kg Sample	DALA	Parent (mean)	Report/Trial No., Reference, analytical method, validation data, storage period
cGAP : USA, 2×0.06 kg ai/ha, 21 day interval, PHI : 1 day								
Panama, San Lorenzo 2015 (MD-2)	0.069 0.069	- 14 d	0.001 0.002	Fruiting	Whole fruit	1 14	<0.01, <0.01[D] <0.01, <0.01	IR-4 PR No. 11398/14-PA01 PYRIP_002 Method: L-00.00-115 Storage period: 6 months
Panama, Veladero 2014 (MD-2)	0.077 0.079	- 14 d	0.002 0.003	Fruiting	Whole fruit Peel Pulp	1 14 1 14 1 14	<0.01, <0.01 <0.01, <0.01 0.024, 0.039 (0.032) <0.01, <0.01 <0.01, <0.01 <0.01, <0.01	IR-4 PR No. 11398/14-PA02 PYRIP_002 Method: L-00.00-115 Storage period: 10 months
Panama, Las Zanguengas 2014 (MD-2)	0.065 0.069 0.067	- 14 d 14 d	0.002 0.002 0.002	Fruiting	Whole fruit Peel Pulp	1 14 1 14 1 14	<0.01, <0.01[D] <0.01, <0.01 0.041, 0.046 (0.043) <0.01, <0.01 <0.01, <0.01 <0.01, <0.01	IR-4 PR No. 11398/14-PA03 PYRIP_002 Method: L-00.00-115 Storage period: 2 months
Panama, Las Zanguengas 2015 (MD-2)	0.069 0.069	- 14 d	0.002 0.002	Fruiting	Whole fruit	1 14	<0.01, <0.01 <0.01, <0.01	IR-4 PR No. 11398/14-PA09 PYRIP_002 Method: L-00.00-115 Storage period: 6 months
Panama, Caño Quebrado 2015 (MD-2)	0.075 0.075	- 14 d	0.002 0.002	Fruiting	Whole fruit Peel Pulp	1 14 1 14 1 14	<0.01, <0.01 <0.01, <0.01 0.032, 0.024 (0.028) <0.01, <0.01[D] <0.01, <0.01 <0.01, <0.01	IR-4 PR No. 11398/14-PA05 PYRIP_002 Method: L-00.00-115 Storage period: 4 months
Panama, Yayas Adentro 2015 (MD-2)	0.070 0.070	- 13 d	0.001 0.001	Fruiting	Whole fruit	0 1 3 5 10 14 21	<0.01, <0.01[D] <0.01, <0.01[D] <0.01, <0.01[D] <0.01, <0.01 <0.01, <0.01 <0.01, <0.01 <0.01, <0.01	IR-4 PR No. 11398/14-PA06 PYRIP_002 Method: L00.00-34 Storage period: 4 months

DALA: days after last treatment

d: days

[D]:Detected, but <LOQ limit

Table 16 Residues of pyriproxyfen following spray treatment on cucumbers in greenhouse

Location, Year (variety)	Application kg ai/ha	Interval days	kg ai/hL	Growth stage	Residues, mg/kg Sample	DALA	Parent (mean)	Report/Trial No., Reference, analytical method, validation data, storage period
cGAP : Italy, 2×0.12 kgai/ha, 14 day interval, PHI : 3 days								
France, Longueville 1999 (Flamengo)	0.11 0.11	- 9	0.008	Fruiting	Whole fruit	0 3 7	0.03 <0.01 <0.01	EA990152/FR01, PYRIP_015 Method: L-00.00-34 Storage period: 5 months
Greece, Profitis 1999 (2-14)	0.11 0.11	- 11	0.008	Fruiting	Whole fruit	3	0.02	EA990152/GR01, PYRIP_015 Method: L-00.00-34 Storage period: 3 months
Spain, Cañada Gallego 1999 (Anico)	0.12 0.11	- 9	0.008	Fruiting	Whole fruit	0 3 6	0.01 <0.01 <0.01	EA990153/SP01, PYRIP_016 Method: L-00.00-34 Storage period: 4 months
Spain, Puerto de Mazzaron 1999 (Anico)	0.11 0.11	- 10	0.008	Fruiting	Whole fruit	3	<0.01	EA990153/SP02, PYRIP_016 Method: L-00.00-34 Storage period: 5 months
France, Longueville 2000 (Toril)	0.11 0.11	- 10	0.008	Fruiting	Whole fruit	3	<0.01	EA000123/FR01, PYRIP_017 Method: L-00.00-34 Storage period: 2 months
Italy, Roncoferraro 2000 (Darina)	0.11 0.12	- 11	0.008	Fruiting	Whole fruit	0 3 7	0.03 <0.01 <0.01	EA000123/IT01, PYRIP_017 Method: L-00.00-34 Storage period: 2 months
Italy, Campofilone 2000 (Darina)	0.11 0.11	- 10	0.008	Fruiting	Whole fruit	3	<0.01	EA000123/IT02, PYRIP_017 Method: L-00.00-34 Storage period: 3 months
Greece, Kato Souli 2000 (G5-1631)	0.12 0.11	- 10	0.008	Fruiting	Whole fruit	0 3 7	0.07 <0.01 <0.01	EA000124/GR01, PYRIP_018 Method: L-00.00-34 Storage period: 3 months

DALA: days after last treatment

Table 17 Residues of pyriproxyfen following spray treatment on cantaloupe in the field

Location, Year (variety)	Application kg ai/ha	Interval days	kg ai/hL	Growth stage	Residues, mg/kg Sample	DALA	Parent (mean)	Report/Trial No., Reference, analytical method, validation data, storage period
cGAP : USA, 2×0.075 kg ai/ha, 14 day interval, PHI : 7 days								
USA, Wallacy (TX) 1999 (Tam Uvalde)	0.076 0.076	- 14	0.08 0.08	Fruiting	Whole fruit	7	0.04, 0.03 (0.035)	200100283/V-20204-A, PYRIP_019 Method: RM 33P 1 3a Storage period: 1 months
USA, Madera (CA)	0.077 0.076	- 14	0.08 0.08	Fruiting	Whole fruit	7	0.02, 0.03 (0.035)	200100283/V-20204-B, PYRIP_019

Location, Year (variety)	Application kg ai/ha	Inter-val days	kg ai/hL	Growth stage	Residues, mg/kg Sample	DALA	Parent (mean)	Report/Trial No., Reference, analytical method, validation data, storage period
1999 (Hales Jumbo)								Method: RM 33P 1 3a Storage period: 1 months
USA, Maricopa (AZ) 1999 (Hy Mark)	0.074 0.074	- 14	0.08 0.08	Fruiting	Whole fruit	3 7 10 14	0.018, 0.017 (0.018) 0.013, 0.02 (<u>0.016</u>) <0.01, 0.011 (0.01) <0.01, <0.01	200100283/V-20204-C, PYRIP_019 Method: RM 33P 1 3a Storage period: 1 months
USA, Martin (NC) 2000 (Ambrosia Hybrid 30510)	0.075 0.079	- 14	0.08 0.08	Fruiting	Whole fruit	7	0.01, 0.02 (<u>0.015</u>)	200100283/V-20204-D, PYRIP_019 Method: RM 33P 1 3a Storage period: 1 months
USA, Pepin (WI) 2000 (Primo Hybrid)	0.0750.076	- 14	0.08 0.08	Fruiting	Whole fruit	7	<0.01, <u>0.01</u>	200100283/V-20204-E, PYRIP_019 Method: RM 33P 1 3a Storage period: 1 months
USA, Madera (CA) 2000 (Top Mark)	0.075 0.075	- 14	0.08 0.08	Fruiting	Whole fruit Peel Pulp	7 7 7	0.017, 0.021 (<u>0.019</u>) 0.034, 0.035 (0.034) <0.01, <u>0.01</u>	200100283/V-20204-F, PYRIP_019 Method: RM 33P 1 3a Storage period: 1 months
USA, Maricopa (AZ) 2000 (Gold Rush)	0.076 0.074	- 14	0.08 0.08	Fruiting	Whole fruit	7	<0.01, <<u>0.01</u>	200100283/V-20204-G, PYRIP_019 Method: RM 33P 1 3a Storage period: 1 months
	0.16 0.14	- 14	0.17 0.15	Fruiting	Whole fruit	7	0.02, 0.02	

DALA: days after last treatment

Remark: all samples were cut into quarters at collection before storing

Table 18 Residues of pyriproxyfen following spray treatment on peppers in greenhouse

Location, Year (variety)	Application kg ai/ha	Inter-val days	kg ai/hL	Growth stage	Residues, mg/kg Sample	DALA	Parent (mean)	Report/Trial No., Reference, analytical method, validation data, storage period
cGAP : Italy, 2×0.12 kgai/ha, 14 day interval, PHI : 3 days								
France, Gontaund de Nogaret 1999 (Volga)	0.11 0.11	- 10	0.008 0.008	Fruiting	Whole fruit	0 3 7	0.10 0.11 0.06	EA990154/FR01, PYRIP_020 Method: L00.00-34 Storage period: 6 months
Greece, Dionisiou 1999	0.11 0.11	- 9	0.008 0.008	Fruiting	Whole fruit	3	0.12	EA990154/GR01, PYRIP_020 Method: L00.00-34 Storage period: 6 months

Location, Year (variety)	Application				Residues, mg/kg			Report/Trial No., Reference, analytical method, validation data, storage period
	kg ai/ha	Inter-val days	kg ai/hL	Growth stage	Sample	DALA	Parent (mean)	
(Dolmi)								
Italy, Cadegli Oppi di Oppeano 1999 (Senior)	0.11 0.11	- 9	0.008 0.008	Fruiting	Whole fruit	4	0.28	EA990154/IT01, PYRIP_020 Method: L00.00-34 Storage period: 6 months
Spain, Ejido 1999 (Cuzco)	0.11 0.11	- 11	0.008 0.008	Fruiting	Whole fruit	0 4 7	0.21 0.21 0.26	EA990155/SP01, PYRIP_021 Method: L00.00-34 Storage period: 6 months
France, Gontaund de Nogaret 2000 (Denvers)	0.11 0.11	- 10	0.008 0.008	Fruiting	Whole fruit	3	0.07	EA000134/FR01, PYRIP_022 Method: L00.00-34 Storage period: 2 months
Italy, San Giorgio di Mantova 2000 (Eldor)	0.12 0.11	- 10	0.008 0.008	Fruiting	Whole fruit	0 3 7	0.24 0.21 0.12	EA000134/IT01, PYRIP_022 Method: L00.00-34 Storage period: 2 months
Greece, Irnathia 2000 (Royal)	0.11 0.11	- 10	0.008 0.008	Fruiting	Whole fruit Canned fruits	0 3 7 3	0.15 0.13 0.08 0.01	EA000135/GR01, PYRIP_023 Method: L00.00-34 Storage period: 5 months
Spain, El Perello 2000 (Mariner)	0.11 0.11	- 9	0.008 0.008	Fruiting	Whole fruit Canned fruits	3 3	0.25 0.02	EA000137/SP01, PYRIP_024 Method: L00.00-34 Storage period: 4 months

DALA: days after last treatment

Table 19 Residues of pyriproxyfen following spray treatment on tomatoes in greenhouse

Location, Year (variety)	Application				Residues, mg/kg			Report/Trial No., Reference, analytical method, validation data, storage period
	kg ai/ha	Interval days	kg ai/hL	Growth stage	Sample	DALA	Parent (mean)	
cGAP : Italy, 2×0.12 kgai/ha, 14 day interval, PHI : 3 days								
Greece, Profitis 1999 (622)	0.11 0.11	- 11	0.008 0.008	Fruiting	Whole fruit	0 3 7	0.06 0.09 0.11	EA990150/GR01, PYRIP_025 Method: L00.00-34 Storage period: 3 months
Greece, Neo Michaniona 1999 (Noa)	0.11 0.11	- 10	0.008 0.008	Fruiting	Whole fruit	3	0.09	EA990150/GR02, PYRIP_025 Method: L00.00-34 Storage period: 3 months
Italy, Grottammare 1999 (Madrila)	0.11 0.11	- 10	0.008 0.008	Fruiting	Whole fruit	3	0.17	EA990150/IT01, PYRIP_025 Method: L00.00-34 Storage period: 7 months
Spain,	0.11	-	0.008	Fruiting	Whole fruit	3	0.11	EA990151/SP01, PYRIP_026

Location, Year (variety)	Application				Residues, mg/kg			Report/Trial No., Reference, analytical method, validation data, storage period
	kg ai/ha	Interval days	kg ai/hL	Growth stage	Sample	DALA	Parent (mean)	
Puerto de Mazarron 1999 (Vica)	0.11	10	0.008					Method: L00.00-34 Storage period: 2 months
Greece, Karlo Souli 2000 (Iron)	0.11 0.11	- 10	0.008 0.008	Fruiting	Whole fruit Juice Puree Canned	3 3 3 3	0.05 <0.01 0.09 <0.01	EA000138/GR01, PYRIP_027 Method: L00.00-34 Storage period: 4 months
Spain, El Perello 2000 (Bodar)	0.11 0.12	- 10	0.008 0.008	Fruiting	Whole fruit Peeled fruit Peels Juice Puree Ketchup Canned	0 3 7 3 3 3 3 3 3	0.06 0.06 0.04 <0.01 0.44 <0.01 0.04 0.04 <0.01	EA000140/SP01, PYRIP_028 Method: L00.00-34 Storage period: 5 months
Italy, Campofilone 2000 (Naomi)	0.11 0.11	- 10	0.008 0.008	Fruiting	Whole fruit	0 3 7	0.15 0.16 0.18	EA990139/IT01, PYRIP_029 Method: L00.00-34 Storage period: 1 months
Italy, Grottammare 2000 (DRC 145)	0.11 0.11	- 10	0.008 0.008	Fruiting	Whole fruit	0 3 6	0.06 0.08 0.06	EA990139/IT02, PYRIP_029 Method: L00.00-34 Storage period: 6 months

DALA: days after last treatment

FATE OF RESIDUES IN STORAGE AND PROCESSING

Nature of residue during processing

The hydrolysis of pyriproxyfen under processing conditions was investigated by Lewis C.J. (2002, PYRIP_030). [U-phenoxyphenyl-^{14}C] pyriproxyfen was incubated in aqueous buffer solutions at a nominal concentration of 0.1 mg/L under three sets of conditions, each designed to simulate an appropriate process: 90 °C (pH 4, 20 minutes) to simulate pasteurisation, 100 °C (pH 5, 60 minutes), to simulate boiling, baking and brewing, and 120 °C (pH 6, 20 minutes) to simulate sterilisation.

Total recovered radioactivity was measured for each test solution before and after incubation. Radioactive components were characterised by fractionation and co-chromatography with authenticated reference compounds using HPLC and TLC.

Table 20 Hydrolysis of [U-phenoxyphenyl-^{14}C] pyriproxyfen under simulated processing conditions

Process represented	% applied radioactivity pH4 (90 °C, 20min) pasteurisation	pH5 (100 °C, 60min) baking/brewing/cooking	pH6 (120 °C, 20min) sterilisation
Prior to incubation (total radioactivity)	96.5	97.6	96.5
Post incubation (total radioactivity)	94.4	93.9	93.4
pyriproxyfen	93.9	93.6	93.2
others	0.4	0.2	0.1

Residues after processing

The fate of pyriproxyfen during processing of raw agricultural commodity (RAC) was investigated in some supervised field trials on pineapple (not further considered since RAC residues were below LOQ), peppers and tomatoes. As a measure of the transfer of residues into processed products, a processing factor was used, which is defined as:

Processing factor = Residue in processed product (mg/kg) ÷ Residue in raw agricultural commodity (mg/kg)

If residues in the RAC were below the LOQ, no processing factor could be derived. In case of residues below the LOQ, but above the LOD in the processed product, the numeric value of the LOQ was used for the calculation. If residues in the processed product were below the LOD, the numeric value of the LOQ was used for the calculation but the PF was expressed as "less than" (e.g. <0.5).

In the following table the processing factors derived from the supervised field trial results (see section Residues from supervised field trials) are summarised:

Table 21 Summary of processing factors for pyriproxyfen based on supervised field trial data

Commodity	Residues in mg/kg	Processing or peeling factor	Median or best estimate	References
Pepper				
Whole fruit	0.13 / 0.25	-	-	PYRIP_023, PYRIP_024
Canned pepper	0.01 / 0.02	0.08 / 0.08	0.08	
Tomato				
Whole fruit	0.05 / 0.06	-	-	PYRIP_027, PYRIP_028
Juice	<0.01 / <0.01	<0.2 / <0.17	<0.18	
Puree	0.09 / 0.04	1.8 / 0.67	1.2	
Canned tomato	<0.01 / <0.01	<0.2 / <0.17	<0.18	
Peeled fruit	- / <0.01	- / <0.17	<0.17	
Peel	- / 0.44	- / 7.3	7.3	
Ketchup	- / 0.04	- / 0.67	0.67	

APPRAISAL

Pyriproxyfen is classified as a juvenile hormone mimic that interferes with normal insect development and reproduction. Metamorphosis of immature life stages is affected, but adults are not directly controlled, although production of viable eggs is affected by transovarial activity. Pyriproxyfen is absorbed through the insect cuticle but may also act by ingestion.

Pyriproxyfen was first evaluated by the JMPR in 1999 and then in 2000 and 2001. In the 1999 evaluation for toxicity and residues an ADI of 0–0.1 mg/kg bw was established. The Meeting concluded that it was not necessary to establish an ARfD due to the low acute toxicity of pyriproxyfen.

The 1999 JMPR recommended the following residue definition for pyriproxyfen:

Definition of the residue for compliance with the MRL and dietary risk assessment in plant and animal commodities: *pyriproxyfen*

The residue is fat-soluble.

Pyriproxyfen was scheduled at the Forty-ninth Session of the CCPR for the evaluation of additional uses by the 2018 JMPR. The current Meeting received new information on use patterns for pyriproxyfen in bananas, mangoes, papayas, pineapples, cucumbers, cantaloupe melons, peppers and tomatoes supported by additional analytical methods, storage stability data, supervised field trials and nature of residues studies simulating typical processing conditions.

Methods of analysis

The current Meeting received additional analytical information for the analysis of pyriproxyfen and 4-OH-pyriproxyfen in plant matrices.

A modification of the QuEChERS-multimethod as well as the applicability of the L-00.00-34 multimethod (formerly "DFG S19") was tested for matrices with high water and high acid (QuEChERS only) content. GC-MS or GC-MS/MS techniques were successfully validated at a LOQ of 0.01 mg/kg while the LC-MS/MS for the QuEChERS-method achieved a LOQ of 0.02 mg/kg.

In addition, the methods RM-33P-1-3 and RM-33P-1-3a were submitted, utilising GC-NPD for the analysis of pyriproxyfen and HPLC-FLD for the analysis of 4-OH-pyriproxyfen in matrices of high water and high acid content. Samples are extracted with acetone and partitioned first with acetonitrile and hexane and afterwards with dichloromethane. Clean-up is performed with silica gel columns. Both test systems were successfully validated with a LOQ of 0.02 mg/kg. The extraction efficiency of method RM-33P-1-3 was also tested against samples from the apple metabolism study (see JMPR Report 1999). For parent pyriproxyfen an extraction efficiency of 78% was achieved while 4-OH-pyriproxyfen showed a slightly higher extraction rate of 87%.

Stability of residues in stored analytical samples

The Meeting received information on the storage stability of pyriproxyfen in pepper, papaya, mango and pineapple. Parent pyriproxyfen was stable in all matrices investigated with maximum storage periods of at least 3 months for peppers, 19 months for papayas, 6 months for mangos and 16 months for pineapples.

Results of supervised residue trials on crops

The Meeting received supervised trial data for applications of pyriproxyfen on various fruit and vegetables crops conducted in Brunei, Costa Rica, France, Greece, Guatemala, Italy, Malaysia, Panama, Philippines, Spain and the USA.

Banana

Pyriproxyfen is registered for use in bananas in the USA with a GAP comprising of three foliar applications at 0.12 kg ai/ha, a 14 day RTI and a PHI of 14 days. Supervised field trials, matching GAP, conducted in Costa Rica and Guatemala were submitted.

Residues of parent pyriproxyfen in whole bananas were (n = 1): 0.036 mg/kg.

Residues of parent pyriproxyfen in bananas pulp were (n = 1): < 0.01 mg/kg.

The Meeting considered one trial insufficient to estimate a maximum residue level for the use of pyriproxyfen on bananas.

Mango

Pyriproxyfen is registered for use on mango in Malaysia with two foliar spraying of 0.05 kg ai/ha each (14 day interval) and a PHI of 1 day. One corresponding supervised field trial conducted in Malaysia was submitted.

Residues of parent pyriproxyfen in mango fruits were (n = 1): < 0.02 mg/kg.

The Meeting considered one trial insufficient to estimate a maximum residue level for the use of pyriproxyfen on mangoes.

Papaya

Pyriproxyfen is registered for use on papaya in the Philippines with two foliar spraying of 0.1 kg ai/ha each (14 day interval) and a PHI of 1 day. Corresponding supervised field trials conducted in Brunei, Malaysia and the Philippine's were submitted.

Residues of parent pyriproxyfen in whole papayas were (n = 6): 0.03, 0.03, 0.04, 0.1, 0.1 and 0.15 mg/kg.

The Meeting estimated a maximum residue level of 0.3 mg/kg and a STMR of 0.07 mg/kg for pyriproxyfen in papaya.

Pineapple

Pyriproxyfen is registered for use on pineapple in the USA with two foliar sprays of 0.06 kg ai/ha each (21 day interval) and a PHI of 1 day. Corresponding supervised field trials conducted in Panama were submitted.

Residues of parent pyriproxyfen in whole pineapple fruits were (n = 6): < 0.01 mg/kg.

The Meeting estimated a maximum residue level of 0.01 mg/kg and a STMR of 0.01 mg/kg for pyriproxyfen in pineapples.

Cucumbers, gherkins and summer squash

Pyriproxyfen is registered for the specific use on protected cucumbers, gherkins and summer squash in Italy with two foliar sprays of 0.12 kg ai/ha each (14 day interval) and a PHI of 3 days. Supervised field trials approximating the GAP (9-11 day intervals) conducted in France, Greece, Italy and Spain were submitted.

Residues of parent pyriproxyfen in cucumbers were (n = 8): < 0.01(7) and 0.02 mg/kg.

The Meeting estimated a maximum residue level of 0.04 mg/kg and a STMR of 0.01 mg/kg for pyriproxyfen in cucumbers, gherkins and summer squash.

Melons, except watermelons

Pyriproxyfen is registered for use in the USA on field grown cucurbits Group 9 (which include melons) with two foliar spraying of 0.075 kg ai/ha each (14 day interval) and a PHI of 7 days. Corresponding supervised field trials conducted in the USA on cantaloupes were submitted.

Residues of parent pyriproxyfen in whole melons were (n = 7): < 0.01, 0.01, 0.015, 0.016, 0.019, 0.035 and 0.035 mg/kg.

In one trial bearing quantifiable residues in whole melons, corresponding pyriproxyfen residues in melon pulp were < 0.01 mg/kg.

The Meeting estimated a maximum residue level of 0.07 mg/kg and a STMR value of 0.016 mg/kg for pyriproxyfen in melons, except watermelons.

Peppers and eggplants

Pyriproxyfen is registered for use on protected peppers and eggplants in Italy with two foliar sprays of 0.12 kg ai/ha each (14 day RTI) and a PHI of 3 days. Supervised field trials approximating the GAP rate (9–11 day re-treatment intervals) conducted in France, Greece, Italy and Spain were submitted.

Residues of parent pyriproxyfen in peppers were (n = 8): 0.07, 0.11, 0.12, 0.13, 0.21, 0.25, 0.26 and 0.28 mg/kg.

The Meeting estimated a maximum residue level of 0.6 mg/kg and a STMR of 0.17 mg/kg for pyriproxyfen in peppers and decided to extrapolate its recommendations to eggplant also.

Based on a default processing factor of 10, the Meeting also estimated a maximum residue level of 6 mg/kg and a STMR of 1.7 mg/kg for pyriproxyfen in peppers chili, dried.

Tomatoes

Pyriproxyfen is registered for use on protected tomatoes in Italy with two foliar sprays of 0.12 kg ai/ha each (14 day interval) and a PHI of 3 days. Supervised field trials approximating the GAP (10–11 day re-treatment intervals) conducted in France, Greece, Italy and Spain were provided.

Residues of parent pyriproxyfen in tomatoes were (n = 8): 0.05, 0.08, 0.06, 0.09, 0.11, 0.11, 0.17 and 0.18 mg/kg.

The Meeting estimated a maximum residue level of 0.4 mg/kg and a STMR of 0.1 mg/kg for pyriproxyfen in tomatoes.

Fate of residues during processing

The Meeting received information on the hydrolysis of radio-labelled pyriproxyfen as well as processing studies using unlabelled material on incurred residues in peppers and tomatoes.

In a hydrolysis study using radio-labelled pyriproxyfen typical processing conditions were simulated (pH 4,5 and 6 with 90 °C, 100 °C and 120 °C for 20, 60 and 20 minutes). In duplicate samples of sterile buffer solution no degradation was observed.

The fate of pyriproxyfen residues has been examined simulating household and commercial processing of peppers and tomatoes.

Estimated processing factors for the commodities considered at this Meeting are summarised below.

Raw commodity	Processed commodity	Pyriproxyfen		
		Individual processing factors	Mean or best estimate processing factor	STMR-P in mg/kg
Pepper	Canned pepper	0.08, 0.08	0.08	0.014
Tomato	Juice	< 0.17, < 0.2	< 0.18	0.018
	Puree	0.67, 1.8	1.2	0.12
	Canned tomato	< 0.17, < 0.2	< 0.18	0.018
	Ketchup	0.67	0.67	0.067

Residues in animal commodities

The Meeting noted that no commodities considered by the current Meeting are relevant for livestock animal feeding.

RECOMMENDATIONS

On the basis of the data from supervised trials the Meeting concluded that the residue levels listed below are appropriate for establishing a maximum residue level and for an IEDI assessment.

Definition of the residue for compliance with the MRL and dietary risk assessment in plant and animal commodities: *pyriproxyfen*

The residue is fat-soluble.

Maximum residue levels and dietary exposure

Commodity		MRL, mg/kg		STMR or STMR-P, mg/kg
CCN	Name	New	Previous	
VC 0424	Cucumbers	0.04	-	0.01
VO 0440	Eggplant	0.6	-	0.17
VC 0425	Gherkins	0.04	-	0.01
VC 0046	Melons, except watermelons	0.07	-	0.016
FI 0350	Papaya	0.3	-	0.07
VO 0051	Peppers	0.6	-	0.17
HS 0444	Peppers chili, dried	6	-	1.7
FI 0353	Pineapple	0.01	-	0.01
VC 0431	Summer squash	0.04	-	0.01
VO 0448	Tomato	0.4	-	0.1

Dietary exposure only

Commodity		STMR-P, mg/kg
CCN	Name	
	Canned pepper	0.014
	Tomato Juice	0.018
	Tomato Puree	0.12
	Canned tomato	0.018
	Ketchup	0.067

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for pyriproxyfen is 0–0.1 mg/kg bw. The International Estimated Daily Intakes (IEDIs) for pyriproxyfen were estimated for the 17 GEMS/Food Consumption Cluster Diets using the STMR or STMR-P values estimated by the JMPR. The results are shown in Annex 3 of the 2018 JMPR Report. The IEDIs ranged from 0–1% of the maximum ADI.

The Meeting concluded that long-term dietary exposure to residues of pyriproxyfen from uses considered by the JMPR is unlikely to present a public health concern.

Acute dietary exposure

The 1999 JMPR decided that an ARfD for pyriproxyfen was unnecessary. The Meeting therefore concluded that the acute dietary exposure to residues of pyriproxyfen from the uses considered is unlikely to present a public health concern.

REFERENCES

Code	Author	Year	Title, Institute, Report reference
PYRIP_001	Borbón Martínez O.	2017	Magnitude of the residues of pyriproxyfen on banana; Report no.: IR-4 PR No. B11399
PYRIP_002	Candanedo Lay E.M.	2016	Pyriproxyfen: Magnitude of the residue on pineapple; Report no.: IR-4 PR No. 11398
PYRIP_003	Keong, N.Ch.	2017	Pyriproxyfen: Magnitude of the residue on mango; Report no.: IR-4 PR No. 10990
PYRIP_004	Chuan Mun Choy, J.	2015	Analytical summary report to: Pyriproxyfen: Magnitude of the residue on mango; Report no.: IR-4 PR No. 10990
PYRIP_005	Fauzan bin Yunus, M.	2017	Analytical summary report to: Pyriproxyfen: Magnitude of the residue on mango; Report no.: IR-4 PR No. 10990
PYRIP_006	Green C.A.	1996	Determination of pyriproxyfen and 4'-OH-pyriproxyfen residues in apples, pears, and citrus fruits;
PYRIP_007	Wood B.	1997	Independent laboratory validation of Valent analytical method RM-33P-1-3 for determining pyriproxyfen and 4'-OH-pyriproxyfen residues in/on apples and oranges;

Code	Author	Year	Title, Institute, Report reference
PYRIP_008	Green C.A.	1998	Determination of pyriproxyfen residues in apples, pears, and citrus fruits;
PYRIP_009	Weber H., Pelz S.	2000	Validation of DFG method S19 (extended revision) for the determination of residues of pyriproxyfen in samples of commodities with high water content (cucumber); Report no.: SUM-9910V
PYRIP_010	Kretschmer S.	2001	Pyriproxyfen: Independent laboratory validation (ILV) of the multi-residue method DFG S19 (extended revision) for the determination of residues of pyriproxyfen in watery crops (cucumber); Report no.: B435G
PYRIP_011	Pensyl J.W.	1997	Radiovalidation of the residue analytical method for determining Residues pyriproxyfen and its degradates in apples;
PYRIP_012	Pensyl, J.W.	1998	Magnitude of the residues of pyriproxyfen and its degradates in peppers; Report no.: VP-11461
PYRIP_013	Samoil, K.S.	2011	Pyriproxyfen: Magnitude of the residue on papaya; Report no.: IR-4 PR 09486
PYRIP_014	Borbón Martínez O.	2017	Magnitude of the residues of pyriproxyfen on banana; Report no.: IR-4 PR No. B11399
PYRIP_015	Grolleau G.	2000	Magnitude of the residue of pyriproxyfen in greenhouse cucumber raw agricultural commodity - Southern France and Greece 1999; Study no.: EA990152; Report no.: NNR-0068
PYRIP_016	Grolleau G.	2000	Magnitude of the residue of pyriproxyfen in greenhouse cucumber raw agricultural commodity - Spain 1999; Study no.: EA990153; Report no.: NNR-0091
PYRIP_017	Grolleau G.	2001	Magnitude of the residue of pyriproxyfen in greenhouse cucumber raw agricultural commodity - Southern France and Italy 2000; Study no.: EA000123; Report no.: NNR-0074
PYRIP_018	Grolleau G.	2001	Magnitude of the residue of pyriproxyfen in greenhouse cucumber raw agricultural commodity - Greece 2000; Study no.: EA000124; Report no.: NNR-0092
PYRIP_019	Green C.A.	2001	Magnitude of the residues of pyriproxyfen on cantaloupe; Report no.: 200100283 (NNR-0098)
PYRIP_020	Grolleau G.	2000	Magnitude of the residue of pyriproxyfen in greenhouse pepper raw agricultural commodity - Southern France, Greece and Italy 1999; Study no.: EA990154; Report no.: NNR-0069
PYRIP_021	Grolleau G.	2000	Magnitude of the residue of pyriproxyfen in greenhouse pepper raw agricultural commodity - Spain - 1999; Study no.: EA990155; Report no.: NNR-0088
PYRIP_022	Grolleau G.	2001	Magnitude of the residue of pyriproxyfen in greenhouse pepper raw agricultural commodity - Southern France and Italy - 2000; Study no.: EA000134; Report no.: NNR-0076
PYRIP_023	Grolleau G.	2001	Magnitude of the residue of pyriproxyfen in greenhouse pepper raw agricultural commodity and processed fractions - Greece - 2000; Study no.: EA000135; Report no.: NNR-0090
PYRIP_024	Grolleau G.	2001	Magnitude of the residue of pyriproxyfen in greenhouse pepper raw agricultural commodity and processed fractions - Spain - 2000; Study no.: EA000137; Report no.: NNR-0089
PYRIP_025	Grolleau G.	2000	Magnitude of the residue of pyriproxyfen in greenhouse tomato raw agricultural commodity - Greece and Italy 1999; Study no.: EA990150; Report no.: NNR-0067
PYRIP_026	Grolleau G.	2000	Magnitude of the residue of pyriproxyfen in greenhouse tomato raw agricultural commodity - Spain - 1999; Study no.: EA990151; Report no.: NNR-0085
PYRIP_027	Grolleau G.	2001	Magnitude of the residue of pyriproxyfen in greenhouse tomato raw agricultural commodity and processed fractions - Greece - 2000; Study no.: EA000138; Report no.: NNR-0086
PYRIP_028	Grolleau G.	2001	Magnitude of the residue of pyriproxyfen in greenhouse tomato raw agricultural commodity and processed fractions - Spain - 2000; Study no.: EA000140; Report no.: NNR-0087
PYRIP_029	Grolleau G.	2001	Magnitude of the residue of pyriproxyfen in greenhouse cherry tomato raw agricultural commodity - Italy - 2000; Study no.: EA000139; Report no.: NNR-0075
PYRIP_030	Lewis C.J.	2002	[^{14}C]Pyriproxyfen: Nature of the residue study; Study no.: 333/204
PYRIP_031	Tejada A.W.	2017	Pyriproxyfen: Magnitude of the residue on papaya; Report no.: IR-4 PR No. 11251