PROTHIOCONAZOLE

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EXPLANATION

Prothioconazole is a systemic triazolinthione fungicide that is used for the control of diseases caused by *Ascomycetes*, *Basidiomycetes* and *Deuteromycetes* in a range of crops. It is registered for use in a variety of crops worldwide.

Prothioconazole was evaluated for the first time by the 2008 JMPR, which established an ADI of 0–0.05 mg/kg bw and an ARfD of 0.8 mg/kg bw for woman of child bearing age for the parent prothioconazole, and an ADI of 0–0.01 mg/kg bw and an ARfD of 0.01 mg/kg bw for woman of child bearing age and 1 mg/kg bw for general population for prothioconazole-desthio. In addition, the Meeting recommended limits for a number of plant commodities and for mammalian meat and fats, edible offal and milks, based on a residue definition of ‘prothioconazole-desthio’.

The 2008 JMPR meeting concluded that the residue definition for plant commodities for enforcement and dietary risk assessment was prothioconazole-desthio. The residue definition for animal commodities for enforcement was prothioconazole-desthio and for dietary risk assessment the sum of prothioconazole-desthio, prothioconazole-desthio-3-hydroxy, prothioconazole-desthio-4-hydroxy and their conjugates, expressed as prothioconazole-desthio. The residue was considered to be not fat-soluble for the purposes of residue definition.

The 2009 JMPR evaluated residue data on additional commodities.

For the 2014 Meeting data were provided on blueberry, corn, cranberry, cucurbits, potato, sweet corn peanut and soya bean together with current use recommendations.

METHODS OF RESIDUE ANALYSIS

A number of validated analytical methods for the determination of residues in plant, animal tissue, milk and soils were evaluated by the 2007 and 2008 JMPR Meetings.

Detailed descriptions of additional analytical methods for crops that had been used in the residue trials evaluated by this Meeting are described below. References to report numbers are in parenthesis. Recovery data obtained during validation of the methods for plant and animal matrices are summarized in Table 1.

Analytical methods for plant matrices

Method RPA JA/03/01

Corn/sweet corn (Krolski & Harbin, 2008. RAJAP004; Krolski & Harbin, 2008. RAJAP001), soya beans (RAJAY026 and RAJAY027 in 2008/2009 JMPR)

Analyte:	Prothioconazole Prothioconazole-desthio Prothioconazole-sulfonic acid	HPLC/MS/MS	Method RPA JA/03/01
LOQ:	0.02 mg/kg		
Description:	Method RPA JA/03/01 was previously evaluated by JMPR in 2008. It has been used for the corn/sweet corn trials evaluated by this Meeting.		

Method JA-001-P04-02

Blueberry (Murphy & Dallstream, 2012, RAJAP022), cranberry (Murphy & Dallstream, 2012, RAJAP023), cucurbits (Murphy & Harbin, 2012, RAJAP024).

Analyte:	Prothioconazole Prothioconazole-desthio Prothioconazole-sulfonic acid	HPLC/MS/MS	Method JA-001-P04-02 in report JA-001-P04-02
LOQ:	0.02 mg/kg		
Description	This is a modification of method RPA JA/03/01 and includes information about potential alternative analytical conditions. The crop matrices are extracted with a mixture of methanol (MeOH), 30% hydrogen peroxide (H_2O_2), and aqueous sodium bicarbonate ($NaHCO_3$) at about 64°C for 2 hours. This extraction procedure converts prothioconazole to a mixture of prothioconazole sulfonic acid and desthio- prothioconazole. Residues of prothioconazole-desthio are extracted without change. Following the addition of a mixture of isotopically labelled prothioconazole sulfonic acid and prothioconazole-desthio internal standards, the sample extracts are purified by octadecyl solid phase extraction (C-18 SPE). The purified analytes are analysed by high performance liquid chromatography-electrospray/tandem mass spectrometry (LC-MS/MS).		

Method 01013

Soya beans (Resende, 2013, F12-012 and Resende, 2013, F13-008), corn (Freitag & Reineke, 2009, 08-2035; Freitag & Reineke, 2009, 08-2036; Freitag, 2008, RA-2675/07; Bomke & Teubner, 2013, 11-2109; Bomke, 2012, 11-2110; Santiago, 2012, F11-034 and Resende, 2013, F12-011), potato (Bomke & Bauer, 2011, 10-2085) and oilseed rape (Freitag, Reineke & Krusell, 2010, 08-2112; Freitag & Hoffmann, 2010, 08-3112)

Analyte:	Prothioconazole Prothioconazole-desthio	HPLC/MS/MS	Method 01013 in reports MR-06/138, VM11-003 and VM12-006.
LOQ:	0.01 mg/kg		
Description:	Bayer method no. 01013 is suitable for the determination of the relevant residues of bixafen (BYF00587), prothioconazole, tebuconazole, trifloxystrobin and the metabolites BYF00587-desmethyl, prothioconazole-desthio (SXX0665) and CGA321113 in plant materials. All analytes are extracted from plant materials using a mixture of acetonitrile/water (4/1; v/v, containing cysteine hydrochloride, by high-speed blending. After filtration of the extract, the stable isotopically labelled analytes are added. The solution is made up to volume, diluted and subjected to reversed phase HPLC-MS/MS without a further clean-up step. Residues are quantified using internal stable labelled standards. Prothioconazole-desthio is detected using electrospray ionization in the positive ion mode (ESI+). The following transition ions are used for prothioconazole-desthio: for quantification $m/z = 312 \rightarrow m/z = 70$ for confirmation $m/z = 312 \rightarrow m/z = 125$ Prothioconazole is detected using electrospray ionization in the negative ion mode (ESI-). The following transition ions are used for prothioconazole:		

for quantification	$m/z = 342 \rightarrow m/z = 100$
for confirmation	$m/z = 342 \rightarrow m/z = 125$

Method 01013 was developed for the determination of residues of prothioconazole and prothioconazole-desthio in/on plant materials, and validated in citrus fruit, pea seed, wheat grain, rape seed and corn green material (MR-06/138). The method has additionally been validated for prothioconazole and prothioconazole-desthio in soya bean seed and wheat grain (Resende, 2013, VM12-006) and also in bean seed, soya bean seed, oat grain, citrus fruit and sugar cane (Santiago, 2012, VM11-003) using the chromatographic conditions described in method 00765 (Sur, 2003, MR-231/02).

Method 00598/M001

Potato (Freitag & Wolters, 2007, RA-2569/05; Freitag & Wolters, 2007, RA-2604/05; Freitag & Wolters, 2007, RA-2570/05 and Freitag & Wolters, 2007, RA-2603/05)

Analyte:	Prothioconazole	HPLC/MS/MS	Method 00598/M001
	Prothioconazole-desthio		in report MR-689/99
LOQ:	Wheat and barley grain, canola seed: 0.01 mg/kg		
	Wheat and barley forage and straw: 0.05 mg/kg		
	Potato tubers: 0.01 mg/kg		

Description: Method 00598/M001 is a modification of method 00598 previously evaluated by JMPR in 2008. Method 00598/M001 was designed to measure prothioconazole and its main metabolite prothioconazole-desthio in cereal and canola matrices. It has been used in potato trials included in this evaluation.

Prothioconazole and prothioconazole-desthio are extracted from the samples with an acetonitrile/water mixture by high-speed blending after addition of cysteine-hydrochloride solution. After filtration the extract is cleaned up by liquid-liquid partition using n-hexane (saturated with acetonitrile) and dichloromethane. The analytes are chromatographed by reversed-phase HPLC on a silica-based C18-column using isocratic acetonitrile/water eluent containing acidic acid. The analytes are determined by HPLC-MS/MS in the MRM mode. Calibration is performed against internal labelled bracketing standards.

Prothioconazole-desthio is detected using electrospray ionization in the positive ion mode (ESI+) using the following transition ions for quantification: $m/z = 312 \rightarrow m/z = 70$.

Prothioconazole is detected using electrospray ionization in the negative ion mode (ESI-), using the following transition ions for quantification: $m/z = 342 \rightarrow m/z = 100$.

Method 00598/M001 was validated in wheat and barley grain, green material and straw, and canola seed, green material, straw and pod (Heinemann, 2000, MR-689/99).

Method ATM-0053

Peanut (Ellis, 2012, BCS-0232 and Ellis, 2012, BCS-0383)

Analyte:	Prothioconazole	HPLC/MS/MS	Method ATM-0053
	Prothioconazole-desthio		in report BCS-0232

LOQ: Peanut Seeds, peanut shells: 0.01 mg/kg

Peanut forage: 0.05 mg/kg

Description: Method ATM-0053 was used to analyse residues of prothioconazole and prothioconazole-desthio in plant material. It has been used for the peanut trials included in this evaluation.

Residues are extracted with 20:80 water/acetonitrile after the addition of 12.5 g/L L-cysteine. The extract is filtered using a 0.45 µm filter. The analytes are determined by HPLC-MS/MS in the MRM mode. Quantitation is achieved using matrix matched analytical standards and a stable labelled internal standard for prothioconazole.

Detection is by turbospray ionization in the positive ion mode using the following transition ions for quantification:

Prothioconazole-desthio: *m/z* = 312 → *m/z* = 70.

Prothioconazole: *m/z* = 344 → *m/z* = 154.

Method ATM-0053 was developed for the determination of residues of prothioconazole and prothioconazole-desthio in/on cereal grain, straw and forage, canola seed and almond hull and shell. Additional recoveries in peanut matrices are provided in reports Ellis, 2012, BCS-0232 and Ellis, 2012, BCS-0383.

Analytical methods for animal matrices

Method 01009

Bovine milk, muscle, liver, kidney and fat, poultry egg (Billian & Wolters, 2006, MR-06/120 and Schulte & Oel, 2013, amendment 1 to MR-06/120)

Analyte:	JAU 6476-desthio	HPLC/MS/MS	Method: 01009
	JAU 6476-3-hydroxy-desthio		in reports MR-06/120 and
	JAU 6476-4-hydroxy-desthio		P 111G
	JAU 6476-3,4-dihydroxy-desthio		
	JAU 6476-4,5-dihydroxy-desthio		

LOQ: 0.01 mg/kg

Description: Method 01009 is for the determination of relevant residues of prothioconazole (JAU 6476-desthio, JAU 6476-3-hydroxy-desthio, JAU 6476-4-hydroxy-desthio, JAU 6476-3,4-dihydroxy-desthio, and JAU 6476-4,5-dihydroxy-desthio) in/on matrices of animal origin. This method has been independently validated (P 1111 G).

Residues are extracted from cattle (milk, muscle, kidney, liver, fat) and poultry (egg) matrices with acetonitrile / water (4/1, v/v) using a high-speed blender. Subsequently, the solutions are refluxed for 2 hours with 5 N HCl. This hydrolysis step cleaves conjugates to agylcones and converts the metabolites with diene structure back to aromatic compounds. Residues of all analytes are determined using HPLC-MS/MS. Residues are quantified against matrix-matched standards.

The following MRM transitions for quantitation and confirmation are used:

JAU 6476-desthio: m/z 312→70 or 312→125

JAU 6476-3-hydroxy-desthio: m/z 328→70 or 328→141

JAU 6476-4-hydroxy-desthio: m/z 328→70 or 328→141

JAU 6476-3,4-hydroxy-desthio: m/z 344→70 or 344→157

JAU 6476-4,5-hydroxy-desthio: m/z 344→70 or 344→157

Bovine meat, milk and egg (Bacher, 2006, P 1111 G)

Analyte: JAU 6476-desthio HPLC/MS/MS Method: 01009

JAU 6476-3-hydroxy-desthio

JAU 6476-4-hydroxy-desthio

JAU 6476-3,4-dihydroxy-desthio

JAU 6476-4,5-dihydroxy-desthio

LOQ: 0.01 mg/kg

Description: This is the independent laboratory validation (ILV) for method 01009, as described above.

Recovery data obtained during the validation of the methods described above are summarized in Table 1. In addition to these data, concurrent recoveries were also determined together with the analysis of field trial samples. Mean recoveries from both method validation and concurrent recoveries were within the acceptable range of 70%-120% with % RSD < 20%.

Multi-residue Methods

A GG/MS multi-residue method has been validated for the analysis of prothioconazole-desthio, and this method was evaluated at the 2008 JMPR Meeting.

A summary of recovery data with the methods used for plant commodities in this submission are presented in the table 1.

Table 1Summary of recovery data obtained during method validation

Commodity	Analyte	Fortification mg/kg	n	Range Recovery (%)	Mean recovery (%)	% RSD	Method	Reference
Plant commodities								
Blueberries	Prothioconazole-desthio	0.02-1.5	14	94-106	101	3.3	JA-001-P04-02	Murphy & Dallstream, 2012, RAJAP022
	Prothioconazole-sulfonic acid	0.02-1.5	14	75-106	97	8.7		
	Prothioconazole [a]	0.02-1.5	14	65-102	87	11.5		
Cranberries	Prothioconazole-desthio	0.02-1.5	11	84-118	103	10.3	JA-001-P04-02	Murphy & Dallstream, 2012, RAJAP023
	Prothioconazole-sulfonic acid	0.02-1.5	11	94-108	101	4.6		
	Prothioconazole [a]	0.02-1.5	12	88-103	96	6.1		
Summer squash	Prothioconazole-desthio	0.02-1.5	13	93-111	102	6.2	JA-001-P04-02	Murphy & Harbin, 2012, RAJAP024
	Prothioconazole-sulfonic acid	0.02-1.5	13	86-102	93	4.6		
	Prothioconazole [a]	0.02-1.5	15	85-105	90	5.4		
Melon	Prothioconazole-desthio	0.02-1.5	12	94-112	103	5.2	JA-001-P04-02	Murphy & Harbin, 2012, RAJAP024
	Prothioconazole-sulfonic acid	0.02-1.5	12	88-100	93	4.1		
	Prothioconazole [a]	0.02-1.5	12	70-105	92	8.9		

Commodity	Analyte	Fortification mg/kg	n	Range Recovery (%)	Mean recovery (%)	% RSD	Method	Reference
Cucumber	Prothioconazole-desthio	0.02-1.5	10	88-113	102	7.3	JA-001-P04-02	Murphy & Harbin, 2012, RAJAP024
	Prothioconazole-sulfonic acid	0.02-1.5	10	87-97	93	3.6		
	Prothioconazole [a]	0.02-1.5	11	83-106	97	8.2		
Maize/Corn stover	Prothioconazole-desthio	0.02-20	16	79-112	93	10.7	RPA JA/03/01	Krolski & Harbin, 2008. RAJAP004
	Prothioconazole-sulfonic acid	0.02-20	16	89-103	98	4.1		
Maize/Corn grain	Prothioconazole-desthio	0.02-0.20	14	89-108	98	5.4	RPA JA/03/01	Krolski & Harbin, 2008. RAJAP004
	Prothioconazole-sulfonic acid	0.02-0.20	14	93-108	101	4.6		
Maize/Corn forage	Prothioconazole-desthio	0.02-10	18	86-110	97	6.5	RPA JA/03/01	Krolski & Harbin, 2008. RAJAP004
	Prothioconazole-sulfonic acid	0.02-10	18	90-108	97	4.7		
Sweetcorn ear without husk	Prothioconazole-desthio	0.02-0.20	13	88-104	96	5.2	RPA JA/03/01	Krolski & Harbin, 2008. RAJAP004
	Prothioconazole-sulfonic acid	0.02-0.20	13	86-103	96	5.8		
Sweetcorn forage	Prothioconazole-desthio	0.02-10	18	86-110	97	6.5	RPA JA/03/01	Krolski & Harbin, 2008. RAJAP004
	Prothioconazole-sulfonic acid	0.02-10	18	90-108	97	4.7		
Sweetcorn stover	Prothioconazole-desthio	0.02-20	16	79-112	93	10.7	RPA JA/03/01	Krolski & Harbin, 2008. RAJAP004
	Prothioconazole-sulfonic acid	0.02-20	16	89-103	98	4.1		
Popcorn stover	Prothioconazole-desthio	0.02-20	16	79-112	93	10.7	RPA JA/03/01	Krolski & Harbin, 2008. RAJAP004
	Prothioconazole-sulfonic acid	0.02-20	16	89-103	98	4.1		
Popcorn grain	Prothioconazole-desthio	0.02-0.20	14	89-108	98	5.4	RPA JA/03/01	Krolski & Harbin, 2008. RAJAP004
	Prothioconazole-sulfonic acid	0.02-20	14	93-108	101	4.6		
Maize/Corn grain	Prothioconazole-desthio	0.02-0.20	10	70-108	93	12.2	RPA JA/03/01	Krolski & Harbin, 2008, RAJAP001
	Prothioconazole-sulfonic acid	0.02-0.20	10	89-105	98	5.4		
Maize/Corn aspirated grain fractions	Prothioconazole-desthio	0.02-5.0	6	74-003	102	14.1	RPA JA/03/01	Krolski & Harbin, 2008, RAJAP001
	Prothioconazole-sulfonic acid	0.02-5.0	6	70-113	93	18.5		
Maize/Corn starch	Prothioconazole-desthio	0.02-0.50	6	77-102	90	11.9	RPA JA/03/01	Krolski & Harbin, 2008, RAJAP001
	Prothioconazole-sulfonic acid	0.02-0.50	6	84-100	90	6.1		
Maize/Corn oil (wet milled)	Prothioconazole-desthio	0.02-0.50	6	77-98	92	9.4	RPA JA/03/01	Krolski & Harbin, 2008, RAJAP001
	Prothioconazole-sulfonic acid	0.02-0.50	6	87-104	96	6.9		
Maize/Corn grits	Prothioconazole-desthio	0.02-0.50	6	80-103	94	10.3	RPA JA/03/01	Krolski & Harbin, 2008, RAJAP001
	Prothioconazole-sulfonic acid	0.02-0.50	6	77-104	90	13.1		
Maize/Corn flour	Prothioconazole-desthio	0.02-0.50	6	86-104	98	6.6	RPA JA/03/01	Krolski & Harbin, 2008, RAJAP001
	Prothioconazole-sulfonic acid	0.02-0.50	6	78-93	87	6.7		
Maize/Corn	Prothioconazole-desthio	0.02-0.50	6	89-113	98	8.5	RPA JA/03/01	Krolski &

Commodity	Analyte	Fortification mg/kg	n	Range Recovery (%)	Mean recovery (%)	% RSD	Method	Reference
meal	Prothioconazole-sulfonic acid	0.02-0.50	6	71-96	79	11.3		Harbin, 2008, RAJAP001
Maize/Corn bran	Prothioconazole-desthio	0.02-0.50	6	80-102	92	10.3	RPA JA/03/01	Krolski & Harbin, 2008, RAJAP001
	Prothioconazole-sulfonic acid	0.02-0.50	6	79-98	86	7.7		
Maize/Corn oil (dry milled)	Prothioconazole-desthio	0.02-0.50	6	80-92	88	4.8	RPA JA/03/01	Krolski & Harbin, 2008, RAJAP001
	Prothioconazole-sulfonic acid	0.02-0.50	6	95-104	99	3.2		
Citrus fruit	Prothioconazole-desthio $m/z = 312 \rightarrow m/z = 70$	0.01-0.10	10	84-107	100	6.6	01013	Brumhard & Stuke, 2007, MR-06/138
	Prothioconazole-desthio $m/z = 312 \rightarrow m/z = 125$	0.01-0.10	10	89-121	103	8.5		
	Prothioconazole $m/z = 342 \rightarrow m/z = 100$	0.01-0.10	10	95-105	99	4.2		
	Prothioconazole $m/z = 342 \rightarrow m/z = 125$	0.01-0.10	10	92-109	101	5.4		
Pea green seed	Prothioconazole-desthio $m/z = 312 \rightarrow m/z = 70$	0.01-0.10	10	96-114	103	6.4	01013	Brumhard & Stuke, 2007, MR-06/138
	Prothioconazole-desthio $m/z = 312 \rightarrow m/z = 125$	0.01-0.10	10	95-113	104	6.1		
	Prothioconazole $m/z = 342 \rightarrow m/z = 100$	0.01-0.10	10	87-96	94	2.9		
	Prothioconazole $m/z = 342 \rightarrow m/z = 125$	0.01-0.10	10	93-110	97	5.4		
Rape seed	Prothioconazole-desthio $m/z = 312 \rightarrow m/z = 70$	0.01-0.10	10	88-107	96	6.2	01013	Brumhard & Stuke, 2007, MR-06/138
	Prothioconazole-desthio $m/z = 312 \rightarrow m/z = 125$	0.01-0.10	10	88-103	95	5.4		
	Prothioconazole $m/z = 342 \rightarrow m/z = 100$	0.01-0.10	10	91-106	97	5.3		
	Prothioconazole $m/z = 342 \rightarrow m/z = 125$	0.01-0.10	10	90-107	97	5.3		
Wheat grain	Prothioconazole-desthio $m/z = 312 \rightarrow m/z = 70$	0.01-0.10	10	91-103	97	3.4	01013	Brumhard & Stuke, 2007, MR-06/138
	Prothioconazole-desthio $m/z = 312 \rightarrow m/z = 125$	0.01-0.10	10	89-104	94	4.9		
	Prothioconazole $m/z = 342 \rightarrow m/z = 100$	0.01-0.10	10	85-101	92	6.5		
	Prothioconazole $m/z = 342 \rightarrow m/z = 125$	0.01-0.10	10	90-101	95	5.1		
Corn green material	Prothioconazole-desthio $m/z = 312 \rightarrow m/z = 70$	0.01-0.10	10	97-119	007	7.9	01013	Brumhard & Stuke, 2007, MR-06/138
	Prothioconazole-desthio $m/z = 312 \rightarrow m/z = 125$	0.01-0.10	10	94-113	101	6.8		
	Prothioconazole $m/z = 342 \rightarrow m/z = 100$	0.01-0.10	10	88-106	99	6.3		

Commodity	Analyte	Fortification mg/kg	n	Range Recovery (%)	Mean recovery (%)	% RSD	Method	Reference
	Prothioconazole $m/z = 342 \rightarrow m/z = 125$	0.01-0.10	10	91-109	101	6.8		
Bean seed	Prothioconazole-desthio $m/z = 312 \rightarrow m/z = 70$	0.01-1.0	10	90-105	99	4.6	01013 + chromatographic conditions from 00765	Santiago, 2012, VM11-003
	Prothioconazole-desthio $m/z = 312 \rightarrow m/z = 125$	0.01-1.0	10	89-107	101	5.0		
	Prothioconazole $m/z = 342 \rightarrow m/z = 100$	0.01-1.0	10	98-115	107	6.8		
	Prothioconazole $m/z = 342 \rightarrow m/z = 125$	0.01-1.0	10	101-120	108	6.2		
Soya bean seed	Prothioconazole-desthio $m/z = 312 \rightarrow m/z = 70$	0.01-1.0	10	97-111	105	4.0	01013 + chromatographic conditions from 00765	Santiago, 2012, VM11-003
	Prothioconazole-desthio $m/z = 312 \rightarrow m/z = 125$	0.01-1.0	10	94-117	109	7.3		
	Prothioconazole $m/z = 342 \rightarrow m/z = 100$	0.01-1.0	10	94-113	103	5.1		
	Prothioconazole $m/z = 342 \rightarrow m/z = 125$	0.01-1.0	10	97-111	104	4.3		
Oat grain	Prothioconazole-desthio $m/z = 312 \rightarrow m/z = 70$	0.01-1.0	10	86-104	93	5.9	01013 + chromatographic conditions from 00765	Santiago, 2012, VM11-003
	Prothioconazole-desthio $m/z = 312 \rightarrow m/z = 125$	0.01-1.0	10	88-111	97	10		
	Prothioconazole $m/z = 342 \rightarrow m/z = 100$	0.01-1.0	10	92-106	98	5.0		
	Prothioconazole $m/z = 342 \rightarrow m/z = 125$	0.01-1.0	10	93-103	98	3.6		
Citrus fruit	Prothioconazole-desthio $m/z = 312 \rightarrow m/z = 70$	0.01-1.0	10	82-102	91	6.5	01013 + chromatographic conditions from 00765	Santiago, 2012, VM11-003
	Prothioconazole-desthio $m/z = 312 \rightarrow m/z = 125$	0.01-1.0	10	84-105	89	7.6		
	Prothioconazole $m/z = 342 \rightarrow m/z = 100$	0.01-1.0	10	88-94	91	2.0		
	Prothioconazole $m/z = 342 \rightarrow m/z = 125$	0.01-1.0	10	87-98	92	3.3		
Sugar cane	Prothioconazole-desthio $m/z = 312 \rightarrow m/z = 70$	0.01-1.0	10	74-92	83	7.8	01013 + chromatographic conditions from 00765	Santiago, 2012, VM11-003
	Prothioconazole-desthio $m/z = 312 \rightarrow m/z = 125$	0.01-1.0	10	75-95	85	8.8		
	Prothioconazole $m/z = 342 \rightarrow m/z = 100$	0.01-1.0	10	68-77	73	3.6		
	Prothioconazole $m/z = 342 \rightarrow m/z = 125$	0.01-1.0	10	70-77	74	3.2		
Soya bean seed	Prothioconazole-desthio $m/z = 312 \rightarrow m/z = 70$	0.01-1.0	10	107-118	112	3.9	01013	Resende, 2013, VM12-006
	Prothioconazole-desthio $m/z = 312 \rightarrow m/z = 125$	0.01-1.0	10	100-122	112	5.2		

Commodity	Analyte	Fortification mg/kg	n	Range Recovery (%)	Mean recovery (%)	% RSD	Method	Reference
	Prothioconazole $m/z = 342 \rightarrow m/z = 100$	0.01-3.0	15	87-120	106	10.3		
	Prothioconazole $m/z = 342 \rightarrow m/z = 125$	0.01-3.0	15	77-118	105	11.7		
Wheat grain	Prothioconazole-desthio $m/z = 312 \rightarrow m/z = 70$	0.01-1.0	10	97-112	103	4.1	01013	Resende, 2013, VM12-006
	Prothioconazole-desthio $m/z = 312 \rightarrow m/z = 125$	0.01-1.0	10	96-110	102	4.0		
	Prothioconazole $m/z = 342 \rightarrow m/z = 100$	0.01-1.0	10	96-105	99	5.0		
	Prothioconazole $m/z = 342 \rightarrow m/z = 125$	0.01-1.0	10	90-103	97	5.0		
Wheat grain	Prothioconazole	0.01-0.10	10	81-106	96	7.4	00598/M001	Heinemann, 2000, MR-689/99
	Prothioconazole-desthio	0.01-0.10	10	71-96	89	7.8		
Wheat green material	Prothioconazole	0.05-5.0	15	81-100	93	5.6	00598/M001	Heinemann, 2000, MR-689/99
	Prothioconazole-desthio	0.05-5.0	15	89-97	94	2.5		
Wheat straw	Prothioconazole	0.05-5.0	14	65-90	85	8.4	00598/M001	Heinemann, 2000, MR-689/99
	Prothioconazole-desthio	0.05-5.0	14	83-96	91	3.9		
Barley grain	Prothioconazole	0.01-0.10	10	72-103	89	8.9	00598/M001	Heinemann, 2000, MR-689/99
	Prothioconazole-desthio	0.01-0.10	10	76-94	90	6.2		
Barley green material	Prothioconazole	0.05-5.0	15	87-100	95	3.6	00598/M001	Heinemann, 2000, MR-689/99
	Prothioconazole-desthio	0.05-5.0	15	87-95	91	2.1		
Barley straw	Prothioconazole	0.05-5.0	15	79-104	91	8.4	00598/M001	Heinemann, 2000, MR-689/99
	Prothioconazole-desthio	0.05-5.0	15	76-93	87	5.1		
Canola seed	Prothioconazole	0.01-0.10	20	69-118	89	14.7	00598/M001	Heinemann, 2000, MR-689/99
	Prothioconazole-desthio	0.01-0.10	20	64-95	83	11.0		
Canola green material	Prothioconazole	0.05-5.0	15	87-97	91	3.1	00598/M001	Heinemann, 2000, MR-689/99
	Prothioconazole-desthio	0.05-5.0	15	86-98	92	3.1		
Canola straw	Prothioconazole	0.05-5.0	15	84-117	101	11.1	00598/M001	Heinemann, 2000, MR-689/99
	Prothioconazole-desthio	0.05-5.0	15	80-95	91	4.4		
Canola pod	Prothioconazole	0.05-5.0	13	74-110	90	13.1	00598/M001	Heinemann, 2000, MR-689/99
	Prothioconazole-desthio	0.05-5.0	13	82-91	97	2.8		
Potato tuber (seed potato)	Prothioconazole	0.01-10	12	76-107	94	8.6	00598/M001	Freitag & Wolters, 2007, RA-2569/05 RA-2604/05 RA-2570/05 RA-2603/05
	Prothioconazole-desthio	0.01-10	12	89-100	94	3.7		
Potato tuber	Prothioconazole	0.01-0.10	18	78-107	97	7.1		
	Prothioconazole-desthio	0.01-0.10	18	80-110	91	6.7		
Triticale straw	Prothioconazole	0.01-1.0	10	72-102	84	12.5	ATM-0053	ATM-0053 in

Commodity	Analyte	Fortification mg/kg	n	Range Recovery (%)	Mean recovery (%)	% RSD	Method	Reference
	Prothioconazole-desthio	0.01-1.0	10	91-119	99	8.4		Ellis, 2012, BCS-0232
Canola seed	Prothioconazole	0.01-1.0	10	84-106	91	6.5	ATM-0053	ATM-0053 in Ellis, 2012, BCS-0232
	Prothioconazole-desthio	0.01-1.0	10	94-102	99	2.6		
Wheat grain	Prothioconazole	0.01-1.0	10	78-106	94	11.0	ATM-0053	ATM-0053 in Ellis, 2012, BCS-0232
	Prothioconazole-desthio	0.01-1.0	10	87-108	93	8.8		
Barley forage	Prothioconazole	0.01-1.0	9	71-99	80	14.0	ATM-0053	ATM-0053 in Ellis, 2012, BCS-0232
	Prothioconazole-desthio	0.01-1.0	10	97-108	102	4.2		
Almond hull and shell	Prothioconazole	0.01-1.0	10	71-91	91	9.6	ATM-0053	ATM-0053 in Ellis, 2012, BCS-0232
	Prothioconazole-desthio	0.01-1.0	10	93-102	98	3.1		
Peanut Seeds	Prothioconazole	0.01-1.0	12	73-88	80	7.1	ATM-0053	Ellis, 2012, BCS-0232
	Prothioconazole-desthio	0.01-1.0	12	76-96	88	8.4		
Peanut shells	Prothioconazole	0.01-1.0	6	73-81	75	4.9	ATM-0053	Ellis, 2012, BCS-0232
	Prothioconazole-desthio	0.01-1.0	6	82-115	98	15.9		
Peanut fodder	Prothioconazole	0.01-1.0	6	70-98	82	16.0	ATM-0053	Ellis, 2012, BCS-0232
	Prothioconazole-desthio	0.01-1.0	6	81-89	84	3.2		
Peanut Seeds	Prothioconazole	0.01-1.0	17	82-109	93	10.2	ATM-0053	Ellis, 2012, BCS-0383
	Prothioconazole-desthio	0.01-1.0	17	74-111	95	11.4		
Peanut shells	Prothioconazole	0.01-1.0	14	74-118	92	13.6	ATM-0053	Ellis, 2012, BCS-0383
	Prothioconazole-desthio	0.01-1.0	17	76-108	95	10.2		
Peanut fodder	Prothioconazole	0.01-1.0	10	60-101	77	21.8	ATM-0053	Ellis, 2012, BCS-0383
	Prothioconazole-desthio	0.01-1.0	12	93-109	102	4.7		
Animal commodities								
Cattle milk	Prothioconazole-desthio $m/z = 312 \rightarrow m/z = 70$	0.01-0.10	10	84-105	94	7.9	01009	Billian & Wolters, 2006, MR-06/120
Cattle muscle		0.01-0.10	10	82-98	91	6.8		
Cattle kidney		0.01-0.10	10	80-97	90	6.0		
Cattle liver		0.01-0.10	10	93-101	97	2.8		
Cattle fat		0.01-0.10	10	83-94	88	3.8		
Poultry egg		0.01-0.10	10	86-94	90	3.0		
Cattle milk	Prothioconazole-desthio $m/z = 312 \rightarrow m/z = 125$	0.01-0.10	10	85-104	93	6.8	01009	Billian & Wolters, 2006, MR-06/120
Cattle muscle		0.01-0.10	10	83-99	92	6.6		
Cattle kidney		0.01-0.10	10	82-100	89	5.9		
Cattle liver		0.01-0.10	10	88-99	95	3.4		
Cattle fat		0.01-0.10	10	84-97	89	5.0		
Poultry egg		0.01-0.10	10	84-93	88	3.0		
Cattle milk	JAU 6476-3-hydroxy-desthio $m/z = 328 \rightarrow m/z = 70$	0.01-0.10	10	80-104	95	9.0	01009	Billian & Wolters, 2006, MR-06/120
Cattle muscle		0.01-0.10	10	82-99	91	6.8		
Cattle kidney		0.01-0.10	10	82-109	92	8.5		
Cattle liver		0.01-0.10	10	88-105	99	5.3		
Cattle fat		0.01-0.10	10	87-97	93	3.1		

Commodity	Analyte	Fortification mg/kg	n	Range Recovery (%)	Mean recovery (%)	% RSD	Method	Reference
Poultry egg		0.01-0.10	10	88-99	93	4.6		
Cattle milk	JAU 6476-3-hydroxy-desthio m/z = 328 → m/z = 141	0.01-0.10	10	79-106	93	9.9	01009	Billian & Wolters, 2006, MR-06/120
Cattle muscle		0.01-0.10	10	82-101	92	7.2		
Cattle kidney		0.01-0.10	10	85-105	94	6.9		
Cattle liver		0.01-0.10	10	94-105	101	3.9		
Cattle fat		0.01-0.10	10	83-102	92	6.3		
Poultry egg		0.01-0.10	10	87-99	93	4.7		
Cattle milk	JAU 6476-4-hydroxy-desthio m/z = 328 → m/z = 70	0.01-0.10	10	76-103	92	10.9	01009	Billian & Wolters, 2006, MR-06/120
Cattle muscle		0.01-0.10	10	83-101	92	7.3		
Cattle kidney		0.01-0.10	10	80-105	90	7.6		
Cattle liver		0.01-0.10	10	91-105	99	5.8		
Cattle fat		0.01-0.10	10	90-100	95	3.1		
Poultry egg		0.01-0.10	10	85-99	91	5.4		
Cattle milk	JAU 6476-4-hydroxy-desthio m/z = 328 → m/z = 141	0.01-0.10	10	78-102	91	9.8	01009	Billian & Wolters, 2006, MR-06/120
Cattle muscle		0.01-0.10	10	82-101	92	7.4		
Cattle kidney		0.01-0.10	10	86-104	93	6.1		
Cattle liver		0.01-0.10	10	90-107	98	5.6		
Cattle fat		0.01-0.10	10	86-103	93	4.8		
Poultry egg		0.01-0.10	10	88-94	91	2.3		
Cattle milk	JAU 6476-3,4-dihydroxy-desthio m/z = 344 → m/z = 70	0.01-0.10	10	78-105	95	10.6	01009	Billian & Wolters, 2006, MR-06/120
Cattle muscle		0.01-0.10	10	66-89	76	10.1		
Cattle kidney		0.01-0.10	10	86-104	93	5.5		
Cattle liver		0.01-0.10	10	82-102	92	8.0		
Cattle fat		0.01-0.10	10	78-117	94	12.8		
Poultry egg		0.01-0.10	10	87-103	92	5.4		
Cattle milk	JAU 6476-3,4-dihydroxy-desthio m/z = 344 → m/z = 157	0.01-0.10	10	75-107	93	12.0	01009	Billian & Wolters, 2006, MR-06/120
Cattle muscle		0.01-0.10	10	66-88	75	9.1		
Cattle kidney		0.01-0.10	10	85-101	93	6.8		
Cattle liver		0.01-0.10	10	90-104	96	4.9		
Cattle fat		0.01-0.10	10	79-115	90	13.5		
Poultry egg		0.01-0.10	10	87-100	92	5.4		
Cattle milk	JAU 6476-4,5-dihydroxy-desthio m/z = 344 → m/z = 70	0.01-0.10	10	77-111	94	11.3	01009	Billian & Wolters, 2006, MR-06/120
Cattle muscle		0.01-0.10	10	77-97	85	7.2		
Cattle kidney		0.01-0.10	10	82-103	92	6.7		
Cattle liver		0.01-0.10	10	88-107	96	5.2		
Cattle fat		0.01-0.10	10	84-124	97	13.4		
Poultry egg		0.01-0.10	10	83-100	89	6.2		
Cattle milk	JAU 6476-4,5-dihydroxy-desthio m/z = 344 → m/z = 157	0.01-0.10	10	81-107	92	9.4	01009	Billian & Wolters, 2006, MR-06/120
Cattle muscle		0.01-0.10	10	76-97	85	7.4		
Cattle kidney		0.01-0.10	10	77-104	92	8.3		
Cattle liver		0.01-0.10	10	89-101	95	3.8		
Cattle fat		0.01-0.10	10	85-123	98	12.7		
Poultry egg		0.01-0.10	10	83-93	88	4.1		

Commodity	Analyte	Fortification mg/kg	n	Range Recovery (%)	Mean recovery (%)	% RSD	Method	Reference
Bovine meat	Prothioconazole-desthio $m/z = 312 \rightarrow m/z = 70$ (quantitation)	0.01-0.10	10	98-100	98	1	01009	Bacher, 2006, P 1111 G
Milk		0.01-0.10	10	98-105	101	2		
Egg		0.01-0.10	10	84-91	88	3		
Bovine meat	Prothioconazole-desthio $m/z = 312 \rightarrow m/z = 125$ (confirmation)	0.01-0.10	10	95-99	97	1	01009	Bacher, 2006, P 1111 G
Milk		0.01-0.10	10	98-106	101	3		
Egg		0.01-0.10	10	82-92	87	3		
Bovine meat	JAU 6476-3-hydroxy-desthio $m/z = 328 \rightarrow m/z = 70$ (quantitation)	0.01-0.10	10	96-102	98	2	01009	Bacher, 2006, P 1111 G
Milk		0.01-0.10	10	96-103	101	3		
Egg		0.01-0.10	10	84-96	89	4		
Bovine meat	JAU 6476-3-hydroxy-desthio $m/z = 328 \rightarrow m/z = 141$ (confirmation)	0.01-0.10	10	96-104	100	3	01009	Bacher, 2006, P 1111 G
Milk		0.01-0.10	10	98-108	103	3		
Egg		0.01-0.10	10	87-98	91	4		
Bovine meat	JAU 6476-4-hydroxy-desthio $m/z = 328 \rightarrow m/z = 70$ (quantitation)	0.01-0.10	10	93-108	99	5	01009	Bacher, 2006, P 1111 G
Milk		0.01-0.10	10	94-109	101	5		
Egg		0.01-0.10	10	85-101	89	5		
Bovine meat	JAU 6476-4-hydroxy-desthio $m/z = 328 \rightarrow m/z = 141$ (confirmation)	0.01-0.10	10	93-108	100	5	01009	Bacher, 2006, P 1111 G
Milk		0.01-0.10	10	94-107	101	4		
Egg		0.01-0.10	10	85-100	89	5		
Bovine meat	JAU 6476-3,4-dihydroxy-desthio $m/z = 344 \rightarrow m/z = 70$ (quantitation)	0.01-0.10	10	86-94	90	4	01009	Bacher, 2006, P 1111 G
Milk		0.01-0.10	10	93-109	99	5		
Egg		0.01-0.10	10	94-102	96	4		
Bovine meat	JAU 6476-3,4-dihydroxy-desthio $m/z = 344 \rightarrow m/z = 157$ (confirmation)	0.01-0.10	10	81-93	88	4	01009	Bacher, 2006, P 1111 G
Milk		0.01-0.10	10	89-107	98	6		
Egg		0.01-0.10	10	91-101	96	3		
Bovine meat	JAU 6476-4,5-dihydroxy-desthio $m/z = 344 \rightarrow m/z = 70$ (quantitation)	0.01-0.10	10	84-93	89	4	01009	Bacher, 2006, P 1111 G
Milk		0.01-0.10	10	94-102	97	2		
Egg		0.01-0.10	10	85-96	90	5		
Bovine meat	JAU 6476-4,5-dihydroxy-desthio $m/z = 344 \rightarrow m/z = 157$ (confirmation)	0.01-0.10	10	84-92	89	3	01009	Bacher, 2006, P 1111 G
Milk		0.01-0.10	10	94-105	97	4		
Egg		0.01-0.10	10	87-99	92	5		

[a] Prothioconazole recovery is the sum of prothioconazole-desthio and prothioconazole-sulfonic acid values from recovery samples fortified with prothioconazole only.

Storage Stability under Frozen Conditions

Plant commodities

Information relating to storage stability of residues in wheat, canola (seed, pod, straw), spinach (leaves), sugar beet (root, leaf with root collar), tomato (fruit) and field pea (dried) was evaluated at the 2008 JMPR Meeting. This showed that residues of prothioconazole-desthio were stable over a frozen storage period of up to 36 months in wheat and at least 24 months for the other crops. Residues of prothioconazole and prothioconazole-desthio were stable in wheat hay and straw, canola seeds, mustard greens, turnip root and tomato fruit over a frozen storage period of 36-42 months.

Subsequently, a freezer storage stability study for residues of prothioconazole and prothioconazole-desthio (JAU6476-desthio) in wheat (forage, straw, grain, bran and flour), canola (seed and oil), mustard greens, tomato (fruit and paste) and turnip roots was performed over a 36-month period (Gould and Timberlake, 2008; Report RAJAY016), and the final results are summarized below.

Wheat, canola, mustard greens, tomato, turnip

Untreated control samples were fortified individually with prothioconazole or its metabolite prothioconazole-desthio at a concentration of 0.25 mg/kg and then frozen at < -10°C. Samples were analysed with method RPA JA/03/01 immediately after fortification (0 day) and after storage intervals up to 36 months. At the day 0, three fortified samples for each compound and one control were analysed. At subsequent analysis intervals, two stored samples and two freshly fortified samples for each compound and one control were analysed.

Prothioconazole is measured indirectly as the sum of the two oxidation products (prothioconazole-sulfonic acid and prothioconazole-desthio). Prothioconazole-desthio is measured directly. The limits of quantification (LOQ) were 0.01 mg/kg for prothioconazole-sulfonic acid and prothioconazole-desthio.

The stability of residues expressed as the mean of two replicate analyses in the stored samples is summarized in Table 2.

Table 2 Storage stability data and concurrent recovery data for prothioconazole and prothioconazole-desthio in various matrices

Storage Interval (days)	Prothioconazole		Prothioconazole-desthio	
	Procedural recovery (%)	Residues remained (%)	Procedural recovery (%)	Residues remained (%)
Canola oil				
0	90	-	95	-
64	92	89	99	99
132	93	86	93	90
190	90	80	93	90
380	91	79	93	89
771	101	74	98	99
1084	92	67	97	99
Canola seed				
0	88	-	91	-
68	87	82	94	96
134	74	66	95	90
197	72	63	93	90
386	71	57	95	90
778	63	53	96	96
1091	89	69	98	96
Mustard greens				
0	82	-	95	-
58	88	78	95	96
127	88	75	99	94

Storage Interval (days)	Prothioconazole		Prothioconazole-desthio	
	Procedural recovery (%)	Residues remained (%)	Procedural recovery (%)	Residues remained (%)
185	82	68	93	90
375	77	61	98	94
766	82	65	99	99
1079	90	71	98	96
Tomato fruit				
0	81	-	93	-
57	83	73	96	95
126	71	62	93	90
184	51	48	91	91
374	62	54	94	92
765	74	51	98	101
1078	89	69	98	98
Tomato paste				
0	91	-	94	-
58	87	84	98	97
126	89	79	92	91
184	86	64	92	90
374	88	59	93	89
765	93	54	96	98
1078	89	49	95	99
Turnip roots				
0	87	-	95	-
58	84	82	93	93
126	74	70	95	93
184	58	63	94	91
374	39	48	96	94
765	80	70	99	101
1078	95	90	98	100
Wheat bran				
0	84	-	96	-
63	89	75	97	99
131	84	69	97	92
189	82	63	94	91
378	86	55	87	88
770	84	57	95	97
1082	87	57	97	98
Wheat flour				
0	88	-	98	-
57	89	88	98	96
127	80	82	95	93
185	83	76	94	92
374	79	70	95	91

Storage Interval (days)	Prothioconazole		Prothioconazole-desthio	
	Procedural recovery (%)	Residues remained (%)	Procedural recovery (%)	Residues remained (%)
766	79	68	98	101
1078	90	73	98	97
Wheat forage				
0	76	-	92	-
59	82	75	96	97
130	78	70	94	90
188	69	62	91	89
377	57	48	93	90
769	53	50	98	99
1078	84	71	99	98
Wheat grain				
0	88	-	96	-
61	90	80	99	94
131	86	76	91	89
189	84	72	94	93
378	89	65	80	78
769	90	59	99	100
1083	91	56	96	98
Wheat straw				
0	92	-	95	-
60	89	85	91	94
130	87	76	91	87
188	84	72	91	88
377	85	73	94	91
769	116	101	122	122
1082	88	74	94	93

USE PATTERN

Prothioconazole is a broad spectrum systemic triazolinthione fungicide that is used for the control of diseases caused by *Ascomycetes*, *Basidiomycetes* and *Deuteromycetes* in a range of crops. It has protective, curative and eradicative activity and can be used as both a seed treatment and a foliar treatment. Its mode of action is interference with the synthesis of ergosterol in the target fungi by inhibition of CYP51, which catalyses demethylation at C14 of lanosterol or 24-methylene dihydrolanosterol, leading to morphological and functional changes in the fungal cell membrane.

Prothioconazole is registered for use in a variety of crops worldwide. In this evaluation the national registered uses are summarised where supervised trials have been conducted or in countries with similar GAPs to where the supervised trials were carried out. Table 3 represents a summary of these relevant GAPs.

Table 3 Registered uses of prothioconazole

Crop	Country	Formulation	Application				PHI days
			Method	Rate, g ai/ha	Spray conc. g ai/hL	No. or max (g ai/ha/ season)	
Berries and small fruit							
Bushberry subgroup (blueberry)[a]	USA	Proline SC 480 (480 g/L SC)	Foliar	200	213	2 (400 g ai/ha)	7
Bushberry subgroup (blueberry)[a]	Canada	Proline SC 480 (480 g/L SC)	Foliar	200	150-200	2 (400 g ai/ha)	7
Low growing berry subgroup, except strawberry (cranberry)[a]	USA	Proline SC 480 (480 g/L SC)	Foliar	175	186	2 (350 g ai/ha)	45
Low growing berry subgroup, except strawberry (cranberry)[a]	Canada	Proline SC 480 (480 g/L SC)	Foliar	175	175	2 (350 g ai/ha)	45
Fruiting vegetables, cucurbits							
Cucurbits[a]	USA	Proline SC 480 (480 g/L SC)	1 Soil + 2 Foliar	200	212	1 soil + 2 foliar (600 g ai/ha)	7
Cucurbits[a]	Canada	Proline SC 480 (480 g/L SC)	1 Soil + 2 Foliar	200 + 2 x 200	200	1 soil + 2 foliar (600 g ai/ha)	7
Cucurbits[a]	Canada	Proline SC 480 (480 g/L SC)	1 Soil + 4 Foliar	200 + 4 x 100	100-200	1 soil + 4 foliar (600 g ai/ha)	7
Fruiting vegetables, other than cucurbits							
Sweet corn	USA	Stratego YLD (125 g/L SC)	Foliar	46	39-246	4	0[e] 14[f]
Sweet corn	USA	Prosaro 421 SC (211 g/L SC)	Foliar	100	106-535	4 (400 g ai/ha)	7[e] 49[f]
Sweet corn	USA	ProlineSC 480	Foliar	200	106-535	4	0[e] 14[e,f]
Sweet corn	Canada	Proline SC 480 (480 g/L SC)	Foliar	200	200-400	4	14[e,f]
Pulses							
Soya bean	USA	Proline SC 480 (480 g/L SC)	Foliar	88-175	94-583	3 (450 g ai/ha)	21
Soya bean	USA	Stratego YLD (125 g/L SC)	Foliar	42	39-225	3	21 [b]
Soya bean	USA	Provost 433 SC (145 g/L SC)	Foliar	32	23-171	3 (95 g ai/ha)	21
Soya bean	Canada	Proline SC 480 (480 g/L SC)	Foliar	100	100-200	1	20
Soya bean	Brazil	FOX SC 325 (175 g/L SC)	Foliar	70	35-350	2	30
Soya bean	Brazil	Proline (250 g/L EC)	Foliar	75	17-250	2	30
Soya bean	Brazil	FOX Xpro (175 g/L SC)	Foliar	70-87.5	46.7-438	4	30
Root and tuber vegetables							
Potato	USA	Emesto Silver (18 g/L SC)	Seed treatment	0.36 g ai/ 100 kg seed pieces	-	3 (160 g ai/ha)	-
Potato	Canada	Emesto Silver (18 g/L SC)	Seed treatment	0.36 g ai/ 100 kg seed pieces	-		-

Crop	Country	Formulation	Application				PHI days
			Method	Rate, g ai/ha	Spray conc. g ai/hL	No. or max (g ai/ha/ season)	
Potato	Germany (EU)	Monceren Pro FS 258 (8 g/L FS)	Seed treatment	0.48-0.64 g ai/100 kg seed pieces (12-16 g ai/ha)	-	1	-
Potato	Netherlands (EU)	Moncereen Pro FS 258 (8 g/L FS)	Seed treatment	0.48 g ai/100 kg seed pieces (12-24 g ai/ha)	-	1	-
Potato	Switzerland	Monceren Pro FS 258 (8 g/L FS)	Seed treatment	0.48 g ai/100 kg seed pieces (12 g ai/ha)	Undiluted or 12-20	1	-
Cereal grains							
Corn (field corn and popcorn)	USA	Proline SC 480 (480 g/L SC)	Foliar	200	213-714	4 (800 g ai/ha)	0[c] 14[d]
Corn	USA	Stratego YLD (125 g/L SC)	Foliar	46	19-246	2	0[c] 14[d]
Corn	USA	Prosaro 421 SC (211 g/L SC)	Foliar	100	106-535	4 (400 g ai/ha)	21[c] 36[d]
Corn	Canada	Proline SC 480 (480 g/L SC)	Foliar	200	200-400	1	14
Corn	Brazil	FOX SC 325 (175 g/L SC)	Foliar	70-87.5	35-438	2	15
Corn	Brazil	FOX XPRO (175 g/L SC)	Foliar	70-87.5	47-438	2	15
Corn	Belarus	Lamador FS 400 (250 g/L SC)	Seed treatment	50 g ai/tonne	-		-
Corn	Belarus	Prosaro 250 EC (125 g/L EC)	Foliar	125	33-63	1	30
Corn	Czech Republic (EU)	Prosaro 250 EC (125 g/L EC)	Foliar	125	14.6-62.5	1	35
Corn	France (EU)	Feuver FS 300 (300 g/L SC)	Seed treatment	4.5 g ai/ 50000 seeds		1	-
Corn/maize	Italy (EU)	Prosaro 250 EC (125 g/L EC)[i]	Foliar	125	25	2	-
Corn/maize	Slovak Republic (EU)	Prosaro 250 EC (125 g/L EC)	Foliar	125		2	-
Oilseeds							
Peanuts [a]	Australia	Brumby 480 SC (480 g/L SC)	Foliar	120-192	27-128	4	28[h]
Peanuts	USA	Proline SC 480 (480 g/L SC)[i]	In-furrow, banded and foliar	175-200	94-426	4 (800 g ai/ha)	14[g]
Peanuts	USA	Provost 433 SC (145 g/L SC)	Foliar	73-112	78-238	4 (450 g ai/ha)	14[g]
Peanuts	Canada	Proline SC 480 (480 g/L SC)	Foliar	175-200	175-200	4 (800 g ai/ha)	14[g]

[a] Proposed new use
[b] For soya bean seed. Do not graze or feed soya bean forage or hay.
[c] For field corn forage
[d] For field corn/popcorn grain and fodder
[e] For sweet corn forage and ears
[f] For sweet corn fodder
[g] Do not feed hay or threshings or allow livestock to graze in treated areas
[h] Do not graze or cut for stock food for 4 weeks after application

The bush berry subgroup includes: Aronia berry; blueberry (highbush and lowbush); Chilean guava; highbush cranberry; currant (black, buffalo, and red); elderberry; European barberry; gooseberry; edible honeysuckle; huckleberry; jostaberry; juneberry (Saskatoon berry); lingonberry; native currant; salal; sea buckthorn; and cultivars, varieties, and/or hybrids of these.

Low growing berry subgroup, except strawberry, includes: Bearberry; bilberry; cloudberry; cranberry; muntries; partridgeberry; and cultivars, varieties, and/or hybrids of these.

RESIDUES RESULTING FROM SUPERVISED TRIALS

Supervised trials were conducted to support MRLs for the following crops or groups of crops: blueberry, cranberry, cucurbits (cucumber, summer squash and melon), sweet corn, soya bean, potato, maize (field corn, popcorn) and peanuts. The results of these supervised trials are summarized in the following tables.

Crop Group	Commodity	Country, year of trials	Table No.
Berries and other small fruits	Blueberry	USA and Canada, 2010	4
	Cranberry	USA, 2010	5
Fruiting vegetables, cucurbits	Cucurbits	USA and Canada, 2010	6
Fruiting vegetables, other than cucurbits	Sweet corn	USA, 2006	7
Pulses	Soya bean	USA, 2004-2005	8
		Brazil, 2012-2013	9
Root and tuber vegetables	Potato	Europe, 2005, 2010	10
Cereal grains	Corn/Maize Popcorn	USA, 2006	11
	Corn/Maize Corn/Maize	USA, 2006	12
		Brazil, 2011-2012	13
		Europe, 2007, 2008, 2011	14
Oilseeds	Peanuts	Australia, 2011-2012	15
Animal feed	Sweetcorn forage and fodder	USA	16
	Field corn forage	USA	17
	Popcorn stover	USA	18
	maize/corn forage	EU	19
	Peanut fodder	Australia	20

In addition to the description and details of the field trials and analytical methods, each report includes procedural recoveries and in some cases a summary of the method validation.

The residue definition for monitoring and risk assessment is prothioconazole-desthio only. However for consistency with previous JMPR evaluations, for trials conducted in the USA, Canada and Brazil, the tables show residues of prothioconazole sulfonic acid (JAU 6476 sulfonic acid), prothioconazole-desthio (JAU 6476 desthio) and total prothioconazole (JAU 6476). For trials conducted in Australia, the tables show residues of prothioconazole (JAU 6476), prothioconazole-desthio (JAU 6476 desthio) and total prothioconazole (JAU 6476). Unless otherwise indicated: the prothioconazole (JAU 6478), JAU 6476 sulfonic acid and total prothioconazole (JAU6478 total) residues are expressed as mg prothioconazole equivalents/kg. The prothioconazole-desthio (JAU 6476 desthio) residue is expressed as mg prothioconazole-desthio/kg.

Only the residues of prothioconazole-desthio have been used for calculation of MRL, HR and STMR values.

The prothioconazole recovery was calculated as the sum of prothioconazole-desthio and prothioconazole-sulfonic acid from recovery samples fortified with prothioconazole only.

In a number of studies, residues of triazole-derived metabolites (TDMs) and hydroxylated metabolites were determined in addition to residues of prothioconazole, prothioconazole desthio and prothioconazole sulfonic acid. The results of these analyses are not summarised here as these analytes are not included in the residue definition for monitoring or risk assessment.

The maximum periods between sampling and analyses were within the tested storage stability period of 3 years for prothioconazole-desthio.

In the trials where multiple analyses were conducted on a single sample, the mean value is reported. Where multiple samples were taken from a single plot, the mean residue 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.
The performance parameters of the methods used in the trials are summarised in section on Methods of residue analysis. Results have not been corrected for concurrent method recoveries. Residues and application rates have generally been rounded to two significant figures or, for residues near the LOQ, to one significant figure. Residue values from the trials conducted according to the maximum GAP were used for the estimation of maximum residue levels. Those results included in the tables are underlined.

Berries and Other Small Fruit

Bushberries - Blueberry

Supervised trials in USA and Canada: Eleven supervised trials on blueberry were conducted in 2010 in the major blueberry-producing states in the USA and Canada (Murphy & Dallstream, 2012, RAJAP022). The trials were carried out according to the GAP in the USA/Canada. Two applications of an SC formulation containing 480 g/L prothioconazole were made at 6–7 day intervals and samples of blueberries were taken from each plot at 5–7 days after last application (DALA). In the two decline trials, additional samples were collected at 0, 3, 13 or 14, and 21 days after the last application.

The blueberries were frozen after collection and stored frozen (< -20 ºC) until extraction and analysis, with the exception of the bulk 'washing' sample from trial JA007-10DA, which was shipped unfrozen. A sub-sample of blueberry RAC was taken from the bulk 'washing' sample and frozen after collection. A second sub-sample was washed with water, drained and then frozen.

Residues of prothioconazole on blueberries were determined by the method, JA-001-P04-02. Procedural recoveries at fortification levels of 0.02–1.5 mg/kg were within 70–120% for prothioconazole-desthio, prothioconazole-sulfonic acid and prothioconazole, with the exception of one recovery for prothioconazole at 0.02 mg/kg which was 65. The results of the trials are summarized in Table 4.

Table 4 Prothioconazole residues in blueberry resulting from supervised trials in the USA and Canada

BLUEBERRY Trial Country, year (Variety)	Application					DAT	Commodity	JAU 6476 sulfonic acid mg/kg	JAU 6476 desthio mg/kg	JAU 6476 total mg/kg	Reference
	Formulation (g ai/L)	g ai/ha	g ai/hL	Water (L/ha)	No	days					
GAP, USA and Canada	480 g/L SC	200			2	7					
JA001-10HA Pennsylvania, USA, 2010 (Bluecrop)	480 g/L SC	197-201	44	449-458	2	6	Berry	0.05	<u>0.28</u>	0.32	Murphy & Dallstream, 2012, RAJAP022
JA002-10HA Pennsylvania, USA, 2010 (Elliot)	480 g/L SC	204-207	54	381-385	2	5	Berry	0.09	<u>0.70</u>	0.79	Murphy & Dallstream, 2012, RAJAP022
JA003-10HA New York, USA, 2010 (Duke)	480 g/L SC	197-199	72	274-276	2	7	Berry	0.16	<u>0.52</u>	0.67	Murphy & Dallstream, 2012, RAJAP022

BLUEBERRY Trial Country, year (Variety)	Application					DAT	Commodity	JAU 6476 sulfonic acid mg/kg	JAU 6476 desthio mg/kg	JAU 6476 total mg/kg	Reference
	Formulation (g ai/L)	g ai/ha	g ai/hL	Water (L/ha)	No	days					
JA004-10DA New York, USA, 2010 (Blue ray)	480 g/L SC	203	72	282-282	2	0 3 7 14 21	Berry	0.22 0.12 0.09 0.06 0.05	0.58 0.42 0.26 0.16 0.11	0.80 0.54 0.35 0.22 0.16	Murphy & Dallstream, 2012, RAJAP022
JA005-10HA North Carolina, USA, 2010 (Blue Haven)	480 g/L SC	194-200	70	276-283	2	7	Berry	0.02	0.15	0.17	Murphy & Dallstream, 2012, RAJAP022
JA006-10HA Georgia, USA, 2010 (Climax)	480 g/L SC	201-203	47	424-429	2	7	Berry	0.05	0.56	0.61	Murphy & Dallstream, 2012, RAJAP022
JA007-10DA Georgia, USA, 2010 (Bright Blue)	480 g/L SC	201	47	423-425	2	0 3 7 13 21 7	Berry Berry, washed	0.06 0.03 0.02 < 0.02 < 0.02 < 0.02	0.40 0.27 0.22 0.12 0.08 0.18	0.46 0.29 0.25 0.13 0.08 0.19	Murphy & Dallstream, 2012, RAJAP022
JA008-10HA Illinois, USA, 2010 (Duke)	480 g/L SC	202-206	106-108	190-192	2	7	Berry	0.18	0.87	1.0	Murphy & Dallstream, 2012, RAJAP022
JA009-10HA Illinois, USA, 2010 (Duke)	480 g/L SC	201	46-47	201	2	7	Berry	0.06	0.60	0.66	Murphy & Dallstream, 2012, RAJAP022
JA010-10HA Ontario, Canada, 2010 (Blue Crop)	480 g/L SC	206-213	80	256-266	2	7	Berry	0.07	0.65	0.72	Murphy & Dallstream, 2012, RAJAP022
JA011-10HA Oregon, USA, 2010 (Blue Crop)	480 g/L SC	198-202	50-57	198-202	2	7	Berry	0.04	0.42	0.45	Murphy & Dallstream, 2012, RAJAP022

Low growing berry subgroup, except strawberry

Cranberry

Six supervised trials were conducted according to the GAP for cranberries in the USA/Canada in the major cranberry-producing states in the USA (Murphy & Dallstream, 2012, RAJAP023) in 2010. Two applications of an SC formulation containing 480 g/L prothioconazole were made at 7–8 day intervals and samples of cranberries were taken from each plot at 43–46 DALA. In the decline trial, additional samples were collected at 35, 40, 50, and 55 days after the last application. The cranberries were frozen after collection and stored frozen (< -20 ºC) until extraction and analysis, with the exception of the bulk ‘washing’ sample from trial JA015-10DA, which was shipped unfrozen. A sub-sample of cranberry RAC was taken from the bulk ‘washing’ sample and frozen after collection. A second sub-sample was washed with water, drained and then frozen.

Residues of prothioconazole on cranberries were determined with method, JA-001-P04-02. Procedural recoveries at fortification levels of 0.02–1.5 mg/kg were within 70–120% for prothioconazole-desthio, prothioconazole-sulfonic acid and prothioconazole. The results of the trials are summarized in Table 5.

Table 5 Prothioconazole residues in cranberry resulting from supervised trials in the USA

CRANBERRY Trial Country, year (Variety)	Application					DAT days	Commodity	JAU 6476 sulfonic acid mg/kg	JAU 6476 desthio mg/kg	JAU 6476 total mg/kg	Reference
	Formulation (g ai/l)	g ai/ha	g ai/hL	Water (L/ha)	No						
GAP, USA and Canada	480 g/L SC	175			2	45					
JA012-10HA New Jersey, USA, 2010 (Stevens)	480 g/L SC	174-176	41	421-430	2	46	Fruit	< 0.02	< 0.02	< 0.04	Murphy & Dallstream, 2012, RAJAP023
JA013-10HA New York, USA, 2010 (Pilgrim)	480 g/L SC	179-180	62-63	281-290	2	45	Fruit	< 0.02	< 0.02	< 0.04	Murphy & Dallstream, 2012, RAJAP023
JA014-10HA Wisconsin, USA, 2010 (Grygleski GHI)	480 g/L SC	169-175	70-75	234-243	2	44	Fruit	< 0.02	0.03	< 0.04	Murphy & Dallstream, 2012, RAJAP023
JA015-10DA Wisconsin, USA, 2010 (Stevens)	480 g/L SC	170-173	70-74	234-243	2	35 40 43 50 55 43	Fruit Whole fruit, washed	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02	0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02	< 0.04 < 0.04 < 0.04 < 0.04 < 0.04 < 0.04	Murphy & Dallstream, 2012, RAJAP023
JA016-10HA British Columbia, USA, 2010 (Pilgrims)	480 g/L SC	168-170	87-90	187-196	2	43	Fruit	< 0.02	0.09	0.09	Murphy & Dallstream, 2012, RAJAP023
JA017-10HA Washington, USA, 2010 (Stevens)	480 g/L SC	172-173	74	232-233	2	46	Fruit	< 0.02	0.03	< 0.04	Murphy & Dallstream, 2012, RAJAP023

Fruiting Vegetables, Cucurbits

Cucumber, melon and summer squash

In 2010, eight supervised trials were conducted on cucumber, eight trials on musk melon and eight trials on summer squash according to the GAP for cucurbits in the USA/Canada in the USA (Murphy & Harbin, 2012, RAJAP024). One application was made by drip irrigation and two foliar applications were made using an SC formulation containing 480 g/L prothioconazole, with a 5-8 days interval between the two foliar applications.

Samples of cucumbers, melon or summer squash were taken from each plot prior to the 3rd application (shown as -0 days in the table below), at 2-3 days and at 5-8 DALA. In the decline trials, additional samples were collected at 0, 13-14 and 19-21 days after the last application. The samples were frozen after collection and stored frozen (< -20ºC) until extraction and analysis, with the exception of the bulk samples from trials JA032-10HA and JA033-10HA (melon) and JA039-10DA (summer squash), which were shipped unfrozen. Sub-samples of melon and summer squash RAC were taken from the bulk samples and frozen after collection. Second sub-samples of melon were peeled and the pulp frozen. A second sub-sample of summer squash was washed with water and cooked, and samples of washed and cooked summer squash were stored frozen.

Residues of prothioconazole on cucurbits were determined with the method JA-001-P04-02. Procedural recoveries at fortification levels of 0.02-1.5 mg/kg were within 70-120% for prothioconazole-desthio, prothioconazole-sulfonic acid and prothioconazole in each matrix. The results of the trials are summarized in Table 6.

Table 6 Prothioconazole residues in cucurbits resulting from supervised trials in the USA and Canada

CUCURBITS Trial Country, year (Variety)	Application					DAT days	Commodity	JAU 6476 sulfonic acid mg/kg	JAU 6476 desthio mg/kg	JAU 6476 total mg/kg	Reference
	Formulation (g ai/l)	g ai/ha	g ai/hL	Water (L/ha)	No						
GAP, USA and Canada	480 g/L SC	200			3	7					
CUCUMBER											
JA019-10DA Georgia, USA, 2010 (Thunder)	480 g/L SC	197-200	86-88	224-230	3	-0 0 3 7 14 21	Fruit	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02	0.02 0.04 0.04 <u>0.03</u> 0.02 < 0.02	< 0.04 0.05 0.04 < 0.04 < 0.04 < 0.04	Murphy & Harbin, 2012, RAJAP024
JA020-10HA North Carolina, USA, 2010 (Long Green)	480 g/L SC	197-204	85-138	146-240	3	-0 3 8	Fruit	< 0.02 < 0.02 < 0.02	0.03 0.04 <u>0.03</u>	< 0.04 0.05 < 0.04	Murphy & Harbin, 2012, RAJAP024
JA021-10HA Florida, USA, 2010 (Alibi)	480 g/L SC	197-201	71	279-281	3	-0 3 7	Fruit	< 0.02 < 0.02 < 0.02	< 0.02 0.02 <u>0.02</u>	< 0.04 < 0.04 < 0.04	Murphy & Harbin, 2012, RAJAP024
JA022-10HA Ohio, USA, 2010 (Intimidator F1)	480 g/L SC	198-200	119	166-168	3	-0 3 7	Fruit	< 0.02 < 0.02 < 0.02	0.04 0.07 <u>0.05</u>	0.04 0.07 0.06	Murphy & Harbin, 2012, RAJAP024
JA023-10HA Illinois, USA, 2010 (Eureka)	480 g/L SC	200-203	66-70	291-303	3	-0 2 7	Fruit	< 0.02 < 0.02 < 0.02	0.02 0.05 <u>0.04</u>	< 0.04 0.05 0.04	Murphy & Harbin, 2012, RAJAP024
JA024-10HA Ontario, Canada, 2010 (Talladega)	480 g/L SC	198-203	84-88	231-235	3	-0 3 6	Fruit	< 0.02 < 0.02 < 0.02	0.06 0.12 <u>0.06</u>	0.06 0.14 0.07	Murphy & Harbin, 2012, RAJAP024

CUCURBITS Trial Country, year (Variety)	Application					DAT days	Commodity	JAU 6476 sulfonic acid mg/kg	JAU 6476 desthio mg/kg	JAU 6476 total mg/kg	Reference
	Formulation (g ai/l)	g ai/ha	g ai/hL	Water (L/ha)	No						
JA025-10HA Illinois, USA, 2010 (Talladega)	480 g/L SC	200-208	62	326-338	3	-0 3 7	Fruit	< 0.02 < 0.02 < 0.02	0.02 0.04 0.02	< 0.04 0.04 < 0.04	Murphy & Harbin, 2012, RAJAP024
JA026-10DA Texas, USA, 2010 (Pointsett)	480 g/L SC	199-202	129-133	152-154	3	-0 0 3 7 14 21	Fruit	< 0.02 0.03 < 0.02 < 0.02 < 0.02 < 0.02	0.04 0.08 0.08 0.05 0.03 0.02	0.05 0.11 0.10 0.05 < 0.04 < 0.04	Murphy & Harbin, 2012, RAJAP024
MELON, NETTED (MUSK)											
JA028-10HA Georgia, USA, 2010 (Athena)	480 g/L SC	200-203	96-99	204-212	3	-0 3 7	Fruit	< 0.02 < 0.02 < 0.02	0.03 0.08 0.03	0.04 0.09 < 0.04	Murphy & Harbin, 2012, RAJAP024
JA029-10HA Illinois, USA, 2010 (Hales Best)	480 g/L SC	200-203	72-73	277-278	3	-0 2 7	Fruit	< 0.02 0.02 < 0.02	0.03 0.13 0.06	< 0.04 0.15 0.07	Murphy & Harbin, 2012, RAJAP024
JA030-10DA Ontario, Canada, 2010 (Earlisweet)	480 g/L SC	198-200	84-86	230-236	3	-0 0 3 7 14 19	Fruit	< 0.02 0.05 < 0.02 < 0.02 < 0.02 < 0.02	0.05 0.18 0.10 0.06 0.06 0.05	0.06 0.23 0.11 0.07 0.07 0.06	Murphy & Harbin, 2012, RAJAP024
JA031-10HA Ontario, Canada, 2010 (Primo)	480 g/L SC	197-201	84	233-235	3	-0 3 7	Fruit	< 0.02 < 0.02 < 0.02	0.07 0.18 0.07	0.08 0.20 0.08	Murphy & Harbin, 2012, RAJAP024
JA032-10HA Texas, USA, 2010 (Mainstream)	480 g/L SC	200-202	133-140	143-152	3	-0 3 7 3	Fruit Pulp	< 0.02 < 0.02 < 0.02 < 0.02	0.03 0.09 0.06 < 0.02	< 0.04 0.10 0.07 < 0.04	Murphy & Harbin, 2012, RAJAP024
JA033-10HA California, USA, 2010 (Jumbo Cantaloupe)	480 g/L SC	200-202	69-71	287-292	3	-0 3 7 3	Fruit Pulp	< 0.02 < 0.02 < 0.02 < 0.02	0.06 0.19 0.15 < 0.02	0.07 0.20 0.17 < 0.04	Murphy & Harbin, 2012, RAJAP024
JA034-10HB California, USA, 2010 (Honeydew)	480 g/L SC	198-204	80-81	248-252	3	-0 3 7	Fruit	< 0.02 < 0.02 < 0.02	0.04 0.10 0.06	0.05 0.11 0.07	Murphy & Harbin, 2012, RAJAP024

CUCURBITS Trial Country, year (Variety)	Application					DAT days	Commodity	JAU 6476 sulfonic acid mg/kg	JAU 6476 desthio mg/kg	JAU 6476 total mg/kg	Reference
	Formulation (g ai/l)	g ai/ha	g ai/hL	Water (L/ha)	No						
JA035-10DA California, USA, 2010 (Hal's Best Jumbo)	480 g/L SC	200-201	59	337-342	3	-0 0 2 7 14 21	Fruit	< 0.02 0.04 < 0.02 0.02 < 0.02 < 0.02	0.07 0.22 0.09 0.15 0.12 0.07	0.08 0.25 0.11 0.17 0.13 0.07	Murphy & Harbin, 2012, RAJAP024
SUMMER SQUASH											
JA036-10HA New York, USA, 2010 (Sunray)	480 g/L SC	200-204	72	282-283	3	-0 3 7	Fruit	< 0.02 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02	< 0.04 < 0.04 < 0.04	Murphy & Harbin, 2012, RAJAP024
JA037-10DA North Carolina, USA, 2010 (Yellow Straight Neck)	480 g/L SC	198-201	111-138	143-181	3	-0 0 3 7 13 20	Fruit	< 0.02 0.02 < 0.02 < 0.02 < 0.02 < 0.02	0.03 0.10 0.02 < 0.02 < 0.02 < 0.02	< 0.04 0.12 0.03 < 0.04 < 0.04 < 0.04	Murphy & Harbin, 2012, RAJAP024
JA038-10HA Florida, USA, 2010 (Early Summer Crookneck)	480 g/L SC	197-201	71	280-281	3	-0 3 7	Fruit	< 0.02 < 0.02 < 0.02	< 0.02 0.02 < 0.02	< 0.04 < 0.04 < 0.04	Murphy & Harbin, 2012, RAJAP024
JA039-10DA Illinois, USA, 2010 (Golden Dawn III)	480 g/L SC	200-201	59-74	271-343	3	-0 0 3 6 13 21 3 3	Fruit Fruit, washed Fruit, cooked	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02	< 0.02 0.07 0.04 0.03 < 0.02 < 0.02 0.02 0.02	< 0.04 0.09 0.05 < 0.04 < 0.04 < 0.04 < 0.04 < 0.04	Murphy & Harbin, 2012, RAJAP024
JA040-10HA Ontario, Canada, 2010 (Senator)	480 g/L SC	197-201	80-84	235-251	3	-0 3 7	Fruit	< 0.02 < 0.02 < 0.02	< 0.02 0.03 < 0.02	< 0.04 0.04 < 0.04	Murphy & Harbin, 2012, RAJAP024
JA041-10HA Ontario, Canada, 2010 (Sunray)	480 g/L SC	199-200	82-84	237-369	3	-0 3 7	Fruit	< 0.02 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02	< 0.04 < 0.04 < 0.04	Murphy & Harbin, 2012, RAJAP024
JA042-10HA California, USA, 2010 (Dark green Zuccini)	480 g/L SC	199-202	54	369-374	3	-0 2 6	Fruit	< 0.02 < 0.02 < 0.02	0.03 0.08 0.05	< 0.04 0.09 0.05	Murphy & Harbin, 2012, RAJAP024

CUCURBITS Trial Country, year (Variety)	Application					DAT days	Commodity	JAU 6476 sulfonic acid mg/kg	JAU 6476 desthio mg/kg	JAU 6476 total mg/kg	Reference
	Formulation (g ai/l)	g ai/ha	g ai/hL	Water (L/ha)	No						
JA043-10HA British Columbia, USA, 2010 (Noche)	480 g/L SC	199-201	100	201	3	-0 2 5	Fruit	< 0.02 < 0.02 < 0.02	< 0.02 0.07 0.06	< 0.04 0.08 0.06	Murphy & Harbin, 2012, RAJAP024

Fruiting Vegetables, Other Than Cucurbits

Sweet corn

Supervised trials in the USA: Twelve supervised trials were conducted in 2006 on sweet corn in the USA (Krolski & Harbin, 2008, RAJAP004). The trials were carried out with 4×200 g ai/ha. The currently supported GAP in the USA is 4×100 g ai/ha. Four foliar applications were made to each treated plot of an SC formulation containing 480 g/L prothioconazole with a 2–11-day intervals between the each application. Each spray included a non-ionic surfactant.

In the sweet corn trials, ears, including seeds plus cob with husk removed, were harvested at 0 and 5–7 DALA (BBCH 73–79). In the decline trial, ears samples were collected at 0, 3, 6, 10 and 14 days. The samples were frozen after collection and stored frozen (< -20 ºC) until extraction and analysis. Residues of prothioconazole were determined with method RPA JA/03/01. Procedural recoveries at fortification levels of 0.02–20 mg/kg were within 70–120% for prothioconazole-desthio, prothioconazole-sulfonic acid and prothioconazole in each matrix. The results of the trials are summarized in Table 7.

Table 7 Prothioconazole residues in sweet corn resulting from supervised trials in the USA

SWEET CORN Trial Country, year (Variety)	Application					DAT days	Commodity	JAU 6476 sulfonic acid mg/kg	JAU 6476 desthio mg/kg	JAU 6476 total mg/kg	Reference
	Formulation (g ai/l)	g ai/ha	g ai/hL	Water (L/ha)	No						
GAP, USA	480 g/L SC	100			4	0/14					
JA002-06HA Virginia, USA, 2006 (Devotion)	480 g/L SC	201-208	128	156-163	4	0 6	Ear Ear	< 0.02 < 0.02	< 0.018 < 0.018	< 0.02 < 0.02	Krolski & Harbin, 2008, RAJAP004
JA003-06HA New York, USA, 2006 (Gourmet Sweet 372A)	480 g/L SC	204-208	122-123	167-170	4	0 7	Ear Ear	< 0.02 < 0.02	< 0.018 < 0.018	< 0.02 < 0.02	Krolski & Harbin, 2008, RAJAP004
JA005-06HA Georgia, USA, 2006 (Sweet G-90)	480 g/L SC	200	142-159	125-141	4	0 7	Ear Ear	< 0.02 < 0.02	< 0.018 < 0.018	< 0.02 < 0.02	Krolski & Harbin, 2008, RAJAP004

SWEET CORN Trial Country, year (Variety)	Application					DAT days	Commodity	JAU 6476 sulfonic acid mg/kg	JAU 6476 desthio mg/kg	JAU 6476 total mg/kg	Reference
	Formulation (g ai/l)	g ai/ha	g ai/hL	Water (L/ha)	No						
JA007-06DA Florida, USA, 2006 (Silver Queen)	480 g/L SC	195-200	167-181	109-120	4	0 3 6 10 14	Ear Ear Ear Ear Ear	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	< 0.018 < 0.018 < 0.018 < 0.018 < 0.018	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	Krolski & Harbin, 2008, RAJAP004
JA008-06HA Missouri, USA, 2006 (Peaches & Cream)	480 g/L SC	200-204	115-149	137-174	4	0 6	Ear Ear	< 0.02 < 0.02	< 0.018 < 0.018	< 0.02 < 0.02	Krolski & Harbin, 2008, RAJAP004
JA009-06HA Illinois, USA, 2006 (BC 0805)	480 g/L SC	196-204	150-160	123-132	4	0 7	Ear Ear	< 0.02 < 0.02	< 0.018 < 0.018	< 0.02 < 0.02	Krolski & Harbin, 2008, RAJAP004
JA010-06HA Nebraska, USA, 2006 (Serendipity)	480 g/L SC	199-204	153-157	129 131	4	0 5	Ear Ear	< 0.02 < 0.02	< 0.018 < 0.018	< 0.02 < 0.02	Krolski & Harbin, 2008, RAJAP004
JA011-06HA Minnesota, USA, 2006 (39H85 Pioneer)	480 g/L SC	196-202	111-118	171-181	4	0 7	Ear Ear	< 0.02 < 0.02	< 0.018 < 0.018	< 0.02 < 0.02	Krolski & Harbin, 2008, RAJAP004
JA012-06HA Iowa, USA, 2006 (Mirai 131)	480 g/L SC	198-206	156-182		4	0 7	Ear Ear	< 0.02 < 0.02	< 0.018 < 0.018	< 0.02 < 0.02	Krolski & Harbin, 2008, RAJAP004
JA031-06HA California, USA, 2006 (Silver Queen)	480 g/L SC	197-201	117-119	168-170	4	0 7	Ear Ear	< 0.02 < 0.02	< 0.018 < 0.018	< 0.02 < 0.02	Krolski & Harbin, 2008, RAJAP004
JA032-06HA Washington, USA, 2006 (Golden Jubilee)	480 g/L SC	202-204	143-145	140-142	4	0 7	Ear Ear	< 0.02 < 0.02	< 0.018 < 0.018	< 0.02 < 0.02	Krolski & Harbin, 2008, RAJAP004
JA033-06HA Oregon, USA, 2006 (Serendipity)	480 g/L SC	195-205	120-126	163-165	4	0 5	Ear Ear	< 0.02 < 0.02	< 0.018 < 0.018	< 0.02 < 0.02	Krolski & Harbin, 2008, RAJAP004

Pulses

Soya beans

Supervised trials in the USA: Residue data on soya beans from 19 trials conducted in the USA were previously evaluated by the 2008 JMPR and 2009 JMPR. The current USA GAP is 3 × 88–175 g ai/ha, PHI 21 days.

Three foliar spray applications of an SC formulation were made using ground-based equipment to soya beans at 145 to 188 g ai/ha. Intervals between applications were from 7 to 11 days (Duah, F. K.; Harbin, A. M, 2006, Report No.: RAJAY026). A non-ionic surfactant was added as a spray adjuvant at the lowest labelled rate. Duplicate treated samples of seed were collected at PHIs ranging from 19 to 23 days. Residues of prothioconazole, prothioconazole-desthio and prothioconazole sulfonic acid were analysed using Method RPA JA/03/01 with LOQ of 0.05 mg/kg.

Table 8 Prothioconazole residues in soya bean seed after three foliar treatments in supervised trials in the USA

SOYA BEAN (Variety) Location Year	Application				Residues (mg/kg) in soya bean seed			Report No Trial No. Doc No
	kg ai/ha	kg ai/hL	GS	DAT	JAU 6476 -sulfonic acid	JAU 6476-desthio	JAU 6476-total	
Soya bean (USA)	3 x 0.15			21	Min 140 L/ha (ground), 47 L/ha (air) Max 0.45 kg ai/ha/year			SC 480 (JMPR08)
Soya (Pioneer 96M20) Molino, Florida 2004	0.145-0.151	0.1-0.103	GS88	7 14 21 28 35	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 <u>< 0.05</u> < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	RAJAY026 JA014-04D-P2 M-270206-01-1
Soya (Stine 2788) Seymour, Illinois 2004	0.151-0.154	0.115-0.117	GS89	7 13 19 27 34	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 <u>< 0.05</u> < 0.05 < 0.05	< 0.05, < 0.05 < 0.05 < 0.05 < 0.05	RAJAY026 JA019-04D-P2 M-270206-01-1
Soya (Pioneer RR 97B52) Tifton, Georgia 2004	0.142-0.15	0.101-0.105	GS80	21	< 0.05	<u>< 0.05, < 0.05</u>	< 0.05, < 0.05	RAJAY026 JA015-04H-P2 M-270206-01-1
Soya (Pioneer 9492RR) Leland, Mississippi 2004	0.15-0.157	0.097-0.102	GS79	20	< 0.05, < 0.05	<u>< 0.05</u>	< 0.05	RAJAY026 JA016-04H-P2 M-270206-01-1
Soya (DP 5915 RR) Washington, Louisiana 2004	0.15-0.157	0.077-0.096	GS81	21	< 0.05, < 0.05	<u>< 0.05</u>	< 0.05	RAJAY026 JA017-04H-P2 M-270206-01-1
Soya (DP5634RR) Proctor, Arkansas 2004	0.149-0.15	0.107-0.108	GS79	21	< 0.05, < 0.05	<u>0.055</u>	0.06	RAJAY026 JA018-04H M-270206-01-1
Soya (Fontanelle 431RR) Stilwell, Kansas 2004	0.149-0.153	0.103-0.109	GS87	23	< 0.05, < 0.05	<u>0.051</u>	0.07	RAJAY026 JA020-04H M-270206-01-1
Soya (NKS28W2) Springfield, Nebraska 2004	0.15-0.15	0.109-0.111	GS80	19	< 0.05, < 0.05	<u>< 0.05</u>	< 0.05	RAJAY026 JA021-04H-P2 M-270206-01-1

SOYA BEAN (Variety) Location Year	Application				Residues (mg/kg) in soya bean seed			Report No Trial No. Doc No
	kg ai/ha	kg ai/hL	GS	DAT	JAU 6476 -sulfonic acid	JAU 6476-desthio	JAU 6476-total	
Soya (Croplan RT0907) Britton, South Dakota 2004	0.149-0.149	0.159-0.159	GS83	19	< 0.05, < 0.05	< 0.05	< 0.05	RAJAY026 JA024-04H-P2 M-270206-01-1
Soya (Croplan RT1447) Dumfries, Minnesota 2004	0.15-0.151	0.085-0.085	GS79	21	< 0.05, < 0.05	< 0.05	< 0.05	RAJAY026 JA025-04H-P2 M-270206-01-1
Soya (SC 9373) New Holland, Ohio 2004	0.151-0.155	0.01-0.105	GS93	20	< 0.05, < 0.05	0.105	0.14	RAJAY026 JA026-04H M-270206-01-1
Soya (92M70) Bagley, Iowa 2004	0.148-0.151	0.11-0.118	GS79	19	< 0.05	< 0.05	< 0.05	RAJAY026 JA027-04H-P2 M-270206-01-1
Soya (DynaGro DG 32M32RR) York, Nebraska 2004	0.149-0.15	0.08-0.08	GS80	19	< 0.05	< 0.05	< 0.05	RAJAY026 JA028-04H-P2 M-270206-01-1
Soya (Pioneer RR) USA Sheridan, Indiana 2004	0.146-0.148	0.094-0.095	GS97	21	< 0.05	< 0.05	< 0.05	RAJAY026 JA029-04H M-270206-01-1
Soya (Pioneer 93M80) USA Richland, Iowa 2004	0.15-0.152	0.093-0.118	GS83	21	< 0.05, < 0.05	< 0.05	< 0.05	RAJAY026 JA030-04H-P2 M-270206-01-1
Soya (Pioneer 91M50) USA Geneva, Minnesota 2004	0.15-0.152	0.094-0.097	GS80	20	< 0.05, < 0.05	< 0.05	< 0.05	RAJAY026 JA031-04H-P2 M-270206-01-1
Soya (Pioneer 93B85) USA Geneva, Minnesota 2004	0.149-0.15	0.088-0.089	GS77	21	< 0.05, < 0.05	< 0.05	< 0.05	RAJAY026 JA032-04H-P2 M-270206-01-1

SOYA BEAN (Variety) Location Year	Application				Residues (mg/kg) in soya bean seed			Report No Trial No. Doc No
	kg ai/ha	kg ai/hL	GS	DAT	JAU 6476 -sulfonic acid	JAU 6476-desthio	JAU 6476-total	
Soya (BT-383CR) USA Carlyle, Illinois 2004	0.15-0.15	0.155-0.161	GS79	21	< 0.05, < 0.05	< 0.05	< 0.05	RAJAY026 JA033-04H M-270206-01-1
Soya (RG 200 RR) USA Sabin, Minnesota 2005	0.148-0.15	0.093-0.099	GS69	19	< 0.05, < 0.05	< 0.05	< 0.05	RAJAY026 JA022-04HA M-270206-01-1

GS = growth stage at last application

Supervised trials in Brazil: Ten supervised trials have been conducted in 2012–2013 on soya bean in Brazil (Resende, 2013, F12-012 and Resende, 2013, F13-008). The trials were carried out according to the GAP for soya bean in Brazil (4 x 87.5 g ai/ha, PHI 30 days). Eight of the trials were decline trials and two were at harvest trials. Four foliar applications were made to each treated plot with an SC formulation containing 175 g/L prothioconazole + 150 g/L trifloxystrobin + 125 g/L bixafen at 87.5 g prothioconazole/ha with a 14 day interval between applications. The applications were made at growth stages BBCH 51-79.

The samples were frozen after collection and stored frozen (< -20ºC) until extraction and analysis. The maximum period of storage was 213 days (7 months). Residues of prothioconazole-desthio and prothioconazole were determined by HPLC/MS/MS method 01013. The LOQ was 0.01 mg/kg for each analyte.

Table 9 Prothioconazole residues in soya bean resulting from supervised trials in Brazil

SOYA BEAN Trial year (Variety)	Application					PHI	Commodity	JAU 6476 mg/kg	JAU 6476 desthio mg/kg	JAU 6476 total mg/kg	Reference
	Formulation (g ai/L)	g ai/ha	g ai/hL	Water (L/ha)	No	days					
GAP, BRAZIL	175 g/L SC	87.5			4	30					
F12-012-01 Brazil, 2012 (TMG 7161 RR)	450 g/L SC [a]	81.7-93.5	41-47	200	4	25 30 35	seed	< 0.01 < 0.01 < 0.01	0.02 0.02 0.01	0.03 0.03 0.02	Resende, 2013, F12-012
F12-012-02 Brazil, 2012 (Monsoy 7808 RR)	450 g/L SC [a]	87.5-107.8	44-54	200	4	25 30 34	seed	< 0.01 < 0.01 < 0.01	0.09 0.10 0.04	0.10 0.11 0.05	Resende, 2013, F12-012
F12-012-03 Brazil, 2012 (M 7908)	450 g/L SC [a]	86.4-95.8	43-48	200	4	25 30 35	seed	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.02 < 0.02 < 0.02	Resende, 2013, F12-012
F12-012-04 Brazil, 2012 (NA 5909 RG)	450 g/L SC [a]	79.0-97.5	40-49	200	4	30	seed	< 0.01	0.01	0.02	Resende, 2013, F12-012

SOYA BEAN Trial year (Variety)	Application					PHI	Commodity	JAU 6476 mg/kg	JAU 6476 desthio mg/kg	JAU 6476 total mg/kg	Reference
	Formulation (g ai/L)	g ai/ha	g ai/hL	Water (L/ha)	No	days					
F12-012-05 Brazil, 2012 (M 7908)	450 g/L SC [a]	91.4-99.5	44-50	200	4	30	seed	< 0.01	0.01	0.02	Resende, 2013, F12-012
F13-008-01 Brazil, 2013 (TMG 7161 RR)	450 g/L SC [a]	87.5-92.4	44-46	200	4	15 20 25 30 35	seed	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.01 0.02 0.02 0.03 0.02	0.02 0.03 0.03 0.04 0.03	Resende, 2013, F13-008
F13-008-02 Brazil, 2013 (TMG 7161 RR)	450 g/L SC [a]	84.7-89.2	42-45	200	4	15 20 25 30 35	seed	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.02 0.02 0.01 < 0.01 < 0.01	0.03 0.03 < 0.02 < 0.02 < 0.02	Resende, 2013, F13-008
F13-008-03 Brazil, 2013 (TMG 7161 RR)	450 g/L SC [a]	81.0-91.5	41-46	200	4	15 20 25 30 35	seed	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	Resende, 2013, F13-008
F13-008-04 Brazil, 2013 (NA 5909)	450 g/L SC [a]	85.9-90.7	43-45	200	4	15 20 25 30 35	seed	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.03 0.05 0.04 0.04 0.03	0.04 0.06 0.05 0.05 0.04	Resende, 2013, F13-008
F13-008-05 Brazil, 2013 (Monsoy 7905)	450 g/L SC [a]	87.5-90.4	44-45	200	4	15 20 25 30 35	seed	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.01 0.03 0.02 < 0.01 < 0.01	0.02 0.04 0.03 < 0.02 < 0.02	Resende, 2013, F13-008

[a] 450 g/L SC formulation containing 175 g/L prothioconazole + 150 g/L trifloxystrobin + 125 g/L bixafen

Root and tuber vegetables

Potato

Supervised trials in Europe (Belgium, UK, Netherlands, Germany, France, Spain and Italy): A total of twenty supervised trials were conducted in 2005 and 2010 on potato (Ref: Bomke & Bauer, 2011, 10-2085; Freitag & Wolters, 2007, RA-2569/05; Freitag & Wolters, 2007, RA-2604/05; Freitag & Wolters, 2007, RA-2570/05 and Freitag & Wolters, 2007, RA-2603/05). All trials were harvest trials.

In the study of Bomke & Bauer, (10-2085), an FS formulation containing 18 g/L prothioconazole and 100 g/L penflufen was applied as a seed treatment to potato tubers at 0.72 g prothioconazole/100 kg seed (equivalent to 36 g prothioconazole/ha). This is a higher application rate than the GAP in the EU (up to 0.64 g ai/100 kg seed, equivalent to 16 g ai/ha), but the trials demonstrate that residues in tubers at harvest are <LOQ, even at the higher application rate. Treated tubers for planting were collected after treatment (shown as 0 or -1 day DAT in the table below, where a 0 day corresponds to the day of planting treated tubers), and then tubers collected at maturity (90 day DAT). Residues of prothioconazole and prothioconazole-desthio were determined by HPLC/MS/MS using method 01013. The LOQ was 0.01 mg/kg for each analyte.

In the studies of Freitag & Wolters, (RA-2569/05) and Freitag & Wolters, (RA-2604/05) an FS formulation containing 8 g/L prothioconazole and 248 g/L penflufen was applied as a seed piece treatment using a roller table prior to planting at 0.60 g prothioconazole/100 kg seed pieces. Treated tubers for planting were collected after treatment (0 day), and then tubers collected at BBCH 43–48 (90–91 DAT) and at maturity/BBCH 49 (110–148 DAT). Residues of prothioconazole and prothioconazole-desthio were determined by HPLC/MS/MS using method 00598/M001. The LOQ was 0.01 mg/kg for each analyte

In the studies of Freitag & Wolters, (RA-2570/05) and Freitag & Wolters (RA-2603/05), an FS formulation containing 8 g/L prothioconazole and 248 g/L penflufen was applied to potato tubers at planting using an in-furrow method at 30.1–32.0 g prothioconazole/ha. Treated tubers were collected after treatment (0 day), then at BBCH 43–48 (90–91 DAT) and at maturity/BBCH 49 (110–148 DAT). Residues of prothioconazole and prothioconazole-desthio were determined by HPLC/MS/MS using method 00598/M001. The LOQ was 0.01 mg/kg for each analyte.

The samples were frozen after collection and stored frozen (< -20 ºC) until extraction and analysis. The maximum period of storage was 318 days (10.6 months).

Table 10 Prothioconazole residues in potato resulting from supervised trials in Europe

POTATO Trial Country, year (Variety)	Application					DAT days	Commodity	JAU 6476 mg/kg	JAU 6476 desthio mg/kg	Reference
	Formulation (g ai/L)	g ai/ha	g ai/hL	Water (L/ha)	No					
GAP, EU	8 g/L FS	12-16	-	-	1	F - The PHI is covered by the vegetation time between application and harvest, no PHI is set.				
10-2085-01 UK, 2010 (Nicola)	118 g/L FS [a]	36 (0.72 g ai/ 100 kg seed)	-	-	1	-1 90	Tuber for planting Tuber	0.89	0.57 < 0.01	Bomke & Bauer, 2011, 10-2085
10-2085-02 France, 2010 (Nicola)	118 g/L FS [a]	36 (0.72 g ai/ 100 kg seed)	-	-	1	0 90	Tuber for planting Tuber	1.21	0.97 < 0.01	Bomke & Bauer, 2011, 10-2085
10-2085-03 Belgium, 2010 (Nicola)	118 g/L FS [a]	36 (0.72 g ai/ 100 kg seed)	-	-	1	0 90	Tuber for planting Tuber	1.17	1.07 < 0.01	Bomke & Bauer, 2011, 10-2085
10-2085-04 Netherlands, 2010 (Nicola)	118 g/L FS [a]	36 (0.72 g ai/ 100 kg seed)	-	-	1	0 90	Tuber for planting Tuber	1.34	0.78 < 0.01	Bomke & Bauer, 2011, 10-2085
R 2005 0466/7 Netherlands, 2005 (Bintje)	258 g/L FS [b]	8.4 (0.60 g ai/ 100 kg seed)	3000	-	1	0 90 124	Tuber for planting Tuber Tuber	0.52 < 0.01 < 0.01	0.95 < 0.01 < 0.01	Freitag & Wolters, 2007, RA-2569/05
R 2005 0873/5 UK, 2005 (Bintje)	258 g/L FS [b]	7.2 (0.60 g ai/ 100 kg seed)	3000	-	1	0 91 133	Tuber for planting Tuber Tuber	0.74 < 0.01 < 0.01	0.33 < 0.01 < 0.01	Freitag & Wolters, 2007, RA-2569/05
R 2005 0874/3	258 g/L FS [b]	5.6 (0.60 g ai/	3000	-	1	0	Tuber for planting	0.38	1.20	Freitag & Wolters,

POTATO Trial Country, year (Variety)	Application Formulation (g ai/L)	g ai/ha	g ai/hL	Water (L/ha)	No	DAT days	Commodity	JAU 6476 mg/kg	JAU 6476 desthio mg/kg	Reference
Germany, 2005 (Bintje)		100 kg seed)				90	Tuber	< 0.01	< 0.01	2007, RA-2569/05
						136	Tuber	< 0.01	< 0.01	
R 2005 0875/1 France, 2005 (Bintje)	258 g/L FS [b]	4.8 (0.60 g ai/ 100 kg seed)	3000	-	1	0	Tuber for planting	0.28	0.97	Freitag & Wolters, 2007, RA-2569/05
						90	Tuber	< 0.01	< 0.01	
						148	Tuber	< 0.01	< 0.01	
R 2005 0881/6 France, 2005 (Bintje)	258 g/L FS [b]	4.0 (0.60 g ai/ 100 kg seed)	3000	-	1	0	Tuber for planting	0.20	0.79	Freitag & Wolters, 2007, RA-2604/05
						90	Tuber	< 0.01	< 0.01	
						122	Tuber	< 0.01	< 0.01	
R 2005 0882/4 Italy, 2005 (Bintje)	258 g/L FS [b]	10.8 (0.60 g ai/ 100 kg seed)	3000	-	1	0	Tuber for planting	0.70	0.48	Freitag & Wolters, 2007, RA-2604/05
						90	Tuber	< 0.01	< 0.01	
						118	Tuber	< 0.01	< 0.01	
R 2005 0883/2 France, 2005 (Bintje)	258 g/L FS [b]	5.7 (0.60 g ai/ 100 kg seed)	3000	-	1	0	Tuber for planting	1.10	0.47	Freitag & Wolters, 2007, RA-2604/05
						90	Tuber	< 0.01	< 0.01	
						110	Tuber	< 0.01	< 0.01	
R 2005 0884/0 Spain, 2005 (Bintje)	258 g/L FS [b]	10.8 (0.60 g ai/ 100 kg seed)	3000	-	1	0	Tuber for planting	0.74	0.34	Freitag & Wolters, 2007, RA-2604/05
						91	Tuber	< 0.01	< 0.01	
						128	Tuber	< 0.01	< 0.01	
R 2005 0467/5 France, 2005 (Bintje)	258 g/L FS [b]	32.0	16.0	200	1	0	Tuber, treated	0.16	0.13	Freitag & Wolters, 2007, RA-2570/05
						90	Tuber	< 0.01	< 0.01	
						110	Tuber	< 0.01	< 0.01	
R 2005 0877/8 Italy, 2005 (Bintje)	258 g/L FS [b]	32.0	10.7	300	1	0	Tuber, treated	0.11	0.11	Freitag & Wolters, 2007, RA-2570/05
						90	Tuber	< 0.01	< 0.01	
						118	Tuber	< 0.01	< 0.01	
R 2005 0878/6 France, 2005 (Bintje)	258 g/L FS [b]	32.0	10.7	300	1	0	Tuber, treated	0.04	0.05	Freitag & Wolters, 2007, RA-2570/05
						90	Tuber	< 0.01	< 0.01	
						122	Tuber	< 0.01	< 0.01	
R 2005 0879/4 Spain, 2005 (Bintje)	258 g/L FS [b]	32.0	10.7	300	1	0	Tuber, treated	0.03	0.03	Freitag & Wolters, 2007, RA-2570/05
						91	Tuber	< 0.01	< 0.01	
						128	Tuber	< 0.01	< 0.01	
R 2005 0880/8	258 g/L FS [b]	32.0	16.0	200	1	0	Tuber, treated	0.09	0.06	Freitag & Wolters,

POTATO Trial Country, year (Variety)	Application					DAT	Commodity	JAU 6476 mg/kg	JAU 6476 desthio mg/kg	Reference
	Formulation (g ai/L)	g ai/ha	g ai/hL	Water (L/ha)	No	days				
Germany, 2005 (Bintje)						90 116	Tuber Tuber	< 0.01 < 0.01	< 0.01 < 0.01	2007, RA-2603/05
R 2005 0885/9 France, 2005 (Bintje)	258 g/L FS [b]	32.0	16.0	200	1	0	Tuber, treated	0.06	0.09	Freitag & Wolters, 2007, RA-2603/05
						90 148	Tuber Tuber	< 0.01 < 0.01	< 0.01 < 0.01	
R 2005 0886/7 Germany, 2005 (Bintje)	258 g/L FS [b]	32.0	16.0	200	1	0	Tuber, treated	0.06	0.05	Freitag & Wolters, 2007, RA-2603/05
						90 136	Tuber Tuber	< 0.01 < 0.01	< 0.01 < 0.01	
R 2005 0887/5 Netherlands, 2005 (Bintje)	258 g/L FS [b]	32.0	16.0	200	1	0	Tuber, treated	0.03	0.04	Freitag & Wolters, 2007, RA-2603/05
						90 124	Tuber Tuber	< 0.01 < 0.01	< 0.01 < 0.01	

[a] 118 g/L FS formulation containing 18 g/L prothioconazole + 100 g/L penflufen.
[b] 258 g/L SE formulation containing 8 g/L prothioconazole + 248 g/L pencycuron.

Cereal grains

Maize (field corn) and popcorn

Supervised trials in the USA: Twenty supervised trials were conducted in 2006 on field corn and three trials on popcorn in the USA (Krolski & Harbin, 2008, RAJAP004). The trials were carried out according to the GAP for corn in the USA. Four foliar applications were made to each treated plot with an SC formulation containing 480 g/L prothioconazole with 2–11-day intervals between the application. Each spray included a non-ionic surfactant.

In the field corn trials, grain was harvested at 14 days (BBCH 89) after last application. In the decline trials, grain samples were collected at PHIs of 0, 7, 13–14, 20–21 and 27–28 days.

In the popcorn trials, grain was sampled at harvest at 14 DAT (BBCH 89).

The samples were frozen after collection and stored frozen (< -20 ºC) until extraction and analysis. The maximum period of storage was 365 days (12 months. Residues were determined by high performance liquid chromatography – triple stage quadrupole mass spectrometry (LC/MS/MS) with LOQ of 0.02 mg/kg equivalent to 0.0018 mg/kg prothioconazole desthio.

Table 11 Prothioconazole residues in field corn (maize) resulting from supervised trials in the USA

FIELD CORN (MAIZE) Trial year (Variety)	Application					DAT days	Commodity	JAU 6476 sulfonic acid mg/kg	JAU 6476 desthio mg/kg	JAU 6476 total mg/kg	Reference
	Formulation (g ai/L)	g ai/ha	g ai/hL	Water (L/ha)	No						
GAP, USA	480 g/L SC	200			4	0/14					
JA004-06HA Pennsylvania, 2006 (TA5750)	480 g/L SC	203-210	111-112	181-197	4	14 14	Grain	< 0.02	< 0.018	< 0.02	Krolski & Harbin, 2008, RAJAP004

FIELD CORN (MAIZE) Trial year (Variety)	Application					DAT days	Commodity	JAU 6476 sulfonic acid mg/kg	JAU 6476 desthio mg/kg	JAU 6476 total mg/kg	Reference
	Formulation (g ai/L)	g ai/ha	g ai/hL	Water (L/ha)	No						
JA006-06HA Georgia, USA, 2006 (31N26)	480 g/L SC	200-201	142-160	125-141	4	14	Grain	< 0.02	< 0.018	< 0.02	Krolski & Harbin, 2008, RAJAP004
JA013-06DA Iowa, USA, 2006 (33N30LL)	480 g/L SC	192-202	160-178	115-120	4	0 7 13 20 27	Grain Grain Grain Grain Grain	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	< 0.018 < 0.018 < 0.018 < 0.018 < 0.018	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	Krolski & Harbin, 2008, RAJAP004
JA014-06DA Minnesota, USA, 2006 (Pioneer 39H85)	480 g/L SC	196 201	110-119	169-178	4	0 7 14 21 28	Grain Grain Grain Grain Grain	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	0.04 0.05 0.05 0.03 0.05	0.05 0.06 0.06 0.03 0.07	Krolski & Harbin, 2008, RAJAP004
JA015-06HA Kansas, USA, 2006 (Garst 8881RR)	480 g/L SC	195-202	149-150	130-136	4	14	Grain	< 0.02	< 0.018	< 0.02	Krolski & Harbin, 2008, RAJAP004
JA016-06HA Illinois, USA, 2006 (Garst 8568 CB/LL)	480 g/L SC	193-209	116-143	145-175	4	13	Grain	< 0.02	< 0.018	< 0.02	Krolski & Harbin, 2008, RAJAP004
JA017-06HA Nebraska, USA, 2006 (NK38B4)	480 g/L SC	197-202	151-154	130-132	4	11	Grain	< 0.02	< 0.018	< 0.02	Krolski & Harbin, 2008, RAJAP004
JA018-06HA Iowa, USA, 2006 (NK65C5)	480 g/L SC	200-202	151-154	130-132	4	12	Grain	< 0.02	< 0.018	< 0.02	Krolski & Harbin, 2008, RAJAP004
JA019-06HA Missouri, USA, 2006 (34B20)	480 g/L SC	200-205	112-118	170-178	4	14	Grain	< 0.02	< 0.018	< 0.02	Krolski & Harbin, 2008, RAJAP004
JA020-06HA North Dakota, USA, 2006 (DKC35-51)	480 g/L SC	197-205	107-131	156-184	4	14	Grain	< 0.02	< 0.018	< 0.02	Krolski & Harbin, 2008, RAJAP004
JA021-06HA North Dakota, USA, 2006 (Pioneer 39D81)	480 g/L SC	193 200	129-142	136-154	4	14	Grain	< 0.02	< 0.018	< 0.02	Krolski & Harbin, 2008, RAJAP004
JA022-06HA Iowa, USA, 2006 (FA6508)	480 g/L SC	204-205	126-143	143-163	4	14	Grain	< 0.02	< 0.018	< 0.02	Krolski & Harbin, 2008, RAJAP004

FIELD CORN (MAIZE) Trial year (Variety)	Application					DAT days	Commodity	JAU 6476 sulfonic acid mg/kg	JAU 6476 desthio mg/kg	JAU 6476 total mg/kg	Reference
	Formulation (g ai/L)	g ai/ha	g ai/hL	Water (L/ha)	No						
JA023-06HA Illinois, USA, 2006 (B-T 6516 RR2YG)	480 g/L SC	198-203	110-120	167-184	4	14	Grain	< 0.02	< 0.018	< 0.02	Krolski & Harbin, 2008, RAJAP004
JA024-06HA Minnesota, USA, 2006 (38G16)	480 g/L SC	196-202	113-114	172-177	4	14	Grain	< 0.02	< 0.018	< 0.02	Krolski & Harbin, 2008, RAJAP004
JA025-06HA Nebraska, USA, 2006 (Pioneer 34N45YG/RR)	480 g/L SC	200-202	107-108	185-188	4	14	Grain	< 0.02	< 0.018	< 0.02	Krolski & Harbin, 2008, RAJAP004
JA026-06HA Indiana, USA, 2006 (DKC 57-59 RR/plus)	480 g/L SC	197-202	109-120	165-182	4	14	Grain	< 0.02	< 0.018	< 0.02	Krolski & Harbin, 2008, RAJAP004
JA027-06HA Iowa, USA, 2006 (Pioneer 34N50)	480 g/L SC	200-204	108-168	122-184	4	14	Grain	< 0.02	< 0.018	< 0.02	Krolski & Harbin, 2008, RAJAP004
JA028-06HA Ohio, USA, 2006 (Crows 7R154)	480 g/L SC	201-205	133-136	149-153	4	14	Grain	< 0.02	< 0.018	< 0.02	Krolski & Harbin, 2008, RAJAP004
JA029-06HA Minnesota, USA, 2006 (Pioneer 38H66)	480 g/L SC	195-203	123-125	156-163	4	14	Grain	< 0.02	< 0.018	< 0.02	Krolski & Harbin, 2008, RAJAP004
JA030-06HA Texas, USA, 2006 (Pioneer 31G97)	480 g/L SC	193-201	146-153	127-137	4	14	Grain	< 0.02	< 0.018	< 0.02	Krolski & Harbin, 2008, RAJAP004

Table 12 Prothioconazole residues in popcorn resulting from supervised trials in the USA

POPCORN Trial Country, year (Variety)	Application					PHI days	Commodity	JAU 6476 sulfonic acid mg/kg	JAU 6476 desthio mg/kg	JAU 6476 total mg/kg	Reference
	Formulation (g ai/L)	g ai/ha	g ai/hL	Water (l/ha)	No						
GAP, USA	480 g/L SC	200			4	0/14					
JA034-06HA Kansas, USA, 2006 (Yellow)	480 g/L SC	195-206	142-145	136-143	4	14	Grain	< 0.02	< 0.018	< 0.02	Krolski & Harbin, 2008, RAJAP004

POPCORN Trial Country, year (Variety)	Application					PHI days	Commodity	JAU 6476 sulfonic acid mg/kg	JAU 6476 desthio mg/kg	JAU 6476 total mg/kg	Reference
	Formulation (g ai/L)	g ai/ha	g ai/hL	Water (l/ha)	No						
JA035-06HA Illinois, USA, 2006 (M2101)	480 g/L SC	197-207	115-143	145-174	4	12 12	Grain	< 0.02	< 0.018	< 0.02	Krolski & Harbin, 2008, RAJAP004
JA036-06HA Nebraska, USA, 2006 (AP2501)	480 g/L SC	198-200	153-155	128-130	4	12 12	Grain	< 0.02	< 0.018	< 0.02	Krolski & Harbin, 2008, RAJAP004

Supervised trials in Brazil: Ten supervised trials were conducted in 2011-2012 on corn in Brazil matching the GAP for corn in Brazil.

In the study of Santiago (F11-034) two foliar applications were made to each treated plot with an SC formulation containing 175 g/L prothioconazole + 150 g/L trifloxystrobin at 87.5 g prothioconazole/ha with a 15-day interval between applications. The applications were made at growth stages BBCH 55–67 and BBCH 60–71.

In the study of Resende (F12-011) two foliar applications were made to each treated plot with an SC formulation containing 175 g/L prothioconazole + 125 g/L bixafen + 150 g/L trifloxystrobin at 87.5 g prothioconazole/ha with a 14-day interval between applications. The applications were made at growth stages BBCH 55-61 and BBCH 65-71.

In the decline trials, seeds were harvested at 10, 14–15 and 19–20 days (BBCH 73–79, 75–79 and 75–79) after last treatment. In the at harvest trials, seeds were harvested at 14–15 days DAT (BBCH 75–79). The samples were frozen after collection and stored frozen (< -20 ºC) until extraction and analysis. The maximum period of storage was 88 days (3 months).

Residues of prothioconazole-desthio and prothioconazole were determined by HPLC/MS/MS using method 01013 with the chromatographic conditions described in method 00765. The LOQ was 0.01 mg/kg for each analyte. Procedural recoveries at fortification levels of 0.01–1.0 mg/kg were within the acceptable range of 70–120%, RSD <20%, for prothioconazole and prothioconazole-desthio.

Table 13 Prothioconazole residues in corn (maize) resulting from supervised trials in Brazil

FIELD CORN Trial Country, year (Variety)	Application					DAT days	Commodity	JAU 6476 mg/kg	JAU 6476 desthio mg/kg	JAU 6476 total mg/kg	Reference
	Formulation (g ai/L)	g ai/ha	g ai/hL	Water (L/ha)	No						
GAP, BRAZIL	175 g/L SC	87.5			2	15					
F11-034-01 Brazil, 2011-2012 (Pioneer 30F35H)	325 g/L SC [a]	87.5	44	200	2	10 15 20	seed	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.02 < 0.02 < 0.02	Santiago, 2012, F11-034
F11-034-02 Brazil, 2011-2012 (Maximus TL)	325 g/L SC [a]	87.5-88.9	44-45	200	2	10 15 20	seed	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.02 < 0.02 < 0.02	Santiago, 2012, F11-034

FIELD CORN Trial Country, year (Variety)	Application					DAT days	Commodity	JAU 6476 mg/kg	JAU 6476 desthio mg/kg	JAU 6476 total mg/kg	Reference
	Formulation (g ai/L)	g ai/ha	g ai/hL	Water (L/ha)	No						
F11-034-03 Brazil, 2012 (DKB 350)	325 g/L SC [a]	86.5-89.4	43-45	200	2	10 15 20	seed	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.02 < 0.02 < 0.02	Santiago, 2012, F11-034
F11-034-04 Brazil, 2012 (Pioneer 30F53)	325 g/L SC [a]	93.3-96.3	47-48	200	2	15	seed	< 0.01	< 0.01	< 0.02	Santiago, 2012, F11-034
F11-034-05 Brazil, 2012 (DKB 350)	325 g/L SC [a]	94.2-95.2	47-48	200	2	15	seed	< 0.01	< 0.01	< 0.02	Santiago, 2012, F11-034
F12-011-01 Brazil, 2012 (DKB 350)	450 g/L SC [b]	77.6-101.7	39-51	200	2	10 14 19	seed	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.02 < 0.02 < 0.02	Resende, 2013, F12-011
F12-011-02 Brazil, 2012 (Pioneer 30F53)	450 g/L SC [b]	93.5-97.5	47-49	200	2	10 14 20	seed	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.02 < 0.02 < 0.02	Resende, 2013, F12-011
F12-011-03 Brazil, 2012 (Maximum TL)	450 g/L SC [b]	95.0-102.8	48-51	200	2	10 15 20	seed	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.02 < 0.02 < 0.02	Resende, 2013, F12-011
F12-011-04 Brazil, 2012 (P30F35)	450 g/L SC [b]	87.5-99.8	44-50	200	2	14	seed	< 0.01	< 0.01	< 0.02	Resende, 2013, F12-011
F12-011-05 Brazil, 2012 (DKB 350)	450 g/L SC [b]	84.7-102.7	42-51	200	2	14	seed	< 0.01	< 0.01	< 0.02	Resende, 2013, F12-011

[a] 325 g/L SC formulation containing 175 g/L prothioconazole + 150 g/L trifloxystrobin
[b] 450 g/L SC formulation containing 175 g/L prothioconazole + 125 g/L bixafen + 150 g/L trifloxystrobin

Supervised trials in Europe (Belgium, UK, Netherlands, Germany, France, Spain, Italy, Portugal and Greece): A total of twenty supervised trials were conducted in 2007, 2008 and 2011 on maize/corn in Europe All trials were decline trials.

In the first set of studies (Freitag & Reineke, 2009, 08-2035 and Freitag & Reineke, 2009, 08-2036) an EC formulation containing 125 g/L prothioconazole and 125 g/L tebuconazole was applied twice to maize at 125 g prothioconazole/ha, with an interval of 23–54 days between applications. The applications were made at growth stages BBCH 33–34 and BBCH 69. Immature (milk ripe) seeds were collected at BBCH 75–79 (8–26 DAT), ear without husk was collected at BBCH 85 (28–56 day PHI) and mature seeds at BBCH 89 (PHI 52–83 DAT). Residues of prothioconazole-desthio were determined by HPLC/MS/MS using method 01013. The LOQ was 0.01 mg/kg. Procedural recoveries at fortification levels of 0.01–3.0 mg/kg were within the acceptable range of 70–120%, RSD < 20%, for prothioconazole-desthio in each matrix.

In the second study (Freitag, 2008, RA-2675/07), an EC formulation containing 250 g/L prothioconazole was applied twice to maize at 200 g prothioconazole/ha, with an interval of 23–43 days between applications. The applications were made at growth stages BBCH 33 and BBCH 69. Ear without husk was collected at BBCH 85 (23–46 DAT) and mature seeds at BBCH 89 (56–69 DAT). Residues of prothioconazole-desthio were determined by HPLC/MS/MS using method 01013 (LOQ was 0.01 mg/kg). Procedural recoveries at fortification levels of 0.01–2.0 mg/kg were within the acceptable range of 70–120%, RSD <20%, for prothioconazole-desthio in each matrix.

In the other studies (Bomke & Teubner, 2013, 11-2109 and Bomke, 2012, 11-2110) an EC formulation containing 125 g/L prothioconazole and 125 g/L fluopyram was applied twice to maize at 130 g prothioconazole/ha, with an interval of 13–16 days between applications. The applications were made at growth stages BBCH 51–65 and BBCH 67–71. Immature (milk ripe) seeds were collected at BBCH 79 (14–31 DAT), ear without husk was collected at BBCH 79 (14–31 DAT) and BBCH 85 (28–56 DAT) and mature seeds and rest of plant at BBCH 89 (48–82 DAT). Residues of prothioconazole-desthio were determined by HPLC/MS/MS using method 01013. The LOQ was 0.01 mg/kg. Procedural recoveries at fortification levels of 0.01–1.0 mg/kg were in the range of 70–120%, RSD < 20%, for prothioconazole-desthio in each matrix.

The samples were frozen after collection and stored frozen (< -20 ºC) until extraction and analysis. The maximum period of storage was 406 days (13.3 months).

Table 14 Prothioconazole residues in maize/corn resulting from supervised trials in Europe

MAIZE Trial Country, year (Variety)	Application					DAT days	Commodity	JAU 6476 desthio mg/kg	Reference
	Formulation (g ai/L)	g ai/ha	g ai/hL	Water (L/ha)	No				
GAP, EU	250 g/L EC	125			2	-			
08-2035-01 UK, 2008 (Cixxom)	250 g/L EC [a]	125	63	200	2	19	seed, immature (milk ripe)	< 0.01	Freitag & Reineke, 2009, 08-2035
						56	ear w/o husk	< 0.01	
						69	seed	<u>< 0.01</u>	
08-2035-02 Germany, 2008 (Justina)	250 g/L EC [a]	125	42	300	2	24	seed, immature (milk ripe)	< 0.01	Freitag & Reineke, 2009, 08-2035
						49	ear w/o husk	< 0.01	
						64	seed	<u>< 0.01</u>	
08-2035-03 Germany, 2008 (Seleno)	250 g/L EC [a]	125	32-42	300-400	2	26			Freitag & Reineke, 2009, 08-2035
							seed, immature (milk ripe)	< 0.01	
						37	ear w/o husk	< 0.01	
						57	seed	<u>< 0.01</u>	
08-2035-04 Netherlands, 2008 (Rosalie)	250 g/L EC [a]	125	32	400	2	18	seed, immature (milk ripe)	< 0.01	Freitag & Reineke, 2009, 08-2035
						40	ear w/o husk	< 0.01	
08-2036-01 France, 2008 (PR33Y74)	250 g/L EC [a]	125	42	300	2	11	seed, immature (milk ripe)	< 0.01	Freitag & Reineke, 2009, 08-2036
						34	ear w/o husk	< 0.01	
						83	seed	<u>< 0.01</u>	
08-2036-02 Spain, 2008 (DKC6451YG)	250 g/L EC [a]	125-132	42	300-316	2	19	seed, immature (milk ripe)	< 0.01	Freitag & Reineke, 2009, 08-2036
						28	ear w/o husk	< 0.01	
						53	seed	<u>< 0.01</u>	

MAIZE Trial Country, year (Variety)	Application					DAT days	Commodity	JAU 6476 desthio mg/kg	Reference
	Formulation (g ai/L)	g ai/ha	g ai/hL	Water (L/ha)	No				
08-2036-03 Italy, 2008 (PR33A46)	250 g/L EC [a]	125	31	400	2	13	seed, immature (milk ripe)	< 0.01	Freitag & Reineke, 2009, 08-2036
						34	ear w/o husk	< 0.01	
						55	seed	< 0.01	
08-2036-04 Portugal, 2008 (A46)	250 g/L EC [a]	125	25-42	300-500	2	8	seed, immature (milk ripe)	< 0.01	Freitag & Reineke, 2009, 08-2036
						30	ear w/o husk	< 0.01	
						52	seed	< 0.01	
R 2007 0829/7 Germany, 2007 (Nescio)	250 g/L EC	200	67	300	2	29 43	ear w/o husk	< 0.01 < 0.01	Freitag, 2008, RA-2675/07
						56	seed	< 0.01	
R 2007 0837/8 Germany, 2007 (DK3745)	250 g/L EC	200	67	300	2	19 46	ear w/o husk	< 0.01 < 0.01	Freitag, 2008, RA-2675/07
						68	seed	< 0.01	
R 2007 0838/6 Spain, 2007 (PR 31G98)	250 g/L EC	186-200	50	372-400	2	16 23	ear w/o husk	< 0.01 < 0.01	Freitag, 2008, RA-2675/07
						64	seed	< 0.01	
R 2007 0839/4 Portugal, 2007 (PR33N09)	250 g/L EC	200	67	300	2	15 33	ear w/o husk	< 0.01 < 0.01	Freitag, 2008, RA-2675/07
						69	seed	< 0.01	
11-2109-01 France, 2011 (Cobalt)	250 g/L SE [b]	125	42	300	2	20	seed, immature	< 0.01	Bomke & Teubner, 2013, 11-2109
						20 40	ear without husk	< 0.01 < 0.01	
						82	seed	< 0.01	
11-2109-02 Germany, 2011 (Saludo)	250 g/L SE [b]	125	42	300	2	31	seed, immature	< 0.01	Bomke & Teubner, 2013, 11-2109
						31 51	ear without husk	< 0.01 < 0.01	
						65	seed	< 0.01	
11-2109-03 UK 2011 (Cougar)	250 g/L SE [b]	125	50	250	2	31	seed, immature	< 0.01	Bomke & Teubner, 2013, 11-2109
						31 49	ear without husk	< 0.01 < 0.01	
						60	seed	< 0.01	
11-2109-04 Belgium, 2011 (Delitop)	250 g/L SE [b]	125	45	275	2	29	seed, immature	< 0.01	Bomke & Teubner, 2013, 11-2109
						29 42	ear without husk	< 0.01 < 0.01	
						64	seed	< 0.01	
11-2110-02	250 g/L SE [b]	125	31	400	2	17	seed, immature	< 0.01	Bomke, 2012,

MAIZE Trial Country, year (Variety)	Application					DAT days	Commodity	JAU 6476 desthio mg/kg	Reference
	Formulation (g ai/L)	g ai/ha	g ai/hL	Water (L/ha)	No				
Italy, 2011 (Constanza)						17 29	ear without husk	< 0.01 < 0.01	11-2110
						62	seed	< 0.01	
11-2110-03 Spain, 2011 (DKC66 67YG)	250 g/L SE [b]	125	42	300	2	20	ear without husk	< 0.01	Bomke, 2012, 11-2110
						69	seed	< 0.01	
11-2110-04 Greece, 2011 (Dekalp, 5276)	250 g/L SE [b]	125	31	400	2	18	seed, immature	< 0.01	Bomke, 2012, 11-2110
						18 31	ear without husk	< 0.01 < 0.01	
						48	seed	< 0.01	
11-2110-05 France, 2011 (PR 33 A46)	250 g/L SE [b]	125	42	300	2	14	seed, immature	< 0.01	Bomke, 2012, 11-2110
						14 29	ear without husk	< 0.01 < 0.01	
						64	seed	< 0.01	

ear w/o husk = ear (Seeds plus cob) without husk

[a] 250 g/L EC formulation containing 125 g/L prothioconazole + 125 g/L tebuconazole

[b] 250 g/L SE formulation containing 125 g/L prothioconazole + 125 g/L AE C656948 (fluopyram)

Oilseeds

Peanuts

Eight supervised trials were conducted in 2011–2012 on peanut in Australia (Ellis, 2012, BCS-0232 and Ellis, 2012, BCS-0383). Four trials included plots which were treated according to the GAP for peanut in Australia (4 times 120–192 g ai/ha at 10–14 days apart, 28-day PHI), and four trials included plots which were treated at total application rates within 25% of the GAP rate, but shorter PHI (up to 21 days). These trials are submitted in support of the use on peanuts as they show that residues in peanut seeds are <LOQ at all sampling intervals. All of the trials were decline trials.

In study of Ellis, (BCS-0232), 3, 3 or 5 foliar applications were made to each treated plot with an SC formulation containing 480 g/L prothioconazole at nominally 180 or 120 g ai/ha with a 10 day interval between applications. Seeds were harvested immediately before the last application and after 0, 7, 14–15 and 20–21 days. For the plot treated at 5 × 120 g ai/ha samples of seeds were collected after field drying following pulling at 7 DAT.

In another study of Ellis, (BSC-0383), each trial consisted of up to 7 treated plots. Plots were treated with an SC formulation containing 480 g/L prothioconazole. One plot received an in-furrow treatment at 192 g ai/ha at planting, and the plants lifted 117–167 DAT. The remaining plots received 3 or 4 foliar applications at 192 or 288 g ai/ha, or an in-furrow application followed by 3 or 4 foliar applications at 192 or 288 g ai/ha, and the plants lifted at 14–15, 21–22 and 27–29 DAT. In furrow applications were made at growth stage BBCH 00, and foliar applications were made at growth stages BBCH 59–89. For all plots, plants were left to dry for 7–13 days before samples of seeds were collected.

Residues of prothioconazole-desthio and prothioconazole were determined by HPLC/MS/MS using method ATM-0053. The LOQ was 0.01 mg/kg for each analyte and matrix in study BCS-0232, and 0.01 mg/kg for each analyte in seeds and shells and 0.05 mg/kg for each analyte in forage in study

BCD-0383. Procedural recoveries at fortification levels of 0.01 (or 0.05)–1.0 mg/kg were within the acceptable range of 70–120%, RSD < 20%, for prothioconazole and prothioconazole-desthio.

The samples were frozen after collection and stored frozen (< -20 ºC) until extraction and analysis. The maximum period of storage was 612 days (20 months), which falls within the confirmed stability period of 3 years.

In peanut seeds, residue levels of prothioconazole-desthio were < 0.01 mg/kg, residues of prothioconazole were < 0.01 mg/kg and residues of total prothioconazole (sum of prothioconazole-desthio and prothioconazole, expressed as prothioconazole) were < 0.02 mg/kg at all sampling intervals in all trials.

The results of the trials relevant to the Australian GAP are summarized in Table 15.

Table 15 Prothioconazole residues in peanut resulting from supervised trials in Australia

PEANUTS	Application					DAT	Commodity	JAU 6476	JAU 6476 desthio	JAU 6476 total	Reference
Trial Country, year (Variety)	Formulation (g ai/L)	g ai/ha	g ai/hL	Water (L/ha)	No	days		mg/kg	mg/kg	mg/kg	
GAP, AUSTRALIA	480 g/L SC	192			4	28					
C351 Australia, 2009 (SO05R)	480 g/L SC	176-177	79-80	221-223	4	-0 0 7 14 21	seed	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	Ellis, 2012, BCS-0232
		176-177	74-80	221-239	5	-0 0 7 14 21	seed	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	
C352 Australia, 2009 (Menzies)	480 g/L SC	175-178	73-79	223-239	4	-0 0 7 14 21	seed	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	Ellis, 2012, BCS-0232
		175-178	73-79	223-239	5	-0 0 7 14 21	seed	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	
C353 Australia, 2009 (Holt)	480 g/L SC	177-178	50-53	334-358	4	-0 0 7 14 21	seed	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	Ellis, 2012, BCS-0232
		177-178	50-54	328-358	5	-0 0 7 14 21	seed	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	

PEANUTS Trial Country, year (Variety)	Application					DAT days	Commodity	JAU 6476 mg/kg	JAU 6476 desthio mg/kg	JAU 6476 total mg/kg	Reference
	Formulation (g ai/L)	g ai/ha	g ai/hL	Water (L/ha)	No						
C354 Australia, 2009 (Walter)	480 g/L SC	176-177	38-45	395-442	4	-0 0 7 15 20	seed	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	Ellis, 2012, BCS-0232
		176-178	38-45	395-442	5	-0 0 7 15 20	seed	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	
C668 Australia 2011-2012 (HOJ-E Holt)	480 g/L SC	193-194	96	201-202	4	15 22 29	Seeds	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.02 < 0.02 < 0.02	Ellis, 2012, BCS-0383
C697 Australia 2012 (Holt)	480 g/L SC	192-193	96	199-201	4	14 21 28	Seeds	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.02 < 0.02 < 0.02	Ellis, 2012, BCS-0383
C680 Australia 2011-2012 (HOJ-L-2)	480 g/L SC	192-194	26-29	673-754	4	15 21 28	Seeds	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.02 < 0.02 < 0.02	Ellis, 2012, BCS-0383
C682 Australia 2011-2012 (WH2 04401)	480 g/L SC	192-193	25-37	517-778	4	14 21 27	Seeds	< 0.01 < 0.01 < 0.01	0.01 < 0.01 < 0.01	< 0.02 < 0.02 < 0.02	Ellis, 2012, BCS-0383

Primary feed commodities

Descriptions of trial conditions are given under the individual food commodities. The residue data relevant to the feed commodities are summarised below.

Sweet corn forage and stover

Supervised trials in the USA: Twelve supervised trials have been conducted in sweet corn in the USA, The trials were carried out according to a GAP for corn in the USA (4 × 200 g ai/ha). Forage and ears were harvested at 0 and 5–7 DALA (BBCH 73–79) and stover was harvested at 12-14 DALA (BBCH 89). In the decline trial, ears and forage samples were collected at 0, 3, 6, 10 and 14 days, and stover was collected at 10, 14, 20, 28 and 34 days.

Twenty supervised trials in field corn and three trials on popcorn have been conducted in the USA, Field corn forage is harvested at a PHI of 0 days, and field corn and popcorn stover is harvested at a PHI of 14 days.

Twenty supervised trials have been conducted in corn in Europe. Forage was harvested at 0 and 5–7 days (BBCH 85–87) and stover was harvested at 14 days (BBCH 89) after last application. In the decline trials, forage, and stover samples were collected at PHIs of 0, 7, 13–14, 20–21 and 27–28 days. Green material was collected prior to the final application (shown as -0 day in the table) and immediately after the final application (0 DAT), and then at grows stages ranging from BBCH 33 to BBCH 85.

Table 16 Prothioconazole residues in sweet corn forage and fodder from US trials

SWEET CORN	Application					DAT	Commodity	JAU 6476 sulfonic acid mg/kg	JAU 6476 desthio mg/kg	JAU 6476 total mg/kg	Reference
Trial Country, year (Variety)	Formulation (g ai/L)	g ai/ha	g ai/hL	Water (L/ha)	No	days					
GAP, USA	480 g/L SC	100			4	0/14					
JA002-06HA Virginia, USA, 2006 (Devotion)	480 g/L SC	201-208	128	156-163	4	0 6 14	Forage Forage Stover	0.95 0.14 0.09	4.08 1.52 0.82	5.45 1.82 0.99	Krolski & Harbin, 2008, RAJAP004
JA003-06HA New York, USA, 2006 (Gourmet Sweet 372A)	480 g/L SC	204-208	122-123	167-170	4	0 7 14	Forage Forage Stover	0.63 0.21 0.11	3.20 1.40 0.71	4.15 1.75 0.89	Krolski & Harbin, 2008, RAJAP004
JA005-06HA Georgia, USA, 2006 (Sweet G-90)	480 g/L SC	200	142-159	125-141	4	0 7 14	Forage Forage Stover	1.09 0.54 0.37	3.75 1.70 1.45	5.22 2.42 1.96	Krolski & Harbin, 2008, RAJAP004
JA007-06DA Florida, USA, 2006 (Silver Queen)	480 g/L SC	195-200	167-181	109-120	4	0 3 6 10 14 10 14 20 28 34	Forage Forage Forage Forage Forage Stover Stover Stover Stover Stover	0.46 0.33 0.18 0.19 0.17 0.30 0.39 0.28 0.37 0.35	2.08 1.56 0.75 0.98 0.80 1.38 1.70 1.48 1.47 1.27	2.76 2.08 1.00 1.28 1.06 1.82 2.26 1.91 1.99 1.75	Krolski & Harbin, 2008, RAJAP004
JA008-06HA Missouri, USA, 2006 (Peaches & Cream)	480 g/L SC	200-204	115-149	137-174	4	0 6 13	Forage Forage Stover	0.51 0.09 0.14		3.13 0.93 1.99	Krolski & Harbin, 2008, RAJAP004
JA009-06HA Illinois, USA, 2006 (BC 0805)	480 g/L SC	196-204	150-160	123-132	4	0 7 14	Forage Forage Stover	0.34 0.04 0.09		2.00 0.43 0.83	Krolski & Harbin, 2008, RAJAP004
JA010-06HA Nebraska, USA, 2006 (Serendipity)	480 g/L SC	199-204	153-157	129 131	4	0 5 13	Forage Forage Stover	0.43 0.12 0.14	1.89 0.83 0.98	2.51 1.04 1.21	Krolski & Harbin, 2008, RAJAP004
JA011-06HA Minnesota, USA, 2006 (39H85 Pioneer)	480 g/L SC	196-202	111-118	171-181	4	0 7 14	Forage Forage Stover	0.56 0.29 0.93	2.12 1.43 5.88	2.95 1.86 7.41	Krolski & Harbin, 2008, RAJAP004

SWEET CORN Trial Country, year (Variety)	Application					DAT	Commodity	JAU 6476 sulfonic acid mg/kg	JAU 6476 desthio mg/kg	JAU 6476 total mg/kg	Reference
	Formulation (g ai/L)	g ai/ha	g ai/hL	Water (L/ha)	No	days					
JA012-06HA Iowa, USA, 2006 (Mirai 131)	480 g/L SC	198-206	156-182		4	0	Forage	0.45	1.98	2.63	Krolski & Harbin, 2008, RAJAP004
						7	Forage	0.07	0.37	0.47	
						12	Stover	0.16	0.90	1.15	
JA031-06HA California, USA, 2006 (Silver Queen)	480 g/L SC	197-201	117-119	168-170	4	0	Forage	0.56	2.82	3.67	Krolski & Harbin, 2008, RAJAP004
						7	Forage	0.34	2.19	2.76	
						12	Stover	0.52	3.61	4.50	
JA032-06HA Washington, USA, 2006 (Golden Jubilee)	480 g/L SC	202-204	143-145	140-142	4	0	Forage	0.39	1.94	2.53	Krolski & Harbin, 2008, RAJAP004
						7	Forage	0.04	0.57	0.67	
						12	Stover	0.08	1.68	1.93	
JA033-06HA Oregon, USA, 2006 (Serendipity)	480 g/L SC	195-205	120-126	163-165	4	0	Forage	< 0.24	1.31	1.69	Krolski & Harbin, 2008, RAJAP004
						5	Forage	0.06	0.65	0.78	
						13	Stover	0.04	0.67	0.78	

Table 17 Prothioconazole residues in field corn (maize) resulting from supervised trials in the USA

FIELD CORN (MAIZE) Trial Country, year (Variety)	Application					DAT	Commodity	JAU 6476 sulfonic acid mg/kg	JAU 6476 desthio mg/kg	JAU 6476 total mg/kg	Reference
	Formulation (g ai/L)	g ai/ha	g ai/hL	Water (L/ha)	No	days					
GAP, USA	480 g/L SC	200			4	0/14					
JA004-06HA Pennsylvania, USA, 2006 (TA5750)	480 g/L SC	203-210	111-112	181-197	4	14	Stover	0.26	2.22	2.71	Krolski & Harbin, 2008, RAJAP004
		208-214	112	185-191	4	0	Forage	0.50	1.90	2.59	
						6	Forage	0.14	1.06	1.30	
JA006-06HA Georgia, USA, 2006 (31N26)	480 g/L SC	200-201	142-160	125-141	4	14	Stover	0.85	4.05	5.31	Krolski & Harbin, 2008, RAJAP004
		200	141-159	125-141	4	0	Forage	0.85	3.44	4.64	
						7	Forage	0.27	1.82	2.27	
JA013-06DA Iowa, USA, 2006 (33N30LL)	480 g/L SC	192-202	160-178	115-120	4	0	Stover	0.97	4.39	5.81	Krolski & Harbin, 2008, RAJAP004
						7	Stover	0.19	1.16	1.47	
						13	Stover	0.32	2.22	2.76	
						20	Stover	< 0.02	0.05	0.06	
						27	Stover	0.37	2.42	3.04	
		195-205	160-176	115-124	4	0	Forage	0.46	2.20	2.89	
						7	Forage	0.15	1.08	1.34	
						14	Forage	0.14	1.04	1.28	
						22	Forage	0.10	0.65	0.81	
						27	Forage	0.10	0.70	0.87	

FIELD CORN (MAIZE) Trial Country, year (Variety)	Application					DAT	Commodity	JAU 6476 sulfonic acid mg/kg	JAU 6476 desthio mg/kg	JAU 6476 total mg/kg	Reference
	Formulation (g ai/L)	g ai/ha	g ai/hL	Water (L/ha)	No	days					
JA014-06DA Minnesota, USA, 2006 (Pioneer 39H85)	480 g/L SC	196 201	110-119	169-178	4	0 7 14 21 28	Stover Stover Stover Stover Stover	3.15 1.37 0.91 0.45 0.71	11.90 5.69 4.66 2.40 3.68	16.27 7.65 6.05 3.11 4.77	Krolski & Harbin, 2008, RAJAP004
		194-204	114-122	164-179	4	0 7 14 22 27	Forage Forage Forage Forage Forage	0.69 0.16 0.16 0.14 0.13	2.15 0.98 1.13 1.00 0.86	3.06 1.24 1.40 1.24 1.08	
JA015-06HA Kansas, USA, 2006 (Garst 8881RR)	480 g/L SC	195-202	149-150	130-136	4	14	Stover	0.58	6.70	7.97	Krolski & Harbin, 2008, RAJAP004
		197-203	149-151	131-137	4	0 7	Forage Forage	0.73 0.25	3.06 2.38	4.11 2.87	
JA016-06HA Illinois, USA, 2006 (Garst 8568 CB/LL)	480 g/L SC	193-209	116-143	145-175	4	13	Stover	0.60	3.18	4.10	Krolski & Harbin, 2008, RAJAP004
		195-208	115-143	142-175	4	0 7	Forage Forage	0.88 0.62	3.60 3.34	4.84 4.30	
JA017-06HA Nebraska, USA, 2006 (NK38B4)	480 g/L SC	197-202	151-154	130-132	4	11	Stover	1.21	6.16	8.01	Krolski & Harbin, 2008, RAJAP004
		200-203	153-155	129-131	4	0 5	Forage Forage	0.54 0.17	2.27 1.11	3.04 1.39	
JA018-06HA Iowa, USA, 2006 (NK65C5)	480 g/L SC	200-202	151-154	130-132	4	12	Stover	0.33	2.32	2.89	Krolski & Harbin, 2008, RAJAP004
		198-201	151-153	130-132	4	0 7	Forage Forage	0.43 0.12	1.84 0.96	2.46 1.17	
JA019-06HA Missouri, USA, 2006 (34B20)	480 g/L SC	200-205	112-118	170-178	4	14	Stover	0.41	3.89	4.69	Krolski & Harbin, 2008, RAJAP004
		188-202	112-155	130-176	4	0 7	Forage Forage	0.45 0.08	2.62 1.20	3.34 1.40	
JA020-06HA North Dakota, USA, 2006 (DKC35-51)	480 g/L SC	197-205	107-131	156-184	4	14	Stover	0.27	2.35	2.87	Krolski & Harbin, 2008, RAJAP004
		198-208	107-138	152-185	4	0 6	Forage Forage	0.42 0.28	2.19 2.12	2.83 2.62	
JA021-06HA North Dakota, USA, 2006 (Pioneer 39D81)	480 g/L SC	193 200	129-142	136-154	4	14	Stover	0.65	3.68	4.71	Krolski & Harbin, 2008, RAJAP004
		189-203	119-142	134-170	4	0 7	Forage Forage	0.53 0.30	1.75 1.25	2.46 1.68	
JA022-06HA Iowa, USA, 2006 (FA6508)	480 g/L SC	204-205	126-143	143-163	4	14	Stover	0.57	3.78	4.74	Krolski & Harbin, 2008, RAJAP004
		200-207	137-138	145-153	4	0 7	Forage Forage	0.36 0.05	1.96 0.61	2.52 0.73	

FIELD CORN (MAIZE) Trial Country, year (Variety)	Application					DAT	Commodity	JAU 6476 sulfonic acid mg/kg	JAU 6476 desthio mg/kg	JAU 6476 total mg/kg	Reference
	Formulation (g ai/L)	g ai/ha	g ai/hL	Water (L/ha)	No	days					
JA023-06HA Illinois, USA, 2006 (B-T 6516 RR2YG)	480 g/L SC	198-203	110-120	167-184	4	14	Stover	0.36	1.09	1.57	Krolski & Harbin, 2008, RAJAP004
		198-203	112-124	163-177	4	0 7	Forage Forage	0.54 0.19	2.65 2.14	3.46 2.54	
JA024-06HA Minnesota, USA, 2006 (38G16)	480 g/L SC	196-202	113-114	172-177	4	14	Stover	0.59	3.59	5.64	Krolski & Harbin, 2008, RAJAP004
		198-216	113-114	174-191	4	0 7	Forage Forage	0.47 0.08	2.58 1.75	3.31 2.01	
JA025-06HA Nebraska, USA, 2006 (Pioneer 34N45YG/RR)	480 g/L SC	200-202	107-108	185-188	4	14	Stover	0.52	2.90	3.71	Krolski & Harbin, 2008, RAJAP004
		200-203	108-110	181-186	4	0 7	Forage Forage	0.31 0.13	1.67 0.93	2.15 1.16	
JA026-06HA Indiana, USA, 2006 (DKC 57-59 RR/plus)	480 g/L SC	197-202	109-120	165-182	4	14	Stover	0.17	0.99	1.27	Krolski & Harbin, 2008, RAJAP004
		197-201	110-118	170-180	4	0 7	Forage Forage	0.16 0.03	0.71 0.12	0.94 0.17	
JA027-06HA Iowa, USA, 2006 (Pioneer 34N50)	480 g/L SC	200-204	108-168	122-184	4	14	Stover	0.84	4.47	5.77	Krolski & Harbin, 2008, RAJAP004
		200-201	128-139	144-156	4	0 7	Forage Forage	0.42 0.14	2.41 1.28	3.08 1.55	
JA028-06HA Ohio, USA, 2006 (Crows 7R154)	480 g/L SC	201-205	133-136	149-153	4	14	Stover	0.31	3.48	4.14	Krolski & Harbin, 2008, RAJAP004
		198-206	135-139	143-152	4	0 7	Forage Forage	0.64 0.18	2.99 1.44	3.94 1.78	
JA029-06HA Minnesota, USA, 2006 (Pioneer 38H66)	480 g/L SC	195-203	123-125	156-163	4	14	Stover	0.48	3.22	4.03 < 0.02	Krolski & Harbin, 2008, RAJAP004
		198-200	125	159-160	4	0 7	Forage Forage	0.40 0.14	2.14 1.16	2.76 1.41	
JA030-06HA Texas, USA, 2006 (Pioneer 31G97)	480 g/L SC	193-201	146-153	127-137	4	14	Stover	0.33	3.42	4.10	Krolski & Harbin, 2008, RAJAP004
		200-205	146-153	131-138	4	0 5	Forage Forage	0.55 0.23	2.99 1.91	3.84 2.34	

Table 18 Prothioconazole residues in popcorn resulting from supervised trials in the USA

POPCORN Trial Country, year (Variety)	Application					DAT days	Commodity	JAU 6476 sulfonic acid mg/kg	JAU 6476 desthio mg/kg	JAU 6476 total mg/kg	Reference
	Formulation (g ai/L)	g ai/ha	g ai/hL	Water (L/ha)	No						
GAP, USA	480 g/L SC	200			4	0/14					
JA034-06HA Kansas, USA, 2006 (Yellow)	480 g/L SC	195-206	142-145	136-143	4	14	Stover	0.40	2.46	3.11	Krolski & Harbin, 2008, RAJAP004
JA035-06HA Illinois, USA, 2006 (M2101)	480 g/L SC	197-207	115-143	145-174	4	12	Stover	1.24	4.43	6.12	Krolski & Harbin, 2008, RAJAP004
JA036-06HA Nebraska, USA, 2006 (AP2501)	480 g/L SC	198-200	153-155	128-130	4	12	Stover	0.36	2.51	3.13	Krolski & Harbin, 2008, RAJAP004

Table 19 Prothioconazole residues in maize/corn forage resulting from supervised trials in Europe

MAIZE Trial Country, year (Variety)	Application					DAT days	Commodity	JAU 6476 desthio mg/kg	Reference
	Formulation (g ai/L)	g ai/ha	g ai/hL	Water (L/ha)	No				
GAP, EU	250 g/L EC	125			2	-			
08-2035-01 UK, 2008 (Cixxom)	250 g/L EC[1]	125	63	200	2	0* 0 19 56	green material	< 0.01 0.64 0.12 0.03	Freitag & Reineke, 2009, 08-2035
08-2035-02 Germany, 2008 (Justina)	250 g/L EC[1]	125	42	300	2	0* 0 24 49	green material	< 0.01 0.31 0.03 < 0.01	Freitag & Reineke, 2009, 08-2035
08-2035-03 Germany, 2008 (Seleno)	250 g/L EC[1]	125	32-42	300-400	2	0* 0 26 37	green material	0.06 0.40 0.04 0.03	Freitag & Reineke, 2009, 08-2035
08-2035-04 Netherlands, 2008 (Rosalie)	250 g/L EC[1]	125	32	400	2	0* 0 18 40	green material	0.05 0.55 0.11 0.05	Freitag & Reineke, 2009, 08-2035
08-2036-01 France, 2008 (PR33Y74)	250 g/L EC[1]	125	42	300	2	0* 0 11 34	green material	0.02 0.42 0.09 0.02	Freitag & Reineke, 2009, 08-2036

MAIZE Trial Country, year (Variety)	Application					DAT days	Commodity	JAU 6476 desthio mg/kg	Reference
	Formulation (g ai/L)	g ai/ha	g ai/hL	Water (L/ha)	No				
08-2036-02 Spain, 2008 (DKC6451YG)	250 g/L EC[1]	125-132	42	300-316	2	0* 0 19 28	green material	0.02 0.43 0.08 0.05	Freitag & Reineke, 2009, 08-2036
08-2036-03 Italy, 2008 (PR33A46)	250 g/L EC[1]	125	31	400	2	0* 0 13 34	green material	0.03 0.35 0.17 0.07	Freitag & Reineke, 2009, 08-2036
08-2036-04 Portugal, 2008 (A46)	250 g/L EC[1]	125	25-42	300-500	2	0* 0 8 30	green material	< 0.01 0.26 0.31 0.07	Freitag & Reineke, 2009, 08-2036
R 2007 0829/7 Germany, 2007 (Nescio)	250 g/L EC	200	67	300	2	-33 0* 0 29 43	green material	0.90 < 0.01 0.76 0.05 0.03	Freitag, 2008, RA-2675/07
R 2007 0837/8 Germany, 2007 (DK3745)	250 g/L EC	200	67	300	2	-39 0* 0 19 46	green material	1.3 < 0.01 0.49 0.03 0.01	Freitag, 2008, RA-2675/07
R 2007 0838/6 Spain, 2007 (PR 31G98)	250 g/L EC	186-200	50	372-400	2	-25 0* 0 16 23	green material	0.94 0.03 0.40 0.18 < 0.01	Freitag, 2008, RA-2675/07
R 2007 0839/4 Portugal, 2007 (PR33N09)	250 g/L EC	200	67	300	2	-43 0* 0 15 33	green material	1.7 0.34 0.42 0.05 0.02	Freitag, 2008, RA-2675/07
11-2109-01 France, 2011 (Cobalt)	250 g/L SE[2]	125	42	300	2	0* 0 20 40	green material	0.18 0.44 0.16 0.08	Bomke & Teubner, 2013, 11-2109
						82	rest of plant	0.06	
11-2109-02 Germany, 2011 (Saludo)	250 g/L SE[2]	125	42	300	2	0* 0 31 51	green material	0.13 0.65 0.05 0.02	Bomke & Teubner, 2013, 11-2109
						65	rest of plant	0.03	
11-2109-03 UK 2011 (Cougar)	250 g/L SE[2]	125	50	250	2	0* 0 31 49	green material	0.34 0.55 0.16 0.12	Bomke & Teubner, 2013, 11-2109
						60	rest of plant	0.22	

MAIZE Trial Country, year (Variety)	Application					DAT days	Commodity	JAU 6476 desthio mg/kg	Reference
	Formulation (g ai/L)	g ai/ha	g ai/hL	Water (L/ha)	No				
11-2109-04 Belgium, 2011 (Delitop)	250 g/L SE[2]	125	45	275	2	0* 0 29 42	green material	0.04 0.28 0.05 0.03	Bomke & Teubner, 2013, 11-2109
						64	rest of plant	0.04	
11-2110-02 Italy, 2011 (Constanza)	250 g/L SE[2]	125	31	400	2	0* 0 17 29	green material	0.04 0.50 0.06 0.03	Bomke, 2012, 11-2110
						62	rest of plant	0.05	
11-2110-03 Spain, 2011 (DKC66 67YG)	250 g/L SE[2]	125	42	300	2	0* 0 20	green material	0.05 0.39 0.06	Bomke, 2012, 11-2110
						69	rest of plant	0.05	
11-2110-04 Greece, 2011 (Dekalp, 5276)	250 g/L SE[2]	125	31	400	2	0* 0 18 31	green material	0.14 0.82 0.27 0.16	Bomke, 2012, 11-2110
						48	rest of plant	0.13	
11-2110-05 France, 2011 (PR 33 A46)	250 g/L SE[2]	125	42	300	2	0* 0 14 29	green material	0.15 0.87 0.24 0.14	Bomke, 2012, 11-2110
						64	rest of plant	0.24	

Soya bean forage and hay

Supervised trials in the USA: Residue data on soya beans from 19 trials conducted in the USA were previously evaluated by the 2008 JMPR and 2009 JMPR. In these trials the time between last application and sampling was too short (7 days) and therefore cannot be used to estimate residue levels.

Peanut fodder

Eight supervised trials were conducted on peanut in Australia. Peanut fodder was analysed in four trials at 28 DAT. The residue values are summarised in Table 20. Residues in hay are not reported because the use instructions indicate: 'Do not feed hay or threshings or allow livestock to graze in treated areas.

Table 20 Prothioconazole residues in peanut resulting from supervised trials in Australia

PEANUTS Trial Country, year (Variety)	Application					DAT days	Commodity	JAU 6476 mg/kg	JAU 6476 desthio mg/kg	JAU 6476 total mg/kg	Reference
	Formulation (g ai/L)	g ai/ha	g ai/hL	Water (l/ha)	No						
GAP, AUSTRALIA	480 g/L SC	192			4	28					
C668 Australia 2011-2012 (HOJ-E Holt)	480 g/L SC	193-194	96	201-202	4	15 22 29	Fodder	0.10 0.07 0.30	3.64 6.86 <u>11.60</u>	4.12 7.64 13.09	Ellis, 2012, BCS-0383
C697 Australia 2012 (Holt)	480 g/L SC	192-193	96	199-201	4	14 21 28	Fodder	< 0.05 < 0.05 < 0.05	2.72 3.43 <u>1.15</u>	3.00 3.78 1.27	Ellis, 2012, BCS-0383
C680 Australia 2011-2012 (HOJ-L-2)	480 g/L SC	192-194	26-29	673-754	4	15 21 28	Fodder	< 0.05 < 0.05 < 0.05	0.52 1.57 <u>1.14</u>	0.58 1.73 1.26	Ellis, 2012, BCS-0383
C682 Australia 2011-2012 (WH2 04401)	480 g/L SC	192-193	25-37	517-778	4	14 21 27	Fodder	0.34 0.26 0.06	17.10 22.10 <u>7.00</u>	19.19 24.62 7.78	Ellis, 2012, BCS-0383

FATE OF RESIDUES IN STORAGE AND IN PROCESSING

Information and Data from Residues in Processed Commodities

Processing studies on corn, peanut and oilseed rape are summarized in this section.

Processing Corn into Corn Flour, Starch, Grits, Oil, Aspirated Grain Fractions and Bran

A corn processing study (Krolski & Harbin, 2008, RAJAP001) has been conducted in which field corn was treated with an SC formulation containing 480 g/L prothioconazole at an exaggerated total application rate of 4.0 kg ai/ha, equivalent to 5× the maximum total seasonal rate for prothioconazole on corn. Bulk samples of corn grain were collected at maturity (BBCH 89) at 13 DAT, and frozen prior to analysis/processing. Subsamples of corn grain were removed for analysis, aspirated grain fractions were generated, and the remaining grain was used to generate the required processed commodities of starch, oil (wet and dry milled, refined, bleached, and deodorized), grits, flour, meal and bran. Processing was performed using procedures that simulated commercial processing practices.

Grain samples were dried in an oven until the moisture content was 10–13%. The corn grain was then aspirated and the material that passed through the 2360 micron sieve was recombined to produce an <u>aspirated grain fraction</u>. After generation of aspirated grain fractions, whole corn was cleaned by aspiration and screening. A subsample of cleaned corn grain was dry milled to form <u>large grits and grits</u>, <u>meal</u>, <u>flour</u> and <u>bran</u>.

The germ material was conditioned and flaked and then extracted with hexane to give crude oil and solvent extracted germ flakes. The crude oil was neutralised with 16 degree Baumé sodium hydroxide to give alkali refined oil and soapstock. The soapstock was discarded. The refined oil was bleached using activated bleaching earth and deodorised using 0.5% citric acid solution to give <u>deodorized oil (dry milled)</u> and distillates.

A second subsample of dried cleaned corn grain was wet milled to form hull, germ, starch and gluten fractions. The germ material was conditioned, flaked and pressed to form crude oil and presscake. The presscake was extracted with hexane to form solvent extracted presscake and crude oil. The combined crude oil was refined using 16 degree Baumé sodium hydroxide to give refined oil and soapstock. The soapstock was discarded. The refined oil was bleached and deodorised as described above to give deodorized oil (wet milled) and distillates.

The samples were frozen after collection and stored frozen (< -12 ºC) until extraction and analysis. The maximum period of storage was 429 days (14 months) for grain and 85 days (3 months) for aspirated grain fractions and processed commodities, which falls within the stability period of 3 years as confirmed by previous studies.

Residues of prothioconazole were determined by high performance liquid chromatography – triple stage quadrupole mass spectrometry (LC/MS/MS). The method, RPA JA/03/01, which was used for the analysis was validated with an LOQ of 0.02 mg/kg for prothioconazole-sulfonic acid, 0.02 mg/kg for prothioconazole-desthio and 0.02 mg/kg for total prothioconazole.

Residues of the metabolites 1,2,4-triazole, triazolylalanine and triazolylacetic acid were also determined in this study, but the results are not summarised here as these analytes are not included in the residue definition for monitoring or risk assessment.

Procedural recoveries at fortification levels of 0.02–5 mg/kg were within the acceptable range of 70–120% for prothioconazole-desthio, prothioconazole-sulfonic acid and prothioconazole in each matrix. The prothioconazole recovery was the sum of prothioconazole-desthio and prothioconazole-sulfonic acid from recovery samples fortified with prothioconazole only.

Residues of prothioconazole were almost exclusively on the outside of the corn grain, and were strongly concentrated (90×) in the aspirated grain fraction, and slightly concentrated in the bran (1.3×). Prothioconazole residues do not concentrate in starch, oil, grits, flour or meal (< 1×) during processing of corn grain. The results are summarised in Table 30.

Table 21 Residues of prothioconazole in corn processed fractions

Processed Fractions	Total prothioconazole residue (mean) mg/kg	Processing factor	Reference
Corn grain	0.072		Krolski & Harbin, 2008, RAJAP001
Aspirated grain fractions	6.50	90	
Starch	< 0.02	< 0.28	
Oil (wet milled)	< 0.02	< 0.28	
Grits	< 0.02	< 0.28	
Flour	0.041	0.57	
Meal	0.031	0.43	
Bran	0.094	1.31	
Oil (dry milled)	< 0.02	< 0.28	

Processing Oilseed Rape to Meal and Refined Oil

A field trial to measure the magnitude of prothioconazole residues in canola seed, canola meal and canola refined oil was previously evaluated by the 2008 JMPR and reported in the 2008 JMPR Evaluation (Prothioconazole – Table 81). The 2008 meeting concluded that no concentration (< 0.7×) of the total prothioconazole derived residues was seen in canola meal and refined oil. The processing factors derived from this study on canola are summarized in Table 22.

Table 22 Residues of prothioconazole in canola processed fractions

Processed Fractions	Total prothioconazole residue mg/kg	Processing factor	Reference
Canola seed	0.03		2008 JMPR
Canola meal	< 0.02	< 0.7	
Refined oil	< 0.02	< 0.7	

An additional oilseed rape processing study (Freitag & Hoffmann, 2010, 08-3112) was submitted. The oilseed rape seed samples originate from a trial (08-2112-02) conducted as part of study of Freitag, Reineke & Krusell, 2010, 08-2112.

Seed samples were processed into screw pressed oil, pomace, meal, solvent extracted oil, crude oil, pre-clarified crude oil, neutralised crude oil and refined oil, simulating commercial practices.

After defrosting, rape seed samples were dried at 35 °C for 30 hours to a moisture content of 6–10%. The conditioned seed was cleaned manually using a sieve to remove parts of coarse stalks and weed seeds. The conditioned and cleaned rape seeds were pressed in a screw press yielding screw-pressed oil, and pomace.

An aliquot of the pomace was milled (distance of the rolls: 2.0 mm) and sub-samples extracted using a small technical extraction device. The extraction was conducted in two steps. The first extraction step started with addition of n-hexane to the pomace. The n-hexane circulated through the pomace for about 2 hours. During the circulation, the n-hexane was heated up to approx. 60 °C. Residual solvent was removed from the solvent-oil-mixture (miscella) using a rotary evaporator at approx. 50 °C, yielding solvent extracted oil. The solvent-extracted meal was sampled after storing at room temperature for approximately one day.

The two oil fractions obtained (screw-pressed oil and solvent extracted oil) were mixed yielding the sample of crude oil. The crude oil was filtered, yielding the sample of pre-clarified crude oil. The pre-clarified crude oil was heated up to 60–70 °C while stirring. After addition of 10% water, the mixture of water and crude oil was heated up to 85–90 °C and maintained at 85 °C for approximately 45 minutes while stirring. After phase separation, the watery phase including mucilage was removed. To the oily phase approximately 1% of concentrated phosphorus acid was added at 60 °C while stirring. The mixture was heated up to approximately 85 °C and maintained at that temperature for approximately 45 minutes while stirring. Immediately before switching off the stirrer, 10% water was added. The mixture of water and oily phases remained at approximately 85 °C until separation of the phases, and the watery phase including flocculated precipitation was then removed. After determination of the acid-number, the oily phase was heated up to approximately 90 °C while stirring. A few grams of a 7% sodium hydroxide solution (amount depending on acidity of the oil phase) were added and the mixture was stirred for approximately 20 minutes. After addition of 10% water and further 5 minutes of stirring the stirrer was switched off, the phases were allowed to separate and the watery phase was removed. The process was repeated as the acid-number of the oil was above 0.12. After the second neutralisation process, the acid value was below 0.12 and an aliquot of neutralised crude oil was sampled.

The remaining neutralised crude oil was heated up to 90 °C while stirring and washed by the addition of 10% water. The mixture was stirred for 20 min at 90 °C. After phase separation the watery phase was removed. These steps were repeated until the pH of the washing water was in the range 7-8 and the soap content of the oil was ≤ 0.01%. The oil was heated up to approximately 95 °C while stirring. After addition of citric acid (60 mg/kg oil), the oil was dried under vacuum. The oil was heated up to 95 °C while stirring. After addition of 1% podsol (referring to the used oil) the oil was bleached for 5 min without vacuum and 20 min with vacuum. The podsol was removed by filtration under vacuum. The oil was heated under vacuum up to 240 °C while stirring. After reaching 160 °C, steam was transferred through the oil in order to expel fatty acids, odour and taste influencing compounds as well as other volatile compounds. The mixture stayed at 240°C for about 20 minutes. After cooling to 160 °C, the steam supply was stopped and then the oil was dried under vacuum until a temperature of ≤ 80 °C was reached. The refined oil was sampled.

All processed fractions were stored frozen (-18 °C) prior to analysis. seed was analysed within 645 days of sampling, and all processed fractions were analysed within 349 days of processing, which fall within the stability period of 3 years as confirmed by previous studies. Residues of prothioconazole-desthio were determined by HPLC/MS/MS using method 01013. The LOQ was 0.01 mg/kg in all matrices. Procedural recoveries in seed and oil at fortification levels of 0.01–0.10 mg/kg were within the acceptable range of 70–120%, RSD < 20%, for prothioconazole-desthio in each matrix.

In this study, samples were also analysed for residues of the hydroxyl metabolites (prothioconazole-alpha-hydroxy-desthio, prothioconazole-3-hydroxy-desthio, prothioconazole-4-hydroxy-desthio, prothioconazole-5-hydroxy-desthio and prothioconazole-6-hydroxy-desthio) and for residues of 1,2,4-triazole, triazole acetic acid, triazole lactic acid and triazole alanine. The results are not summarised here as none of these analytes are included in the residue definition for monitoring or risk assessment.

Results show that prothioconazole-desthio residues do not concentrate in meal, screw-pressed oil or refined oil (≤1×), but do concentrate in crude oil and solvent extracted oil (2×) during processing of rape seed. The results are summarised in Table 23.

Table 23 Residues of prothioconazole in oilseed rape processed fractions

Processed Fractions	Prothioconazole-desthio residue mg/kg	Processing factor	Reference
Oilseed rape seed	0.01		Freitag & Hoffmann, 2010, 08-3112
Screwpressed oil	0.01	1	
Pomace	0.01	1	
Meal	< 0.01	<1	
Solvent extracted oil	0.02	2	
Crude oil	0.02	2	
Preclarified crude oil	0.02	2	
Neutralised crude oil	0.01	1	
Refined oil	< 0.01	<1	

Based on the available processing studies, the processing factors that have been calculated are summarized in Table 24.

Table 24 Summary of processing factors

Commodity	Processed fraction	Processing factor	Reference
Peanut	Peanut meal	1.8	2008 JMPR
	Refined oil	< 0.1	
	Roasted peanuts	0.5	
	Peanut butter	0.6	
Wheat	Aspirated grain fraction	250	2008 JMPR
	Bran	2.4	
	Flour	< 0.4	
	Middlings	0.6	

Commodity	Processed fraction	Processing factor	Reference
	Shorts	1	
	Wheat germ	2	
Soya bean	Aspirated grain fraction	75	2008 JMPR
	Meal	0.2	
	Hull	0.5	
	Refined oil	< 0.2	
Oilseed rape	Meal	<1	Freitag & Hoffmann, 2010, 08-3112
	Refined oil	<1	
Corn	Aspirated grain fractions	90	Krolski & Harbin, 2008, RAJAP001
	Starch	<1	
	Oil (wet milled)	<1	
	Grits	<1	
	Flour	<1	
	Meal	<1	
	Bran	1.3	
	Oil (dry milled)	<1	

RESIDUE ON ANIMAL PRODUCTS

Residues in Animal Commodities

In addition to those commodities which had been evaluated by the previous Meetings, the following commodities could result in residues in animal tissues, milk, and eggs: sweet corn stover, field corn stover, popcorn stover, soya bean hay and peanut fodder.

APPRAISAL

Prothioconazole was evaluated first time by the 2008 JMPR. The residue definition for plant commodities for enforcement and dietary risk assessment was prothioconazole-desthio. The residue definition for animal commodities for enforcement was prothioconazole-desthio and for dietary risk assessment was the sum of prothioconazole-desthio, prothioconazole-desthio-3-hydroxy, prothioconazole-desthio-4-hydroxy and their conjugates, expressed as prothioconazole-desthio. The residue was considered to be not fat-soluble for the purposes of residue definition.

The 2008 JMPR established, for prothioconazole-desthio, an ADI of 0–0.01 mg/kg bw and ARfD of 0.01 mg/kg bw for women of child bearing age and 1 mg/kg bw for the general population.

The Forty-fifth Session of CCPR scheduled prothioconazole for periodic re-evaluation of residues by the 2014 JMPR. For the 2014 Meeting data were provided on blueberry, corn, cranberry, cucurbits, potato, sweet corn peanut and soya bean together with current use recommendations and analytical methods used in supervised trials.

Methods of analysis

A number of validated analytical methods for the determination of residues in plant, animal tissue, milk and soils were evaluated by the 2007 and 2008 JMPR Meetings. The Meeting received the basic analytical methods with minor modifications which were validated to determine residues in crops that had been used in the residue trials. An analytical method was also provided for the determination of residues of prothioconazole in animal matrices.

The trials carried out in the USA/Canada used method RPA JA/03/01 or the modified method JA-001-P04-02 which involve extraction with methanol, hydrogen peroxide and sodium bicarbonate at an elevated temperature. This converts prothioconazole to a mixture of prothioconazole-sulfonic acid, while prothioconazole-desthio is extracted unchanged. After purification steps, the residues are analysed by LC-MS/MS. The LOQ of these methods is 0.02 mg/kg for prothioconazole-sulfonic acid and 0.02 mg/kg for prothioconazole-desthio.

The trials carried out in Brazil, EU and Australia used methods 01013, 00598/M001 and ATM-0053 in which residues of prothioconazole and prothioconazole-desthio are extracted with acetonitrile/water containing cysteine hydrochloride. After purification steps, dependent on the method, the analytes are analysed by LC-MS/MS. The LOQ of these methods is 0.01 mg/kg (or 0.05 mg/kg for forage and straw matrices) for prothioconazole and prothioconazole-desthio.

Storage Stability under Frozen Conditions

Information relating to storage stability of residues in wheat, canola (seed, pod, straw), spinach (leaves), sugar beet (root, leaf with root collar), tomato (fruit) and field pea (dried) was evaluated at the 2008 JMPR Meeting. This showed that residues of prothioconazole-desthio were stable over a frozen storage period of up to 36 months in wheat and at least 24 months for the other crops. Residues of prothioconazole and prothioconazole-desthio were stable in wheat hay and straw, canola seeds, mustard greens, turnip root and tomato fruit over a frozen storage period of 36–42 months.

An additional freezer storage stability study for residues of prothioconazole and prothioconazole-desthio (JAU 6476-desthio) in wheat (forage, straw, grain, bran and flour), canola (seed and oil), mustard greens, tomato (fruit and paste) and turnip roots was performed.

No significant degradation of prothioconazole-desthio was observed in any matrix analysed over the 3-year frozen storage period.

The stability of parent prothioconazole varied from 57 days up to 378 days without showing any dependency on the sample matrix. However, metabolism studies indicated that after foliar application prothioconazole rapidly degraded to prothioconazole-desthio and polar metabolites being converted to the sulfonic acid derivative, which is measured by the analytical method. Therefore the relative instability of parent compound does not affect the validity of the desthio residues measured in supervised trials.

Results of supervised residue trials on crops

Supervised trials have been conducted to support MRLs for the following crops or groups of crops: blueberry, cranberry, cucurbits (cucumber, summer squash and melon), sweet corn, soya bean, potato, maize (field corn, popcorn) and peanuts.

In trials conducted in the USA, Canada and Brazil, residues of prothioconazole sulfonic acid (JAU 6476 sulfonic acid), prothioconazole-desthio (JAU 6476 desthio) and total prothioconazole (JAU 6476) were determined and reported. In trials conducted in Australia, residues of prothioconazole (JAU 6476), prothioconazole-desthio (JAU 6476 desthio) and total prothioconazole (JAU 6476) were determined and reported. However, only the residues of prothioconazole-desthio have been used for calculation of MRL, HR and STMR values in accordance with the residue definition. The residues of prothioconazole-desthio are expressed as mg prothioconazole-desthio equivalents/kg.

Bush berries

Blueberry

Eleven supervised trials were conducted in 2010 in the USA and Canada matching the US GAP (2 × 200 g ai/ha nominal rate at 6–7 days intervals, 7-day PHI) for the bush berry subgroup.

Prothioconazole-desthio residues from trials on blueberries were: 0.15, 0.22, 0.26, 0.28, 0.42, 0.52, 0.56, 0.60, 0.65, 0.70 and 0.87 mg/kg.

Taking into account that blueberry is the representative commodity for the subgroup; the Meeting estimated a maximum residue level of 1.5 mg/kg, a HR of 0.87 mg/kg and a STMR of 0.52 mg/kg for prothioconazole-desthio residues for the bush berry subgroup.

Low growing berries

Cranberry

Six supervised trials were conducted in 2010 in the USA according to the US GAP (2 × 186 g ai/ha nominal rate at 7-10 days intervals, 45-day PHI)

Prothioconazole-desthio residues from trials on cranberries were: < 0.02 (3), 0.03 (2) and 0.09 mg/kg.

The Meeting estimated maximum residue level of 0.15 mg/kg, HR of 0.09 mg/kg and STMR of 0.025 mg/kg for prothioconazole-desthio residues for cranberry.

Fruiting vegetables, Cucurbits

Eight supervised trials were conducted in 2010 on *cucumber*, eight trials on *musk melon* and eight trials on *summer squash* in the USA according to US GAP (one soil +2 × 200 g ai/kg nominal rate at 7-10 days intervals, max 600 g ai/season,, 7-day PHI).

Prothioconazole-desthio residues from trials on cucumber were: 0.02, 0.02, 0.03, 0.03, 0.04, 0.05, 0.05, 0.06 mg/kg.

Prothioconazole-desthio residue concentrations from trials on musk melon were: 0.03, 0.06, 0.06, 0.06, 0.06, 0.07, 0.15, 0.15 mg/kg.

Prothioconazole-desthio residue concentrations from trials on summer squash were: < 0.02 (5), 0.03, 0.05 and 0.06 mg/kg.

The Meeting noted that the GAP in USA is for cucurbit vegetables, the residue populations were not significantly different and the median residues were within the 5 times range, and agreed to combine the datasets. Residues in the 24 trials were: < 0.02 (5), 0.02 (2), 0.03 (4), 0.04, 0.05 (3), 0.06 (6), 0.07 and 0.15 (2) mg/kg.

The Meeting noted that the ARfD would be exceeded by 150% for watermelon. As no alternative GAP for fruiting vegetables was available, watermelon was not included in the recommendation for the crop group fruiting vegetables, cucurbits.

The Meeting estimated a maximum residue level of 0.2 mg/kg, HR of 0.15 mg/kg and STMR of 0.045 mg/kg prothioconazole-desthio residues for fruiting vegetables, cucurbits (except watermelon).

Fruiting vegetables, Other Than Cucurbits

Sweet corn

Twelve supervised trials were conducted on sweet corn in the USA at 4 × 200 g ai/ha matching the current US GAP. The PHI for sweet corn forage and ears is 0 days and 14 days for fodder.

Prothioconazole-desthio residue concentrations in corn ears were: < 0.018 in all samples.

The Meeting estimated a maximum residue level of 0.02 mg/kg, and HR and STMR values of 0.018 mg/kg for sweet corn.

Pulses

Soya beans

Residue data on soya beans from 19 trials conducted in the USA were previously evaluated by the 2008 and 2009 JMPR Meetings. The current US GAP for soya beans permit 3 applications at 88–175 g ai/ha rate at 10–21 day intervals with a PHI of 21 days.

The average prothioconazole-desthio residues in soya bean samples derived from trials corresponding to the current US GAP were: < 0.05 (16), 0.051, 0.055 and 0.105 mg/kg

Ten supervised trials were conducted in 2012–2013 season on soya bean in Brazil matching the GAP for Brazil (4 × 87.5 g ai/ha, PHI 30 days). The prothioconazole-desthio residues were reported as the parent compound. Applying the conversion factor of 0.907 (312.20/344.26) the residues expressed as prothioconazole-desthio were: < 0.009 × 4, 0.009 × 2, 0.018, 0.027, 0.036 and 0.091 mg/kg.

The Meeting noted that the US trials resulted in higher residues and used those values. The meeting estimated a maximum residue level of 0.2 mg/kg and STMR value of 0.05 mg/kg.

Root and tuber vegetables

Potato

A total of twenty supervised trials were conducted in 2005 and 2010 as potato seed-piece treatments in Belgium, France, Germany, Italy, the Netherlands, Spain and the UK at maximum GAP (up to 0.64 g ai/100 kg seed, equivalent to 16 g ai/ha) or higher rate. The potatoes were harvested at maturity. The prothioconazole-desthio residues were < 0.01 mg/kg in all samples take 90–136 days after seed treatment.

Taking into account that the LOQ of the enforcement method is 0.02 mg/kg, the Meeting estimated a maximum residue level of 0.02* mg/kg, HR and STMR values of 0.01 mg/kg for potato.

Cereal grains

Maize

Twenty supervised trials were conducted in 2006 on field corn and three trials on popcorn in the USA (4 × 200 g ai/ha at 7–14 days apart, 0 day PHI for forage and 14 days for grain and fodder).

The prothioconazole-desthio residues in maze grain samples collected at 14 days after last application were: < 0.018 (17), and 0.05 mg/kg,

Ten supervised trials were conducted in 2011-2012 on corn in Brazil according to the GAP (2 × 70-87.5 g ai/ha, at minimum 15 days, 15 days PHI).

The prothioconazole-desthio residues in maize grain samples collected at 14-15 days after last application were: < 0.01 (10) mg/kg.

A total of twenty supervised trials were conducted in 2007, 2008 and 2011 on maize/corn in Europe matching the GAP of the Czech Republic (2 × 125 g ai/ha, PHI 35 days) and Italy (2 × 125 g ai/ha, retreatment at a minimum of 14 days, 35 days or non-specified PHI).

The prothioconazole-desthio residues in all of immature and mature maize grain samples collected from 17 days onward were: < 0.01 (20) mg/kg.

The Meeting considered that all maize grain/seed samples (47) contained non-detectable resides regardless of maturity and time between last application and sampling, except in one trial where residues of up to 0.05 mg/kg residue was measured.

The Meeting estimated a maximum residue level of 0.1 mg/kg and STMR value of 0.018 mg/kg for maize and popcorn.

Oilseeds

Peanuts

Residue data on peanuts from trials conducted in the USA had been evaluated by the 2009 JMPR. Residues of prothioconazole-desthio were < 0.02 mg/kg in 12 trials matching the USA GAP (4 × 200 g ai/ha, PHI 14 days).

Eight supervised trials were conducted in 2011–2012 in Australia. Four trials included plots which were treated according to the GAP for peanut in Australia (4 times 120–192 g ai/ha at 10–14 days apart, 28-day PHI), and four trials included plots which were treated with higher number of applications and a shorter PHI (up to 21 days). Samples were taken immediately before the last application and 0, 7, 14–15 and 20–21 days after.

The prothioconazole-desthio residues in all samples collected from Day 0 to day 29 days were: < 0.01 mg/kg.

The Meeting confirmed its previous recommendations of 0.01* mg/kg.

Primary feed commodities

Descriptions of trial conditions and residues are described under relevant food commodities. The residue data relevant to the feed commodities are summarized below.

Maize and sweet corn forage and fodder

Prothioconazole-desthio residue concentrations in sweet corn forage from trials conducted at 4 × 200 g ai/ha were: 1.31, 1.50, 1.89, 1.94, 1.98, 2.08, 2.12, 2.37, 2.82, 3.20, 3.75 and 4.08 mg/kg.

Prothioconazole-desthio residue concentrations in field corn forage from trials based on the USA GAP for corn are: 0.71, 1.67, 1.75, 1.84, 1.90, 1.96, 2.14, 2.15, 2.19, 2.20, 2.27, 2.41, 2.58, 2.62, 2.65, 2.99 (2), 3.06, 3.44 and 3.60 mg/kg.

The Meeting noted that the GAP in USA for sweet corn and maize is the same and that the residue populations were not significantly different, and agreed to combine the datasets.

Residues in the trials were: 0.71, 1.31, 1.50, 1.67, 1.75, 1.84, 1.89, 1.90, 1.94, 1.96, 1.98, 2.08, 2.12, 2.14, 2.15, 2.19, 2.20, 2.27, 2.37, 2.41, 2.58, 2.62, 2.65, 2.82, 2.99 (2), 3.06, 3.44, 3.60, 3.75 and 4.08 mg/kg.

Residues in forage from the European trials were much lower (< 0.2 mg/kg) than from the trials conducted according to the USA GAP, and therefore the Meeting used the US trial data for the estimation of residue levels.

The Meeting estimated highest residue of 4.08 mg/kg and median residue of 2.15 mg/kg for sweet corn and filed maize forage.

Prothioconazole-desthio residue concentrations in sweet corn stover from trials conducted at 4 × 200 g ai/ha were: 0.67 (2), 0.71, 0.82, 0.90, 0.98, 1.45, 1.68 (2), 1.70, 3.61 and 5.88 mg/kg.

Prothioconazole-desthio residue concentrations in field corn stover from trials based on the USA GAP for corn were: 0.67 (2), 0.71, 0.82, 0.90, 0.98, 0.99, 1.09, 1.45, 1.68 (2), 1.70, 2.22, 2.32, 2.35, 2.42, 2.90, 3.18, 3.22, 3.42, 3.48, 3.59, 3.61, 3.68, 3.78, 3.89, 4.05, 4.47, 4.66, 5.88, 6.16 and 6.70 mg/kg.

Prothioconazole-desthio residue concentrations in popcorn stover from trials based on the USA GAP for corn were: 2.46, 2.51 and 4.43 mg/kg.

The Meeting noted that the GAP in the USA is for sweet corn and maize is the same and the residue populations were not significantly different, and agreed to combine the datasets. Residues in the trials were: 0.99, 1.09, 2.22, 2.32, 2.35, 2.42, 2.46, 2.51, 2.90, 3.18, 3.22, 3.42, 3.48, 3.59, 3.68, 3.78, 3.89, 4.05, 4.43, 4.47, 4.66, 6.16 and 6.70 mg/kg.

The Meeting estimated a maximum residue level of 15 mg/kg (dry weight basis) highest residue of 6.7 mg/kg and median residue of 3.48 mg/kg for sweet corn and field maize stover.

Soya bean forage and hay

The Meeting received information on residues in soya bean forage and hay. However, the samples were taken at 7 days PHI, which did not correspond to the current US GAP and therefore could not be used for the estimation of residue levels.

Peanut fodder

Prothioconazole-desthio residue concentrations in peanut hay from trials based on the Australian GAP for peanuts were: 1.14, 1.15, 7.00 and 11.60 mg/kg.

The Meeting estimated highest and median residues of 11.6 mg/kg and 4.08 mg/kg, respectively, for peanut hay.

Fate of residue during processing

Processing Corn

Field corn was treated at 5× total seasonal rate. Subsamples of corn grain were removed for analysis, aspirated grain fractions were generated, and the remaining grain was used to generate the required processed commodities of starch, oil (wet and dry milled, refined, bleached, and deodorized), grits, flour, meal and bran. Processing was performed using procedures that simulated commercial practices.

The processing factors calculated from total prothioconazole residues were: starch (< 0.28), oil, wet milled (< 0.28, grits (< 0.28), flour (0.57), meal (0.43), bran (1.31), oil, dry milled (< 0.28).

The Meeting noted that the major part of the residue is prothioconazole-desthio and concluded that the results of processing study can be used for estimation of the processing factors for prothioconazole-desthio. The Meeting estimated STMR values (mg/kg) for maize starch (0.0050), maize flour (0.010), and refined maize oil (0.0050).

Processing rape seed

Rape seed was sampled following treatment with prothioconazole, 2 × 120 g/ha. The conditioned and cleaned rape seeds were pressed in a screw press, yielding screw pressed oil, and pomace. An aliquot of pomace was extracted twice with hexane. The screw pressed oil and extracted oil were combined to obtain crude oil, which was neutralized and processed to obtain refined oil.

The processing factors calculated were: screw pressed oil (1), pomace (1), meal (< 1), solvent extracted oil (2), crude oil (2), pre-clarified crude oil (2), neutralized crude oil (1) and refined oil (< 1)

The Meeting estimated STMR-P values for rape seed oil, edible of 0.02 mg/kg based on the STMR of 0.02 estimated by the 2009 JMPR

Residues in Animal Commodities

The following commodities evaluated by the present and previous Meetings could result in residues in animal tissues, milk, and eggs: sweet corn forage and stover, field corn forage and stover, popcorn forage and stover, soya bean hay and peanut fodder (dry), grains, straw and fodder (dry) of cereal grains.

Livestock dietary burden

The maximum and mean dietary burdens were calculated using the highest residues or median residues of prothioconazole-desthio estimated by the current Meeting on a basis of the OECD Animal Feeding Table. The calculated maximum and mean animal burdens are summarized below

Summary of livestock dietary burdens (ppm of dry matter diet)

	US-Canada		EU		Australia		Japan	
	Max	Mean	Max	Mean	Max	Mean	Max	Mean
Beef cattle	4.04	3.32	12.36	5.47	21.6[a]	5.50[c]	3.15	3.15
Dairy cattle	10.63	4.85	10.89	4.94	18.42[b]	5.50[d]	7.66	5.25
Broilers	2.86	2.86	1.17	1.17	1.18	1.18	0.29	0.29
Layers	2.86	2.86[f]	3.33[e]	1.71	1.18	1.18	1.72	1.72

[a] Highest maximum beef or dairy cattle dietary burden suitable for MRL estimates for mammalian meat
[b] Highest maximum dairy cattle dietary burden suitable for MRL estimates for mammalian milk
[c] Highest mean beef or dairy cattle dietary burden suitable for STMR estimates for mammalian meat.
[d] Highest mean dairy cattle dietary burden suitable for STMR estimates for milk.
[e] Highest maximum poultry dietary burden suitable for MRL estimates for poultry meat and eggs.
[f] Highest mean poultry dietary burden suitable for STMR estimates for poultry meat and eggs.

Animal commodity maximum residue levels

The 2009 JMPR summarized the total prothioconazole desthio residues (mg/kg) in edible tissues of dairy cattle after 28 days of dosing with prothioconazole-desthio:

Tissue	4 ppm dose		25 ppm dose		100 ppm dose	
	Range	Mean	Range	Mean	Range	Mean
Liver	0.02-0.05	0.04	0.18-0.26	0.22	0.61-1.6	0.95
Kidney	0.01-0.04	0.02	0.11-0.17	0.14	0.41-1.1	0.65
Muscle	< 0.01	< 0.01	< 0.01	< 0.01	0.01-0.03	0.02
Fat	< 0.01	< 0.01	0.01-0.02	0.01	0.03-0.14	0.07
Milk	< 0.004	< 0.004	< 0.004	< 0.004	0.013-0.02	0.017

The maximum dietary burden for beef cattle remained the same as was estimated by the 2009 JMPR. The maximum dietary burden for dairy cattle is 18.42 ppm which is smaller than the 25 ppm dose and no detectable residue can be expected in milk.

The mean dietary burden for beef and dairy cattle is 5.5 ppm, which is higher than the 4.8 ppm estimated by the 2009 JMPR. However the STMR values estimated by the 2009 JMPR cover the likely median residues.

The Meeting confirmed its previous recommendations for maximum residue levels, HR and median residues for mammalian meat, and edible offal and milk.

The 2008 Meeting concluded that the feeding study on poultry did not reflect the residue composition in feed and could not be used for estimation of residue levels.

RECOMMENDATIONS

On the basis of the data from supervised trials and farm animal feeding studies reported by the 2006 JMPR, the Meeting concluded that the residue levels listed below are appropriate for establishing maximum residue limits and for IEDI assessment.

Definition of the residue (for enforcement and dietary risk assessment) for plant commodities: *prothioconazole-desthio*.

Definition of the residue (for enforcement and dietary risk assessment) for animal commodities: *the sum of prothioconazole-desthio. (M04), prothioconazole-desthio-3-hydroxy (M14), prothioconazole-desthio-4-hydroxy (M15), expressed as prothioconazole-desthio after correction for molecular weight.*

Commodity		MRL mg/kg	STMR mg/kg	HR mg/kg
CCN	Name			
FB 2006	Bush berries	1.5	0.52	0.87
FB0265	Cranberry	0.15	0.025	0.9
VC 0046	Fruiting vegetables, Cucurbits, except watermelon	0.2	0.045	0.15
VO 0447	Sweet corn	0.02	0.018	0.018
VD 0541	Soya bean	0.2	0.05	
GC 0656	Popcorn	0.1	0.018	
VR 0589	Potato	0.02(*)	0.01	0.01
GC 0645	Maize	0.1	0.01	
AF 0645	Maize forage		2.15	4.08
	Sweet corn forage		2.15	4.08
AS 0645	Maize fodder	15	3.48	6.7
	Sweet corn fodder	15	3.48	6.7
AL 0697	Peanut fodder	15	4.08	11.6
CF 1255	Maize flour		0.0057	
OR 0645	Maize oil		0.0028	
	Maize starch		0.0028	
OR 0495	Rape oil refined		0.02	

DIETARY RISK ASSESSMENT

Long-term intake

The International Estimated Daily Intake (IEDI) for prothioconazole-desthio was calculated from the recommendations for STMR-s for raw agricultural commodities in combination with consumption data for corresponding food commodities. The results are shown in Annex 3 to the 2014 Report.

The IEDI of the 17 GEMS/Food cluster diets were in the range of 0–3 % of the maximum ADI of 0.01 mg/kg bw. The Meeting concluded that the long-term intake of residues from uses of prothioconazole considered by the Meeting is unlikely to present a public health concern.

Short-term intake

The International Estimated Short-term Intake (IESTI) for prothioconazole-desthio was calculated from the recommendations for HR and STMR-s for raw agricultural commodities in combination with consumption data for corresponding food commodities. The results are shown in Annex 4 to the 2014 Report.

The IESTI for women of child bearing age is 0–100% of the ARfD of 0.01 mg/kg bw. The IESTI for the rest of the population is 0–1 % of the ARfD of 1 mg/kg bw.

For watermelon the IESTI represented 150% of the ARfD of 0.01 mg-kg bw. No alternative GAP was available. On the basis of information provided to the JMPR it was not possible to conclude that the estimate of short-term intake of prothioconazole, from the consumption of watermelon was less than the ARfD. The Meeting did not estimate MRL for watermelon.

The other commodities considered by the JMPR were within 0–100% of ARfD for women of child bearing age and 1% of general population. The Meeting concluded that the short-term intake of prothioconazole when used in ways that have been considered by the MPR is unlikely to present public health concern.

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