

THIAMETHOXAM (245)

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EXPLANATION

Thiamethoxam was evaluated for toxicology and residues as a new compound in 2010, with many MRL recommendations.

The data from passion fruit trials in Kenya have been provided to support a thiamethoxam MRL on passion fruit. The data had previously been generated within the COLEACP (Comité de Liaison Europe-Afrique-Caraïbes-Pacifique) program to seek an EU import tolerance (Henning-Helbig, thiamethoxam, import tolerance request).

METHODS OF RESIDUE ANALYSIS

Analytical methods

The Meeting received descriptions of the thiamethoxam analytical methods used for analysis of passion fruit samples from the trials. The descriptions were supported by procedural recoveries and representative chromatograms.

Low-fat products (Wasser, 2005, R A6003)

Analyte:	thiamethoxam	LC-MS-MS	Method M1 ⁷
LOQ:	0.01 mg/kg.		
Description	Residues are extracted with acetonitrile + acetic acid (999+1) with added magnesium sulphate and sodium chloride. Triphenyl phosphate is used as an internal standard. After cleanup by solid-phase dispersion followed by centrifuging, the analytes are measured by LC-MS-MS (precursor ion m/z = 291.8).		

Passion fruit (Leak, 2007, PGD-274)

Analyte:	thiamethoxam	LC-MS-MS	Method PGD-274
Reporting limit:	0.02 mg/kg.		
Description	Whole fruit, flesh or peel samples are homogenized in the presence of solid carbon dioxide. A portion of homogenized sample is then extracted and homogenized with ethyl acetate and sodium sulphate. After filtration the extract may be stored in a freezer. An aliquot of the extract is concentrated to dryness under nitrogen. The residue is then dissolved in methanol-water (50:50, v/v) and the solution filtered ready for analysis by LC-MS-MS (precursor ion m/z = 292). The method relies on a calibration with matrix-matched external standards.		

In Method M1, peak area ratio (thiamethoxam: internal standard) was proportional to concentration ratio (thiamethoxam: internal standard) over the working range, demonstrating linearity of response.

In method PGD-274, peak area for thiamethoxam was proportional to amount injected in matrix-matched standards over the working range, also demonstrating adequate linearity of response.

Table 1 Procedural analytical recoveries for spiked thiamethoxam in passion fruit

Commodity	Spiked compound	Spike conc., mg/kg	n	Recoveries %	Method	Ref
Passion fruit	thiamethoxam	0.01	3	87 95 87	M1	R A6003
Passion fruit	thiamethoxam	0.1	1	103	M1	R A6003
Passion fruit	thiamethoxam	3	3	87 89 93	M1	R A6003

⁷ Method M1: QuEChERS, A multi-residue method for the analysis of pesticide residues in low-fat products.

Commodity	Spiked compound	Spike conc., mg/kg	n	Recoveries %	Method	Ref
Passion fruit peel	thiamethoxam	6	1	93	M1	R A6003
Passion fruit	thiamethoxam	0.1	2	121 169	PGD-274	PGD-274

Thiamethoxam residue levels in the control samples were at or below LOD (0.001 mg/kg) in study R A6003 and below LOQ (0.02 mg/kg) in study PGD-274.

USE PATTERN

No labels or information on the registered uses of thiamethoxam on passion fruit have been received.

A proposal for critical GAP in Kenya described the use of thiamethoxam on passion fruit as: application rate 100 g ai/ha, PHI 3 days, 3 treatments with 14 days intervals between treatments (Anon, 2011). The thiamethoxam product, Actara 25 WG, was not registered for use on passion fruit in Kenya as of February 2011 (Anon, 2011).

The intended use pattern for passion fruit in Kenya was provided as part of the field trials conducted within the Pesticide Initiative Programme of COLEACP⁸ aiming to provide data for establishing import MRLs in the European Union. The application conditions were based on the requirement of appropriate control of diseases, but they were not supported by label or official declaration of the approved use.

RESIDUES RESULTING FROM SUPERVISED TRIALS

The Meeting received information on supervised field trials for thiamethoxam foliar use on passion fruit in Kenya. Summaries and laboratory and field reports were provided.

Trials were generally well documented with laboratory and field reports.

The trial design was unreplicated single plot. The pesticide was applied by knapsack sprayer. Plot sizes were: 6–8 vines in 2005 and 10 vines in 2007. Field sample size was 22–25 fruits or at least 1 kg. Samples were stored frozen before analysis for approximately 9 months in 2006 and 1 month in 2007.

The thiamethoxam trials were part of a residue program with a number of pesticides. In the trials commenced in 2005, thiamethoxam was applied as a foliar spray in a mixture with four other pesticides: lambda-cyhalothrin, sulphur, azoxystrobin and malathion. In the trials of 2007, thiamethoxam was applied as a foliar spray with two other pesticides: azoxystrobin and chlorothalonil.

Laboratory reports included procedural analytical recoveries. Dates of analyses were also provided. Although trials included control plots, no control data are recorded in the tables. Residues in samples from control plots did not exceed the LOQ.

Samples were analysed for thiamethoxam, but not for metabolite CGA 322704 (*N*-(2-chlorothiazol-5-ylmethyl)-*N'*-methyl-*N''*-nitroguanidine).

Analytical method M1 was used for the trials in 2005 (KE/PF/2005). Analytical method PGD-274 was used for the trials in 2007 (KE/PF/2007). Procedural recovery tests at the same times as the analyses of the supervised field trial samples are summarised in Table 2.

The two procedural recoveries for analytical method PGD-274, 121 % and 169 %, suggest that the method was not under control and that the residue data generated by this method cannot be accepted as valid.

Residue data are recorded unadjusted for analytical recovery.

⁸ The COLEACP is a professional association with the objective to harmonise the relationships between producers and exporters from African, Caribbean and Pacific countries (ACP) and EU importers.

A practical PHI of 3 days is needed for passion fruit, which must be regularly harvested over a production season that may last for several months. All of the trials included data for a PHI of 3 days.

Table 2 Thiamethoxam residues in passion fruit resulting from supervised trials in Kenya

PASSION FRUIT country, year (variety)	Application				PHI days	Commodity	Residue, mg/kg thiamethoxam	Ref
	Form	kg ai/ha	water (l/ha)	no. interval				
Kenya, 2005 (Purple variety grafted on yellow rootstock)	WG	0.1	1000	3 14 days	1	whole fruit	0.15	KE/PF/2005/1 Site: Thika
					3	peel	0.33	
					3	flesh	0.01	
					3	whole fruit	0.087	
					7	whole fruit	0.051	
					14	whole fruit	0.029	
21	whole fruit	0.020						
Kenya, 2005 (Purple variety grafted on yellow rootstock)	WG	0.1	1000	3 14 days	1	whole fruit	0.090	KE/PF/2005/2 Site: Embu
					3	peel	0.094	
					3	flesh	0.016	
					3	whole fruit	0.10	
					7	whole fruit	0.049	
					14	whole fruit	0.026	
21	whole fruit	0.019						
Kenya, 2005 (Purple variety grown from seedlings)	WG	0.1	1000	3 14 days	1	whole fruit	0.11	KE/PF/2005/3 Site: Karatina
					3	peel	0.14	
					3	flesh	0.02	
					3	whole fruit	0.087	
					7	whole fruit	0.024	
					14	whole fruit	0.014	
21	whole fruit	<0.01						
Kenya, 2007 (Purple variety grafted on yellow rootstock)	WG	0.1	1020	3 14 days	1	whole fruit	0.51	KE/PF/2007/01 Site: Thika ^a
					3	peel	0.41	
					3	flesh	0.094	
					3	whole fruit	0.23	
					7	whole fruit	0.28	
					14	whole fruit	0.34	
21	whole fruit	0.15						
Kenya, 2007 (Purple variety grafted on yellow rootstock)	WG	0.1	1020	3 14 days	1	whole fruit	0.42	KE/PF/2007/02 Site: Embu ^a
					3	peel	0.39	
					3	flesh	0.076	
					3	whole fruit	0.30	
					7	whole fruit	0.18	
					14	whole fruit	0.16	
21	whole fruit	0.080						

^a Residue data not accepted as valid because of aberrant recoveries. The two procedural recoveries were 121 % and 169 % for method PGD-274 used in generating the data for the trials in 2007 (KE/PF/2007).

APPRAISAL

Thiamethoxam was evaluated as a new compound in 2010. Thiamethoxam residue data for a use on passion fruit have been provided in 2011 to support an MRL on passion fruit.

The Meeting received descriptions of the thiamethoxam analytical methods used for analysis of passion fruit samples from the trials.

The analytical methods used in the supervised trials relied on LC-MS-MS and achieved LOQs of 0.01 and 0.02 mg/kg for thiamethoxam. Metabolite CGA 322704 (*N*-(2-chlorothiazol-5-ylmethyl)-*N'*-methyl-*N''*-nitroguanidine) was not analysed with these methods.

The Meeting has not received labels or information on the registered uses of thiamethoxam on passion fruit.

The Meeting received information on supervised field trials for thiamethoxam foliar use on passion fruit in Kenya. Samples were analysed for thiamethoxam, but not for metabolite CGA 322704.

Thiamethoxam was applied at 0.1 kg ai/ha in 3 supervised trials in 2005 and 2 from 2007. The residue data from the trials in 2007 were not accepted as valid because of the aberrant procedural recoveries (> 120%) of the analytical method.

The application conditions were based on the requirement of appropriate control of diseases of passion fruit, but they were not supported by label or official declaration of approved use. Therefore, the Meeting could not estimate a maximum residue level for thiamethoxam in passion fruit.

REFERENCES

Code	Author	Year	Title
	Anon	2011	Thiamethoxam. Passion fruit. Request for Codex MRLs. On behalf of COLEACP-PIP. Unpublished.
KE/PF/2005	Henning-Helbig S Delhove G	no date 2006	Thiamethoxam. Import tolerance request. COLEACP-PIP, Belgium. Report on residues trials in Kenya on passion fruit. PIP Pesticide Residues Trials Programme. Trial KE/PF/2005/01-02 and 03. COLEACP (MU/PIP). Belgium. Unpublished.
KE/PF/2007	Delhove G	2007	Report on residues trials in Kenya on passion fruit. PIP Pesticide Residues Trials Programme. Trial KE/PF/2007/01-02. COLEACP (MU/PIP). Belgium. Unpublished.
PGD-274	Leak J	2007	Analysis for residues of pesticide combination number one in passion fruit from Kenya in support of the Pesticides Initiative Programme. Study PGD-274. Central Science Laboratory, UK. Unpublished.
R A6003	Wasser C	2005	Magnitude of the residue of: thiamethoxam, acetamiprid, deltamethrin, lambda-cyhalothrin, beta-cyfluthrin, acrinathrin, azoxystrobin, trifloxystrobin, malathion (and its metabolite malaaxon), acephate (and its metabolite methamidophos), dimethoate (and its metabolite omethoate), buprofezin, fenpyroximate, propineb, pyrethrins, spinosad, hexythiazox, sulfur, captan, (and its metabolite tetrahydrophthalimide), thiocyclam (and its metabolite nereistoxine), thiophanate methyl and its metabolite carbendazim, spiromesifen, and copper in passion fruit raw agricultural commodity. Kenya 2005. Report R A6003. COLEACP (MU/PIP). Belgium. Anadiag, France. Unpublished.