SPIROMESIFEN (294)

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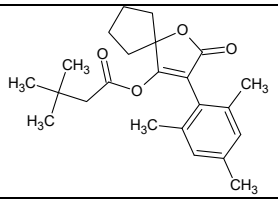
EXPLANATION

Spiromesifen (ISO common name) is a contact insecticide-acaricide belonging to the titronic acid class of compounds. The pesticidal mode of action is inhibition of lipid biosynthesis, especially triglycerides and free fatty acids. It is registered in multiple countries for control of white flies and mites. Spiromesifen was considered for the first time for toxicology and residues by the 2016 JMPR.

Note: Throughout this document, values are listed to the precision provided in the submitted reports, except for values calculated by the JMPR (2 significant figures). All rounding was in accordance with ISO standards.

IDENTITY

ISO common name	Spiromesifen
Chemical Name	
IUPAC	3-mesityl-2-oxo-1-oxaspiro[4.4]non-3-en-4-yl 3,3-dimethylbutyrate
CAS	2-oxo-3-(2,4,6-trimethylphenyl)-1-oxaspiro[4.4]non-3-en-4-yl 3,3-dimethylbutanoate
CIPAC No.	747
CAS No.	283594-90-1
Structural Formula	
Molecular formula	C23H30O4
Molecular mass	370.5

Physical and chemical properties

Physical and chemical properties of spiromesifen:

Property	Guideline and method	Test material specification and purity	Findings	Reference/ Remarks
Technical Grade Active Ingredient				
No Data Submitted				
Pure Active Ingredient				
Melting, freezing or solidification point	OECD 102 meltmicro-scope	Spiromesifen PAI Lot M00391 99.5%	Mean melting point 98.7 ± 0.12 °C (96.7 ± 0.15 °C after recrystallization)	Study ID 14 120 0949
Boiling point	EC 92/69/EWG A2 Photo cell detection	Spiromesifen PAI Lot M00391 99.5%	> 350 °C; not determinable due to decomposition	Study ID 14 120 0949
Relative density of purified active substance	OECD 109, EC A3	Spiromesifen PAI Lot M00391 99.5%	1.13 g/cm³ (20 °C)	Study ID 14 120 0949
Vapour pressure of	OECD 104, EC A4	Spiromesifen PAI Lot M00391	7×10^{-6} Pa (20 °C) 1×10^{-5} Pa (25 °C)	Study ID 14 120 0949

Property	Guideline and method	Test material specification and purity	Findings	Reference/ Remarks
purified active substance		99.5%		
Physical state and colour	Visual	Spiromesifen PAI Lot M00391 99.5%	Colourless crystal	Study ID 14 120 0949
Dissociation Constant	None	Spiromesifen PAI Lot M00391 99.5%	No acidic or basic properties in water of pH values between 4 and 9. Dissociation is not expected to occur.	Study ID 14 120 0949
Solubility in organic solvents	Flask method	Spiromesifen PAI Lot M00391 99.5%	n-Heptane 23 g/L Xylene > 250 g/L Dichloromethane > 250 g/L 2-Propanol 110 g/L 1-Octanol 60 g/L Polyethylenglycol 400 22 g/L Acetone > 250 g/L Ethylacetate > 250 g/L Acetonitrile > 250 g/L Dimethylsulfoxide 55 g/L all measurements at 20 °C	Study ID 14 120 0949
Solubility in water	OECD 105, EC A 6, column method	Spiromesifen PAI Lot M00391 99.5%	0.13 mg/L (20 °C)	Study ID 14 120 0949
n-octanol/ water partition coefficient	OECD 117 column method	Spiromesifen PAI Lot M00391 99.5%	Log P_{ow} = 4.55 (ambient temp.)	Study ID 14 120 0949
Radiolabel Purified Active Ingredient				
Direct phototrans-formation of purified active substance in water	EPA 161-2 ECC 95/36/EC	[dihydrofuranone-3-^{14}C] Spiromesifen Batch vial C-798A 99.2%	$T_{1/2}$ = 1.7 days (pH 4, 25 °C) Degradation products at end of study M01, 12.3% appl. radioactivity (AR) M16, 35.8% AR M17, 36.6% AR All others < 10% AR	Study ID 110962-1
Hydrolysis at pH 4, 7, and 9	EPA 161-1, SETAC	[dihydrofuranone-3-^{14}C] Spiromesifen Batch vial C-798A 99.2%	Hydrolysis half lives (d = days, h = hrs) Temp., °C pH 4 pH 7 pH 9 20 (calc'ed.) 107 d 45 d 4.8 d 25 53 d 4.3 d 50 2.2 d 2.6 h Degradation product M01 was the predominant hydrolysis product.	Study ID 109481-1

Spiromesifen is registered as a suspension concentrate (SC) formulation containing 240 g ai/L and as a wettable powder (WP) formulation containing 30% ai (by weight).

METABOLISM AND ENVIRONMENTAL FATE

Metabolism and environmental fate studies in target crops, rotational crops, goats, hens, rats, soils, and water were conducted with spiromesifen labelled in the 3-dihydrofuranone moiety (Figure 1). Studies were also conducted with spiromesifen labelled in the cyclopentyl ring (soil aerobic degradation and aqueous photolysis) or in the trimethyl phenyl ring (soil aerobic degradation).

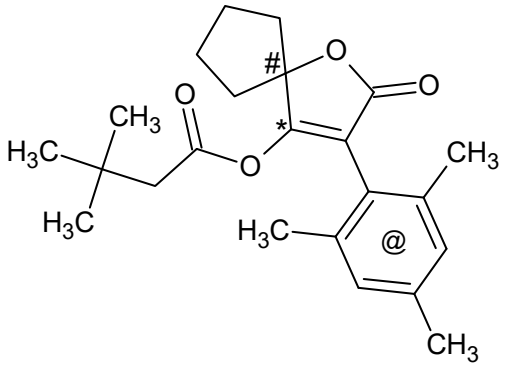
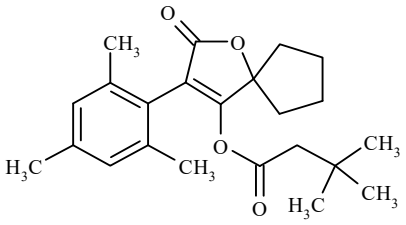
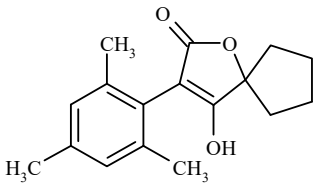
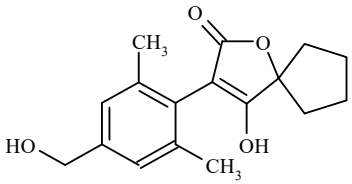


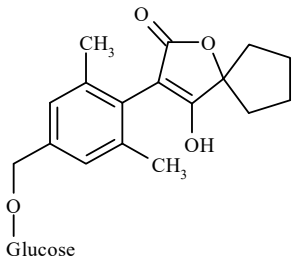
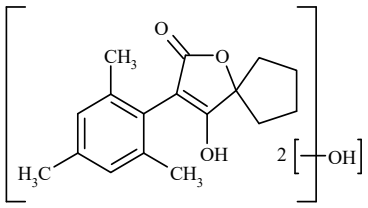
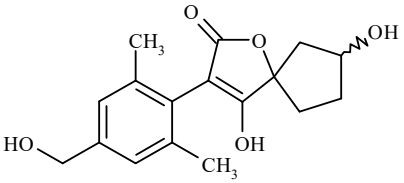
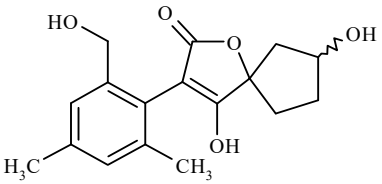
Figure 1 [^{14}C]Spiromesifen * = 3-dihydrofuranone, # = 1-cyclopentyl, @ = UL-trimethylphenyl

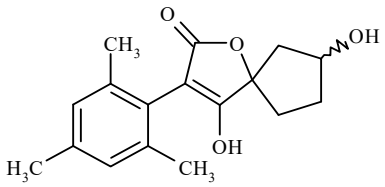
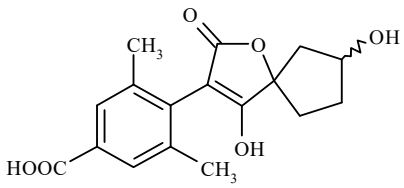
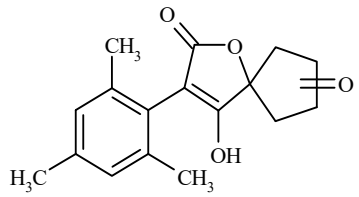
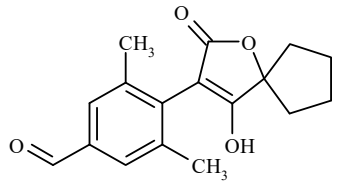
Chemical names, structures, and code names of metabolites and degradation products of spiromesifen are shown below. All the compounds were identified in at least one matrix in studies with radiolabelled spiromesifen.

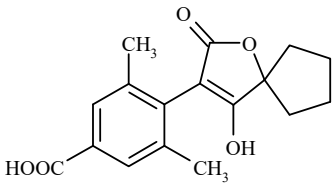
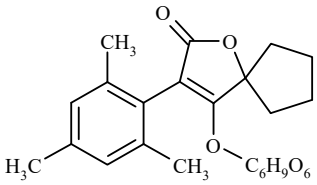
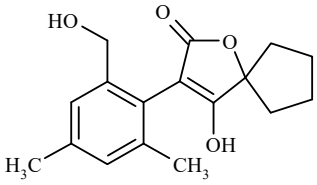
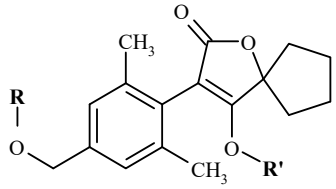
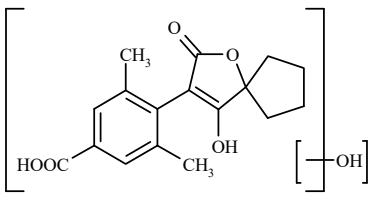
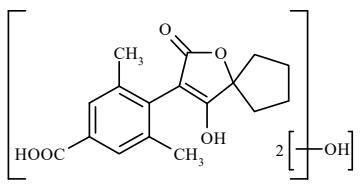
Known metabolites and degradation products of spiromesifen:

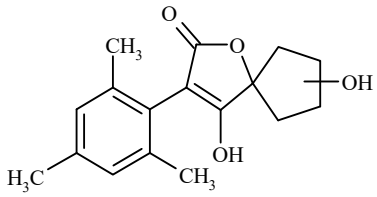
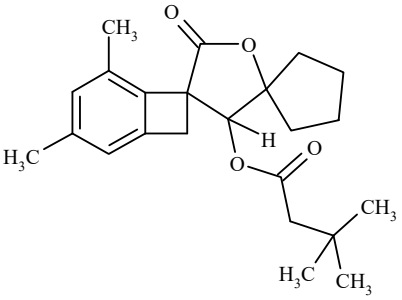
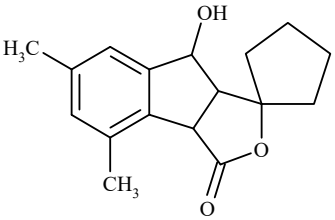
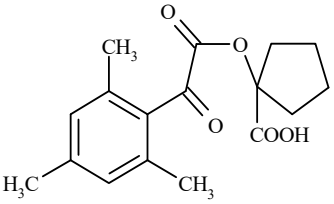
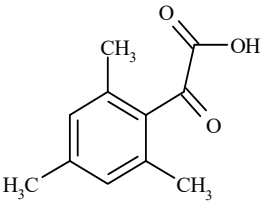
No.	Formula Names (Name used in the summary in bold)	Cited by	Codes used	Recovered in [metabolite postulated in the pathway]
Parent	 1: Spiromesifen 2: Butanoic acid, 3,3-dimethyl-, 2 oxo-3-(2,4,6-trimethylphenyl)-1-oxaspiro[4.4]non-3-en-4-yl ester 3: BSN 2060	all reports		tomato cotton lettuce rat goat hen rotational crops soil soil photolysis column leaching aged column leaching hydrolysis aqueous photolysis water/sediment aerobic

No.	Formula Names (Name used in the summary in bold)	Cited by	Codes used	Recovered in [metabolite postulated in the pathway]
M01	 1: Sp-enol 2: BSN 2060-enol	Haynes, 2001a	TF5 BSN 2060-enol	tomato
		Langford-Pollard, 2000	CS7 CG7 BSN 2060-enol	cotton
		Haynes, 2001b	L8 BSN 2060-enol	lettuce
		Shaw, 2000	RU18 BSN 2060-enol	rat
		Dean, 2001	BSN 2060-enol	goat
		Corden, 2001	BSN 2060-enol HL12	hen
		Aikens, 2002	BSN 2060-enol	rotational crops soil
		Brumhard & Elke, 2001	Enol	soil
		Ripperger & Hall, 2001	Enol	soil
		Babczinski, 2001a	Enol	soil
		Shephard *et al.* 2001	BSN 2060-enol	column leaching
		Babczinski, 2001b	Enol	aged column leaching
		Malekani & Ripperger, 2000	Enol	hydrolysis aqueous
		Arthur *et al.*, 2001a	BSN 2060-enol	photolysis
		Arthur & Shephard, 2001	BSN 2060-enol	water/sediment aerobic
		Desmarteau *et al.*, 2001a and 2001b	BSN 2060-enol	water/sediment anaerobic
		Arthur *et al.*, 2001b	BSN 2060-enol	
M02	 4-hydroxymethyl-Sp-enol	Haynes, 2001a	BSN 2060-4-hydroxymethyl TF4	tomato
		Langford-Pollard, 2001	BSN 2060-4-hydroxymethyl CG4b	cotton
		Haynes, 2001b	BSN 2060-4-hydroxymethyl L4F L5E	lettuce
		Shaw, 2000	BSN 2060-4-hydroxymethyl RU14	rat
		Dean, 2001	BSN 2060-4-hydroxymethyl	goat
		Corden, 2001	BSN 2060-4-hydroxymethyl HL8	hen
		Aikens, 2002	BSN 2060-4-hydroxymethyl	rotational crops

No.	Formula Names (Name used in the summary in bold)	Cited by	Codes used	Recovered in [metabolite postulated in the pathway]
M03	 4-hydroxymethyl-glucoside-Sp-enol	Haynes, 2001a Langford-Pollard, 2001 Haynes, 2001b Aikens, 2002	BSN 2060-4-hydroxymethyl-glucoside TF3 BSN 2060-4-hydroxymethyl-glucoside CG3b BSN 2060-4-hydroxymethyl-glucoside L4D BSN 2060-4-hydroxymethyl-glucoside	tomato cotton lettuce rotational crops
M04	 Dihydroxy-Sp-enol	Langford-Pollard, 2001 Haynes, 2001b Aikens, 2002	BSN 2060-dihydroxy-enol CG2d BSN 2060-dihydroxy-enol L3C BSN 2060-dihydroxy-enol	cotton lettuce rotational crops
M05	  1: cis- or trans-4-hydroxymethyl-3-pentanol-Sp-enol 2: cis- or trans-4-hydroxymethyl-3-hydroxy-Sp-enol 3: cis- or trans-4- or 2-hydroxy-methyl-3-hydroxy-Sp-enol	Langford-Pollard, 2001 Shaw, 2000 Dean, 2001 Corden, 2001	cis- or trans-BSN 2060-4-hydroxymethyl-3-pentanol CG3a cis- or trans-BSN 2060-4- or –2-hydroxymethyl-3-pentanol RU1 BSN 2060-hydroxy-3-pentanol BSN 2060-hydroxymethyl-3-pentanol cis- or trans-BSN 2060-4- or –2-hydroxymethyl-3-pentanol BSN 2060-4- or –2-hydroxymethyl-3-pentanol HL3	cotton rat goat hen

No.	Formula Names (Name used in the summary in bold)	Cited by	Codes used	Recovered in [metabolite postulated in the pathway]
M06	 1: 3-pentanol-Sp-enol 2: cis- or trans-3-hydroxy-Sp-enol	Langford-Pollard, 2001	cis- or trans- BSN 2060-3-pentanol CG4a	cotton
		Haynes, 2001b	cis- or trans- BSN 2060-3-pentanol L5D	lettuce
		Shaw, 2000	cis- or trans- BSN 2060-3-pentanol RU13 and RU17	rat
		Dean, 2001	cis- or trans- BSN 2060-3-pentanol	goat
		Corden, 2001	cis- or trans- BSN 2060-3-pentanol BSN 2060-3-pentanol HL11	hen
		Aikens, 2002	cis- or trans- BSN 2060-3-pentanol	rotational crops
M07	 cis- or trans-4-carboxy-3-hydroxy-Sp-enol	Shaw, 2000	cis- or trans- BSN 2060-4-carboxy-3-pentanol RU2	rat
		Dean, 2001	cis- or trans- BSN 2060-4-carboxy-3-pentanol	goat
		Corden, 2001	cis- or trans- BSN 2060-4-carboxy-3-pentanol BSN 2060-4-carboxy-3-pentanol HL4a	hen
M08	 1: oxo-cyclopentyl-Sp-enol  2: 4-formyl-Sp-enol	Shaw, 2000	BSN 2060-4-aldehyde or BSN 2060-pentanone RU3	rat
		Corden, 2001	BSN 2060-4-aldehyde or BSN 2060-4-carboxy-3-pentanone BSN 2060-aldehyde HL4b Ja03MP2D Keto-Enol (M3)	hen
		Brumhard & Elke, 2001 Ripperger & Hall, 2001 Babczinski, 2001a	Pentanone	soil soil soil

No.	Formula Names (Name used in the summary in bold)	Cited by	Codes used	Recovered in [metabolite postulated in the pathway]
M09	 1: 4-carboxy-Sp-enol 2: KTS 9439	Shaw, 2000 Dean, 2001 Brumhard & Elke, 2001 Ripperger & Hall, 2001 Babczinski, 2001a	BSN 2060-4-carboxylic acid RU16 BSN 2060-4-carboxylic acid 4-carboxylic acid 4-carboxy 4-Carboxy 4-Carboxy	rat goat soil soil soil
M10	 1: Sp-enol-glucuronide 2: BSN 0546-glucuronide	Dean, 2001 Corden, 2001	BSN 2060-enol-glucuronide BSN 2060-enol-glucuronide HL7	goat hen
M11	 2-hydroxymethyl-Sp-enol	Dean, 2001 Corden, 2001 Ripperger & Hall, 2001 Aikens, 2002	BSN 2060-2-hydroxymethyl BSN 2060-2-hydroxymethyl HL10 2-Hydroxymethyl BSN 2060-2-hydroxymethyl	goat hen soil rotational crops
M12	 where **R** = H and **R'** = $C_6H_9O_6$ or where **R'** = H and **R** = $C_6H_9O_6$ 4-hydroxymethyl-glucuronide-Sp-enol	Dean, 2001	BSN 2060-4-hydroxymethyl-glucuronide	goat
M13	 4-carboxy-hydroxy-Sp-enol	Corden, 2001	BSN 2060-hydroxy-4-carboxy HL6a HL6b	hen
M14	 4-carboxy-dihydroxy-Sp-enol	Corden, 2001	BSN 2060-dihydroxy-4-carboxy HL2	hen

No.	Formula Names (Name used in the summary in bold)	Cited by	Codes used	Recovered in [metabolite postulated in the pathway]
M15	 hydroxy-cyclopentyl-Sp-enol	Ripperger & Hall, 2001	Hydroxy-Enol (M2)	soil
M16	 Spiromesifen-cyclobutyl photoisomer	Arthur & Shephard, 2001	BSN 2060-cyclobutyl photoisomer	aqueous photolysis
M17	 Sp-enol photoisomer	Arthur & Shephard, 2001	BSN 2060-enol photoisomer	aqueous photolysis
M18	 1st compound metabolite production M18	Brumhard & Elke, 2001	Ja03MP2C 1st compound	soil (metabolite production)
M19	 2nd compound metabolite production M19	Brumhard & Elke, 2001	Ja03MP2C 2nd compound	soil (metabolite production)

Plant metabolism

The Meeting received studies depicting the metabolism of spiromesifen in tomato, lettuce, and cotton.

Tomato

The metabolism of spiromesifen in tomatoes was investigated by Haynes (2001, Report 200332). Spiromesifen, radiolabelled at the 3-hydrofuranone carbon, was prepared as a suspension concentrate formulation and applied twice to three mature tomato plants grown in a polytunnel at application rates

of 439 g ai/ha (31 days prior to harvest) and 378 g ai/ha (7 days prior to harvest). Treatments were made to additional plants with protected fruits and to fruits directly to evaluate translocation. Application directly to the fruits was equivalent to ca. a 3× exaggerated application rate.

Ripe and non-ripe tomato fruits and foliage were harvested from all plants 7 days after the second application. Fruits and leaves were surface washed (three times) with acetonitrile (ACN), and aliquots of the wash solution were subjected to radio-assay and chromatographic analysis. After washing, fruits from the non-translocation plants were homogenized, extracted sequentially with ACN (2×) and ACN:water (1:1, v/v, 2×), and centrifuged. Aliquots of the supernatant were analysed by radio-assay and prepared for chromatographic analysis. Residues remaining after extraction were determined by combustion/liquid scintillation counting (LSC) of the solids. Leaves and fruits which were directly treated to assess translocation were treated in the same manner as described for the non-translocation plants. Protected fruits from the translocation plants were homogenized and a portion of the homogenized material taken for combustion and radio-assay. Extracts of fruits were concentrated by rotary film evaporation, and the concentrate was sorbed to a C_{18} solid-phase extraction column. The column was eluted with water and then hexane. The water fraction was concentrated prior to analysis by thin-layer chromatography (TLC) and high-performance liquid chromatography (HPLC), and the hexane fraction was concentrated prior to analysis by HPLC. Metabolites were isolated from the hexane fraction by preparative TLC and HPLC and analysed by HPLC and mass spectrometry techniques. A portion of the extract from non-ripe tomato fruits underwent incubation with ß-glucosidase and analysed by HPLC. All samples were stored frozen (< –15 °C) and were analysed within 1 month of harvest for ripe fruits and within 4 months of harvest for non-ripe fruits and leaves.

Total radioactive residues (TRR) were relatively low in protected fruit from the translocation samples (0.021 mg eq/kg) and in leaves (residues not reported), and further analysis of these samples was not done. The TRR in ripe fruits (0.88 mg eq/kg) and non-ripe fruits (0.50 mg eq/kg) consisted primarily of surface residues (0.67 mg eq/kg, 79% TRR ripe fruit; 0.36 mg eq/kg, 74% TRR non-ripe fruit). Residues extracted sequentially with ACN and ACN:H_2O (1:1, v/v) accounted for ca. 0.13 mg eq/kg (17% TRR ripe fruit, 25% TRR non-ripe fruit), with lesser amounts as unextracted residues (0.032 mg eq/kg, 3.8% TRR ripe fruit; 0.009 mg eq/kg, 1.8% TRR non-ripe fruit).

The only major residue (≥ 10% TRR) in both ripe fruit and non-ripe fruit surface washes and extracts was spiromesifen (Table 1), which accounted for ca. 86% TRR overall in both ripe (0.73 mg/kg) and non-ripe fruit (0.43 mg/kg).

Table 1 Summary of radioactive residues in extracts of tomato following treatment with [dihydrofuranone-3-^{14}C]spiromesifen (Report 200332)

	Surface wash		Extracts		Total	
Fraction/Residue	mg eq./kg	% TRR	mg eq./kg	% TRR	mg eq./kg	% TRR
Ripe Fruit, 1× Nominal Application						
Total extracted	0.67	79	0.14	17	0.81	96
TF1	< 0.002	< 0.2	0.003	0.3	0.003	0.3
TF2	< 0.002	< 0.2	0.003	0.3	0.003	0.3
4-hydroxymethyl-glucoside-Sp-enol	< 0.002	< 0.2	0.046	5.4	0.046	5.4
4-hydroxymethyl-Sp-enol	< 0.002	< 0.2	0.004	0.5	0.004	0.5
Sp-enol	0.002	0.2	0.004	0.5	0.006	0.7
TF6	0.008	0.9	0.001	0.1	0.008	1
Spiromesifen	0.65	77	0.076	9.0	0.73	86
TF8	< 0.002	< 0.2	0.002	0.2	0.002	0.2
Others	0.008	1.0	0.005	0.6	0.014	1.6
Unextracted	–	–	–	–	0.032	3.8
Total	–	–	–	–	0.84	100
Non-Ripe Fruit, 1× Nominal Application						
Total extracted	0.36	74	0.12	25	0.49	98
Polar	< 0.001	< 0.1	< 0.001	< 0.1	< 0.001	< 0.2
TF1	< 0.001	< 0.1	< 0.001	< 0.1	< 0.001	< 0.2
TF2	< 0.001	< 0.1	< 0.001	< 0.1	< 0.001	< 0.2
4-hydroxymethyl-glucoside-Sp-	< 0.001	< 0.1	0.035	7.0	0.035	7.0

	Surface wash		Extracts		Total	
Fraction/Residue	mg eq./kg	% TRR	mg eq./kg	% TRR	mg eq./kg	% TRR
enol						
4-hydroxymethyl-Sp-enol	< 0.001	< 0.1	< 0.001	< 0.1	< 0.001	< 0.2
Sp-enol	0.001	0.3	0.002	0.4	0.003	0.70
TF6	0.005	1.1	0.001	0.3	0.007	1.4
Spiromesifen	0.35	71	0.080	16	0.43	87
TF8	< 0.001	< 0.1	< 0.001	< 0.1	< 0.001	< 0.2
Others	0.006	1.3	0.004	0.8	0.010	2.1
Unextracted	–	–	–	–	0.009	1.8
Total	–	–	–	–	0.50	100

Following treatment with ß-glucosidase, residues of spiromesifen-4-hydroxymethyl-glucoside were decreased and there was a corresponding increase in spiromesifen-4-hydroxymethyl (Table 2)

Table 2 Effects of treatment with ß-glucosidase on organosoluble spiromesifen residues from non-ripe tomato fruit (Report 200332)

	Control		ß-Glucosidase-treated	
Fraction/Residue	mg eq./kg	% TRR	mg eq./kg	% TRR
Solvent-extracted	0.079	16	0.079	16
TF1	0.003	0.6	0.001	0.3
TF2	0.004	0.9	0.006	1.2
4-hydroxymethyl-glucoside-Sp-enol	0.046	9.3	0.005	1.0
4-hydroxymethyl-Sp-enol	0.005	1.1	0.050	10.0
Sp-enol	0.012	2.4	0.013	2.6
TF6	0.002	0.5	0.002	0.5
Spiromesifen	0.004	0.5	0.001	0.2
TF8	< 0.001	< 0.1	< 0.001	< 0.1
Others	0.001	0.3	0.001	0.2

Lettuce

The metabolism of spiromesifen in lettuce was investigated by Hanes (2001, Report BAG 318/994567). Spiromesifen, radiolabelled at the 3-hydrofuranone carbon, was prepared as a suspension concentrate formulation and applied twice to four lettuce plants/treatment rate growing in a protected environment (plastic tunnel). Applications were made 26 days after sowing of the lettuce seeds and 1 week prior to harvest at application rates of ca. 300 g ai/ha (1× nominal rate), ca 0.75× and ca. 1.25× the nominal application rate; only results from the 1× rate were reported.

Harvested lettuce plants were extracted using ACN and ACN:water (1:1, v/v), and the extracts were assayed for radioactivity via LSC; PES were also assayed for radioactivity. Aliquots of pooled extract were analysed by HPLC and TLC to characterize and identify extracted residues. For two fractions from the analysis, three aliquots were partitioned against dichloromethane, with one aliquot adjusted to pH 1 using 6 M HCl and another adjusted to pH 12 using 6 M NaOH. Aliquots from the resulting aqueous and organic phases were assayed for radioactivity and by TLC when warranted by the results from the radio-analysis.

Total radioactive residue in harvested lettuce, defined as the sum of extracted and unextracted radioactivity, was 0.41 mg eq/kg. Of that, over 98% was extracted (0.40 mg eq/kg), and 1.4% (0.006 mg eq/kg) remained in the PES. Parent spiromesifen was the predominant residue, accounting for 58% TRR (0.24 mg/kg; Table 3). At pH 1 and pH 7 the radioactivity in the L3 and L4 extracts partitioned primarily into the aqueous fractions (ca. 86% for L3 and ca. 75% for L4). At pH 12, all the radioactivity in the L3 and L4 extracts portioned into the aqueous phase. Within the aqueous and organic phases, the residue profiles were not significantly affected by pH; therefore, detailed results are not presented in this evaluation.

Table 3 Summary of radioactive residues in extracts of lettuce following treatment with [dihydrofuranone-3-^{14}C]spiromesifen (Report BAG 318/994567)

Fraction/Residue	mg eq./kg	% TRR
Total extracted	0.405	98.6
L1	< 0.001	< 0.2
L2	0.002	0.4
L3	0.038	9.3
Spiromesifen-dihydroxy-enol	0.025	6.2
Unidentified	0.013	3.2
L4	0.094	22.8
4-hydroxymethyl-glucoside-Sp-enol	0.049	11.9
4-dihyroxymethyl-Sp-enol	0.005	1.1
Unidentified	0.040	9.8
L5	0.029	7.1
3-pentanol-Sp-enol	0.009	2.1
4-hydroxymethyl-Sp-enol	0.012	2.8
Unidentified	0.008	2.2
L6 Sp-enol	0.006	1.5
L7	< 0.001	< 0.2
L8 Spiromesifen	0.237	57.6
L9	< 0.001	< 0.2

Cotton

The metabolism of spiromesifen in cotton was investigated by Langford-Pollard (2001, Report BAG 287/993894). Spiromesifen, radiolabelled at the 3-hydrofuranone carbon, was prepared as a suspension concentrate formulation and applied three times, on a 7-day interval during boll-set to boll-split growth stages, with the last application occurring 21 days before harvest. Nine plants were treated at 300 g ai/A/application (1.5× nominal rate) and two plants were treated at 1000 g ai/A/application (5× nominal rate).

At maturity, cotton was harvested and separated into gin trash, lint, and undelinted seed. The undelinted seed was then further processed into delinted seed and seed lint. In addition, samples of bolls that were protected from direct application of the test material were harvested to assess translocation of residues. Delinted seed, seed lint, and gin trash were extracted using ACN:water (1:1, v/v) and analysed for radioactivity by LSC. Post-extraction solids (PES) were analysed for radioactivity by combustion and LSC. Extracts from each matrix were also analysed chromatographically to characterize and identify residues. For gin trash, aliquots of extract were adjusted to pH 1, 3, or 12; unadjusted extract was pH 7. These extracts were partitioned against dichloromethane, and the aqueous and organic phases were analysed by high-performance liquid chromatography (HPLC). An aliquot of extract from gin trash was also subjected to enzyme treatment with cellulase, pectinase, and ß-glucosidase, followed by HPLC analysis. PES from the gin trash samples were also treated with the same enzyme mixture. Portions of the PES from gin trash samples was also extracted with 0.1 M HCl or 0.1 M NaOH at ambient temperature and by refluxing for 2 hours with 0.1 M HCl or 0.1 M NaOH, or 1 hour with 6 M HCl or 2 M NaOH.

HPLC was run on a C_{18} column at 40 °C. Mobile phases consisted of acetonitrile and either 0.2% phosphoric acid or 0.2% formic acid at different gradients. Eluate collection was of 30-second fractions for radio-analysis. Thin-layer chromatography (TLC) on silica gel plates was used to assess chemical purity and to compare chromatographic behaviour of metabolites against reference substances.

Of the total radioactive residues (TRR) in undelinted cotton seed (seed lint + delinted seed; 0.051 mg eq/kg) from treated bolls, 94% (0.048 mg eq/kg) was extracted, with 73% (0.037 mg eq/kg) being associated with the seed lint (Table 4). Total radioactive residues were much lower in protected bolls than treated bolls, indicating that only a small amount of the residue in cotton came from translocation. Further analysis of protected cotton bolls was not done.

Table 4 Total radioactive residues in cotton seed and gin trash following application of spiromesifen at ca. 300 g/ha (Report BAG 287/993894)

		Undelinted seed [a]		Seed lint		Delinted seed		Gin trash	
Bolls	Fraction	mg eq./kg	% TRR	mg eq./kg	% TRR	mg eq./kg	% TRR	mg eq./kg	% TRR
Treated	Extracted	0.048	94.2	0.037	73.3	0.011	20.9	6.30 [b]	99.5 [b]
	Solvent	–	–	–	–	–	–	5.84	92.2
	2 M NaOH reflux	–	–	–	–	–	–	0.46	7.3
	PES	0.003	5.9	0.002	3.7	0.001	2.2	0.03	0.5
	Total	0.051	100.1	0.039	77.0	0.012	23.1	6.33	100
Protected	Extract	0.0033	71.2	0.0023	49.3	0.0010	21.9	–	–
	PES	0.013	28.9	0.0009	20.5	0.0004	8.4	–	–
	Total	0.0046	100.1	0.0032	69.8	0.0014	30.3	–	–

[a] Sum of seed lint and delinted seed.

[b] Sum of solvent and NaOH reflux. Direct combustion of gin trash resulted in 5.57 mg eq/kg.

In cotton seed components, only two residues occurred at levels > 0.001 mg/kg: parent spiromesifen and the -enol metabolite, and each were at greater levels in seed lint than in the delinted seed. Spiromesifen made up ca. 52% of the TRR (0.020 mg/kg) in seed lint and ca. 82% of the TRR (0.009 mg/kg) in delinted seed. The enol metabolite made up ca. 48% of the TRR (0.018 mg/kg) in seed lint and ca. 18% of the TRR (0.002 mg/kg) in delinted seed. Analysis of gin trash extracts (Table 5) show that the parent compound and the enol metabolite are the major residues (i.e., > 10% TRR and > 0.01 mg/kg) in cotton.

Table 5 Summary of radioactive residues in extracts of cotton gin trash following treatment of cotton with [dihydrofuranone-3-^{14}C]spiromesifen (Report BAG 287/993894)

	Solvent-extracted		2 M NaOH reflux		Total	
Fraction/Residue	mg eq./kg	% TRR	mg eq./kg	% TRR	mg eq./kg	% TRR
CG1	0.01	0.1	< 0.02	< 0.3	< 0.02	< 0.3
CG2	0.31	4.9	0.04	0.7	0.35	5.6
Dihydroxy-Sp-enol	0.23	3.6	Not calc'd	Not calc'd	0.23	3.6
Unidentified	0.08	1.3	Not calc'd	Not calc'd	0.08	1.3
CG3	0.06	0.9	0.06	0.9	0.11	1.8
4-hydroxymethyl-3-pentanol-Sp-enol	0.02	0.3	Not calc'd	Not calc'd	0.02	0.3
4-hydroxymethyl-glucoside-Sp-enol	0.02	0.3	Not calc'd	Not calc'd	0.02	0.03
Unidentified	0.02	0.03	Not calc'd	Not calc'd	0.02	0.03
CG4	0.61	9.6	0.09	1.5	0.70	11.1
3-pentanol-Sp-enol	0.05	0.8	Not calc'd	Not calc'd	0.05	0.8
4-hydroxymethyl-Sp-enol	0.44	6.9	Not calc'd	Not calc'd	0.44	6.9
Unidentified	0.13	2.0	Not calc'd	Not calc'd	0.13	2.0
CG5	0.08	1.3	< 0.02	< 0.3	0.08	1.3
CG6	0.03	0.5	< 0.02	< 0.3	0.03	0.5
Sp-enol	2.89	45.6	0.24	3.8	3.13	49.4
CG8	0.05	0.8	< 0.02	< 0.3	0.05	0.8
CG9	0.05	0.8	< 0.02	< 0.3	0.05	0.8
CG10	0.03	0.5	< 0.02	< 0.3	0.03	0.5
Spiromesifen	1.66	26.3	< 0.02	< 0.3	1.66	26.3
CG12	0.03	0.4	< 0.02	< 0.3	0.03	0.4
Total	5.84	92.2	0.46	7.3	6.30	99.5

Treatment of cotton gin trash extracts with enzymes did not result in substantial changes in the metabolite profiles in the samples, indicating that residues were not hydrolysed by cellulase, pectinase or ß-glucosidase.

Conclusions—Primary crops

Metabolism studies in tomato, lettuce, and cotton consistently show spiromesifen to be a major residue. The available data indicate that spiromesifen is not significantly translocated following foliar application. The enol metabolite and the 4-hydroxymethyl-glucoside metabolite may be major residues in certain crop matrices; however, a consistent pattern of occurrence or relative levels was not observed.

Rotational crops

The Meeting received data from a radiolabelled, confined study in rotational crops (spinach, turnip, and spring wheat) as well as field accumulation studies for bulb onion, green onion, sugar beet, barley, wheat, sugarcane, and alfalfa, and greenhouse accumulation studies for carrot, lettuce, and tomato.

In a study examining residues in confined rotational crops (Aikens, Report 200337), [dihydrofuanone-3-^{14}C]spiromesifen was applied to bare soil in environmentally controlled rooms at a rate of 800 g ai/ha. The treated soil received regular watering and was maintained on a diurnal cycle with artificial light. Rotational crops of spring wheat, spinach, and turnip were planted into the treated soil 30, 120–187, and 365 days after application.

Spring wheat was harvested as forage (ca. 8 weeks after sowing), hay, grain (at maturity), and straw (at maturity); spinach and turnip (including root and foliage) were harvested at maturity. In addition, soil cores were collected immediately after application and at the time of sowing for each crop. Total radioactivity in the sampled matrices was measured by combustion/LSC Crop samples were extracted with ACN and/or ACN:water (1:1, v/v). For wheat matrices, additional work was done with acidic and alkaline extractions. Extracts were assayed for radioactivity and partitioned against ethyl acetate to characterize aqueous and organosoluble residues. The aqueous and organic fractions were further analysed by TLC to identify residues. Samples of wheat grain were assayed to determine the extent of incorporation of radioactivity in to starch, and PES of wheat grain and wheat straw were assayed to determine the extent of incorporation of radioactivity into natural constituents.

Total radioactive residues in soil in the spinach and turnip pots indicate a bi-phasic dissipation, with a rapid initial loss followed by much slower decline (Figure 2). Soil TRR in the wheat pot was reported to have an initial increase between the 0-day sample and the 30-day sample before a nearly linear decline through the remainder of the study. This may be due to a mix-up between the 0-day and 30-day samples; however, there was no discussion of this in the report. Given that the reported data are TRR and not specific residues of interest, they are not adequate to determine whether residues of interest may accumulate from multi-year applications of spiromesifen.

Except for wheat grain, TRR in rotational crops decreased with increasing plant-back interval (Table 6). Thirteen components were resolved from the rotational crop commodity extracts. Of those, seven were identified by co-chromatography with known standards: spiromesifen, Sp-enol, 2-hydroxymethyl-Sp-enol, 4-hydroxymethyl-Sp-enol, 3-pentanol-Sp-enol, dihydroxy-Sp-enol, and 4-hydroxymethyl-glucoside-Sp-enol. Spiromesifen was not a major residue in any commodity. The major residues in most commodities were the 4-hydroxymethyl metabolite in free and conjugated forms. Except for the 2-hydroxymethyl residue, the other identified metabolites occurred as major residues (≥ 10% TRR, ≥ 0.01 mg/kg) in at least one matrix (Table 7). Analysis of the PES of wheat grain (187-day PBI) and wheat straw (187- and 365-day PBIs) indicated that of the unextracted residue, nearly 80% in grain and over 40% in straw consisted of polysaccharides, cellulose, and lignin.

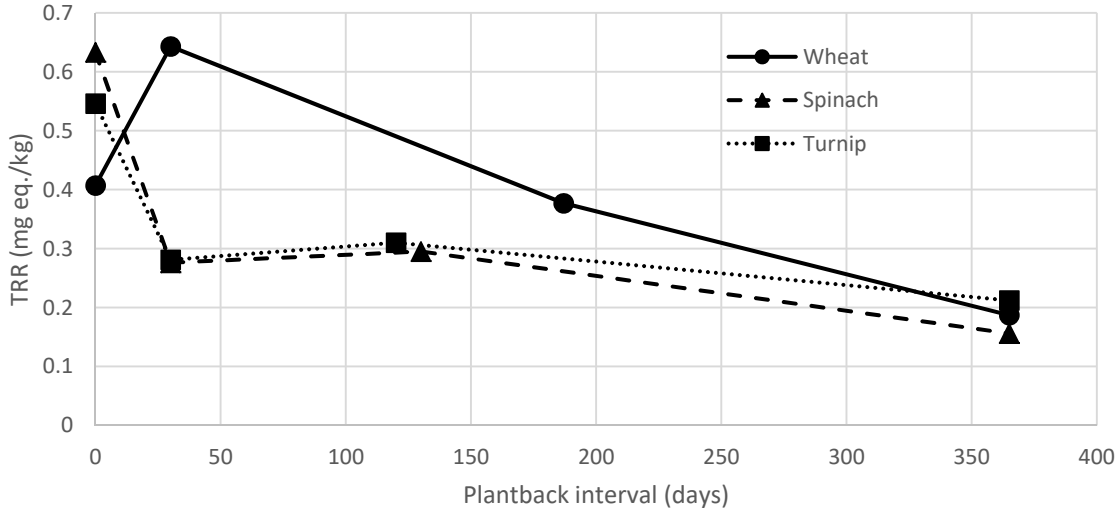


Figure 2 Total radioactive residues in soil from the confined rotational crop study with spiromesifen

The metabolism of spiromesifen in target and rotational crops appears to be similar. The proposed metabolic pathway for spiromesifen in those crops is shown in Figure 3.

Table 6 Summary of total radioactive residues in rotational crop matrices following application of [^{14}C]spiromesifen at 800 g ai/ha

Commodity	Total radioactive residue, mg eq/kg		
	30-day PBI	Nominal 120-day PBI [actual PBI]	365-day PBI
Wheat forage	0.640	0.062 [187 days]	0.033
Wheat grain	0.027	0.180 [187 days]	0.082
Wheat hay	0.542	0.316 [187 days]	0.279
Wheat straw	1.149	0.520 [187 days]	0.353
Spinach	0.315	0.198 [130 days]	0.043
Turnip foliage	0.169	0.129 [120 days]	0.025
Turnip root	0.079	0.034 [120 days]	0.010

Table 7 Profile of radioactive residues in rotational crops planted into soil treated with [^{14}C]spiromesifen

Matrix	Metabolite	30-day PBI		120-day PBI (nominal)		365-day PBI	
		mg eq./kg	% TRR	mg eq./kg	% TRR	mg eq./kg	% TRR
Spinach							
Leaves	Spiromesifen	< 0.001	< 0.32	0.005	2.53	0.001	2.33
	Sp-enol	0.002	0.63	0.005	2.53	0.002	4.65
	2-hydroxymethyl-Sp-enol	0.004	1.27	0.001	0.51	0.001	2.33
	4-hydroxymethyl-Sp-enol [a]	0.146	46.35	0.035	17.68	< 0.004	< 9.31
	-4-hydroxymethyl-Sp-enol	0.004	1.27	–	–	–	–
	-glucoside	0.126	40.00	0.030	15.15	0.003	6.98
	-unknown conj.	0.016	5.08	0.005	2.53	< 0.001	< 2.33
	3-pentanol-Sp-enol	0.022	6.98	0.057	28.79	0.009	20.93
	Dihydroxy-Sp-enol	0.026	8.25	0.038	19.19	0.003	6.98
	Total identified	0.186	63.80	0.135	71.23	0.014	46.53
	Total extracted	0.291	92.5	0.189	95.5	0.030	70.7
	PES	0.024	7.5	0.009	4.5	0.013	29.3
	Total	0.315	100	0.198	100	0.043	100
Turnip							
Leaves	Spiromesifen	< 0.0005	–	< 0.001	< 0.78	< 0.0005	–
	Sp-enol	0.005	2.96	0.003	2.33	< 0.001	< 4.00

Matrix	Metabolite	30-day PBI		120-day PBI (nominal)		365-day PBI	
		mg eq./kg	% TRR	mg eq./kg	% TRR	mg eq./kg	% TRR
	2-hydroxymethyl-Sp-enol	0.002	1.18	0.002	1.55	< 0.0005	–
	4-hydroxymethyl-Sp-enol [a]	0.096	56.81	0.049	37.99	< 0.0035	< 12.00
	-4-hydroxymethyl-Sp-enol	0.052	30.77	0.014	10.85	–	–
	-glucoside	0.035	20.71	0.030	23.26	0.002	8.00
	-unknown conj.	0.009	5.33	0.005	3.88	< 0.001	< 4.00
	3-pentanol-Sp-enol	0.025	14.79	0.023	17.83	0.002	8.00
	Dihydroxy-Sp-enol	0.006	3.55	0.007	5.43	< 0.005	–
	Total identified	0.126	79.29	0.079	65.91	0.003	24.00
	Total extracted	0.159	94.3	0.120	92.7	0.014	57.3
	PES	0.010	5.7	0.009	7.3	0.011	42.7
	Total	0.169	100	0.129	100	0.025	100
Roots	Spiromesifen	< 0.0005	–	< 0.001	< 2.94	n.a.	–
	Sp-enol	0.008	10.13	0.003	8.82	n.a.	–
	2-hydroxymethyl-Sp-enol	0.002	2.53	0.002	5.88	n.a.	–
	4-hydroxymethyl-Sp-enol [a]	0.042	53.19	< 0.008	< 23.53	n.a.	–
	-4-hydroxymethyl-Sp-enol	0.040	50.63	0.007	20.59	n.a.	–
	-glucoside	0.002	2.53	< 0.001	< 2.94	n.a.	–
	-unknown conj.	–	–	–	–	n.a.	–
	3-pentanol-Sp-enol	0.010	12.66	0.003	8.82	n.a.	–
	Dihydroxy-Sp-enol	0.004	5.06	0.003	8.82	n.a.	–
	Total identified	0.062	84.81	0.017	61.75	n.a.	–
	Total extracted	0.073	93.0	0.028	82.3	0.004	38.7
	PES	0.005	6.9	0.006	17.7	0.006	61.3
	Total	0.079	100	0.034	100	0.010	100
Wheat							
Forage	Spiromesifen	< 0.003	< 0.047	0.001	1.61	< 0.0005	–
	Sp-enol	0.006	0.94	< 0.001	< 1.61	< 0.0005	–
	2-hydroxymethyl-Sp-enol	0.011	1.72	< 0.001	< 1.61	< 0.0005	–
	4-hydroxymethyl-Sp-enol [a]	0.413	64.53	< 0.022	< 35.48	0.007	21.21
	-4-hydroxymethyl-Sp-enol	0.016	2.50	< 0.001	< 1.61	–	–
	-glucoside	0.341	53.28	0.017	27.42	0.005	15.15
	-unknown conj.	0.056	8.75	0.004	6.45	0.002	6.06
	3-pentanol-Sp-enol	0.019	2.97	< 0.0005	< 0.0005	0.001	3.03
	Dihydroxy-Sp-enol	0.071	11.09	0.007	11.29	0.002	6.06
	Total identified	0.512	81.72	0.025	51.6	0.006	30.3
	Total extracted	0.626	97.8	0.048	77.1	0.021	62.3
	PES	0.014	2.2	0.014	22.9	0.012	37.7
	Total	0.640	100	0.062	100	0.03	100
Grain	Spiromesifen	< 0.001	< 3.70	0.003	1.67	< 0.001	< 1.22
	Sp-enol	< 0.0005	–	< 0.001	< 0.56	< 0.002	< 2.44
	2-hydroxymethyl-Sp-enol	< 0.0005	–	< 0.0005	–	< 0.0005	–
	4-hydroxymethyl-Sp-enol [a]	< 0.005	< 18.51	< 0.003	< 1.68	< 0.005	< 6.10
	-4-hydroxymethyl-Sp-enol	< 0.001	< 3.7	< 0.001	< 0.56	–	–
	-glucoside	0.003	11.11	< 0.001	< 0.56	< 0.003	< 3.66
	-unknown conj.	< 0.001	< 3.7	< 0.001	< 0.56	< 0.002	< 2.44
	3-pentanol-Sp-enol	0.001	3.7	< 0.0005	–	< 0.0005	–
	Dihydroxy-Sp-enol	0.003	11.11	< 0.001	< 0.56	< 0.002	< 2.44
	Total identified	0.014	70.3 [b]	0.072	62.8	0.029	52.5
	Total extracted	0.020	75.8	0.114	63.1	0.056	68.6
	PES	0.007	24.2	0.066	36.9	0.026	31.4
	Total	0.027	100	0.180	100	0.082	100
Hay	Spiromesifen	0.003	0.55	0.001	0.32	< 0.002	< 0.72
	Sp-enol	0.003	0.55	0.003	0.95	< 0.003	< 1.08
	2-hydroxymethyl-Sp-enol	0.006	1.11	0.003	0.95	0.003	1.08
	4-hydroxymethyl-Sp-enol [a]	0.349	64.39	0.079	25.00	0.050	17.93
	-4-hydroxymethyl-Sp-enol	0.005	0.92	0.003	0.95	0.003	1.08

Matrix	Metabolite	30-day PBI		120-day PBI (nominal)		365-day PBI	
		mg eq./kg	% TRR	mg eq./kg	% TRR	mg eq./kg	% TRR
	-glucoside	0.266	49.08	0.054	17.09	0.032	11.47
	-unknown conj.	0.078	14.39	0.022	6.96	0.015	5.38
	3-pentanol-Sp-enol	0.005	0.92	0.003	0.95	0.022	7.89
	Dihydroxy-Sp-enol	0.040	7.38	0.018	5.70	0.032	11.47
	Total identified	0.376	74.9	0.091	33.9	0.100	40.2
	Total extracted	0.508	93.8	0.269	85.2	0.249	89.1
	PES	0.034	6.2	0.047	14.8	0.030	10.8
	Total	0.542	100	0.316	100	0.279	100
Straw	Spiromesifen	0.004	0.35	0.009	1.73	< 0.001	< 0.28
	Sp-enol	0.009	0.78	0.012	2.31	0.003	0.85
	2-hydroxymethyl-Sp-enol	0.022	1.91	0.018	3.46	0.006	1.70
	4-hydroxymethyl-Sp-enol [a]	0.607	52.83	0.136	26.15	0.067	18.98
	-4-hydroxymethyl-Sp-enol	0.017	1.48	0.023	4.42	0.003	0.85
	-glucoside	0.437	38.03	0.079	15.19	0.046	13.03
	-unknown conj.	0.153	13.32	0.034	6.54	0.018	5.1
	3-pentanol-Sp-enol	0.080	6.96	0.016	3.08	0.040	11.33
	Dihydroxy-Sp-enol	0.094	8.18	0.033	6.35	0.046	13.03
	Total identified		71.01		43.1 [c]		46.2 [c]
	Total extracted	1.128	98.2	0.433	83.2	0.301	85.4
	PES	0.021	1.8	0.088	16.9	0.052	14.6
	Total	1.149	100	0.521	100	0.353	100

[a] Sum of the 4-hydroxymethyl, glucoside and unknown conjugate residues.

[b] Includes TRR in starch (33.3% at 30 days, 58.3% at 187 days, and 40.3% at 365 days).

[c] Including TRR incorporated into natural plant constituents, TRR identified is 87.5% at 187 days and 87.3% at 365 days, and TRR extracted is 92.4% at 187 days and 88.9% at 365 days.

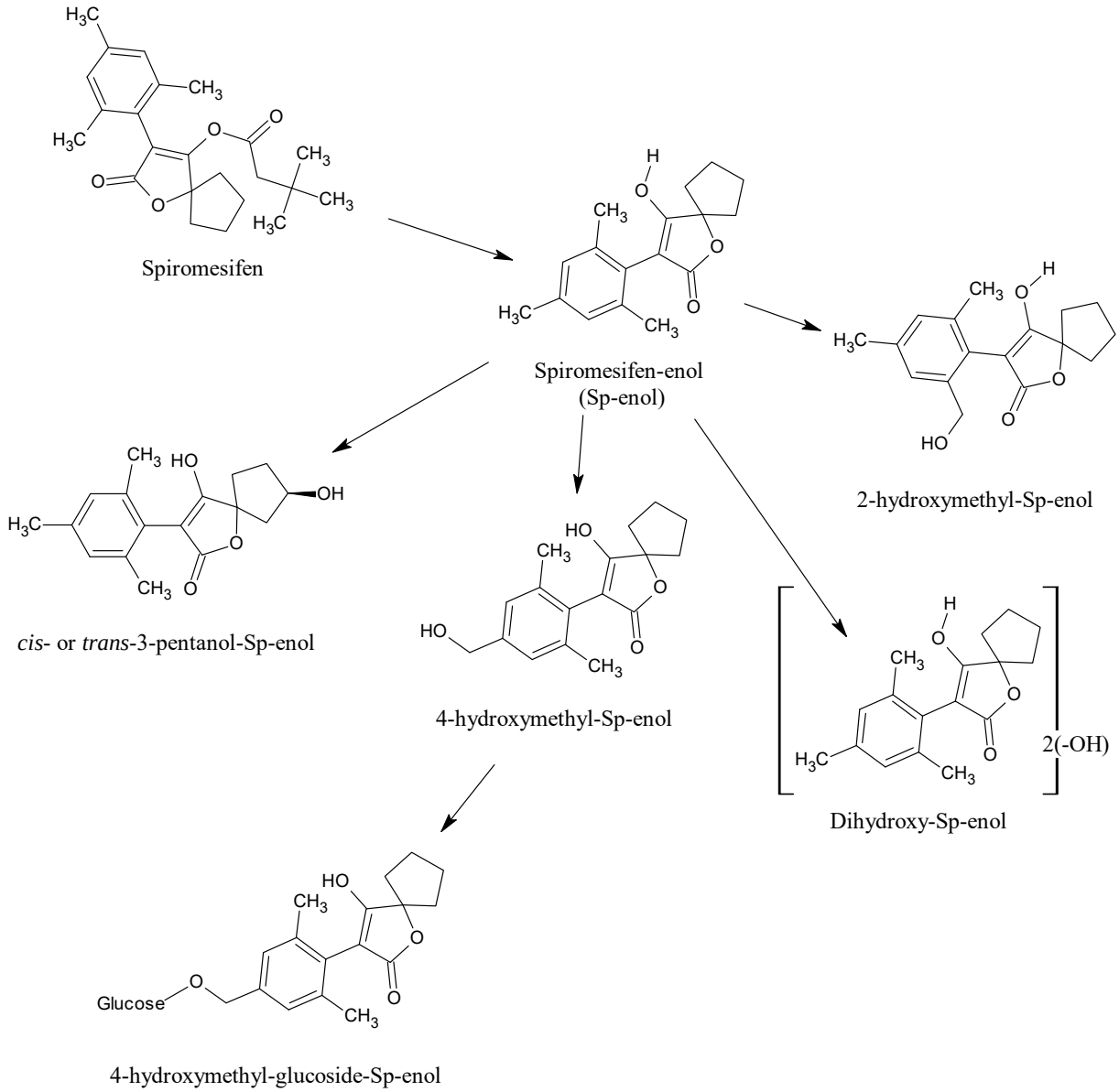


Figure 3 Proposed metabolic pathway of spiromesifen in target and rotational crops.

In a study examining field accumulation in rotational bulb onion and green onion (Gould and Harbin, 2009; Report RABSP001-1), spiromesifen was applied three times on a 4- to 7-day retreatment interval to a vegetable cover crop or to bare soil at a target rate of 280 g ai/ha per application (totalling 836–864 g ai/ha) to nine sites. Following the third application, the plot was tilled and onions were planted 28 to 31 days after the last application (DALA). Samples were harvested at maturity and shipped, frozen, to the analytical laboratory. Samples were homogenized in the presence of dry ice and returned to frozen storage until analysis.

The analytical method (Method 00631/M001; Report MR-193/02) uses ACN/water extraction with reversed-phase HPLC-MS/MS quantification to determine residues of spiromesifen and its –enol metabolite. Residues of 4-hydroxymethyl-Sp-enol and its conjugates were analysed per analytical method 00914 (Report-No.: 110333-1). The residues were extracted with water using a Dionex Accelerated Solvent Extractor (ASE200). To hydrolyse glucoside conjugates from the 4-hydroxymethyl-Sp-enol metabolite, the extracts were acidified with glacial acetic acid, cleaned-up on a strong cation exchange column, acidified with concentrated HCl, and heated to 95 °C for four hours. The methods for all analytes included the use of isotopically labelled internal standards. All residues

were reported in spiromesifen equivalents. The residues of the three analytes obtained from two extractions/analyses were summed to obtain the total spiromesifen residue. The LOQ was 0.02 mg/kg for the total residue (see Residue Analysis, below); the limit of detection (LOD) of each analyte varied from 0.0035 to 0.006 mg/kg, depending on the analyte. The total residue was obtained by calculating the sum of the individual spiromesifen residues (sum of spiromesifen, Sp-enol and 4-hydroxymethyl-Sp-enol, all expressed as parent equivalents). Values below the LOD were summed into the total residue value as ½ the LOD.

Samples were stored frozen for a maximum of 543 days prior to analysis of spiromesifen and Sp-enol, and for a maximum of 1050 days prior to analysis of 4-hydroxymethyl-Sp-enol. Stability during storage was demonstrated for at least 679 days for spiromesifen and its –enol metabolite and for at least 1180 days for 4-hydroxymethyl-Sp-enol residues.

Residues of spiromesifen and its metabolites were generally low in both bulb and green onion at the 30-day plant-back interval (Table 8).

Table 8 Residues of spiromesifen in rotational bulb onion and green onion planted ca. 30 days after application to a cover crop or to bare soil

Trial ID and Location (Year)	Applic., g ai/ha	DAP [b]	Variety	Commodity sampled	Residue, mg/kg [mean] [a]			
					Spiromesifen	Sp-enol	4-hydroxy-methyl-Sp-enol	Total
BS001-06RA Germansville, PA US (2006)	289 + 289 + 286	77	Stuttgard (bulb)	Bulbs	< 0.0035, < 0.0035 [< 0.01]	0.0062, 0.0058 [< 0.01]	< 0.006, < 0.006 [< 0.01]	< 0.02, < 0.02 [< 0.02]
BS002-06RA Raymondville, TX US (2006)	282 + 288 + 287	115	Yellow Granex F1 (bulb)	Bulbs	< 0.0035, < 0.0035 [< 0.01]	0.0050, 0.0044 [< 0.01]	< 0.006, < 0.006 < 0.01]	< 0.02, < 0.02 [< 0.02]
BS003-06RA Levelland, TX US (2006)	280 + 282 + 278	148	Yellow Spanish (bulb)	Bulbs	< 0.0035, < 0.0035 [< 0.01]	< 0.0038, < 0.0038 [< 0.01]	< 0.006, < 0.006 [< 0.01]	< 0.02, < 0.02 [< 0.02]
BS004-06RA Fresno, CA US (2006)	281 + 281 + 280	225	Stockton red (bulb)	Bulbs	< 0.0035, < 0.0035 [< 0.01]	0.0038, 0.0041 [< 0.01]	No Sample	< 0.02, < 0.02 [< 0.02]
BS005-06RA Chico, CA US (2006)	282 + 279 + 280	195	Yellow Dulce (bulb)	Bulbs	< 0.0035, < 0.0035 [< 0.01]	0.048, 0.018 [0.033]	0.0072, < 0.006 [< 0.01]	0.059, 0.028 [0.043]
BS006-06RA Ephrata, WA US (2006)	278 + 279 + 279	110	Colorado #6 (bulb)	Bulbs	0.0060, < 0.0035 [0.0048]	0.0060, < 0.0038 [< 0.01]	< 0.006, < 0.006 [< 0.01]	< 0.02, < 0.02 [< 0.02]
BS007-06RA Raymondville, TX US (2006)	284 + 287 + 287	127	Yellow Granex F1 (bulb)	Whole plant w/o roots	< 0.0035, < 0.0035 [< 0.01]	< 0.0038, < 0.0038 [< 0.01]	< 0.007, < 0.007 [< 0.01]	< 0.02, < 0.02 [< 0.02]
BS008-06RB Fresno, CA US (2006)	282 + 280 + 280	56	Southport white (green)	Whole plant w/o roots	< 0.0035, < 0.0035 [< 0.01]	0.036, 0.042 [0.039]	0.039, 0.032, 0.039, 0.016 [0.032]	0.078, 0.070 [0.074]
BS009-06RA Hickman, CA US (2006)	280 + 279 + 277	60	Southport white (green)	Whole plant w/o roots	< 0.0035, < 0.0035 [< 0.01]	< 0.0038, 0.0076 [< 0.01]	< 0.007, 0.0113 [< 0.01]	< 0.02, < 0.02 [< 0.02]

[a] For purposes of calculating the mean, residues reported as < LOD (0.0035 to 0.006 mg/kg depending on the analyte) were assumed to be at the LOD. For total residue, residues are reported as being < LOQ (0.02 mg/kg) rather than the LOD.

[b] DALA = Days after planting until harvest

In a field rotational sugar beet study (Harbin, 2002; Report 200161), three broadcast applications, each at ca. 280 g ai/ha, were made to bare soil on a 0- to 10-day interval to 12 sites. Sugar beets were planted into the treated soil 24 to 36 DALA. Sugar beet roots and tops were harvested at maturity and shipped, frozen, to the analytical laboratory. Upon receipt at the laboratory, the samples were homogenized in the presence of dry ice and returned to frozen storage. Samples were stored frozen for up to 531 days prior to analysis of spiromesifen and Sp-enol, and for up to 453 days prior to analysis of 4-hydroxymethyl-Sp-enol residues. Residues of spiromesifen, Sp-enol, and 4-hydroxymethyl-Sp-enol (including glucoside conjugates) were determined using the same analytical methods described for rotational onions, above.

Residues in rotational sugar beet roots were below the limit of detection 24 to 36 days after application in all samples for each analyte (spiromesifen = 0.001 mg/kg, Sp-enol = 0.002 mg/kg, 4-hydroxymethyl-Sp-enol = 0.006 mg/kg). Total residues were < 0.02 mg/kg in all samples of sugar beet root.

Total residues in sugar beet tops were < 0.02 mg/kg in all samples except one, for which the average residue was 0.16 mg/kg. Residues in that sample were from the 4-hydroxymethyl metabolite.

Table 9 Residues of spiromesifen in rotational sugar beet tops planted ca. 30 days after application to bare soil

					Residue, mg/kg [mean] [a]			
Trial ID and Location (Year)	Applic., g ai/ha	DALA [b]	Variety	Commodity sampled	Spiromesifen	Sp-enol	4-hydroxy-methyl-Sp-enol	Total
BS159-00R Ephrata, WA US (2000)	283 + 280 + 280	153	Oasis	Tops	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	< 0.004, < 0.004 [< 0.01]	< 0.02 < 0.02 [< 0.02]
BS160-00R American Falls, ID US (2000)	283 + 291 + 287	152	Beta 4006R	Tops	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	0.007, 0.005 [< 0.01]	< 0.02 < 0.02 [< 0.02]
BS161-00R El Centro, CA US (2000)	274 + 282 + 274	171	Rival	Tops	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	0.007, 0.005 [< 0.01]	< 0.02 < 0.02 [< 0.02]
BS162-00R Brawley, CA US (2000)	292 + 286 + 278	255	4430R	Tops	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	< 0.004, < 0.004 [< 0.01]	< 0.02 < 0.02 [< 0.02]
BS163-00R New Holland, OH US (2000)	281 + 280 + 281	151	Crystal 319	Tops	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	0.004, 0.005 [< 0.01]	< 0.02 < 0.02 [< 0.02]
BS164-00R Williamston, MI US (2000)	281 + 286 + 279	162	E-17	Tops	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	< 0.004, < 0.004 [< 0.01]	< 0.02 < 0.02 [< 0.02]
BS165-00R [c] Northwood, ND US (2000)	282 + 276 + 278	147	Crystal 222	Tops	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	< 0.004, 0.005 [< 0.01]	< 0.02 < 0.02 [< 0.02]
BS166-00R Northwood, ND US (2000)	279 + 287 + 287	150	Crystal 222	Tops	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	0.004, 0.007 [< 0.01]	< 0.02 < 0.02 [< 0.02]
BS167-00R East Grand Forks, MN US. (2000)	280 + 281 + 289	154	Maribo 9369	Tops	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	0.007, 0.010 [< 0.01]	< 0.02 < 0.02 [< 0.02]
BS168-00R Wilcox, AZ US (2000)	290 + 284 + 281	244	Rival	Tops	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	0.171, 0.153 [0.162]	0.171, 0.153 [0.16]
BS169-00R Claude, TX US (2000)	281 + 282 + 284	140	Giant Western	Tops	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	< 0.004, < 0.004 [< 0.01]	< 0.02 < 0.02 [< 0.02]
BS170-00R New Rockford, ND US (2000)	282 + 280 + 284	162	Crystal 222	Tops	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	0.008, 0.005 [< 0.01]	< 0.02 < 0.02 [< 0.02]

[a] For purposes of calculating the mean, residues reported as < LOD (0.0035 to 0.006 mg/kg depending on the analyte) were assumed to be at the LOD. For total residue, residues are reported as being < LOQ (0.02 mg/kg) rather than the LOD.

[b] DALA = Days after last application until harvest

In a field rotational barley study (Lemke, 2002; Report 200186), three broadcast applications, each at ca. 280–290 g ai/ha, were made to bare soil on a 0- to 8-day interval to 12 sites. Barley was planted into the treated soil 27 to 31 DALA. Barley grain, hay, and straw were harvested at normal harvest times and shipped, frozen, to the analytical laboratory. Upon receipt at the laboratory, the samples were homogenized in the presence of dry ice and returned to frozen storage. Samples were stored frozen for up to 649 days prior to analysis of spiromesifen and metabolite residues. Residues of spiromesifen, Sp-enol, and 4-hydroxymethyl-Sp-enol (including glucoside conjugates) were determined using the same analytical methods described for rotational onions, above.

Summed residues of spiromesifen, the Sp–enol metabolite, and the 4-hydroxymethyl-Sp-enol metabolite were < 0.02 mg/kg in barley grain. The highest total residues in hay and straw were 0.18 mg/kg and 0.11 mg/kg, respectively (Table 10).

Table 10 Residues of spiromesifen in rotational barley planted ca. 30 days after application to bare soil

					Residue, mg/kg [mean] [a]			
Trial ID and Location (Year)	Applic., g ai/ha	Variety	DALA [b]	Commodity sampled	Spiromesifen	Sp-enol	4-hydroxy-methyl-Sp-enol	Total
BS147-00R North Rose, NY US (2000)	274 + 269 + 292	AC Stephen	108	Grain	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	< 0.0040, 0.0054 [< 0.005]	< 0.02, < 0.02 [< 0.02]
			86	Hay	< 0.002, < 0.002 [< 0.01]	0.0014, < 0.001 [< 0.01]	0.1148, 0.1320 [0.12]	0.116, 0.132 [0.12]
			108	Straw	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	0.0300, 0.0206 [0.025]	0.03, 0.021 [0.026]
BS148-00R Centerville, SD US (2000)	278 + 280 + 282	Stark	112	Grain	n.a., < 0.001 [< 0.01]	n.a., < 0.001 [< 0.01]	n.a., < 0.004 [< 0.01]	n.a., < 0.02 [< 0.02]
			85	Hay	< 0.002, < 0.002 [< 0.01]	< 0.001, < 0.001 [< 0.01]	0.1588, 0.1428 [0.15]	0.159, 0.143 [0.15]
			112	Straw	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	0.0551, 0.0658 [0.060]	0.055, 0.066 [0.060]
BS149-00R Lesterville, SD US (2000)	279 + 280 + 280	Robust	109	Grain	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	< 0.004, < 0.004 [< 0.01]	< 0.02, < 0.02 [< 0.02]
			78	Hay	< 0.002, < 0.002 [< 0.002]	< 0.001, < 0.001 [< 0.01]	0.0436, 0.0694 [0.056]	0.044, 0.069 [0.056]
			109	Straw	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	0.0239, 0.0204 [0.022]	0.024, 0.02 [0.022]
BS150-00R Garner, ND US (2000)	293 + 291 + 291	Robust	110	Grain	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	< 0.004, < 0.004 [< 0.01]	< 0.02, < 0.02 [< 0.02]
			85	Hay	< 0.002, < 0.002 [< 0.01]	< 0.001, < 0.001 [< 0.01]	0.0292, 0.0402 [0.035]	0.029, 0.04 [0.034]
			110	Straw	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	0.0144, 0.0127 [0.014]	0.014, 0.013 [0.014]

					Residue, mg/kg [mean] [a]			
Trial ID and Location (Year)	Applic., g ai/ha	Variety	DALA [b]	Commodity sampled	Spiromesifen	Sp-enol	4-hydroxy-methyl-Sp-enol	Total
BS151-00R Velva, ND US (2000)	286 + 283 + 280	Robust	119	Grain	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	0.0041, 0.0074 [0.0058]	< 0.02, < 0.02 [< 0.02]
			84	Hay	< 0.002, < 0.002 [< 0.002]	< 0.001, < 0.001 [< 0.01]	0.0762, 0.0433 [0.060]	0.076, 0.043 [0.060]
			119	Straw	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	0.0538, 0.0570 [0.055]	0.054, 0.057 [0.056]
BS152-00R New Rockford, ND US. (2000)	271 + 272 + 279	Robust	123	Grain	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	< 0.004, < 0.004 [< 0.01]	< 0.02, < 0.02 [< 0.02]
			93	Hay	< 0.002, < 0.002 [< 0.01]	0.0019, 0.0011 [< 0.01]	< 0.016, < 0.016 [< 0.016]	< 0.02, < 0.02 [< 0.02]
			123	Straw	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	< 0.005, < 0.005 [< 0.01]	< 0.02, < 0.02 [< 0.02]
BS153-00R Ellendale, ND US (2000)	281 + 287 + 279	Robust	119	Grain	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	< 0.004, < 0.004 [< 0.01]	< 0.02, < 0.02 [< 0.02]
			92	Hay	< 0.002, < 0.002 [< 0.01]	< 0.001, < 0.001 [< 0.01]	0.0948, 0.1263 [0.11]	0.095, 0.126 [0.11]
			119	Straw	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	0.0224, 0.0262 [0.024]	0.022, 0.026 [0.024]
BS154-00R Lake Andes, SD US (2000)	277 + 282 + 274	Bowman	109	Grain	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	< 0.004, < 0.004 [< 0.01]	< 0.02, < 0.02 [< 0.02]
			90	Hay	< 0.002, < 0.002 [< 0.01]	< 0.001, < 0.001 [< 0.01]	0.0319, 0.0419 [0.037]	0.032, 0.042 [0.037]
			109	Straw	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	0.0293, 0.0325 [0.031]	0.029, 0.032 [0.030]
BS155-00R Smithfield, UT US (2000)	276 + 278 + 280	Baronesse	172	Grain	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	< 0.004, < 0.004 [< 0.01]	< 0.02, < 0.02 [< 0.02]
			126	Hay	< 0.002, < 0.002 [< 0.002]	< 0.001, < 0.001 [< 0.01]	< 0.016, < 0.016 [< 0.016]	< 0.02, < 0.02 [< 0.02]
			172	Straw	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	0.0281, 0.0224 [0.025]	0.028, 0.022 [0.025]
BS156-00R Maricopa, AZ US (2000)	259 + 287 + 290	Baretta	195	Grain	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	0.0053, 0.0100 [< 0.01]	< 0.02, < 0.02 [< 0.02]
			159	Hay	< 0.002, < 0.002 [< 0.01]	< 0.001, < 0.001 [< 0.01]	0.0675, 0.0428 [0.055]	0.068, 0.043 [0.056]
			195	Straw	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	0.1200, 0.0979 [0.11]	0.120, 0.098 [0.11]
BS157-00R Hermiston, OR US (2000)	287 + 283 + 277	Steptoe	120	Grain	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	0.0102, 0.0119 [0.011]	< 0.02, < 0.02 [< 0.02]
			85	Hay	< 0.002, < 0.002 [< 0.01]	< 0.001, < 0.001 [< 0.01]	0.1220, 0.2311 [0.18]	0.122, 0.231 [0.18]

					Residue, mg/kg [mean] [a]			
Trial ID and Location (Year)	Applic., g ai/ha	Variety	DALA [b]	Commodity sampled	Spiromesifen	Sp-enol	4-hydroxy-methyl-Sp-enol	Total
			120	Straw	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	0.0986, 0.0829 [0.091]	0.099, 0.083 [0.091]
BS158-00R American Falls, ID US (2000)	280 + 286 + 287	Ida Gold	152	Grain	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	0.0047, 0.0049 [< 0.01]	< 0.02, < 0.02 [< 0.02]
			105	Hay	< 0.002, < 0.002 [< 0.01]	< 0.001, < 0.001 [< 0.01]	< 0.016, 0.0732 [< 0.045]	< 0.02, 0.073 [< 0.047]
			152	Straw	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	0.0392, 0.0454 [0.042]	0.039, 0.045 [0.042]

[a] For purposes of calculating the mean, residues reported as < LOD (0.001 to 0.016 mg/kg depending on the analyte) were assumed to be at the LOD. For total residue, residues are reported as being < LOQ (0.02 mg/kg) rather than the LOD.

[b] DALA = Days after last application until harvest

In a field rotational wheat study (Lemke, 2002; Report 200185), three broadcast applications, each at ca. 280–290 g ai/ha, were made to bare soil on a 0- to 8-day interval to 20 sites. Wheat was planted into the treated soil 27 to 39 DALA. Wheat forage, grain, hay, and straw were harvested at normal harvest times and shipped, frozen, to the analytical laboratory. Upon receipt at the laboratory, the samples were homogenized in the presence of dry ice and returned to frozen storage. Samples were stored frozen for up to 668 days prior to analysis of spiromesifen and metabolite residues. Residues of spiromesifen, Sp-enol, and 4-hydroxymethyl-Sp-enol (including glucoside conjugates) were determined using the same analytical methods described for rotational onions, above.

Summed residues of spiromesifen, the Sp–enol metabolite, and the 4-hydroxymethyl-Sp-enol metabolite were < 0.01 mg/kg in wheat grain. The highest total residues in forage, hay and straw were 0.15 mg/kg, 0.10 mg/kg and 0.21 mg/kg, respectively (Table 11).

Table 11 Residues of spiromesifen in rotational wheat planted ca. 30 days after application to bare soil

					Residue, mg/kg [mean] [a]			
Trial ID and Location (Year)	Applic., g ai/ha	Variety	DALA [b]	Commodity sampled	Spiromesifen	Sp-enol	4-hydroxy-methyl-Sp-enol	Total
BS126-00R Tifton, GA US (2001)		Coker 9663	185	Forage	< 0.003, < 0.003 [< 0.01]	< 0.004, < 0.004 [< 0.01]	0.0205, 0.0068 [0.0136]	0.02, < 0.02 [< 0.02]
			196	Hay	< 0.003, < 0.003 [< 0.01]	< 0.003, < 0.003 [< 0.01]	0.0175, 0.0154 [0.0164]	< 0.02, < 0.02 [< 0.02]
			217	Grain	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	< 0.004, < 0.004 [< 0.01]	< 0.01, < 0.01 [< 0.01]
			217	Straw	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	0.0225, 0.0189 [0.0207]	0.022, < 0.02 [< 0.021]
BS127-00R Benoit, MS US (2001)		NK Coker 9663	167	Forage	< 0.003, < 0.003 [< 0.01]	< 0.004, < 0.004 [< 0.01]	0.0170, 0.0095 [0.0132]	< 0.02, < 0.02 [< 0.02]
			196	Hay	< 0.003, < 0.003 [< 0.01]	0.0033, < 0.003 [< 0.01]	0.0456, 0.0356 [0.0406]	0.049, 0.036 [0.043]
			238	Grain	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	< 0.004, < 0.004 [< 0.01]	< 0.01, < 0.01 [< 0.01]

					Residue, mg/kg [mean] [a]			
Trial ID and Location (Year)	Applic., g ai/ha	Variety	DALA [b]	Commodity sampled	Spiromesifen	Sp-enol	4-hydroxy-methyl-Sp-enol	Total
			238	Straw	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	0.0348, 0.0460 [0.0404]	0.035, 0.046 [0.041]
BS128-00R Stilwell, KS US (2001)		Karl 92	225	Forage	< 0.003, < 0.003 [< 0.01]	< 0.004, < 0.004 [< 0.01]	< 0.006, < 0.006 [< 0.01]	< 0.02, < 0.02 [< 0.02]
			274	Hay	< 0.003, < 0.003 [< 0.01]	< 0.003, < 0.003 [< 0.01]	0.0108, 0.0103 [0.0106]	< 0.02, < 0.02 [< 0.02]
			292	Grain	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	< 0.004, < 0.004 [< 0.01]	< 0.01, < 0.01 [< 0.01]
			292	Straw	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	< 0.013, < 0.013 [< 0.013]	< 0.02, < 0.02 [< 0.02]
BS129-00R Louisville, NE US (2001)		Arapahoe	231	Forage	< 0.003, < 0.003 [< 0.01]	0.0046, 0.0051 [< 0.01]	0.0791, 0.0736 [0.0764]	0.084, 0.079 [0.082]
			283	Hay	< 0.003, < 0.003 [< 0.01]	< 0.003, < 0.003 [< 0.01]	0.0702, 0.0561 [0.0632]	0.07, 0.056 [0.063]
			311	Grain	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	0.0064, 0.0044 [0.0054]	< 0.01, < 0.01 [< 0.01]
			311	Straw	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	0.0759, 0.0671 [0.0715]	0.076, 0.067 [0.072]
BS130-00R Oxford, IN US (2001)		BECKS 107	224	Forage	< 0.003, < 0.003 [< 0.01]	< 0.004, < 0.004 [< 0.01]	0.0172, 0.0149 [0.0161]	< 0.02, < 0.02 [< 0.02]
			259	Hay	< 0.003, < 0.003 [< 0.01]	< 0.003, < 0.003 [< 0.01]	0.0373, < 0.009 [< 0.023]	0.037, < 0.02 [< 0.029]
			308	Grain	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	< 0.004, < 0.004 [< 0.01]	< 0.01, < 0.01 [< 0.01]
			308	Straw	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	0.0155, < 0.013 [< 0.014]	< 0.02, < 0.02 [< 0.02]
BS131-00R New Holland, OH US (2001)		Hopewell	247	Forage	< 0.003, < 0.003 [< 0.01]	< 0.004, < 0.004 [< 0.01]	< 0.006, < 0.006 [< 0.01]	< 0.02, < 0.02 [< 0.02]
			280	Hay	< 0.003, < 0.003 [< 0.01]	< 0.003, < 0.003 [< 0.01]	0.0262, < 0.009 [< 0.018]	0.026, < 0.02 [< 0.023]
			306	Grain	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	< 0.004, < 0.004 [< 0.01]	< 0.01, < 0.01 [< 0.01]
			306	Straw	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	< 0.013, 0.0196 [< 0.016]	< 0.02, < 0.02 [< 0.02]
BS132-00R Centerville, SD US (2000)		Oxen	62	Forage	< 0.003, < 0.003 [< 0.01]	< 0.004, < 0.004 [< 0.01]	0.0837, 0.0878 [0.0857]	0.084, 0.088 [0.086]
			85	Hay	< 0.003, < 0.003 [< 0.01]	< 0.003, < 0.003 [< 0.01]	0.0671, 0.0719 [0.0695]	0.067, 0.072 [0.070]
			112	Grain	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	< 0.004, < 0.004 [< 0.01]	< 0.01, < 0.01 [< 0.01]

					Residue, mg/kg [mean] [a]			
Trial ID and Location (Year)	Applic., g ai/ha	Variety	DALA [b]	Commodity sampled	Spiromesifen	Sp-enol	4-hydroxy-methyl-Sp-enol	Total
			112	Straw	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	0.0288, 0.0269 [0.0278]	0.029, 0.027 [0.028]
BS133-00R Eakly, OK US (2001)		Tonkawa	191	Forage	< 0.003, < 0.003 [< 0.01]	< 0.004, < 0.004 [< 0.01]	0.0357, 0.0226 [0.0292]	0.036, 0.023 [0.030]
			229	Hay	< 0.003, < 0.003 [< 0.01]	< 0.003, < 0.003 [< 0.01]	0.0438, 0.0531 [0.0484]	0.044, 0.053 [0.049]
			265	Grain	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	< 0.004, < 0.004 [< 0.01]	< 0.01, < 0.01 [< 0.01]
			265	Straw	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	0.0436, 0.0413 [0.0424]	0.044, 0.041 [0.043]
BS134-00R Velva, ND US (2000)		2375	61	Forage	< 0.003, < 0.003 [< 0.01]	< 0.004, < 0.004 [< 0.01]	0.0221, 0.0390 [0.0306]	0.022, 0.039 [0.031]
			80	Hay	< 0.003, < 0.003 [< 0.01]	< 0.003, < 0.003 [< 0.01]	0.0786, 0.0988 [0.0887]	0.079, 0.099 [0.089]
			119	Grain	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	0.0042, 0.0041 [< 0.01]	< 0.01, < 0.01 [< 0.01]
			119	Straw	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	0.0758, 0.0968 [0.0863]	0.076, 0.097 [0.087]
BS135-00R Ellendale, ND US (2000)		Forge	69	Forage	< 0.003, < 0.003 [< 0.01]	< 0.004, < 0.004 [< 0.01]	0.0322, 0.0315 [0.0319]	0.032, 0.032 [0.032]
			92	Hay	< 0.003, < 0.003 [< 0.01]	< 0.003, < 0.003 [< 0.01]	0.1043, 0.0912 [0.0978]	0.104, 0.091 [0.098]
			120	Grain	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	0.0042, < 0.004 [< 0.01]	< 0.01, < 0.01 [< 0.01]
			120	Straw	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	0.0401, 0.0573 [0.0487]	0.040, 0.057 [0.049]
BS136-00R Lake Andes, SD US (2000)		Forge	62	Forage	< 0.003, < 0.003 [< 0.01]	< 0.004, < 0.004 [< 0.01]	0.1468, 0.1354 [0.141]	0.147, 0.135 [0.141]
			81	Hay	< 0.003, < 0.003 [< 0.01]	< 0.003, < 0.003 [< 0.01]	0.0928, 0.1003 [0.0966]	0.093, 0.100 [0.097]
			109	Grain	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	< 0.004, < 0.004 [< 0.01]	< 0.01, < 0.01 [< 0.01]
			109	Straw	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	0.0505, 0.0373 [0.0439]	0.050, 0.037 [0.044]
BS137-00R New Rockford, ND US. (2000)		Russ Wheat	66	Forage	< 0.003, < 0.003 [< 0.01]	< 0.004, < 0.004 [< 0.01]	0.0340, 0.0358 [0.0349]	0.034, 0.036 [0.035]
			101	Hay	< 0.003, < 0.003 [< 0.01]	< 0.003, < 0.003 [< 0.01]	< 0.009, < 0.009 [< 0.01]	< 0.02, < 0.02 [< 0.02]
			123	Grain	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	< 0.004, < 0.004 [< 0.01]	< 0.01, < 0.01 [< 0.01]

					Residue, mg/kg [mean] [a]			
Trial ID and Location (Year)	Applic., g ai/ha	Variety	DALA [b]	Commodity sampled	Spiromesifen	Sp-enol	4-hydroxy-methyl-Sp-enol	Total
			123	Straw	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	< 0.013, < 0.013 [< 0.013]	< 0.02, < 0.02 [< 0.02]
BS138-00R Barnard, SD US (2000)		Alsen	70	Forage	< 0.003, < 0.003 [< 0.01]	< 0.004, < 0.004 [< 0.01]	0.0238, 0.0311 [0.0274]	0.024, 0.031 [0.028]
			90	Hay	< 0.003, < 0.003 [< 0.01]	< 0.003, < 0.003 [< 0.01]	0.0500, 0.0831 [0.0666]	0.050, 0.083 [0.067]
			117	Grain	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	< 0.004, < 0.004 [< 0.01]	< 0.01, < 0.01 [< 0.01]
			117	Straw	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	< 0.013, < 0.013 [< 0.013]	< 0.02, < 0.02 [< 0.02]
BS139-00R Uvalde, TX US (2001)		Ogallala	133	Forage	< 0.003, < 0.003 [< 0.01]	< 0.004, < 0.004 [< 0.01]	< 0.006, < 0.006 [< 0.006]	< 0.02, < 0.02 [< 0.02]
			169	Hay	< 0.003, < 0.003 [< 0.01]	< 0.003, < 0.003 [< 0.01]	< 0.009, < 0.009 [< 0.009]	< 0.02, < 0.02 [< 0.02]
			205	Grain	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	< 0.004, < 0.004 [< 0.01]	< 0.01, < 0.01 [< 0.01]
			205	Straw	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	< 0.013, < 0.013 [< 0.013]	< 0.02, < 0.02 [< 0.02]
BS140-00R Claude, TX US (2001)		Jagger	272	Forage	< 0.003, < 0.003 [< 0.01]	< 0.004, < 0.004 [< 0.01]	0.0080, 0.0165 [0.0122]	< 0.02, < 0.02 [< 0.02]
			295	Hay	< 0.003, < 0.003 [< 0.01]	0.0037, < 0.003 [< 0.01]	0.0381, 0.0278 [0.0330]	0.042, 0.028 [0.035]
			326	Grain	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	0.0086, 0.0084 [< 0.01]	< 0.01, < 0.01 [< 0.01]
			326	Straw	< 0.001, < 0.001 [< 0.01]	< 0.001, 0.0064 [< 0.01]	0.0521, 0.1628 [0.107]	0.052, 0.169 [0.110]
BS141-00R Dill City, OK US (2001)		Jagger	199	Forage	< 0.003, < 0.003 [< 0.01]	< 0.004, < 0.004 [< 0.01]	0.0147, < 0.006 [< 0.01]	< 0.02, < 0.02 [< 0.02]
			231	Hay	< 0.003, < 0.003 [< 0.01]	< 0.003, < 0.003 [< 0.01]	0.0376, < 0.009 [< 0.023]	0.038, < 0.02 [< 0.029]
			267	Grain	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	< 0.004, < 0.004 [< 0.01]	< 0.01, < 0.01 [< 0.01]
			267	Straw	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	0.0563, 0.0566 [0.0564]	0.056, 0.057 [0.057]
BS142-00R Frederick, OK US (2001)		Custer	236	Forage	< 0.003, < 0.003 [< 0.01]	< 0.004, < 0.004 [< 0.01]	0.017, < 0.006 [< 0.012]	< 0.02, < 0.02 [< 0.02]
			266	Hay	< 0.003, < 0.003 [< 0.01]	< 0.003, < 0.003 [< 0.01]	0.0292, < 0.009 [< 0.019]	0.029, < 0.02 [< 0.025]
			292	Grain	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	< 0.004, < 0.004 [< 0.01]	< 0.01, < 0.01 [< 0.01]

					Residue, mg/kg [mean] [a]			
Trial ID and Location (Year)	Applic., g ai/ha	Variety	DALA [b]	Commodity sampled	Spiromesifen	Sp-enol	4-hydroxy-methyl-Sp-enol	Total
			292	Straw	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	< 0.013, < 0.013 [< 0.013]	< 0.02, < 0.02 [< 0.02]
BS143-00R Hart, TX US (2001)		TAM 200	214	Forage	< 0.003, < 0.003 [< 0.01]	< 0.004, < 0.004 [< 0.01]	0.0076, 0.0061 [< 0.01]	< 0.02, < 0.02 [< 0.02]
			252	Hay	< 0.003, < 0.003 [< 0.01]	< 0.003, < 0.003 [< 0.01]	< 0.009, 0.0094 [< 0.01]	< 0.02, < 0.02 [< 0.02]
			278	Grain	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	< 0.004, < 0.004 [< 0.01]	< 0.01, < 0.01 [< 0.01]
			278	Straw	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	0.0160, 0.0215 [0.0188]	< 0.02, 0.022 [< 0.021]
BS144-00R Halfway, TX US (2001)		TAM 200	196	Forage	< 0.003, < 0.003 [< 0.01]	< 0.004, < 0.004 [< 0.01]	0.0763, 0.0761 [0.0762]	0.076, 0.076 [0.076]
			233	Hay	< 0.003, < 0.003 [< 0.01]	< 0.003, < 0.003 [< 0.01]	0.0996, 0.0948 [0.0972]	0.100, 0.095 [0.098]
			249	Grain	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	0.0048, 0.0051 [0.0050]	< 0.01, < 0.01 [< 0.01]
			249	Straw	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	0.2109, 0.2133 [0.212]	0.211, 0.213 [0.212]
BS145-00R Hermiston, OR US (2001)		Penawawa	85	Forage	< 0.003, < 0.003 [< 0.01]	< 0.004, < 0.004 [< 0.01]	0.0096, 0.0143 [0.0119]	< 0.02, < 0.02 [< 0.02]
			92	Hay	< 0.003, < 0.003 [< 0.01]	< 0.003, < 0.003 [< 0.01]	0.0286, 0.0404 [0.0345]	0.029, 0.040 [0.035]
			120	Grain	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	0.0047, < 0.004 [< 0.01]	< 0.01, < 0.01 [< 0.01]
			120	Straw	< 0.001, < 0.001 [< 0.01]	< 0.001, < 0.001 [< 0.01]	0.0298, 0.0334 [0.0316]	0.030, 0.033 [0.032]

[a] For purposes of calculating the mean, residues reported as < LOD (0.001 to 0.004 mg/kg depending on the analyte) were assumed to be at the LOD. For total residue, residues are reported as being < LOQ (0.02 mg/kg) rather than the LOD.

[b] DALA = Days after last application until harvest

In a field rotational sugarcane study (Krolski *et al*., 2012; Report RARVP030-02), a single broadcast application at a target rate of 840 g ai/ha was made to bare soil at six sites. Sugarcane was planted into the treated soil 13–14 days after application. Sugarcane stalks were harvested at crop maturity and shipped, frozen, to the analytical laboratory. Samples were homogenized in the presence of dry ice and returned to frozen storage. Samples were stored frozen for up to 590 days prior to analysis of spiromesifen and metabolite residues. Residues of were extracted from hydrated sugarcane stalk "by vortexing in the presence of ACN. QuCHERS salts were added, and the samples were vortexed again." After centrifugation, an aliquot of the supernatant was diluted, filtered, and then analysed by LC-MS/MS. The method did not include hydrolysis procedures necessary to determine the glucoside conjugates of the 4-hydroxymethyl-Sp-enol metabolite.

Residues of spiromesifen, the Sp–enol metabolite, and the 4-hydroxymethyl-Sp-enol metabolite (no conjugates) were each < 0.01 mg/kg (< 0.03 mg/kg total) in all samples of sugarcane stalks planted 14 days after application of spiromesifen.

In a field rotational alfalfa study (Beedle, 2002; Report 200112), three broadcast applications, each at ca. 270–290 g ai/ha, were made to bare soil on a 0- to 13-day interval to 12 sites. Alfalfa was planted into the treated soil 24 to 31 DALA. Alfalfa forage and hay were collected from the first cutting at normal harvest times and shipped, frozen, to the analytical laboratory. Upon receipt at the laboratory, the samples were homogenized in the presence of dry ice and returned to frozen storage. Samples were stored frozen for up to 649 days prior to analysis of spiromesifen and metabolite residues. Residues of spiromesifen, Sp-enol, and 4-hydroxymethyl-Sp-enol (including glucoside conjugates) were determined using the same analytical methods described for rotational onions, above.

Spiromesifen and the Sp–enol metabolite were < LOD in all forage and hay samples. The maximum summed residue of spiromesifen, the Sp-enol metabolite, and the 4-hydroxymethyl-Sp-enol metabolite in alfalfa forage was 0.85 mg/kg (22% dry matter; equivalent to 1.35 mg/kg at 35% dry matter). The maximum summed residue in alfalfa hay was 2.2 mg/kg (70% dry matter; equivalent to 2.8 mg/kg at 89% dry matter; Table 12).

Table 12 Residues of spiromesifen in rotational alfalfa planted ca. 30 days after application to bare soil

					Residue, mg/kg [mean] [a]			
Trial ID and Location (Year)	Applic., g ai/ha	Variety	DALA [b]	Commodity sampled	Spiromesifen	Sp-enol	4-hydroxy-methyl-Sp-enol	Total
BS114-00R North Rose, NY US 2000	286 + 278 + 274	Centurion	146	Forage	< 0.001, < 0.001 [< 0.01]	< 0.002, < 0.002 [< 0.01]	0.0212, 0.0185 [0.0198]	0.021, < 0.02 [< 0.021]
				Hay	< 0.005, < 0.005 [< 0.005]	< 0.006, < 0.006 [< 0.01]	0.0321, 0.0346 [0.0334]	0.032, 0.035 [0.034]
BS115-00R Suffolk, VA US 2000	281 + 283 + 281	Clean Sweep 1000	303	Forage	< 0.001, < 0.001 [< 0.01]	< 0.002, < 0.002 [< 0.01]	0.0051, 0.0054 [< 0.01]	< 0.02, < 0.02 [< 0.02]
				Hay	< 0.005, < 0.005 [< 0.01]	< 0.006, < 0.006 [< 0.01]	< 0.013, < 0.013 [< 0.013]	< 0.02, < 0.02 [< 0.02]
BS116-00R Oxford, ID US 2000	284 + 283 + 286	CPS 939	299	Forage	< 0.001, < 0.001 [< 0.01]	< 0.002, < 0.002 [< 0.01]	< 0.004, < 0.004 [< 0.01]	< 0.02, < 0.02 [< 0.02]
				Hay	< 0.005, < 0.005 [< 0.01]	< 0.006, < 0.006 [< 0.01]	0.0157, < 0.013 [< 0.014]	< 0.02, < 0.02 [< 0.02]
BS117-00R York, NE US 2000	280 + 281 + 279	Alfaleaf II	78	Forage	< 0.001, < 0.001 [< 0.01]	< 0.002, < 0.002 [< 0.01]	0.2139, 0.2197 [0.217]	0.214, 0.220 [0.217]
				Hay	< 0.005, < 0.005 [< 0.01]	< 0.006, < 0.006 [< 0.01]	0.6747, 0.6072 [0.641]	0.675, 0.607 [0.641]
BS118-00R Stilwell, KSUS 2000	282 + 280 + 280	Kanza	379	Forage	< 0.001, < 0.001 [< 0.01]	< 0.002, < 0.002 [< 0.01]	< 0.004, 0.006 [< 0.01]	< 0.02, < 0.02 [< 0.02]
				Hay	< 0.005, < 0.005 [< 0.01]	< 0.006, < 0.006 [< 0.01]	< 0.013, < 0.013 [< 0.013]	< 0.02, < 0.02 [< 0.02]
BS119-00R New Holland, OH US 2000	271 + 274 + 281	Vernal	80	Forage	0.001, < 0.001 [< 0.01]	< 0.002, < 0.002 [< 0.01]	0.0445, 0.0508 [0.0476]	0.044, 0.051 [0.048]
				Hay	< 0.005, < 0.005 [< 0.01]	< 0.006, < 0.006 [< 0.01]	0.1079, 0.1212 [0.115]	0.108, 0.121 [0.114]
BS120-00R Smithfield, UT US 2000	281 + 279 + 278	Broadleaf	112	Forage	< 0.001, < 0.001 [< 0.01]	< 0.002, < 0.002 [< 0.01]	0.1025, 0.0685 [0.0855]	0.102, 0.068 [0.085]

					Residue, mg/kg [mean] [a]			
Trial ID and Location (Year)	Applic., g ai/ha	Variety	DALA [b]	Commodity sampled	Spiromesifen	Sp-enol	4-hydroxy-methyl-Sp-enol	Total
				Hay	< 0.005, < 0.005 [< 0.01]	< 0.006, < 0.006 [< 0.01]	0.2149, 0.2241 [0.220]	0.215, 0.224 [0.220]
BS121-00R Fresno, CA US 2000	280 + 277 + 281	Germains WL 525	76	Forage	< 0.001, < 0.001 [< 0.01]	< 0.002, < 0.002 [< 0.01]	0.1456, 0.127 [0.136]	0.146, 0.127 [0.136]
				Hay	< 0.005, < 0.005 [< 0.01]	< 0.006, < 0.006 [< 0.01]	0.4218, 0.3652 [0.394]	0.422, 0.365 [0.393]
BS122-00R Velva, ND US 2000	287 + 284 + 283	Vernal	80	Forage	< 0.001, < 0.001 [< 0.01]	< 0.002, < 0.002 [< 0.01]	0.3905, 0.3380 [0.364]	0.390, 0.338 [0.364]
				Hay	< 0.005, < 0.005 [< 0.01]	< 0.006, < 0.006 [< 0.01]	0.8449, 0.6568 [0.751]	0.845, 0.657 [0.751]
BS123-00R Jerome, ID US 2000	274 + 279 + 281	Pioneer 54Q53-n221	80	Forage	< 0.001, < 0.001 [< 0.01]	< 0.002, < 0.002 [< 0.1]	0.2628, 0.1979 [0.230]	0.263, 0.198 [0.230]
				Hay	< 0.005, < 0.005 [< 0.01]	< 0.006, < 0.006 [< 0.01]	0.7248, 0.8794 [0.802]	0.725, 0.879 [0.802]
BS124-00R Hillsboro, ND US 2000	278 + 281 + 282	TMF 42J	85	Forage	< 0.001, < 0.001 [< 0.01]	< 0.002, < 0.002 [< 0.01]	0.6328, 1.0744 [0.854]	0.633, 1.074 [0.854]
				Hay	< 0.005, < 0.005 [< 0.01]	< 0.006, < 0.006 [< 0.01]	2.1554, 2.2765 [2.22]	2.155, 2.276 [2.22]
BS125-00R Gardner, ND US 2000	287 + 281 + 273	TMF 42J	85	Forage	< 0.001, < 0.001 [< 0.01]	< 0.002, < 0.002 [< 0.01]	0.3748, 0.2796 [0.327]	0.375, 0.280 [0.328]
				Hay	< 0.005, < 0.005 [< 0.01]	< 0.006, < 0.006 [< 0.01]	0.9084, 1.1417 [1.03]	0.921, 1.142 [1.03]

[a] For purposes of calculating the mean, residues reported as < LOD (0.005 to 0.013 mg/kg depending on the analyte) were assumed to be at the LOD. For total residue, residues are reported as being < LOQ (0.02 mg/kg) rather than the LOD.

[b] DALA = Days after last application until harvest

In a study examining residues of spiromesifen in rotational carrot and lettuce grown under greenhouse conditions (Braune 2015, Report 14-2550), spiromesifen was applied four times (216 g ai/ha) on a 10–11-day interval to tomato as the primary crop. The trials were conducted in Belgium, France, Greece, and Italy. After tomato fruits were harvested, the tomato plants were removed, and carrot and lettuce were planted as rotational crops 26 to 35 days after the last application of spiromesifen. Carrot and lettuce samples were harvested at immature and mature stages. In addition, soil samples were collected 16 DALA (Greece) or 26–35 DALA (other locations). Samples were frozen within one day of collection and shipped frozen to the analytical laboratory. Samples were homogenized in the presence of dry ice prior to analysis. Soil samples were stored frozen for up to 271 days prior to analysis of spiromesifen and Sp-enol residues. Plant samples were stored for up to 230 days prior to determination of spiromesifen and Sp-enol, and up to 221 day prior to determination of 4-hydroxymethyl-Sp-enol residues. Residues of spiromesifen, Sp-enol, and 4-hydroxymethyl-Sp-enol (including glucoside conjugates) were determined using the same analytical methods described for rotational onions, above.

Residues of spiromesifen and Sp-enol were < 0.01 mg/kg, and residues of 4-hydroxymethyl-Sp-enol were below the LOQ (0.02 mg/kg) in all carrot samples and most lettuce samples (maximum in lettuce = 0.029 mg/kg; Table 13). Residues of spiromesifen were < 0.01 mg/kg in soils, except for

the sample from Greece (0.02 mg/kg). Residues of Sp-enol in soils ranged from < 0.01 to 0.041 mg/kg.

Table 13 Residues of spiromesifen in soil and rotational carrot and lettuce planted ca. 30 days after application to tomato plants

					Residue, mg/kg [mean] [a]			
Trial ID and Location (Year)	Applic., g ai/ha	Variety	DALA [b]	Commodity sampled	Spiromesifen	Sp-enol	4-hydroxy-methyl-Sp-enol	Total
14-2550-01 Villers-Perwin, Belgium 2014	216 + 216 + 216 + 216	Soil	32	0–30 cm	< 0.01	< 0.01	Not analysed	< 0.02
		Carrot/ Romance	118	Root	< 0.01	< 0.01	< 0.02	< 0.05
			204	Root	< 0.01	< 0.01	< 0.02	< 0.05
		Lettuce/ Sansula loose leaf variety	49	Leaves	< 0.01	< 0.01	0.029	0.06
			60	Leaves	< 0.01	< 0.01	0.022	0.05
14-2550-02 Trani, Italy 2014	216 + 216 + 216 + 216	Soil	26	0–30 cm	< 0.01	0.041	Not analysed	0.051
		Carrot/ Nantes 2-forto	99	Root	< 0.01	< 0.01	< 0.02	< 0.05
			110	Root	< 0.01	< 0.01	< 0.02	< 0.05
		Lettuce/ Silice Crisp head variety	64	Leaves	< 0.01	< 0.01	< 0.02	< 0.05
			74	Leaves	< 0.01	< 0.01	< 0.02	< 0.05
14-2550-03 Toulouse, France 2014	216 + 216 + 216 + 216	Soil	35	0–30 cm	< 0.01	0.018	Not analysed	< 0.03
		Carrot/ Chambord F1	119	Root	< 0.01	< 0.01	< 0.02	< 0.05
			133	Root	< 0.01	< 0.01	< 0.02	< 0.05
		Lettuce/ Millennia Butter head variety	67	Leaves	< 0.01	< 0.01	0.028	0.06
			81	Leaves	< 0.01	< 0.01	< 0.02	< 0.05
14-2550-04 Aronas Katerini, Greece 2014	194 + 216 + 227 + 238	Soil	16	0–30 cm	0.020	0.033	Not analysed	0.053
		Carrot/ Nantes 2	119	Root	< 0.01	< 0.01	< 0.02	< 0.05
			129	Root	< 0.01	< 0.01	< 0.02	< 0.05
		Lettuce/ Manchester loose leaf variety	73	Leaves	< 0.01	< 0.01	< 0.02	< 0.05
			84	Leaves	< 0.01	< 0.01	< 0.02	< 0.05

[a] For purposes of calculating the mean, residues reported as < LOD (0.005 to 0.013 mg/kg depending on the analyte) were assumed to be at the LOD. For total residue, residues are in terms of spiromesifen and are reported as being <LOQ (0.02 mg/kg) rather than the LOD.

[b] DALA = Days after last application until harvest

In studies examining spiromesifen residues in soil and rotational tomatoes in greenhouses in Spain (Schöning and Wolters 2005, Reports RA-2200/04 and RA-2201/04), four applications of spiromesifen, each at 216 g ai/ha, were made to growing tomatoes on an 8- or 10-day retreatment interval (RA-2200/04) or a 10- or 11-day retreatment interval (RA-2201/04). In Study RA-2200/04, the soil profile was set to model the Almeria area of Spain, where ca. 20 cm of soil is used to level the stony ground, the soil is covered by a layer of dung (2–3 cm) which, in turn, is covered by a 10 cm layer of gravelly sand. Natural soil was used in the second study. Tomatoes were harvested three days after the last application and discarded. New tomato plants were planted into the treated plots and soil samples were collected beginning ca. 30 days (RA-2200/04) or ca. 45 days (RA-2201/04) after the last application and extending out to 140 days (RA-2200/04) or 127 days (RA-2201/04). Soil samples were frozen within 24 hours of collection and shipped, frozen, to the analytical laboratory. Rotational tomatoes were harvested at maturity, were frozen within 24 hours of collection and shipped, frozen, to the analytical laboratory. Prior to extraction, soil samples were stored for up to 240 days and tomato samples were stored for 149 days. Soils were analysed for residues of spiromesifen, Sp-enol, and 4-hydroxymethyl-Sp-enol; tomato fruit samples were analysed for residues of 4-hydroxymethyl-Sp-enol only. Analysis of 4-hydroxymethyl-Sp-enol included a hydrolysis step to quantify the conjugated forms of that metabolite.

Residues of 4-hydroxymethyl-Sp-enol were < 0.01 mg/kg in tomato fruits from both studies (n = 4). Residues of spiromesifen, Sp-enol, and 4-hydroxymethyl-Sp-enol were < 0.01 in the lower soil layers (0–10 cm and 10–20 cm in RA-2200/04, and 10–20 cm and 20–30 cm in RA-2201/04). In the gravelly sand layer (RA-2200/04), spiromesifen residues ranged from < 0.01 mg/kg to 0.14 mg/kg, with the higher concentrations occurring at the 30-DALA and 45-DALA samples. Residues of Sp-enol in the gravelly sand ranged from 0.014 mg/kg to 0.24 mg/kg and remained relatively constant throughout the 30 to 140 DALA sampling period. In the top soil layer from Study RA-2201/04, spiromesifen residues ranged from < 0.01 mg/kg to 0.089 mg/kg and were all < 0.01 mg/kg by the 127-DALA sampling interval. Residues of Sp-enol ranged from 0.018 mg/kg to 0.17 mg/kg and showed a slight trend for higher residues at the 127-DALA sampling interval.

Conclusions—Rotational crops

Parent spiromesifen was < 0.01 mg/kg in all rotational crop samples and, therefore, was not a major residue in rotational crops. While the free and conjugated forms of the 4-hydroxymethyl metabolite tended to be the major residue in rotational crops, the overall residue profile in rotational crops is dependent on the elapsed time since the last application of spiromesifen. Most the radioactivity in the PES, which tended to be < 10% of the extracted residues from the confined rotational crop study was incorporated into naturally occurring plant constituents.

Animal metabolism

The Meeting received metabolism studies on laboratory animals, lactating goats, and laying hens. All the studies were conducted with spiromesifen radiolabelled at the 3-dihydrofuranone moiety.

Laboratory animals

Metabolism in laboratory animals was evaluated by the WHO Panel of the 2016 Meeting.

Lactating goats

The metabolism of spiromesifen in lactating goats was investigated by Dean (2001, Study BAG 290/992828). In this study, a target dose of 10 mg/kg body weight/day was given to a single goat on three consecutive days via gavage. The dose was approximately equivalent to 344 ppm in the diet. Milk was collected twice daily during the dosing period, and the goat was sacrificed ca. six hours after the final dose.

Total radioactive residues were determined by LSC directly for bile, urine, cage wash, and milk, and by combustion followed by LSC for faeces, blood, and tissues. For determination of residues, bile and urine samples were analysed directly using TLC and HPLC. Liver, kidney, and

muscle samples were extracted with ACN and ACN:water (1:1, v/v), and milk and fat samples were extracted with hexane followed by acetonitrile. Milk and tissue extracts were analysed for residues by TLC and HPLC. Additional work was done on bile samples, which were incubated with ß-glucuronidase or hydrolysed with acid (1 M HCl) or base (1 M NaOH).

Of the applied dose (AD), 50% was recovered; approximately 33% was found in urine, 15% in faeces, and 1% in the cage wash. Less than 1% of the AD was found in tissues and was distributed as follows: fat 0.20%, kidney 0.10%, liver 0.31%, milk 0.02%, and muscle 0.23%.

In milk, TRR may have reached a plateau by the third dosing based on the levels measured in the afternoon collections (Table 14); however, the short duration of the study prohibits a definitive conclusion. Total radioactive residues in other matrices are summarized in Table 15. Of the edible tissues, concentrations were highest in kidney and liver, and approximately two-fold greater in fat than in muscle.

Table 14 Total radioactive residues of spiromesifen in milk of a lactating goat following dosing for three days at 10 mg/kg body weight/day

Sampling time (hours after first dose)	Total radioactive residue, mg eq./L		
	Afternoon collection	Morning collection [a]	Daily mean
0–24	0.08	0.09	0.08
24–48	0.14	0.09	0.11
48–54	0.16	n.s.[b]	–

[a] Collected prior to administration of daily dose

[b] Not sampled

Table 15 Total radioactive residues and extraction efficiency of spiromesifen in tissues and body fluids of a lactating goat following dosing for three days at 10 mg/kg body weight/day

		Extraction efficiency, % of TRR by combustion + LSC			
Organ / Tissue	Residue levels, mg eq./kg	Hexane	ACN	ACN/H_2O	Total
Liver	3.82	–	62.8	34.2	97.0
Kidney	8.41	–	89.5	9.2	98.7
Round muscle	0.22	–	85.8	11.2	97.0
Flank muscle	0.26				
Loin muscle	0.21				
Subcutaneous fat	0.44	55.6	39.9	4.5	100
Perirenal fat	0.57				
Omental fat	0.48				
Milk	0.10	8.6	85.0	4.5	98.1
Whole blood	0.98	Not extracted			
Bile	78.9				

Table 16 Profile of radioactive residues in lactating goat following administration of [^{14}C]spiromesifen for three days at 10 mg/kg body weight/day

Residue	Liver		Kidney		Fat [a]		Muscle [a]		Milk [a]	
	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/L
TRR (mg/kg)	3.59		8.92		0.47		0.26		0.11	
Spiromesifen	< 0.8 [b]	< 0.03	– [b]	–	9.2 [b]	0.04	0.7 [b]	< 0.01	32.8 [b]	0.03
Sp-enol	29.1	1.04	77.2	6.90	64.5	0.30	71.5	0.17		
4-hydroxymethyl-Sp-enol	5.9	0.21	7.5	0.67	–	–	6.3	0.02	24.3	0.02
4- or 2-hydroxy methyl-3-hydroxy-Sp-enol [e]	1.4	0.05	–	–	–	–	–	–	–	–
4-carboxy-3-hydroxy-Sp-enol [e]			–	–	–	–	–	–	–	–

Residue	Liver		Kidney		Fat [a]		Muscle [a]		Milk [a]	
	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/L
3-hydroxy-Sp-enol	6.4	0.24	1.8	0.16	–	–	1.1	< 0.01	–	–
4-carboxy-Sp-enol	2.1	0.07	–	–	–	–	2.3	< 0.01	9.2	0.01
2-hydroxymethyl-Sp-enol			2.0	0.18	–	–				
Sp-enol-glucuronide	21.4	0.77	1.3	0.12	–	–	–	–	–	–
4-hydroxymethyl-glucuronide-Sp-enol	9.3	0.33	1.1	0.10	–	–	–	–	–	–
Others [d]	4.7	0.17	7.4	0.66	13.6	0.06	7.7	0.02	23.8	0.02
Sum identified	75.6	2.71	90.9	8.13	73.7	0.40	81.9	0.21	66.3	0.8
Unidentified	10.2	0.36	1.6	0.14	12.7	0.08	1.2	< 0.01	–	–
Extracted	90.5	3.25	99.9	8.92	99.8	0.47	90.7	0.24	90.0	0.10
Not extracted	9.5	0.34	0.1	0.01	0.02	< 0.01	2.3	< 0.01	2.9	< 0.01
Total [c]	100.0	3.59	100.0	8.92	100.0	0.47	100.0	0.26	100.0	0.11

[a] Composite samples

[b] In additional samples analysed ca. 2 months after sacrifice and ca. 10–11 months after sacrifice, % TRR were as follows:

	Liver	Kidney	Fat	Muscle	Milk
Spiromesifen (2 months/11 months)	4.4/3.5	1.5/68.3	22.8/11.9	1.7/1.1	28.6/13.5
Sp-enol (2 months/11months)	34.6/26.9	77/?	47.2/60.4	70.3/68.8	17.8/21.1

Percent TRR (2 months/11 months) were: liver =

[c] Any lack of correspondence between the sum of the individual values and the "total" values is due to rounding.

[d] Radioactivity not in discrete fractions

[e] Tentatively identified

Extracts of fat, muscle, and milk samples were reanalysed ca. 11 months after sacrifice due to a lack of chromatographic resolution between same residue components. Storage stability showed some degradation of the parent compound to the enol metabolite during that time frame. Based on the initial analysis, spiromesifen was a major residue in milk only (29% TRR, 0.03 mg/L). In other goat matrices, the Sp–enol metabolite was the only major residue (plus the 4-hydroxymethyl glucuronide-Sp-enol metabolite in liver). The proposed metabolic pathway in goats is summarized in Figure 4.

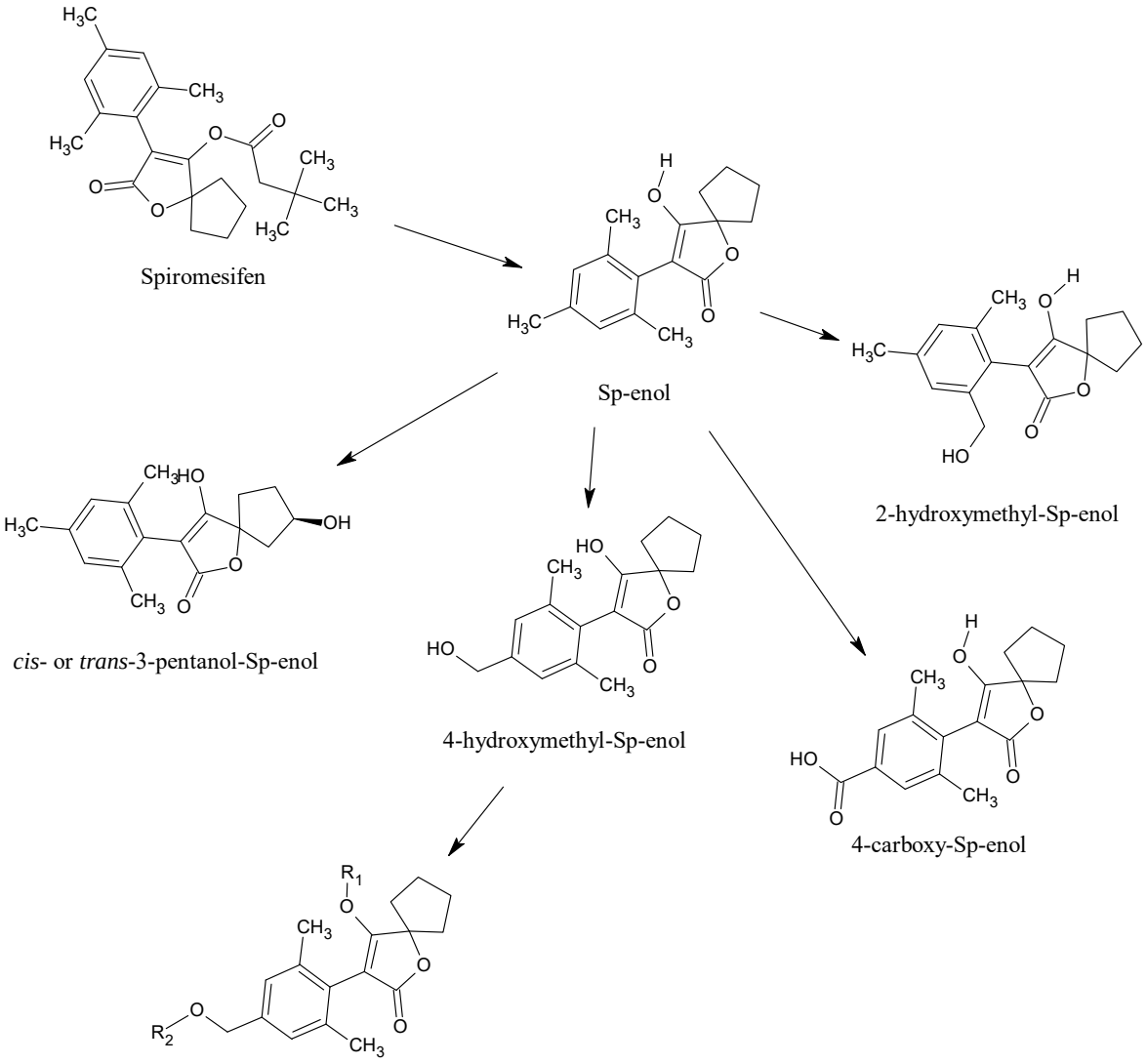


Figure 4 Proposed metabolic pathway of spiromesifen in lactating goat

Laying hens

The metabolism of spiromesifen in laying hens was investigated by Corden (2001, Study BAG 327/003887). Seven laying hens were given a target dose of 10 mg/kg body weight/day on three consecutive days via gavage. The dose was equivalent to 190 ppm in the feed. Cage washings, excreta and eggs were collected daily. Hens were sacrificed ca. five hours after the final dose, and tissues (liver, breast muscle, thigh muscle, fat, skin, and partially formed eggs) were harvested. Tissue and egg samples were homogenized prior to freezing for later analysis.

Total radioactive residues were measured by LSC for samples of cage wash and by combustion/LSC for tissues, eggs, and excreta. For characterization and identification of residues, eggs, fat, and skin were extracted, sequentially with hexane and ACN; skin was additionally extracted with ACN:H_2O (1:1, v/v) followed by water. For eggs, fat, and skin, the hexane extracts were partitioned against ACN; similarly, the original ACN extracts were partitioned against hexane. The extracts from each solvent were then pooled (i.e., original hexane plus partitioned hexane). For fat, a portion of the post-extraction solids was treated with either 0.1 M HCl, 0.1 M NaOH, or protease

enzyme to further characterize the residues. Samples of excreta, liver, and muscle were extracted, sequentially with ACN and ACN:H_2O (1:1, v/v). The level of radioactivity in the various extracts was determined by LSC, and in the post-extraction solids by combustion/LSC. Extracts were analysed by TLC and HPLC. In the case of liver, aliquots of extract were incubated with acid or with ß-glucuronidase to evaluate the presence of conjugated residues. Furthermore, portions of the liver extract were partitioned against ethyl acetate and analysed by LC-MS to further characterize the nature of those extracts.

Of the applied dose (AD), 63% was recovered; approximately 58% was found in excreta and 4% in the cage wash. Approximately 0.2% of the AD was found in tissues and was distributed as follows: fat, skin, and eggs < 0.1%; and liver and muscle 0.1%.

In eggs, TRR did not appear to have reached a plateau by the third dosing (Table 17); however, the short duration of the study prohibits a definitive conclusion. The TRR in partially formed eggs at 53 hours after the initial dosing was 0.13 mg eq/kg, and the weight of the partially formed eggs was like that of the laid eggs, indicating that residues in eggs may have still been increasing. Total radioactive residues in other matrices are summarized in Table 18. Of the edible tissues, concentrations were highest in liver and skin, and comparable in fat and muscle.

Table 17 Total radioactive residues of spiromesifen in eggs of laying hens following dosing for three days at 10 mg/kg body weight/day

Sampling time (hours after first dose)	Total radioactive residue, mg eq/kg
0–24	< 0.006
24–48	0.020
48–53	0.032

Table 18 Total radioactive residues and extraction efficiency of spiromesifen in tissues and excreta of laying hens following dosing for three days at 10 mg/kg body weight/day

		Extraction efficiency, % of TRR by combustion/LSC				
Organ / Tissue	Residue levels, mg eq./kg	Hexane	ACN	ACN/H_2O	H2O	Total
Egg (Days 2 & 3)	0.026	–	77.0	–	–	77.0
Fat	0.090	3.0	81.4	–	–	84.4
Liver	1.68	–	50.6	45.2	–	95.8
Skin	0.32	0.6	57.0	32.0	7.5	97.1
Muscle	0.067	–	70.3	16.8	–	87.1
Excreta (Day 3)	211	–	94.0	5.5	–	99.5

Table 19 Profile of radioactive residues in laying hens following administration of [^{14}C]spiromesifen for three days at 10 mg/kg body weight/day

Residue	Muscle [a]		Fat		Skin		Liver		Egg [a]	
	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/L
TRR (mg/kg)	0.067		0.090		0.32		1.68		0.026	
Spiromesifen	3.7	0.002	51.2	0.046	< 0.4	< 0.01	< 0.3	< 0.01	28.4	0.007
Sp-enol	38.5	0.026	3.7	0.003	18.0	0.06	17.6	0.30	43.6	0.011
4-hydroxymethyl-Sp-enol	5.9	0.004	1.8	0.002	7.7	0.02	11.1	0.19	0.8	< 0.001
4- or 2-hydroxymethyl-3-pentanol-Sp-enol	2.4	0.002	2.0	0.002	2.5	0.01	4.8	0.08	0.8	< 0.001
4-carboxy-3-hydroxy-Sp-enol	3.6 [e]	0.002	2.4 [e]	0.002	41.9 [e]	0.13	20.0	0.34	< 0.6 [e]	< 0.001
Oxo-cyclopentyl-Sp-enol							4.6	0.08		
Sp-enol-glucuronide	2.2	0.001	< 1.1	< 0.001	0.8	< 0.01	3.0	0.05	< 0.6	< 0.001
4-carboxy-hydroxy-Sp-enol	2.4	0.002	1.0	0.001	12.5	0.04	15.7	0.26	< 0.6	< 0.001
2-hydroxymethyl-Sp-enol	1.8	0.001	1.2	0.001	6.4	0.02	4.8	0.08	< 0.6	< 0.001
4-carboxy-dihydroxy-Sp-enol	9.1	0.006	2.8	0.003	2.1	0.01	4.3	0.07	0.8	< 0.001

Residue	Muscle [a]		Fat		Skin		Liver		Egg [a]	
	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/L
TRR (mg/kg)	0.067		0.090		0.32		1.68		0.026	
Others [d]	3.6	0.002	9.0	0.008	3.5	0.01	3.3	0.06	1.9	< 0.001
Sum identified [c]	69.6	0.046	77.2	0.069	95.9	0.31	89.0	1.49	74.4	0.019
Unidentified	13.9	0.009	4.2	0.004	0.6	< 0.01	3.5	0.06	0.7	< 0.001
Extracted	87.1	0.058	81.4	0.073	96.5	0.31	95.8	1.61	77.0	0.020
Not extracted	12.9	0.009	15.6[2]	0.014	2.8	0.01	4.2	0.07	23.1	0.006
Total	100	0.067	97.0	0.087	99.3	0.32	100	1.68	100.1	0.026

[a] Composite samples

[b] Includes protease-released residues

[c] Any lack of correspondence between the sum of the individual values and the "total" values is due to rounding

[d] Radioactivity not in discrete fractions

[e] Not resolved

Spiromesifen was a major residue in egg and fat only (Table 19). Sp-enol was a major residue in muscle, skin, liver, and egg. No other major residues were identified in muscle, fat, or egg. In skin, other residues which occurred at > 10% TRR and > 0.01 mg/kg were unresolved peaks consisting of the 4-carboxy-3-hydroxy-Sp-enol and oxo-cyclopentyl-Sp-enol metabolites and the 4-carboxy-hydroxy-Sp-enol metabolite. In liver, the other major residues were the 4-hydroxymethyl-Sp-enol metabolite, the 4-carboxy-3-hydroxy-Sp-enol metabolite, and the 4-carboxy-hydroxy-Sp-enol metabolite. The proposed metabolic pathway in laying hens is shown in Figure 5.

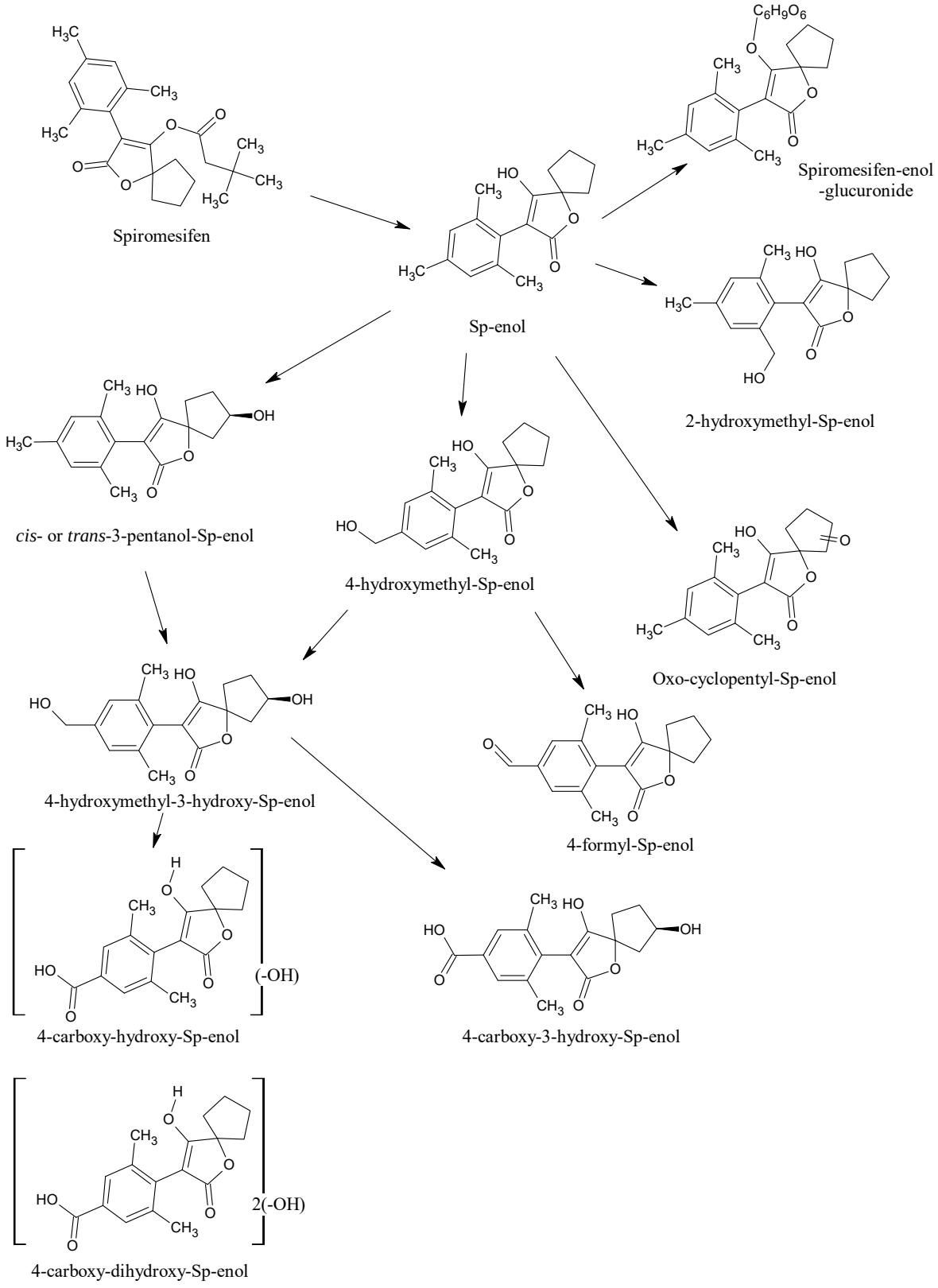


Figure 5 Proposed metabolic pathway of spiromesifen in laying hens

Conclusions—Animals

Metabolism of spiromesifen in animals is generally similar, with desterification leading to spiromesifen-enol, followed by multiple oxidations to the alkane portions of the molecule. Some differences, however, are noted. While both goats and hens form glucuronide conjugates of metabolized spiromesifen, the main conjugate in the hen was identified as the spiromesifen-enol compound whereas in goats it was the 4-hydroxymethyl-enol compound, reflecting an additional oxidation step; glucuronide conjugates were not identified in rats.

Spiromesifen plus spiromesifen-enol (free and glucuronide conjugate) made up most residues in all matrices except poultry skin and liver. In those matrices, the principal residues were comprised of carboxylated and hydroxylated forms of spiromesifen-enol.

Environmental fate

The Meeting received studies depicting the environmental fate of spiromesifen in soils and in water. Soil studies included laboratory and field dissipation studies and a photolysis study. Aqueous studies included hydrolysis and photolysis.

Aqueous hydrolysis and photolysis

Hydrolysis of spiromesifen in aqueous buffers (pH 4, 7, and 9) was investigated by Arthur *et al.* (2001, rev. 2005, Study 109481-1). Spiromesifen, radiolabelled at the 3-dihydrofuranone moiety, was incubated at 25 °C or 50 °C for varying time periods, depending on temperature and pH, and aliquots of the test system were analysed by HPLC-MS throughout the time course of the study.

At all pH levels and at both temperatures, spiromesifen was hydrolysed to Sp-enol. Hydrolysis was more rapid at the higher temperature and at higher pH levels (Figure 6). When modelled using first-order kinetics, half-lives of spiromesifen ranged from 2.6 hours (pH 9) to 2.2 days (pH 4) at 50 °C and from 4.3 days (pH 9) to 53 days (pH 4) at 25 °C. When modelled at 20 °C, half-lives were 4.8 days at pH 9, 45 days at pH 7, and 107 days at pH 4.

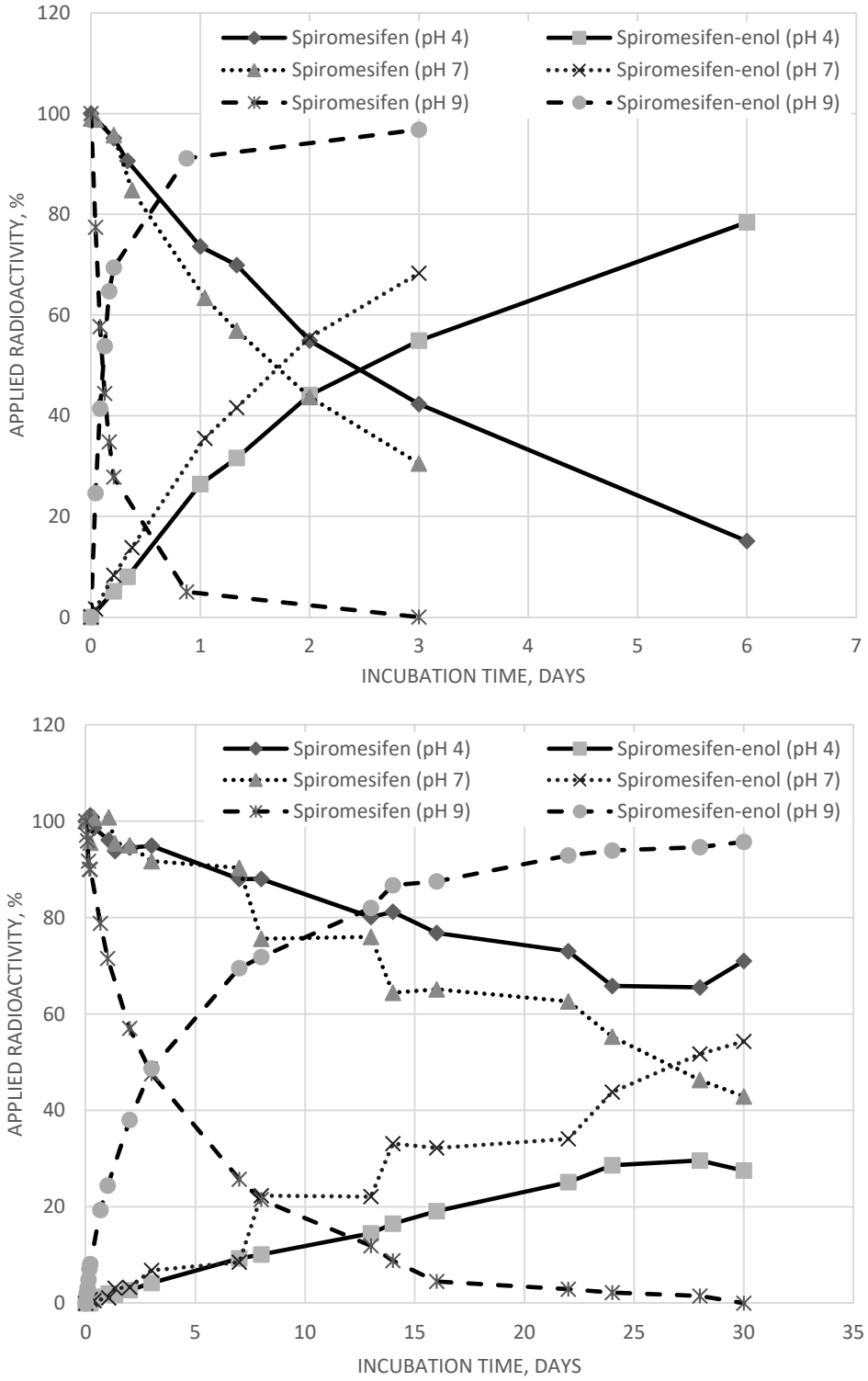


Figure 6 Aqueous hydrolysis of spiromesifen at 50 °C (upper) and 25 °C (lower)

Quantum yield of direct photodegradation of spiromesifen was investigated by Hellpointner (2001; Study MR-120/01) using unlabelled parent compound illuminated with polychromatic light for 480 minutes. Spiromesifen concentration was determined by HPLC. Quantum yield and UV absorption data were used to estimate environmental direct half-lives of 2 to 11 days for the periods of main use.

Based on the results of the hydrolysis study, a photolysis study was also conducted at pH 4 (Arthur and Shepherd; 2001, rev. 2003; Study 110962-1). Radiolabelled spiromesifen

(dihydrofuranone-3-^{14}C) in pH 4 acetate buffer was irradiated for 120 hours under conditions mimicking midsummer sunlight at 40° latitude. The test system was maintained at 25 °C and samples were taken for HPLC followed by LC-MS/MS or NMR analysis at varying intervals from 0 to 5 days post-treatment. Photolytic loss of spiromesifen was accompanied by increases in Sp-enol, a spiromesifen-cyclobutyl photoisomer, and an Sp-enol photoisomer (Table 20). Based on results from the dark control samples, the study indicates that most the dissipation was due to photolysis.

Table 20 Photolysis of spiromesifen at pH 4

Irradiation time, days	Spiromesifen	Sp-enol	Sp-cyclo-butyl photoisomer	Sp-enol photoisomer	Unknown	Others	Total
0	100.0	0.0	0.0	0.0	0.0	0.0	100.0
0.1	91.2	0.4	1.2	0.0	2.5	0.0	95.3
0.2	87.9	0.0	2.4	0.0	5.8	0.0	96.0
1.0	59.0	2.9	10.3	12.3	8.4	0.0	92.9
1.2	58.8	3.0	10.3	12.0	7.8	0.0	91.9
2.0	53.7	4.0	12.8	16.5	7.2	0.0	94.3
3.7	24.9	9.6	28.4	31.0	4.7	1.2	99.8
5.0	11.1	12.3	35.8	36.6	0.0	1.2	97.0
Dark control							
0	100.0	0.0	n.d.	n.d.	n.d.	n.d.	100.0
2	95.5	4.6	n.d.	n.d.	n.d.	1.2	101.2
3	86.7	5.4	n.d.	n.d.	n.d.	n.d.	92.1
6	81.8	8.3	n.d.	n.d.	n.d.	n.d.	90.1
9	79.7	13.9	n.d.	n.d.	n.d.	n.d.	93.5

n.d. = Not detected

A third photolysis study was conducted by Shephard and Arthur (2001; Study 110596) using [cyclopentyl-1-^{14}C]spiromesifen-enol which was continuously irradiated for 9 days under conditions equivalent to approximately 31 solar days at Phoenix, AZ USA. Samples were taken throughout the test period and residues analysed by LC-MS.

Spiromesifen-enol was stable to photolysis under the experimental conditions (100% of applied radioactivity at Day 0 and 93.4% of applied radioactivity at Day 9).

Aerobic soil metabolism and dissipation

The Meeting received studies depicting metabolism of spiromesifen in soil under laboratory conditions (n = 3) and dissipation in the field (n = 4). All studies were conducted under aerobic conditions. For the laboratory studies, each study was conducted with spiromesifen radiolabelled in a different moiety. The study parameters are summarized in Table 21. Residues were extracted with water followed by acetonitrile. Residues were analysed by TLC, HPLC-UV, HPLC-MS, NMR and LC-MS/MS. For the radiolabelled studies, radioactivity was measured by combustion/LSC for solids and by LSC for liquids. CO_2 was trapped in soda lime and analysed by LSC. In the field studies, soils were analysed for residues of spiromesifen, cyclobutyl photoisomer of spiromesifen, Sp-enol, 4-carboxy-Sp-enol, and the photoisomer of Sp-enol. Recoveries from method validation and concurrent recovery samples ranged from 86 to 122% and from 87 to 124%, respectively, across all soils and analytes.

Table 21 Summary of soil metabolism and dissipation studies conducted with spiromesifen

Study ID	Radiolabel position	Application, g ai/ha	Soil types	Soil moisture, % of water holding capacity at 1/3 bar	Conditions
Laboratory Studies					
Brumhard and Elke; 2001; Study MR229/01	Dihydro-furanone-3-^{14}C	1× 180	Claude (silty clay loam) Fresno (sandy loam) Höfchen (silt)	75 75 40	Dark, 20 ± 1 °C

Study ID	Radiolabel position	Application, g ai/ha	Soil types	Soil moisture, % of water holding capacity at 1/3 bar	Conditions
			Laacher Hof AXXa (sandy loam)	40	
Ripperger and Hall' 2001; Study 110450	Cyclopentyl-1-^{14}C	1× 900	Fresno (sandy loam)	75	Dark, 20 ± 1 °C
Babczinski; 2001; Study MR-361/00	Phenyl-UL-^{14}C	1× 900	Fresno (sandy loam)	75	Dark, 20 ± 1 °C
Field Studies					
Dyer *et al.*; 2002; Study 110327	unlabelled	3× 330, not incorporated	Levelland (loam)	n.a.	Ambient field
Dyer *et al.*; 2002; Study 110348	unlabelled	3× 330, not incorporated	Fresno (sandy loam)	n.a.	Ambient field
Dyer *et al.*; 2002; Study 110978	unlabelled	3× 330, not incorporated	Hawthorne (sand)	n.a.	Ambient field
Dyer *et al.*; 2002; Study 110986	unlabelled	2× 308, not incorporated	Ephrata (loamy sand)	n.a.	Ambient field

The results of the laboratory studies demonstrate that spiromesifen is rapidly degraded in soil and thoroughly metabolized to the final degradation product carbon dioxide. Major metabolites involved in the degradation are Sp-enol and 4-carboxy-Sp-enol (Table 22). Modelling by using the metabolic pathway of spiromesifen in these soils (Schad; 2001; Study MR-501/01) gives half-life estimates of 5 days for spiromesifen, 22 days for Sp-enol, and 26 days for 4-carboxy-Sp-enol.

In field studies (Dyer *et al.*; 2002; Reports 110327, 110348, 110978, and 110986), first-order half-life estimates based on residues following either three applications at ca. 330 g ai/ha or two applications at 308 g ai/ha indicate that spiromesifen, its enol metabolite, and total residues (spiromesifen, Sp-enol, 4-carboxy Sp-enol, cyclobutyl photoisomer of spiromesifen, and photoisomer of Sp-enol) are not expected to persist (Table 23).

Table 22 Spiromesifen metabolite profile in soil from laboratory studies (Report MR-229/01)

Soil	Days after applic.	Spiromesifen	Sp-enol	Oxo-cyclopentyl-Sp-enol	4-carboxy-Sp-enol	Unknown	$^{14}CO_2$	Extracted	Unextracted	Total
Höfchen	0	80.8	10.4	n.d.	0.4	n.d.	–	95.2	4.8	100.0
	1	64.7	26.7	n.d.	1.4	n.d.	0.5	94.7	6.5	101.7
	3	40.1	43.5	n.d.	3.0	n.d.	2.1	90.2	7.1	99.4
	7	19.8	48.5	1.4	7.3	0.9	7.6	81.6	11.5	100.6
	14	11.0	37.6	1.2	10.6	0.7	19.7	62.7	17.6	100.0
	30	3.1	16.2	1.1	9.4	1.1	47.3	32.5	22.9	102.7
	62	1.4	2.1	0.2	0.8	0.3	67.6	6.0	21.5	95.1
	90	1.0	0.7	0.1	0.1	0.1	71.0	2.6	18.9	92.5
	120	1.0	0.4	n.d.	0.1	n.d.	70.4	2.2	18.7	91.3
Laacher Hof	0	81.6	10.8	n.d.	0.7	n.d.	–	95.7	4.3	100.0
AXXa	1	62.0	30.6	n.d.	1.5	n.d.	0.3	96.1	4.9	101.4
	3	37.2	51.0	n.d.	2.6	n.d.	1.3	94.4	6.3	102.0
	7	15.6	57.5	1.9	6.6	0.7	5.0	86.4	9.9	101.3
	14	7.7	49.3	1.6	10.6	0.7	12.8	72.1	14.8	99.7
	30	2.1	25.3	1.7	11.4	1.4	32.3	44.8	21.9	99.1
	62	1.0	7.7	0.5	5.4	0.7	54.2	17.7	24.1	96.0
	90	0.8	3.6	0.3	2.5	0.4	65.8	8.9	22.9	97.6
	120	0.7	1.8	0.1	0.8	0.4	67.8	4.6	21.2	93.8
Claude	0	86.6	5.1	n.d.	0.4	0.3	–	95.9	4.1	100.0
Texas, USA	1	78.7	11.5	n.d.	1.2	n.d.	0.3	93.1	5.2	98.5
	3	66.1	22.3	n.d.	1.7	n.d.	0.8	93.6	5.6	100.1
	7	49.7	29.7	3.1	3.1	0.5	2.8	89.1	7.2	99.1

Soil	Days after applic.	Spiromesifen	Sp-enol	Oxo-cyclopentyl-Sp-enol	4-carboxy-Sp-enol	Unknown	$^{14}CO_2$	Extracted	Unextracted	Total
	14	37.0	31.8	4.8	4.7	0.5	7.6	80.6	8.8	97.0
	30	17.5	26.8	6.7	7.5	1.8	21.5	63.2	13.4	98.0
	62	5.8	13.1	4.0	7.3	2.0	41.5	35.7	16.8	94.0
	90	3.1	7.2	2.5	6.7	1.5	50.5	23.3	17.7	91.4
	120	1.8	3.7	1.4	5.0	1.4	58.4	15.1	17.4	91.0
	181	0.9	1.3	0.4	3.5	1.1	66.9	7.9	15.8	90.6
	272	0.6	0.8	0.2	2.6	0.4	72.3	5.8	14.6	92.8
	365	0.4	0.8	0.3	1.7	0.7	72.1	4.7	13.9	90.7
Fresno	0	88.4	3.9	n.d.	n.d.	n.d.	–	96.2	3.8	100.0
California	1	82.5	9.3	n.d.	0.9	n.d.	0.2	95.1	4.7	100.0
	3	75.8	17.5	n.d.	1.4	n.d.	0.6	98.9	4.9	104.4
	7	59.0	25.8	1.6	2.2	0.6	2.4	93.1	5.9	101.3
	14	48.0	28.4	2.3	2.8	0.8	7.8	84.1	8.1	100.0
	30	27.4	25.8	3.8	2.5	1.7	24.3	64.0	12.5	100.7
	62	11.0	12.9	2.4	1.7	1.4	46.8	34.0	15.8	96.6
	90	6.6	7.1	1.7	1.2	1.0	62.7	19.7	16.2	98.6
	120	4.0	3.6	1.0	1.1	0.9	64.8	12.4	16.4	93.6
	181	1.6	1.5	0.5	0.6	0.6	78.9	5.8	14.6	99.4
	272	1.0	1.0	0.3	0.5	0.4	77.9	4.5	13.3	95.7
	365	0.7	0.8	0.3	0.4	0.4	79.0	3.7	13.3	96.0

n.d. = Not detected

Table 23 Dissipation kinetic estimates for spiromesifen from field studies

Analyte	Study no.	Trial location	DT_{50}, days	DT_{90}, days	r^2
Spiromesifen	110327	Levelland, Texas, USA	4.6	15.4	0.962
	110348	Fresno, California, USA	2.1	6.9	0.997
	110978	Hawthorne, Florida, USA	6.4	21.1	0.991
	110986	Ephrata, Washington, USA	4.5	15.0	0.997
Spiromesifen-enol	110327	Levelland, Texas, USA	20.7	68.9	0.958
	110348	Fresno, California, USA	16.9	56.2	0.985
	110978	Hawthorne, Florida, USA	5.8	19.4	0.991
	110986	Ephrata, Washington, USA	17.5	58.0	0.995
Total residues	110327	Levelland, Texas, USA	15.2	50.5	0.964
	110348	Fresno, California, USA	13.9	46.3	0.965
	110978	Hawthorne, Florida, USA	6.6	21.8	0.993
	110986	Ephrata, Washington, USA	8.9	29.6	1.000

The proposed metabolic pathway for spiromesifen in soils is shown in Figure .

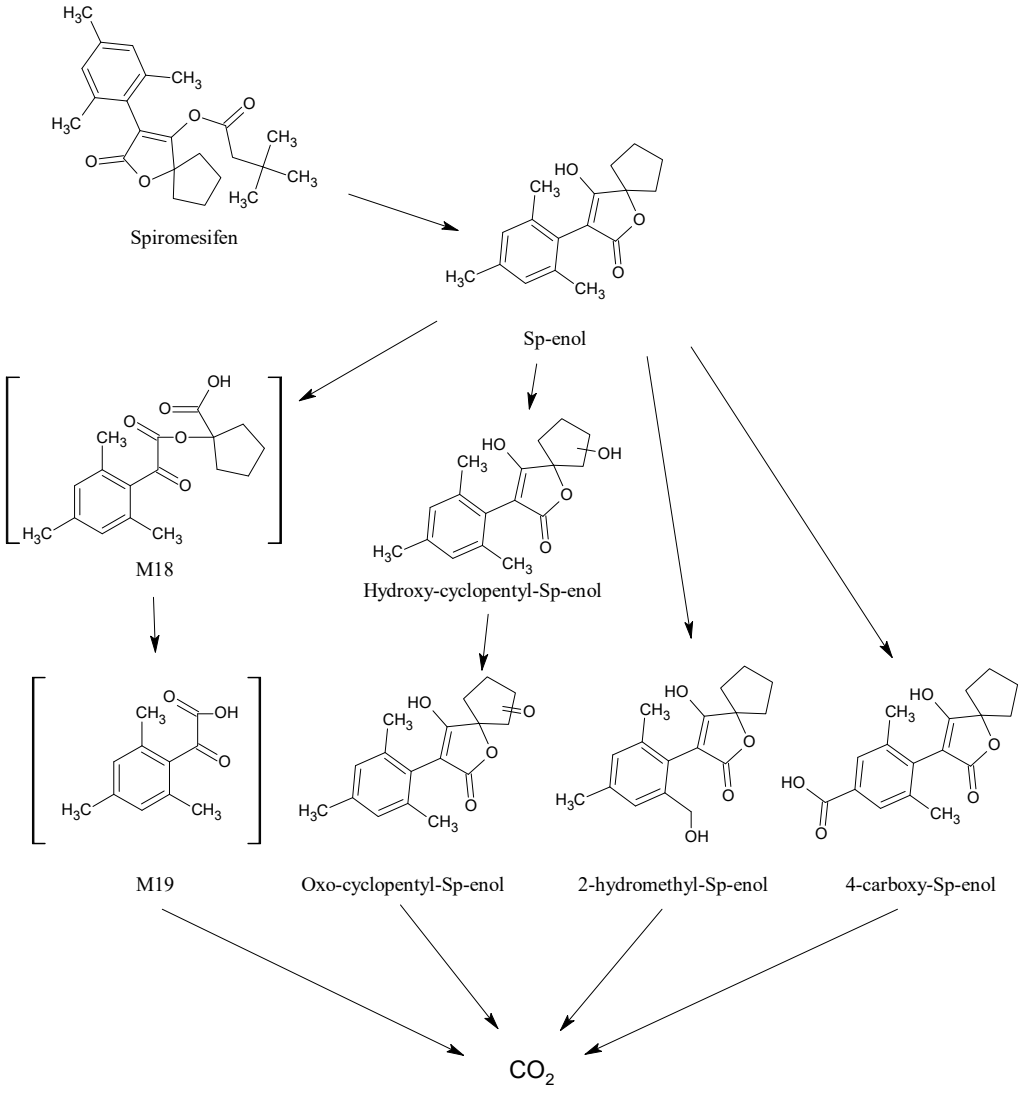


Figure 7 Proposed metabolic pathway of spiromesifen in soil

Conclusions–Environmental fate

Spiromesifen can be expected to dissipate rather rapidly in the environment. It is susceptible to hydrolysis and photolysis, especially under more alkaline conditions. Mineralisation to CO_2 appears to be a significant dissipation pathway in soil.

RESIDUE ANALYSIS

Summary of analytical methods

Table 24 Overview of the analytical methods submitted for spiromesifen and metabolites.

Report ID Method ID	Matrix	Analytes	Extraction	Clean-up	Separation/ Analysis/LOQ [a]
Data Gathering					
MR-190/00 Method 00631	Broccoli, bean, corn, cotton, cucumber, pepper, melon,	Spiromesifen, spiromesifen-enol	ACN:H_2O (4:1, v/v)	For lower-sensitivity HPLC-MS/MS instruments: Partition against cyclohexane/ethyl	HPLC-MS/MS with deuterated internal standards/0.01 mg/kg

Report ID Method ID	Matrix	Analytes	Extraction	Clean-up	Separation/ Analysis/LOQ [a]
	strawberry, sugar beet, tea, tomato			acetate on a ChemElut® column	
MR-193/02 Method 00631/M001	Tomato, corn (green material)	Spiromesifen, spiromesifen-enol	ACN:H_2O (4:1, v/v)	Partition against cyclohexane/ethyl acetate on a ChemElut® column	HPLC-MS/MS with deuterated internal standards/0.01 mg/kg
RABSP012 Method BS001-P09-01	Wheat (grain aspirated grain fractions, bran, flour, germ, middlings, shorts), sorghum (grain, aspirated grain fractions)	Spiromesifen, spiromesifen-enol	ACN:H_2O (4:1, v/v)	Partition against cyclohexane/ethyl acetate on a ChemElut® column	HPLC-MS/MS with deuterated internal standards/0.01 mg/kg
110333, 111035, 200250 Method 00914	Rotational crop matrices	4-hydroxymethyl-Sp-enol (including conjugates)	Water at 125 °C, 1500 psi (ca. 10300 KPa) followed by conc. HCl at 95 °C for 4 hrs.	SCX solid-phase extraction (not grains), C_{-18} solid-phase extraction	HPLC-MS/MS with deuterated internal standards/0.01 mg/kg
PGD-223	Papaya	Spiromesifen	ACN:H_2O (4:1, v/v)	None	HPLC-MS/MS with matrix-matched standards/0.02 mg/kg
MR-07/222 Method 01038	Tea (green and black)	Spiromesifen, spiromesifen-enol	ACN:H_2O (4:1, v/v)	Partition against cyclohexane/ethyl acetate on a ChemElut® column	HPLC-MS/MS with matrix-matched standards/0.02 mg/kg
M-387601-01-2 Method from JPPA (non-GLP)	Tea (green)	Spiromesifen, spiromesifen-enol	ACN:H_2O (4:1, v/v) under addition of 1% HCOOH	Partition against hexane/ethyl acetate and purification on silica gel mini column	HPLC/MS with standards in solvent/0.05 mg/kg
RARVP030-02 Method 00631 (modified)	Sugarcane	Spiromesifen, spiromesifen-enol	ACN:H_2O (4:1, v/v)	C_{-18} solid-phase extraction	HPLC-MS/MS with deuterated internal standards/0.01
110878-01 Method BS110201	Muscle, fat, kidney, liver, milk	Spiromesifen, spiromesifen-enol, 4-hydroxymethyl-Sp-enol (including conjugates)	ACN:H_2O (4:1, v/v) at 70 °C, 1500 psi (ca. 10300 KPa)	Liver and Kidney: SCX followed by conc. HCl (3 hr @ 90 °C), then 3 M NaOH. Fat and Muscle: 6 M NH_4OH (30 min), then 1 M HCl. All matrices: C_{-8} solid-phase extraction	HPLC-MS/MS with deuterated internal standards/0.01
Enforcement					
MR-07/222 Method 01038	Orange fruit, rape seed, tomato fruit, wheat grain	Spiromesifen, spiromesifen-enol	ACN:H_2O (4:1, v/v)	None	HPLC-MS/MS with matrix-matched standards/0.02 mg/kg
M-13/048 Method 01038/M001	Tea (fermented), coffee (green bean and roasted bean)	Spiromesifen, spiromesifen-enol	ACN:H_2O (4:1, v/v)	None	HPLC-MS/MS with matrix-matched standards/0.02 mg/kg
110878-01 Method BS110201	Muscle, fat, kidney, liver, milk	Spiromesifen, spiromesifen-enol, 4-hydroxymethyl-Sp-enol (including conjugates)	ACN:H_2O (4:1, v/v) at 70 °C, 1500 psi (ca. 10300 KPa)	Liver and Kidney: SCX followed by conc. HCl (3 hr @ 90 °C), then 3 M NaOH. Fat and Muscle: 6 M NH_4OH (30 min), then 1 M HCl. All matrices: C_{-8} solid-	HPLC-MS/MS with deuterated internal standards/ 0.005 mg/kg (milk), 0.01 mg/kg (muscle, fat), 0.05 mg/kg (kidney, liver)

Report ID Method ID	Matrix	Analytes	Extraction	Clean-up	Separation/ Analysis/LOQ [a]
				phase extraction	

[a] Defined by the lowest limit of method validation

Plant materials

Methods for the analysis of spiromesifen and metabolites used in the rotational crop residue trials generally fall into two groups: those that use a hydrolysis step to capture residues of free and conjugated 4-hydroxymethyl-Sp-enol and those that do not. Aside from that, the methods are all very similar and consist of extraction with an acetonitrile-based solvent and analysis by HPLC-MS/MS. Some methods include clean-up steps and some include the use of deuterated internal standards. The methods are summarized in Table 24. Variations to the method that are not shown in Table 24 include timing of the addition of internal standards, modifications to HPLC solvent gradient or characteristics, changes in injection volume, and changes to ion mass transitions being used for quantification and confirmation. Radiovalidation work demonstrated that the solvents used in the analytical methods can extract 95+% of the TRR from the plant metabolism samples.

The initial method, Method 00631, did not use a clean-up step if analysis was occurring on an HPLC-MS/MS instrument with high sensitivity. For other instruments, a clean-up and concentration step was added just after extraction, whereby formic acid was added to the extract and residues were partitioned onto a ChemElut® column and eluted with cyclohexane/ethyl acetate. The eluate was evaporated to dryness, deuterated internal standards were added, and the residues were reconstituted with ACN:H_2O (2:3, v/v). Mass transitions for quantification were m/z [M + H] + 371 → 273 for spiromesifen and m/z 273 → 187 for spiromesifen-enol. Recovery of spiromesifen at fortifications of 0.01 mg/kg and 0.10 mg/kg ranged from 60% to 118% (mean: 92%, relative standard deviation (RSD): 8.4%, n = 292; Tables 25 and 27). For spiromesifen-enol the recoveries ranged from 63% to 106% (mean: 93%, RSD: 6.8%, n = 292; Table 26 and Table 28). Modifications to Method 00631, resulting in Method 00631/M001, were the inclusion of the clean-up step regardless of the instrumentation being used, with the addition of the deuterated internal standard occurring immediately after extraction, an increase in the injection volume, and the addition of mass transitions for residue confirmation: m/z 371 → 255 for spiromesifen and m/z 273 → 227 for Sp-enol. Recoveries from crude and cleaned-up extracts are shown in Table 29 through Table 32.

Table 25 Method validation recoveries for spiromesifen using Method 00631

Sample material	Fortification level, mg/kg	Recovery, %	Mean recovery, %	RSD, %	Mean overall	RSD overall	LOQ, mg/kg
Broccoli (head)	0.01	82, 84, 84, 86, 93	86	5.0	87	3.6	0.01
	0.1	85, 87, 88, 88, 89	87	1.7			
Broccoli (plant)	0.01	86, 88, 91	88	2.8	91	3.8	0.01
	0.1	94, 94, 94	94	0.0			
Bean (bean with pod)	0.01	82, 91, 92	88	6.2	84	9.4	0.01
	0.1	75, 75, 90	80	10.8			
Corn (grain)	0.01	85, 86, 86, 88, 88	87	1.5	86	3.4	0.01
	0.1	78, 86, 87, 87, 87	85	4.6			
Corn (green material)	0.01	86, 89, 94	90	4.5	90	2.9	0.01
	0.1	90, 91, 91	91	0.6			
Corn (straw)	0.01	90, 95, 118	101	14.8	93	14.7	0.01
	0.1	78, 87, 88	84	6.5			
Cotton (seed)	0.01	74, 76, 89, 92, 94	85	11. 0	79	14.1	0.01
	0.1	60, 67, 73, 79, 86	73	13.9			
Cucumber (fruit)	0.01	89, 91, 92	91	1.7	92	3.6	0.01
	0.1	87, 94, 96	92	5.1			
Pepper (fruit)	0.01	87, 88, 91, 92, 101	92	6.0	97	9.1	0.01
	0.1	96, 98, 98, 104, 117	103	8.4			
Melon (fruit)	0.01	84, 87, 87	86	2.0	89	4.3	0.01
	0.1	90, 93, 94	92	2.3			
Melon	0.01	74, 80, 83	79	5.8	83	7.1	0.01

Sample material	Fortification level, mg/kg	Recovery, %	Mean recovery, %	RSD, %	Mean overall	RSD overall	LOQ, mg/kg
(pulp)	0.1	85, 87, 91	88	3.5			
Melon (peel)	0.01	72, 75, 81, 85, 86	80	7.7	80	5.9	0.01
	0.1	77, 77, 83, 83, 84	81	4.3			
Strawberry (fruit)	0.01	82, 85, 85	84	2.1	82	3.4	0.01
	0.1	78, 80, 82	80	2.5			
Strawberry (jam)	0.01	91, 93, 94	93	1.6	90	4.1	0.01
	0.1	85, 86, 89	87	2.4			
Strawberry (preserve)	0.01	92, 92, 94	93	1.2	93	2.3	0.01
	0.1	89, 94, 95	93	3.5			
Sugar beet (body)	0.01	72, 75, 80	76	5.3	78	4.3	0.01
	0.1	78, 80,80	79	1.5			
Sugar beet (leaf)	0.01	86, 90, 93	90	3.9	90	3.1	0.01
	0.1	87, 89, 92	89	2.8			
Tomato (fruit)	0.01	75, 79, 84	79	5.7	82	5.3	0.01
	0.1	84, 84, 87	85	2.0			
Tomato (juice)	0.01	83, 85, 87	85	2.4	83	8.5	0.01
	0.1	74, 75, 92	80	12.6			
Tomato (preserve)	0.01	82, 83, 86	84	2.5	85	5.4	0.01
	0.1	80, 83, 93	85	8.0			
Tomato (puree)	0.01	83, 87, 88	86	3.1	88	3.7	0.01
	0.1	87, 88, 93	89	3.6			
Alfalfa (forage)	0.01	87, 92, 92, 88, 88, 93	90	2.9	90	2.9	0.01
Alfalfa (hay)	0.01	91, 89, 95, 98, 97	94	4.1	94	4.1	0.01
Bean (seed)	0.01 0.05 0.10	80, 75, 88 85, 89, 95 77, 85, 92	81 90 85	8.1 5.6 8.9	85	7.9	0.01
Cabbage (head)	0.01	79, 91, 114, 73, 92, 89	90	15.7	84	17.0	0.01
	2.0	72, 72, 72	72	0			
Cotton (gin by-products)	0.01	83, 78, 79, 70, 84, 99, 88 86, 74, 84	83	10.9	81	7.3	0.01
	10.0		81	7.9			
Lettuce (leaf)	0.01	77, 95, 80, 91, 97	88	10.2	88	10.1	0.01
	10.0	99, 78, 85	87	12.2			
Lettuce (head)	0.01	81, 92, 70, 92, 87, 115, 89	89	15.3	91	13.6	0.01
	5.0	105, 91, 85	94	11.0			
Manioc (root)	0.01	93, 92, 96, 92, 90	93	2.4	95	3.5	0.01
	1.0	101, 97, 94, 96	97	3.0			
Mustard (leaf)	0.01	97, 96, 113, 106, 104, 107	104	6.2	101	5.2	0.01
	0.1	100, 107, 106	104	3.6			
	1.0	97, 102, 97, 100, 104	100	3.1			
	10	93, 99, 97	96	3.2			
Soya bean (seed)	0.01	83, 81, 90, 86, 84	85	4.0	86	3.7	0.01
	0.10	91, 86, 88, 83, 87	87	3.4			
Squash (fruit)	0.01	95, 99, 97, 88, 71, 78	88	12.9	87	10.7	0.01
	0.15	91, 81, 85	84	6.7			
Spinach (leaf)	0.01	88, 81, 88, 114, 85, 91, 88, 82, 115, 105	94	13.6	90	14.4	0.01
	10.0	78, 89, 72, 81	80	8.8			
Potato (tuber)	0.01	90, 92, 100, 97, 76, 82	90	10.1	91	8.4	0.01
	0.01	93, 93, 101	96	4.8			
	1.0	92, 84, 96	91	6.7			
Wheat (grain)	0.01	92, 93, 92, 87, 94, 90, 93, 87	91	3.0	91	3.0	0.01
Wheat (forage)	0.01	88, 113, 89, 91, 94, 90, 85	93	10.0	93	10.0	0.01
Wheat (hay)	0.01	90, 98, 107, 90, 93, 85, 87	93	8.1	93	8.1	0.01
Wheat (straw)	0.01	85, 81, 90, 86, 88, 82, 81	85	4.2	85	4.2	0.01

Table 26 Method validation recoveries for spiromesifen-enol using Method 00631

Sample material	Fortification level, mg/kg	Recovery, %	Mean recovery, %	RSD, %	Mean overall	RSD overall	LOQ, mg/kg
Broccoli (head)	0.01	85, 89, 89, 87, 100	92	6.8	93	4.6	0.01
	0.1	92, 94, 94, 94, 94	94	1.0			
Broccoli (plant)	0.01	88, 89, 95	91	4.2	93	3.6	0.01
	0.1	94, 94, 96	95	1.2			
Bean (bean with pod)	0.01	94, 95, 100	96	3.3	94	3.3	0.01
	0.1	91, 92, 94	92	1.7			
Corn (grain)	0.01	91, 92, 93, 94, 98	94	2.9	93	2.2	0.01
	0.1	91, 92, 93, 94, 94	93	1.4			
Corn (green material)	0.01	95, 97, 99	97	2.1	97	2.4	0.01
	0.1	94, 98, 100	97	3.1			
Corn (straw)	0.01	83, 93, 94	90	6.8	92	4.9	0.01
	0.1	92, 92, 96	93	2.5			
Cotton (seed)	0.01	72, 81, 86, 87, 93	84	9.4	78	11.9	0.01
	0.1	63, 68, 73, 77, 78	72	8.8			
Cucumber (fruit)	0.01	84, 98, 99	94	9.0	85	13.3	0.01
	0.1	70, 79, 81	77	7.6			
Pepper (fruit)	0.01	87, 87, 91, 93, 94	90	3.6	81	12.4	0.01
	0.1	69, 70, 71, 75, 76	72	4.3			
Melon (fruit)	0.01	92, 94, 95	94	1.6	94	2.9	0.01
	0.1	92, 92, 99	94	4.3			
Melon (pulp)	0.01	84, 84, 96	88	7.9	90	5.5	0.01
	0.1	92, 92, 93	92	0.6			
Melon (peel)	0.01	89, 91, 92, 94, 95	92	2.6	92	2.3	0.01
	0.1	90, 91, 91, 93, 95	92	2.2			
Strawberry (fruit)	0.01	88, 90, 95	91	4.0	88	4.9	0.01
	0.1	83, 85, 86	85	1.8			
Strawberry (jam)	0.01	88, 90, 91	90	1.7	87	5.5	0.01
	0.1	80, 81, 89	83	5.9			
Strawberry (preserve)	0.01	84, 85, 87	85	1.8	84	3.2	0.01
	0.1	79, 84, 85	83	3.9			
Sugar beet (body)	0.01	94, 94, 98	95	2.4	95	1.7	0.01
	0.1	94, 94, 95	94	0.6			
Sugar beet (leaf)	0.01	94, 95, 100	96	3.3	95	3.3	0.01
	0.1	92, 92, 97	94	3.1			
Tomato (fruit)	0.01	93, 93, 95	94	1.2	94	1.0	0.01
	0.1	93, 94, 95	94	1.1			
Tomato (juice)	0.01	90, 91, 97	93	4.1	92	4.2	0.01
	0.1	86, 91, 95	91	5.0			
Tomato (preserve)	0.01	94, 94, 98	95	2.4	93	4.2	0.01
	0.1	87, 90, 95	91	4.5			
Tomato (puree)	0.01	95, 97, 101	98	3.1	94	5.0	0.01
	0.1	89, 89, 95	91	3.8			
Alfalfa (forage)	0.01	87, 91, 95, 90, 94, 93	92	3.2	92	3.2	0.01
Alfalfa (hay)	0.01	85, 78, 91, 94, 104	90	10.8	90	10.8	0.01
Cabbage (head)	0.01	95, 95, 114, 88, 87, 99	96	10.2	96	8.0	0.01
Cotton (gin by-products)	0.01	75, 89, 101, 75, 80, 105	88	15.0	92	13.5	0.01
	4.08	101, 94, 105	100	5.6			
Lettuce (leaf)	0.01	103, 113, 100,100, 106	104	5.2	108	6.5	0.01
	0.75	107, 112, 120	113	5.8			
Lettuce (head)	0.01	102, 106, 107, 106, 93, 110, 110	105	5.6	107	6.0	0.01
	0.50	116, 114, 105	112	5.2			
Manioc (root)	0.01	89, 87, 88, 92, 90	89	2.2	92	5.4	0.01
	1.0	103, 97, 92, 94	97	5.0			
Mustard (leaf)	0.01	101, 96, 103, 107, 101, 105, 102	102	3.4	101	3.2	0.01
	0.10	96, 101, 102	100	3.2			

Sample material	Fortification level, mg/kg	Recovery, %	Mean recovery, %	RSD, %	Mean overall	RSD overall	LOQ, mg/kg
	1.0	97, 101, 99, 101, 97	99	2.0			
Soya bean (seed)	0.01	81, 89, 86, 91, 85	86	4.5	87	3.8	0.01
	0.10	91, 84, 88, 90, 86	88	3.3			
Squash (fruit)	0.01	92, 100, 96, 101, 92, 88	95	5.4	96	4.5	0.01
	0.15	99, 96, 97	97	1.6			
Spinach (leaf)	0.01	114, 110, 100, 114, 102, 101, 115, 118, 110, 84	107	9.6	107	8.4	0.01
	3.0	109, 104, 109	107	2.7			
Potato (tuber)	0.01	104, 111, 104, 97, 92, 97	101	6.7	98	6.9	0.01
	0.01	104, 93, 103	100	6.1			
	1.0	95, 87, 94	92	4.7			
Wheat (grain)	0.01	85, 88, 87, 89, 90, 85, 92, 87	88	2.9	88	2.9	0.01
Wheat (forage)	0.01	87, 115, 91, 90, 88, 91, 89	93	10.6	93	10.6	0.01
Wheat (hay)	0.01	90, 97, 105, 92, 87, 88, 93	93	6.7	93	6.7	0.01
Wheat (straw)	0.01	85, 83, 88, 82, 85, 81, 84	84	2.7	84	2.7	0.01

Table 27 Results of independent laboratory validation of spiromesifen using Method 00631

Sample material	Fortification level, mg/kg	Recovery, %	Mean recovery, %	RSD, %	Mean overall	RSD overall	LOQ, mg/kg
Tomato (fruit)	0.01	94, 102, 93, 86, 95	94	6.1	93	4.3	0.01
	0.5	94, 91, 92, 93, 92	92	1.2			
Cotton (seed)	0.01	92, 96, 99, 99, 109	99	6.3	95	6.6	0.01
	0.5	87, 95, 88, 93, 94	91	3.6			

Table 28 Results of independent laboratory validation of spiromesifen-enol using Method 00631

Sample material	Fortification level, mg/kg	Recovery, %	Mean recovery, %	RSD, %	Mean overall	RSD overall	LOQ, mg/kg
Tomato (fruit)	0.01	104, 106, 100, 94, 102	101	4.5	103	4.4	0.01
	0.1	105, 109, 101, 109, 101	105	3.8			
Cotton (seed)	0.01	102, 110, 96, 110, 108	105	5.8	103	5.3	0.01
	0.1	103, 105, 101, 98, 95	100	4.0			

Table 29 Method validation recoveries for spiromesifen using crude extracts from Method 00631/M001

Sample material	Fortification level, mg/kg	Recovery, %	Mean recovery, %	RSD, %	Mean overall	RSD overall	LOQ, mg/kg
371 m/z → 273 m/z (quantification)							
Corn (green material)	0.01	86, 91, 72, 86, 90	85	9.0	84	7.9	0.01
	0.1	82, 89, 78, 92, 78	84	7.7			
Tomato (fruit)	0.01	99, 104, 96, 97, 96	98	3.4	97	3.5	0.01
	0.1	96, 91, 99, 94, 97	95	3.2			
371 m/z → 255 m/z (confirmation)							
Corn (green material)	0.01	97, 100, 74, 86, 84	88	11.9	85	10.8	0.01
	0.1	81, 80, 79, 93, 74	81	8.6			
Tomato (fruit)	0.01	89, 86, 89, 87, 88	88	1.5	92	7.4	0.01

Sample material	Fortification level, mg/kg	Recovery, %	Mean recovery, %	RSD, %	Mean overall	RSD overall	LOQ, mg/kg
	0.1	104, 90, 102, 85, 97	96	8.4			

Table 30 Method validation recoveries for spiromesifen-enol using crude extracts from Method 00631/M001

Sample material	Fortification level, mg/kg	Recovery, %	Mean recovery, %	RSD, %	Mean overall	RSD overall	LOQ, mg/kg
273 m/z → 187 m/z (quantification)							
Corn (green material)	0.01	91, 82, 83, 80, 82	84	5.1	83	7.2	0.01
	0.1	81, 84, 75, 96, 80	83	9.4			
Tomato (fruit)	0.01	95, 99, 89, 86, 98	93	6.1	93	6.5	0.01
	0.1	91, 85, 90, 104, 95	93	7.6			
273 m/z → 227 m/z (confirmation)							
Corn (green material)	0.01	104, 89, 72, 85, 73	85	15.5	85	13.3	0.01
	0.1	86, 81, 80, 105, 79	86	12.6			
Tomato (fruit)	0.01	95, 98, 92, 86, 90	92	5.0	93	4.1	0.01
	0.1	91, 93, 94, 98, 97	95	3.0			

Table 31 Method validation recoveries for spiromesifen using cleaned-up extracts from Method 00631/M001

Sample material	Fortification level, mg/kg	Recovery, %	Mean recovery, %	RSD, %	Mean overall	RSD overall	LOQ, mg/kg
371 m/z → 273 m/z (quantification)							
Corn (green material)	0.01	84, 79, 87, 85, 77	82	5.1	84	6.2	0.01
	0.1	85, 89, 85, 93, 77	86	6.9			
Tomato (fruit)	0.01	109, 90, 86, 99, 97	96	9.2	96	6.6	0.01
	0.1	95, 93, 97, 99, 91	95	3.3			
371 m/z → 255 m/z (confirmation)							
Corn (green material)	0.01	87, 82, 71, 83, 68	78	10.5	79	10.2	0.01
	0.1	90, 81, 73, 84, 68	79	11.1			
Tomato (fruit)	0.01	100, 93, 90, 103, 109	99	7.7	98	5.9	0.01
	0.1	96, 91, 101, 97, 98	97	3.8			

Table 32 Method validation recoveries for spiromesifen-enol using cleaned-up extracts from Method 00631/M001

Sample material	Fortification level, mg/kg	Recovery, %	Mean recovery, %	RSD, %	Mean overall	RSD overall	LOQ, mg/kg
273 m/z → 187 m/z (quantification)							
Corn (green material)	0.01	86, 93, 75, 91, 80	85	8.8	83	9.6	0.01
	0.1	82, 82, 75, 92, 70	80	10.4			
Tomato (fruit)	0.01	93, 100, 83, 90, 95	92	6.8	93	5.1	0.01
	0.1	96, 89, 94, 94, 97	94	3.3			
273 m/z → 227 m/z (confirmation)							
Corn (green material)	0.01	82, 73, 72, 77, 75	76	5.2	77	5.0	0.01
	0.1	74, 78, 76, 84, 75	77	5.1			
Tomato (fruit)	0.01	91, 91, 88, 104, 100	95	7.2	96	5.7	0.01
	0.1	101, 92, 95, 97, 102	97	4.3			

Method 00914 was developed for the analysis of free and conjugated 4-hydroxymethyl spiromesifen-enol and was used in the field rotational crop studies. The method uses accelerated solvent extraction with water (125 °C, 1500 psi; ca. 10,300 KPa) to extract residues and acid digestion

at 95 °C for 4 hours with concentrated HCl to hydrolyse conjugates form the free analyte. After hydrolysis, deuterated internal standards are added and the extracts cleaned-up using C_{-18} solid-phase extraction. Analysis is by HPLC-MS/MS using the mass transition of M/z 287 → 207 for quantification and m/z 289 → 259 for confirmation. Results of method validation samples and independent laboratory validation are shown in Tavle 33 through Table 35.

Table 33 Method validation results for 4-hydroxymethyl-Sp-enol using Method 00914

Crop	Limit of quantitation (LOQ)	Recovery fortification level, mg/kg	Recoveries % range (mean)	RSD, % (n)
Wheat (forage)	0.01	0.01 0.02 0.20	95–102 (99) 91–94 (95) 94–101 (97)	3.8 (3) 4.3 (3) 3.9 (3)
Wheat (hay)	0.02	0.02 0.02 0.20	85–91 (88) 84–88 (86) 101–107 (103)	3.5 (3) 2.4 (3) 3.1 (3)
Wheat (straw)	0.02	0.02 0.02 0.25	86–94 (94) 86–104 (95) 100–108 (104)	9.0 (3) 9.5 (3) 3.8 (3)
Wheat (grain)	0.01	0.01 0.01 0.01	75–89 (81) 93–119 (105) 105–110 (107)	8.7 (3) 10.5 (8) 2.3 (3)
Wheat (bran)	0.01	0.01 0.04	101–103 (102) 85–100 (92)	1.1 (3) 8.3 (3)
Wheat (flour)	0.01	0.01	70–112 (95)	23.3 (3)
Wheat (germ)	0.01	0.01	73–100 (87)	15.5 (3)
Wheat (middlings)	0.01	0.01	104–112 (109)	4.0 (3)
Wheat (shorts)	0.01	0.01 0.02	111–124 (118) 82–103 (93)	5.6 (3) 11.3 (3)
Alfalfa (forage)	0.02	0.02 1.25	83–91 (87) 113; 113	4.0 (4) –
Alfalfa (hay)	0.02	0.02 2.5	82–111 (93) 78–105 (93)	14.0 (4) 14.7 (3)
Barley (hay)	0.02	0.02 0.25	78–124 (100) 70–109 (94)	20.6 (5) 22.2 (3)
Barley (straw)	0.02	0.02 0.20	87–100 (94) 92–93 (92)	5.7 (4) 0.6 (3)
Barley (grain)	0.01	0.01 0.02	98–116 (106) 104–109 (106)	7.7 (4) 2.5 (3)
Sugar beet (tops)	0.02	0.02 0.20	89–99 (92) 77–116 (90)	6.6 (7) 24.6 (3)
Sugar beet (roots)	0.02	0.02 0.20	69–82 (76) 65–95 (80)	7.4 (4) 18.7 (3)
Turnip (tops)	0.02	0.02 0.10	92–104 (99) 93–98 (95)	6.3 (3) 2.8 (3)
Turnip (roots)	0.02	0.02 0.10	88–102 (94) 88–100 (94)	7.9 (3) 6.4 (3)
Onion (green onion)	0.02	0.02	93–96 (95)	1.8 (3)
Onion (bulb onion)	0.02	0.02	89–103 (95)	6.1 (5)

Table 34 Independent laboratory validation of Method 00914 for analysis of spiromesifen-enol

Sample material	Fortification Level [a], mg/kg	Individual values, %	Mean value, %	RSD, %	LOQ, mg/kg
Wheat straw	0.020	77; 108	92	33	0.02
	0.040	87; 78	82	11	
	0.20	88; 86	87	2	

[a] Fortification and calculated concentrations are expressed in spiromesifen equivalents

Table 35 Independent laboratory validation of Method 00914 for analysis of 4-hydroxymethyl spiromesifen-enol

Sample material	Fortification Level [a], mg/kg	Individual values, %	Mean value, %	RSD, %	LOQ, mg/kg
Wheat straw	0.020	110; 96	103	14	0.02
	0.040	100; 97	98	2	
	0.20	99; 92	95	8	

[a] Fortification and calculated concentrations are expressed in spiromesifen equivalents

Two different methods were used for the analysis of tea. Samples analysed by the sponsor, Bayer Crop Science, used a method essentially identical to Method 00631/M001. Overall, recoveries ranged from 77 to 115% (mean ± RSD = 90 ± 12%) for spiromesifen, and from 72 to 93% (85 ± 10%) for Sp-enol.

Tea samples analysed by the Japan Plant Protection Association were extracted with ACN:H_2O:formic acid (80:20:1, v/v/v), filtered, and transferred to hexane/ethyl acetate (95:5, v/v) by liquid-liquid partitioning. Additional clean-up was by silica gel mini columns. Residues were analysed by HPLC-MS, monitoring ion mass:charge ratios of 273.2 for spiromesifen, 271.9 for Sp-enol, and 287.3 for 4-hydroxymethyl-Sp-enol. Recovery results for this method are shown in Table 36.

Table 36 Method validation results for spiromesifen and metabolites in tea analysed using the Japan Plant Protection Association method

Sample Material	Fortification Level [a], mg/kg	Individual values, %	Mean value, %	RSD, %	Mean overall	RSD overall	LOQ, mg/kg
Spiromesifen							
Tea (green, dried)	0.05	116, 114, 114, 114, 110, 100	111	5.3	101	9.2	0.05
	2	104, 97, 95, 104, 102, 99	100	3.8			
	25	93, 85, 83, 112, 107, 92	95	12.3			
	80	102, 102, 99, 100, 92, 90	98	5.3			
Spiromesifen-enol							
Tea (green, dried)	0.04	105, 100, 95, 93, 90, 88	95	6.7	93	5.0	0.04
	2	95, 92, 91, 92, 87, 84	90	4.4			
	25	95, 93, 91, 95, 94, 92	93	1.7			
4-hydroxymethyl-Sp-enol							
Tea (green, dried)	0.04	110, 108, 108, 108, 108, 108	108	0.8	93	12.8	0.04
	2	88, 82, 81, 92, 91, 91	88	5.5			
	10	82, 79, 78, 88, 86, 84	83	4.7			

[a] Fortification and calculated concentrations are expressed in spiromesifen equivalents

For sugarcane, samples were extracted with acetonitrile and then mixed with “QuEChERS salts” (magnesium sulfate, sodium chloride, sodium citrate, and disodiumsulfate sesquihydrate). The extracts were centrifuged, the supernatant diluted with water, and an aliquot analysed by LC-MS/MS. Method validation recoveries ranged from 75 to 114 percent across all analytes and concentration levels (Table 37).

Table 37 Method validation results for residues of spiromesifen and metabolites in sugarcane

Sample Material	Fortification Level [a], mg/kg	Individual values, %	Mean value, %	RSD, %	Mean overall	RSD overall	LOQ, mg/kg
Spiromesifen							
Sugarcane stalk	0.01	109, 89, 95, 114, 96, 106, 91	100	9.5	101	8.0	0.01

Sample Material	Fortification Level [a], mg/kg	Individual values, %	Mean value, %	RSD, %	Mean overall	RSD overall	LOQ, mg/kg
	0.10	104, 105, 101	103	1.9			
Spiromesifen-enol							
Sugarcane stalk	0.01	83, 75, 79, 79, 93, 91, 84	83	6.6	86	7.7	0.01
	0.10	90, 92, 92	91	1.4			
4-hydroxymethyl-Sp-enol							
Sugarcane stalk	0.01	82, 82, 80, 85, 84, 88, 84	84	2.4	85	3.4	0.01
	0.10	86, 87, 89	87	1.7			

[a] Fortification and calculated concentrations are expressed in spiromesifen equivalents

Livestock materials

Method 110878-1 was developed and validated for the analysis of spiromesifen residues in livestock commodities. Residues in tissues are extracted with ACN:H_2O (4:1, v/v) using accelerated solvent extraction (70 °C, 1500 psi, ca. 10,300 KPa); internal standards are added at the completion of the extraction process. Radiovalidation data showed extraction efficiencies of 96, 100, and 69% of the total residue (defined as spiromesifen, Sp-enol, and 4-hydroxymethyl-Sp-enol) from milk, fat, and liver, respectively. Extracts from liver and kidney are cleaned-up using a strong cation exchange column and then digested with HCl to hydrolyse conjugated residues. Resulting residues are cleaned-up using C_{-8} solid-phase extraction. Fat and muscle extracts are treated with ammonium hydroxide to hydrolyse spiromesifen to Sp-enol followed by clean-up using C_{-8} solid-phase extraction. Milk samples are not subjected to liquid extraction. Rather, internal standards are added to the milk sample, and residues in milk are isolated using C_{-8} solid-phase extraction. For all samples, analysis is by HPLC-MS/MS. Method validation and independent laboratory validation results are shown in Table 38.

Table 38 Method validation and independent laboratory validation results for residues of spiromesifen and metabolites in livestock commodities

Sample Material	Fortification Level [a], mg/kg	Individual values, %	Mean value, %	RSD, %	Mean overall	RSD overall	LOQ, mg/kg
Spiromesifen: Method Validation							
Muscle	0.01	85, 81, 88	84	4	–	–	0.01
Fat	0.01	92, 98, 96	96	3	–	–	0.01
Kidney	0.05	87, 80, 91	86	6	–	–	0.05
Liver	0.05	70, 66, 73	70	5	–	–	0.05
Milk	0.005	108, 101, 84	98	13	96	9.0	0.005
	0.025	100, 93, 90	94	6			
Spiromesifen: Independent Laboratory Validation							
Milk	0.005	107, 102	104	5	94	11	0.005
	0.01	94, 96	95	2			
	0.05	86, 78	82	11			
Spiromesifen-enol: Method Validation							
Muscle	0.01	88, 108, 76, 76, 73	84	17	86	13	0.01
	0.05	89, 90, 89	90	1			
Fat	0.01	98, 93, 98	96	3	96	2.4	0.01
	0.05	96, 97, 93	95	2			
Kidney	0.05	106, 110, 110	109	2	105	4.8	0.05
	0.25	100, 98, 106	101	4			
Liver	0.05	103, 95, 102	100	4	97	4.8	0.05
	0.25	94, 92, 94	93	1			
Milk	0.005	110, 108, 107	108	1	108	1.3	0.005
	0.025	107, 106, 108	107	1			
Spiromesifen-enol: Independent Laboratory Validation							
Milk	0.005	106, 98	102	8	95	6.8	0.005
	0.010	90, 95	93	5			
	0.050	89, 91	90	2			

Sample Material	Fortification Level [a], mg/kg	Individual values, %	Mean value, %	RSD, %	Mean overall	RSD overall	LOQ, mg/kg
Liver	0.05	91, 89	90	3	92	6.5	0.05
	0.10	96, 83	89	14			
	0.50	97, 99	98	2			
4-hydroxymethyl-Sp-enol: Method Validation							
Muscle	0.01	82, 85, 104, 92, 95, 95	90	9	91	7.3	0.01
	0.05	87, 92, 87	89	3			
Fat	0.01	97, 95, 93	95	2	96	2.1	0.01
	0.05	97, 97, 99	98	1			
Kidney	0.05	103, 92, 96	97	6	97	3.9	0.05
	0.25	95, 99, 96	96	2			
Liver	0.05	98, 93, 94	95	2	93	3.5	0.05
	0.25	93, 88, 92	91	3			
Milk	0.005	102, 103, 102	102	< 1	104	2.6	0.005
	0.025	107, 107, 101	105	4			
4-hydroxymethyl-Sp-enol: Independent Laboratory Validation							
Milk	0.005	97, 99	98	2	94	6.8	0.005
	0.010	84, 94	89	11			
	0.050	88, 100	94	13			
Liver	0.05	107, 107	107	1	103	3.2	0.05
	0.10	101, 104	103	4			
	0.50	102, 99	100	3			

Conclusions—Residue Analysis

The provided analytical methods are suitable for the analysis of spiromesifen, spiromesifen-enol, and 4-hydroxymethyl-Sp-enol (including the conjugates). Recoveries were within the generally acceptable range of 70–120% and relative standard deviations were less than 20%.

Stability of residues in stored samples

The Meeting received studies depicting the stability of residues of spiromesifen and Sp-enol in cucumber, melon (peel), and beans with pods (Schöning, 2002; Report MR-513/01); corn forage, corn grain, corn stover, mustard green whole leaves, cotton undelinted seed, cotton gin trash, potato tuber, potato chips, potato granules/flakes, potato wet peel, tomato whole fruit, tomato paste, and tomato puree (Duah, 2002; Report 200178); wheat forage, wheat straw, wheat grain, sugar beet roots and sugar beet tops (Lemke, 2002; Report 200185); and spiromesifen, Sp-enol, and 4-hydroxymethyl-Sp-enol in wheat forage, grain, and hay, and turnip root (Murphy, 2007; Report RABSY015). For all matrices, control samples were fortified with a mixed standard of spiromesifen and spiromesifen-enol (in the case of Report RABSY015, separate samples were fortified with 4-hydroxy-Sp-enol). Fortified samples were stored frozen (at least –10 °C) and residues were analysed using either Method 00631 or Method 00631/M001.

Parent spiromesifen frequently showed decomposition, with a concomitant increase in the -enol metabolite. The Sp-enol metabolite does appear to be more stable during storage, and when taken together, the sum of spiromesifen and Sp-enol residues account for 83 to 110% of the initial amount of spiromesifen and Sp-enol added to the storage stability samples (Table 39).

Table 39 Storage stability results for spiromesifen and its metabolites in various matrices

			Mean ± RSD % remaining			
Report	Matrix	Storage time, days	Spiromesifen	Sp-enol	4-hydroxy-Sp-enol	Total [a] (normalized to Day 0)
MR-513/01	Cucumber fruit	0	87 ± 1.5	102 ± 1.8	–	100
		30	69 ± 3.8	100 ± 2.1	–	89
		60	78 ± 0.7	95 ± 1.6	–	91
		91	71 ± 2.1	101 ± 1.0	–	90
		190	82 ± 2.1	112 ± 3.6	–	102

			Mean ± RSD % remaining			
Report	Matrix	Storage time, days	Spiromesifen	Sp-enol	4-hydroxy-Sp-enol	Total [a] (normalized to Day 0)
		367	69 ± 2.2	124 ± 1.6	–	100
		546	57 ± 0.4	124 ± 1.2	–	94
		727	53 ± 13.3	127 ± 4.2	–	93
	Melon peel	0	90 ± 1.0	100 ± 2.3	–	100
		30	79 ± 2.2	101 ± 1.5	–	94
		60	81 ± 1.9	94 ± 1.6	–	92
		91	92 ± 9.8	81 ± 2.5	–	92
		190	93 ± 4.3	106 ± 4.1	–	105
		367	79 ± 3.3	119 ± 0.8	–	103
		546	82 ± 8.3	109 ± 1.8	–	100
		727	72 ± 5.0	113 ± 4.9	–	97
	Climbing French bean	0	83 ± 0.7	110 ± 1.4	–	100
		30	61 ± 1.9	112 ± 3.4	–	88
		60	73 ± 1.4	98 ± 1.2	–	89
		91	66 ± 2.3	105 ± 2.0	–	87
		190	76 ± 9.0	124 ± 6.1	–	102
		367	57 ± 9.9	129 ± 5.0	–	93
		374	58 ± 4.3	130 ± 3.5	–	94
		546	56 ± 10.9	132 ± 4.6	–	94
		727	45 ± 4.4	137 ± 2.2	–	89
RABSY015	Wheat forage	0	95 ± 0.8	93 ± 2.7	100 ± 2.0	100
		91	93 ± 0.3	94 ± 0.9	97 ± 0.9	100
		372	93 ± 0.6	112 ± 3.4	104 ± 1.4	109
		679	81 ± 4.2	117 ± 1.4	95 ± 3.4	106
	Wheat grain	0	96 ± 2.2	95 ± 3.7	85 ± 9.9	100
		91	97 ± 5.7	91 ± 7.0	78 ± 10.3	99
		372	95 ± 3.4	96 ± 1.7	90 ± 6.5	101
		679	94 ± 4.1	102 ± 1.1	76 ± 5.0	103
	Wheat hay	0	94 ± 1.9	97 ± 1.7	100 ± 0.8	100
		91	95 ± 0.5	100 ± 4.8	100 ± 3.4	102
		372	87 ± 3.9	113 ± 2.6	107 ± 0.9	105
		679	79 ± 6.9	116 ± 3.7	101 ± 2.7	102
	Turnip root	0	92 ± 1.7	92 ± 0.6	99 ± 2.3	100
		91	94 ± 0.5	94 ± 4.1	102 ± 2.5	103
		372	90 ± 7.4	96 ± 3.7	105 ± 1.2	102
		679	82 ± 6.0	97 ± 6.7	101 ± 2.7	98
200178	Corn forage	0	115 ± 2.7	111 ± 2.3	–	100
		157	88 ± 3.3	132 ± 4.6	–	98
		318	95 ± 1.0	103 ± 1.5	–	88
	Corn grain	0	118 ± 5.8	107 ± 4.4	–	100
		157	81 ± 1.2	120 ± 4.3	–	90
		318	97 ± 0.3	118 ± 3.4	–	96
	Corn stover	0	107 ± 6.1	111 ± 3.2	–	100
		156	69 ± 6.8	124 ± 1.7	–	88
		326	71 ± 8.3	104 ± 4.0	–	80
	Mustard greens	0	115 ± 4.4	106 ± 4.8	–	100
		159	79 ± 11.5	117 ± 2.1	–	89
		347	56 ± 18.3	138 ± 9.5	–	90
	Cotton, undelinted seed	0	102 ± 2.0	96 ± 10.2	–	100
		156	75 ± 4.0	117 ± 1.8	–	98
		318	55 ± 14.9	127 ± 6.6	–	93
	Cotton, gin trash	0	98 ± 5.7	97 ± 4.8	–	100
		159	67 ± 2.7	119 ± 8.8	–	95
		322	58 ± 8.0	150 ± 7.3	–	107
	Potato tubers	0	109 ± 2.3	110 ± 2.4	–	100
		159	68 ± 7.7	127 ± 2.1	–	89
		320	44 ± 8.8	153 ± 5.7	–	90
	Potato chips	0	77 ± 6.2	101 ± 1.0	–	100

			Mean ± RSD % remaining			
Report	Matrix	Storage time, days	Spiromesifen	Sp-enol	4-hydroxy-Sp-enol	Total [a] (normalized to Day 0)
		157	85 ± 4.3	111 ± 2.1	–	110
		322	83 ± 1.9	93 ± 1.5	–	99
	Potato granules/flakes	0	85 ± 3.8	90 ± 4.3	–	100
		157	75 ± 3.5	93 ± 5.3	–	96
		322	64 ± 11.4	107 ± 10.1	–	97
	Potato wet peel	0	91 ± 2.9	112 ± 3.1	–	100
		157	93 ± 8.3	111 ± 3.2	–	100
		323	83 ± 0.8	112 ± 5.4	–	96
	Tomato	0	117 ± 5.2	111 ± 2.4	–	100
		160	96 ± 5.9	110 ± 2.8	–	90
		316	79 ± 4.9	110 ± 2.8	–	83
	Tomato paste	0	122 ± 7.4	114 ± 2.2	–	100
		156	81 ± 9.8	116 ± 5.7	–	84
		318	87 ± 3.7	117 ± 3.4	–	87
	Tomato puree	0	125 ± 3.2	110 ± 2.3	–	100
		159	94 ± 6.5	108 ± 3.2	–	87
		322	84 ± 1.4	115 ± 4.5	–	86
200185	Wheat forage	434	–	–	80.7 ± 1.9	–
	Wheat grain	468	–	–	70.0 ± 9.4	–
	Wheat straw	434	–	–	72.3 ± 7.6	–
	Sugar beet root	436	–	–	67.7 ± 23.6	–
	Sugar beet tops	465	–	–	70.3 ± 2.2	–

[a] Total = the weighted sum of the % remaining for each residue, normalized to its zero-day result, expressed as a percentage (e.g., [(69%÷87%)/2 + (100%÷102%)/2] = 89%. The total for Study RABSY015 includes only spiromesifen and Sp-enol since the 4-hydroxy metabolite was not co-applied with spiromesifen or Sp-enol to the same sample.

Conclusions—Storage stability

Spiromesifen is not stable under frozen storage conditions. The amount remaining in stored samples fell to below 70% across many matrices by the 30-day time point. The loss of spiromesifen is accompanied by an increase in spiromesifen-enol. As storage samples were fortified with both spiromesifen and spiromesifen-enol, the stability of spiromesifen-enol, *per se*, could not be determined directly; however, the sum of spiromesifen and spiromesifen-enol, when compared to the sum of the fortification levels, indicates that spiromesifen-enol was stable throughout the periods of the storage stability studies. When evaluated as the sum of spiromesif and spiromesifen-enol, residues were stable throughout the periods of the storage stability studies.

USE PATTERN

Spiromesifen is registered for use on numerous crops in multiple countries. Information on registered uses that was provided to the meeting is summarized in Table 40.

Table 40 Summary of registered use patterns for spiromesifen. For all uses, application timing coincides with threshold pest pressure. n.s. = not specified; n.a. = not applicable.

			Formulation		Application			Applic. rate per trtmt.				
Crop	Country	Site	Type	Conc., g ai/L	Method	Max No.	RTI, days	Conc., kg ai/hL	Spray rate, L/ha	Rate, kg ai/ha	PHI, days	Remarks
Fruits												
Berries and other small fruits												
Low growing berry [a]	USA	F	SC	240	Broadcast (ground)	3	7	0.03	958	280	3	0.84 kg ai/ha /season max
Strawberry	Belgium	G	SC	240	Broadcast	2	7	n.s.	n.s.	0.120	1	
Strawberry	Canada	F	SC	240	Broadcast (ground)	3	7	0.28	100	0.278	3	0.84 kg ai/ha /season max

			Formulation		Application			Applic. rate per trtmt.				
Crop	Country	Site	Type	Conc., g ai/L	Method	Max No.	RTI, days	Conc., kg ai/hL	Spray rate, L/ha	Rate, kg ai/ha	PHI, days	Remarks
Strawberry	Canada	F	SC	240	Broadcast (aerial)	3	7	0.56	50	0.278	3	
Strawberry	Colombia	F	SC	240	Broadcast	1	5	0.012	n.s.	0.144	3	
Strawberry	Ecuador	F	SC	240	Broadcast (ground)	2	10	n.s.	n.s.	0.072	3	
Strawberry	Greece	G	SC	240	Broadcast	4	10	0.01	1500	0.144	3	
Strawberry	Italy	G	SC	240	Broadcast	4	10	0.01	1000	0.144	3	
Strawberry	Mexico	F	SC	240	Broadcast	n.s.	n.s.	n.s.	n.s.	0.288	3	
Strawberry	Netherlands	G	SC	240	Broadcast	n.s.	7	0.01	1000	0.120	1	
Strawberry	Peru	F	SC	240	Broadcast	2	n.s.	0.03	500	0.144	3	
Strawberry	Spain	G	SC	240	Broadcast	4	10	n.s.	n.s.	0.072	3	
Strawberry	United Arab Emirates	F	SC	240	Broadcast	n.s.	n.s.	n.s.	n.s.	0.144	3	
Assorted tropical and sub-tropical fruit—inedible peel												
Papaya	Colombia	F	SC	240	Broadcast	1	5	n.s.	n.s.	0.120	7	
Papaya	Ecuador	F	SC	240	Broadcast (ground)	2	10	n.s.	n.s.	0.120	7	
Papaya	Mexico	F	SC	240	Broadcast	n.s.	n.s.	n.s.	n.s.	0.180	SL	Do not apply during the fruiting stage
Fruiting vegetables, Cucurbits												
Cucurbit vegetables [b]	Canada	F	SC	240	Broadcast (ground)	3	7	0.29	50	0.144	1	0.43 kg ai/ha season max
Cucurbit vegetables [b]	Canada	F	SC	240	Broadcast (aerial)	3	7	0.29	50	0.144	1	
Cucurbit vegetables [c]	USA	F	SC	240	Broadcast (ground)	3	7	0.16	96	0.149	7	0.45 kg ai/ha season max
Cucurbit vegetables [c]	USA	F	SC	240	Broadcast (aerial)	3	7	0.32	48	0.149	7	
Fruiting vegetables [d]	Belgium	G	SC	240	Broadcast	2	7	n.s.	n.s.	0.072	1	
Fruiting vegetables [e]	Belgium	G	SC	240	Broadcast	2	7	n.s.	n.s.	0.120	1	
Courgette	Belize	F	SC	240	Broadcast	4	7	0.02	600	0.120	7	
Courgette	Dominican Republic	F	SC	240	Broadcast	4	7	0.02	600	0.120	7	
Courgette	El Salvador	F	SC	240	Broadcast	4	7	0.02	600	0.120	7	
Courgette	Guatemala	F	SC	240	Broadcast	4	7	0.02	600	0.120	7	
Courgette	Honduras	F	SC	240	Broadcast	4	7	0.02	600	0.120	7	
Courgette	Italy	G	SC	240	Broadcast	4	10	0.01	1500	0.216	3	
Courgette	Mexico	F	SC	240	Broadcast	n.s.	n.s.	n.s.	n.s.	0.144	7	
Courgette	Mexico	G	SC	240	Broadcast	2	7	0.024	n.s.	n.s.	7	
Courgette	Netherlands	G	SC	240	Broadcast	n.s.	7	0.01	1000	0.120	1	
Courgette	Spain	G	SC	240	Broadcast	4	10	n.s.	n.s.	0.072	3	
Cucumber	Belize	F	SC	240	Broadcast	4	7	0.02	600	0.120	7	
Cucumber	Costa Rica	F	SC	240	Broadcast	4	7	0.02	600	0.120	7	
Cucumber	Dominican Republic	F	SC	240	Broadcast	4	7	0.02	600	0.120	7	
Cucumber	El Salvador	F	SC	240	Broadcast	4	7	0.02	600	0.120	7	
Cucumber	Greece	G	SC	240	Broadcast	4	10	0.01	1500	0.144	3	
Cucumber	Guatemala	F	SC	240	Broadcast	4	7	0.02	600	0.120	7	
Cucumber	Honduras	F	SC	240	Broadcast	4	7	0.02	600	0.120	7	
Cucumber	India	F	SC	240	Broadcast	n.s.	n.s.	0.03	500	0.144	5	
Cucumber	Italy	G	SC	240	Broadcast	4	10	0.01	1500	0.216	3	
Cucumber	Mexico	F	SC	240	Broadcast	n.s.	n.s.	n.s.	n.s.	0.144	7	
Cucumber	Mexico	G	SC	240	Broadcast	2	7	0.024	n.s.	n.s.	7	

			Formulation		Application			Applic. rate per trtmt.				
Crop	Country	Site	Type	Conc., g ai/L	Method	Max No.	RTI, days	Conc., kg ai/hL	Spray rate, L/ha	Rate, kg ai/ha	PHI, days	Remarks
Cucumber	Netherlands	G	SC	240	Broadcast	n.s.	7	0.01	1000	0.120	1	
Cucumber	Nicaragua	F	SC	240	Broadcast	4	7	0.02	600	0.120	7	
Cucumber	Panama	F	SC	240	Broadcast	4	7	0.02	600	0.120	7	
Cucumber	Spain	G	SC	240	Broadcast	4	10	n.s.	n.s.	0.072	3	
Cucumber	United Arab Emirates	F	SC	240	Broadcast	n.s.	n.s.	n.s.	n.s.	0.144	3	
Cucumbers	New Zealand	G	SC	240	Broadcast	2	7	n.s.	n.s.	0.144	n.s.	
Gherkin	Netherlands	G	SC	240	Broadcast	n.s.	7	0.01	1000	0.120	1	
Gourd	Netherlands	G	SC	240	Broadcast	n.s.	7	0.01	1000	0.120	1	
Melon	Belize	F	SC	240	Broadcast	4	7	0.02	600	0.120	7	
Melon	Brazil	F	SC	240	Broadcast (ground)	4	5	0.04	400	0.144	14	
Melon	Brazil	F	SC	240	Broadcast (aerial)	4	5	0.36	40	0.144	14	
Melon	Costa Rica	F	SC	240	Broadcast	4	7	0.02	600	0.120	7	
Melon	Dominican Republic	F	SC	240	Broadcast	4	7	0.02	600	0.120	7	
Melon	El Salvador	F	SC	240	Broadcast	4	7	0.02	600	0.120	7	
Melon	Greece	G	SC	240	Broadcast	4	10	0.01	1500	0.144	3	
Melon	Guatemala	F	SC	240	Broadcast	4	7	0.02	600	0.120	7	
Melon	Honduras	F	SC	240	Broadcast	4	7	0.02	600	0.120	7	
Melon	Italy	G	SC	240	Broadcast	4	10	0.01	1000	0.144	3	
Melon	Mexico	F	SC	240	Broadcast (ground)	n.s.	n.s.	n.s.	n.s.	0.144	7	
Melon	Mexico	G	SC	240	Broadcast	2	7	0.024	n.s.	n.s.	7	
Melon	Netherlands	G	SC	240	Broadcast	n.s.	7	0.01	1000	0.120	1	
Melon	Nicaragua	F	SC	240	Broadcast	4	7	0.02	600	0.120	7	
Melon	Panama	F	SC	240	Broadcast	4	7	0.02	600	0.120	7	
Melon	Spain	G	SC	240	Broadcast	4	10	n.s.	n.s.	0.072	3	
Melon	United Arab Emirates	F	SC	240	Broadcast	n.s.	n.s.	n.s.	n.s.	0.144	3	
Melon (horizontal cultivation)	Belgium	G	SC	240	Broadcast	2	7	n.s.	n.s.	0.120	1	
Melon (vertical cultivation)	Belgium	G	SC	240	Broadcast	2	7	n.s.	n.s.	0.120	1	max 0.3 L product/ha row
Pumpkin	Belgium	G	SC	240	Broadcast	2	7	n.s.	n.s.	0.120	1	
Pumpkin	Mexico	F	SC	240	Broadcast (ground)	n.s.	n.s.	n.s.	n.s.	0.144	7	
Pumpkin	Mexico	G	SC	240	Broadcast	2	7	0.024	n.s.	n.s.	7	
Pumpkin	Netherlands	G	SC	240	Broadcast	n.s.	7	0.01	1000	0.120	1	
Squash	Italy	G	SC	240	Broadcast	2	10	0.01	1000	0.120	3	
Squash	Netherlands	G	SC	240	Broadcast	n.s.	7	0.01	1000	0.120	1	
Squash	United Arab Emirates	F	SC	240	Broadcast	n.s.	n.s.	n.s.	n.s.	0.144	3	
Summer squash	Netherlands	G	SC	240	Broadcast	n.s.	7	0.01	1000	0.120	1	
Watermelon	Belize	F	SC	240	Broadcast	4	7	0.02	600	0.120	7	
Watermelon	Costa Rica	F	SC	240	Broadcast	4	7	0.02	600	0.120	7	
Watermelon	Dominican Republic	F	SC	240	Broadcast	4	7	0.02	600	0.120	7	
Watermelon	El Salvador	F	SC	240	Broadcast	4	7	0.02	600	0.120	7	
Watermelon	Greece	G	SC	240	Broadcast	4	10	0.01	1500	0.144	3	
Watermelon	Guatemala	F	SC	240	Broadcast	4	7	0.02	600	0.120	7	
Watermelon	Honduras	F	SC	240	Broadcast	4	7	0.02	600	0.120	7	

			Formulation		Application			Applic. rate per trtmt.				
Crop	Country	Site	Type	Conc., g ai/L	Method	Max No.	RTI, days	Conc., kg ai/hL	Spray rate, L/ha	Rate, kg ai/ha	PHI, days	Remarks
Watermelon	Italy	G	SC	240	Broadcast	4	10	0.01	1000	0.144	3	
Watermelon	Mexico	F	SC	240	Broadcast (ground)	n.s.	n.s.	n.s.	n.s.	0.144	7	
Watermelon	Mexico	G	SC	240	Broadcast	2	7	0.024	n.s.	n.s.	7	
Watermelon	Nicaragua	F	SC	240	Broadcast	4	7	0.02	600	0.120	7	
Watermelon	Panama	F	SC	240	Broadcast	4	7	0.02	600	0.120	7	
Watermelon	Spain	G	SC	240	Broadcast	4	10	n.s.	n.s.	0.072	3	
Watermelon	United Arab Emirates	F	SC	240	Broadcast	n.s.	n.s.	n.s.	n.s.	0.144	3	
Zucchini	Greece	G	SC	240	Broadcast	4	10	0.01	1500	0.144	3	
Fruiting vegetables, other than cucurbits												
Fruiting vegetables [f]	Canada	F	SC	240	Broadcast (ground)	3	7	0.29	50	0.144	7	0.43 kg ai/ha season max
Fruiting vegetables [f]	Canada	F	SC	240	Broadcast (aerial)	3	7	0.29	50	0.144	7	
Fruiting vegetables [g]	USA	F	SC	240	Broadcast (ground)	3	7	0.16	96	0.146	1	0.45 kg ai/ha season max
Fruiting vegetables [g]	USA	F	SC	240	Broadcast (aerial)	3	7	0.32	48	0.146	1	
Pepper (sweet and chilli)	Belgium	G	SC	240	Broadcast	2	7	n.s.	n.s.	0.180	1	
Tomatoes, eggplant (aubergine), pepino (melon pear)	Belgium	G	SC	240	Broadcast	2	7	n.s.	n.s.	0.072	1	
Eggplant	United Arab Emirates	F	SC	240	Broadcast	n.s.	n.s.	n.s.	n.s.	0.180	3	
Eggplant (aubergine)	Greece	G	SC	240	Broadcast	4	10	0.01	1500	0.144	3	
Eggplant (aubergine)	Italy	G	SC	240	Broadcast	4	10	0.01	1500	0.216	3	
Eggplant (aubergine)	Mexico	F	SC	240	Broadcast	n.s.	7	n.s.	n.s.	0.144	1	
Eggplant (aubergine)	Mexico	G	SC	240	Broadcast	2	7	0.024	n.s.	n.s.	3	
Eggplant (aubergine)	Netherlands	G	SC	240	Broadcast	n.s.	7	0.01	1000	0.120	1	
Eggplant (aubergine)	Spain	G	SC	240	Broadcast	4	10	n.s.	n.s.	0.072	3	
Eggplant (Brinjal)	India	F	SC	240	Broadcast (ground)	n.s.	n.s.	0.02	500	0.096	5	
Husk tomato	Mexico	F	SC	240	Broadcast	n.s.	7	n.s.	n.s.	0.144	1	
Naranjilla	Colombia	F	SC	240	Broadcast	2	5	0.018	n.s.	0.144	3	
Okra	India	F	SC	240	Broadcast (ground)	n.s.	n.s.	0.02	500	0.120	3	
Pepper	Italy	G	SC	240	Broadcast	4	10	0.01	1500	0.216	3	
Pepper	Spain	G	SC	240	Broadcast	4	10	n.s.	n.s.	0.072	3	
Pepper	United Arab Emirates	F	SC	240	Broadcast	n.s.	n.s.	n.s.	n.s.	0.180	3	
Pepper (bell)	Greece	G	SC	240	Broadcast	4	10	0.01	1500	0.144	3	
Pepper (capsicums)	Colombia	F	SC	240	Broadcast	2	5	n.s.	n.s.	0.144	3	
Pepper (capsicums)	New Zealand	F	SC	240	Broadcast (ground)	2	7	0.01	1000	0.144	1	
Pepper (chilli)	Belize	F	SC	240	Broadcast	4	7	0.02	600	0.120	7	
Pepper (chilli)	Colombia	F	SC	240	Broadcast	4	5	n.s.	n.s.	0.144	3	

			Formulation		Application			Applic. rate per trtmt.				
Crop	Country	Site	Type	Conc., g ai/L	Method	Max No.	RTI, days	Conc., kg ai/hL	Spray rate, L/ha	Rate, kg ai/ha	PHI, days	Remarks
Pepper (chilli)	Dominican Republic	F	SC	240	Broadcast	4	7	0.02	600	0.120	7	
Pepper (chilli)	El Salvador	F	SC	240	Broadcast	4	7	0.02	600	0.120	7	
Pepper (chilli)	Guatemala	F	SC	240	Broadcast	4	7	0.02	600	0.120	7	
Pepper (chilli)	Honduras	F	SC	240	Broadcast	4	7	0.02	600	0.120	7	
Pepper (chilli)	India	F	SC	240	Broadcast (ground)	n.s.	n.s.	0.01	750	0.096	7	
Pepper (chilli)	Mexico	F	SC	240	Broadcast	n.s.	7	n.s.	n.s.	0.144	1	
Pepper (chilli)	Mexico	G	SC	240	Broadcast	2	7	0.024	n.s.	n.s.	3	
Pepper (chilli)	Netherlands	G	SC	240	Broadcast	n.s.	7	0.01	1000	0.120	1	
Pepper (paprika)	Peru	F	SC	240	Broadcast	2	n.s.	0.03	500	0.144	3	
Pepper (sweet chilli)	Nicaragua	F	SC	240	Broadcast	4	7	0.01	600	0.072	7	
Pepper (sweet)	Netherlands	G	SC	240	Broadcast	n.s.	7	0.01	1000	0.120	1	
Sweet corn	USA	F	SC	240	Broadcast (ground)	1 2	14	0.32	96	0.280 0.302	See remarks	0.30 kg ai/ha /season max PHI = 5 days for forage, silage, sweet corn for fresh consumption PHI = 30 days for grain and stover)
Sweet corn	USA	F	SC	240	Broadcast (aerial)	1 2	14	0.64	48	0.280 0.302		
Tomato	Belize	F	SC	240	Broadcast	4	7	0.02	600	0.120	7	
Tomato	Brazil	F	SC	240	Broadcast (ground)	4	5	0.01	1000	0.144	3	
Tomato	Brazil	F	SC	240	Broadcast (aerial)	4	5	0.36	40	0.144	3	
Tomato	Colombia	F	SC	240	Broadcast	2	5	n.s.	n.s.	0.144	3	
Tomato	Costa Rica	F	SC	240	Broadcast	4	7	0.02	600	115	7	
Tomato	Dominican Republic	F	SC	240	Broadcast	4	7	0.02	600	0.120	7	
Tomato	El Salvador	F	SC	240	Broadcast	4	7	0.02	600	0.120	7	
Tomato	France	G	SC	240	Broadcast	4	10	n.s.	n.s.	0.216	3	
Tomato	Greece	G	SC	240	Broadcast	4	10	0.01	1500	0.144	3	
Tomato	Guatemala	F	SC	240	Broadcast	4	7	0.02	600	0.120	7	
Tomato	Honduras	F	SC	240	Broadcast	4	7	0.02	600	0.120	7	
Tomato	India	F	SC	240	Broadcast (ground)	n.s.	n.s.	0.03	500	0.150	3	
Tomato	Italy	G	SC	240	Broadcast	4	10	0.01	1500	0.216	3	
Tomato	Japan	F	WP	229	Broadcast	2		0.01	3000	0.360	1	
Tomato	Kenya	F	SC	240	Broadcast (ground)	4	10	0.01	1000	0.144	n.s.	
Tomato	Mexico	F	SC	240	Broadcast	n.s.	7	n.s.	n.s.	0.144	1	
Tomato	Mexico	G	SC	240	Broadcast	2	7	0.024	n.s.	n.s.	3	

			Formulation		Application			Applic. rate per trtmt.				
Crop	Country	Site	Type	Conc., g ai/L	Method	Max No.	RTI, days	Conc., kg ai/hL	Spray rate, L/ha	Rate, kg ai/ha	PHI, days	Remarks
Tomato	Netherlands	G	SC	240	Broadcast	n.s.	7	0.01	1000	0.120	1	
Tomato	New Zealand	F	SC	240	Broadcast (ground)	2	7	0.01	1000	0.144	1	
Tomato	New Zealand	G	SC	240	Broadcast (ground)	2	7	0.01	1000	0.144	7	
Tomato	Panama	F	SC	240	Broadcast	4	7	0.02	600	0.115	7	
Tomato	Peru	F	SC	240	Broadcast	2	n.s.	0.03	500	0.144	7	
Tomato	Spain	G	SC	240	Broadcast	4	10	n.s.	n.s.	0.072	3	
Tomato	United Arab Emirates	F	SC	240	Broadcast	n.s.	n.s.	n.s.	n.s.	0.180	3	
Tree tomato	Colombia	F	SC	240	Broadcast	2	5	n.s.	n.s.	0.144	3	
Leafy vegetables (including Brassica leafy vegetables)												
Leafy greens vegetables [h]	Canada	F	SC	240	Broadcast (ground)	3	7	0.14	100	0.144	7	0.43 kg ai/ha season max
Leafy greens vegetables [h]	Canada	F	SC	240	Broadcast (aerial)	3	7	0.29	50	0.144	7	
Leafy greens vegetables [i]	USA	F	SC	240	Broadcast (ground)	3	7	0.16	96	0.149	7	0.45 kg ai/ha season max
Leafy greens vegetables [i]	USA	F	SC	240	Broadcast (aerial)	3	7	0.32	48	0.149	7	
Brassica leafy vegetables [j]	Canada	F	SC	240	Broadcast (ground)	3	7	0.14	100	0.144	7	0.43 kg ai/ha season max
Brassica leafy vegetables [j]	Canada	F	SC	240	Broadcast (aerial)	3	7	0.29	50	0.144	7	
Brassica leafy vegetables [k]	USA	F	SC	240	Broadcast (ground)	3	7	0.16	96	0.149	7	0.45 kg ai/ha season max
Brassica leafy vegetables [k]	USA	F	SC	240	Broadcast (aerial)	3	7	0.32	48	0.149	7	
Legume vegetables												
Bean	Belize	F	SC	240	Broadcast	4	7	0.02	600	0.120	7	
Bean	Colombia	F	SC	240	Broadcast	2	5	0.018	n.s.	0.144	3	
Bean	Dominican Republic	F	SC	240	Broadcast	4	7	0.02	600	0.120	7	
Bean	El Salvador	F	SC	240	Broadcast	4	7	0.02	600	0.120	7	
Bean	Greece	G	SC	240	Broadcast (ground)	4	10	0.01	1500	0.144	3	
Bean	Guatemala	F	SC	240	Broadcast	4	7	0.02	600	0.120	7	
Bean	Honduras	F	SC	240	Broadcast	4	7	0.02	600	0.120	7	
Beans	Netherlands	G	SC	240	Broadcast	n.s.	7	0.01	1000	0.120	1	
Beans	United Arab Emirates	F	SC	240	Broadcast	n.s.	n.s.	n.s.	n.s.	0.180	3	
Dwarf French bean (horizontal cultivation)	Belgium	G	SC	240	Broadcast	2	7	n.s.	n.s.	0.120	1	
Dwarf French bean (vertical cultivation)	Belgium	G	SC	240	Broadcast	2	7	n.s.	n.s.	0.120	1	max 0.3 L product/ha row
French beans	Kenya	F	SC	240	Broadcast (ground)	4	10	0.01	1000	0.144	n.s.	
Green bean	Brazil	F	SC	240	Broadcast (ground)	3	5	0.04	400	0.144	21	
Green bean	Brazil	F	SC	240	Broadcast (aerial)	3	5	0.36	40	0.144	21	
Green bean	Italy	G	SC	240	Broadcast	4	10	0.01	1000	0.144	3	
Green bean	Spain	G	SC	240	Broadcast	4	10	n.s.	n.s.	0.072	3	
Root and tuber vegetables												
Tuberous and	Canada	F	SC	240	Broadcast	2	7	0.14	100	0.144	7	0.29 kg ai/ha

			Formulation		Application			Applic. rate per trtmt.				
Crop	Country	Site	Type	Conc., g ai/L	Method	Max No.	RTI, days	Conc., kg ai/hL	Spray rate, L/ha	Rate, kg ai/ha	PHI, days	Remarks
corm vegetables [l]					(ground)							/season max
Tuberous and corm vegetables [l]	Canada	F	SC	240	Broadcast (aerial)	2	7	0.29	50	0.144	7	
Tuberous and corm vegetables [m]	USA	F	SC	240	Broadcast (ground)	2	7	0.30	96	0.280	7	0.56 kg ai/ha /season max
Tuberous and corm vegetables [m)]	USA	F	SC	240	Broadcast (aerial)	2	7	0.60	48	0.280	7	
Potato	Belize	F	SC	240	Broadcast	4	7	0.02	600	0.120	7	
Potato	Dominican Republic	F	SC	240	Broadcast	4	7	0.02	600	0.120	7	
Potato	El Salvador	F	SC	240	Broadcast	4	7	0.02	600	0.120	7	
Potato	Guatemala	F	SC	240	Broadcast	4	7	0.02	600	0.120	7	
Potato	Honduras	F	SC	240	Broadcast	4	7	0.02	600	0.120	7	
Potato	Mexico	F	SC	240	Broadcast	n.s.	7	n.s.	n.s.	0.144	7	
Potato	New Zealand	F	SC	240	Broadcast (ground)	2	7	n.s.	n.s.	0.144	7	
Potato	Peru	F	SC	240	Broadcast	2	n.s.	0.03	500	0.144	7	
Potato	United Arab Emirates	F	SC	240	Broadcast	n.s.	n.s.	n.s.	n.s.	0.180	7	
Cereal grains												
Maize (field corn)	Canada	F	SC	240	Broadcast (ground)	2	14	0.14	100	0.144	See remarks	0.29 kg ai/ha/season max PHI = 5 days for green forage, silage PHI = 30 days for grain and stover)
Maize (field corn)	Canada	F	SC	240	Broadcast (aerial)	2	14	0.29	50	0.144		
Maize (field corn, popcorn)	USA	F	SC	240	Broadcast (ground)	1 2	14	0.32	96	0.280 0.302	See remarks	0.30 kg ai/ha/season max PHI = 5 days for forage, silage PHI = 30 days for grain and stover)
Maize (field corn, popcorn)	USA	F	SC	240	Broadcast (aerial)	1 2	14	0.64	48	0.280 0.302		
Maize	Mexico	F	SC	240	Broadcast	n.s.	n.s.	0.04	500	0.192	5 (feed), 10 (grain)	
Oilseeds												
Cotton	Brazil	F	SC	240	Broadcast (ground)	2	5	0.05	300	0.144	21	
Cotton	Brazil	F	SC	240	Broadcast (aerial)	2	5	0.36	40	0.144	21	

			Formulation		Application			Applic. rate per trtmt.				
Crop	Country	Site	Type	Conc., g ai/L	Method	Max No.	RTI, days	Conc., kg ai/hL	Spray rate, L/ha	Rate, kg ai/ha	PHI, days	Remarks
Cotton	Colombia	F	SC	240	Broadcast	2	5	n.s.	n.s.	0.144	21	
Cotton	Costa Rica	F	SC	240	Broadcast	4	7	0.02	600	0.120	7	
Cotton	India	F	SC	240	Broadcast (ground)	n.s.	n.s.	0.03	500	0.144	10	
Cotton	Mexico	F	SC	240	Broadcast	n.s.	n.s.	n.s.	n.s.	0.144	30	
Cotton	Panama	F	SC	240	Broadcast	4	7	0.02	600	0.120	7	
Cotton	USA	F	SC	240	Broadcast (ground)	3	7	0.30	96	0.280	30	0.56 kg ai/ha /season max
Cotton	USA	F	SC	240	Broadcast (aerial)	3	7	0.60	48	0.280	30	0.56 kg ai/ha /season max
Seed for beverages and sweets												
Coffee	Brazil	F	SC	240	Broadcast	2	n.s.	n.s.	n.s.	0.144	21	
Coffee	Colombia	F	SC	240	Broadcast	1	5	n.s.	n.s.	0.120	35	
Processed foods of plant origin												
Derived products of plant origin												
Tea	India	F	SC	240	Broadcast (ground)	n.s.	n.s.	0.02	400	0.096	7	
Tea	Japan	F	SC	300	Broadcast	1	n.a.	0.00	4000	0.63	7	
Legume animal feeds												
Alfalfa (grown for seed)	Canada	F	SC	240	Broadcast (ground)	3	7	0.24	100	0.240	n.s.	0.72 kg ai/ha /season max
Alfalfa (grown for seed)	Canada	F	SC	240	Broadcast (aerial)	3	7	0.48	50	0.240	n.s.	0.72 kg ai/ha /season max

[a] Bearberry, Bilberry, Blueberry (lowbush), Cloudberry, Cranberry, Lingonberry, Muntries, Partridgeberry, Strawberry

[b] Balsam apples, balsam pears, cantaloupes, chayote fruit, Chinese cucumbers, Chinese wax gourds, citron melons, cucumbers, edible gourds (other than those listed in this item), muskmelons (other than those listed in this item), West Indian gherkins, winter squash, muskmelon (includes cantaloupe), pumpkin, summer squash, winter squash (includes butternut squash, calabaza, hubbard squash, acorn squash, spaghetti squash), watermelon

[c] Chayote (fruit), Chinese wax gourd (Chinese preserving melon), citron melon, cucumber, gherkin, edible gourd (includes, hyotan, cucuzza, hechima, Chinese okra), Momordica spp. (includes balsam apple, balsam pear, bitter melon, Chinese cucumber), muskmelon (includes cantaloupe), pumpkin, summer squash, winter squash (includes butternut squash, calabaza, hubbard squash, acorn squash, spaghetti squash), watermelon

[d] Vertical cultivation: Cucumber, gherkin, courgette/summer squash

[e] Horizontal cultivation: Gherkin, courgette/summer squash

[f] Bell peppers, eggplants, groundcherries, non-bell peppers, pepinos, pepper hybrids, tomatillos, tomatoes

[g] Eggplant, groundcherry (Physabis sp.), pepino, pepper (includes: bell pepper, chilli pepper, cooking pepper, pimento, sweet pepper), tomatillo, and tomato

[h] Amaranth, arugula, corn salad, dandelion leaves, dock, edible leaved chrysanthemum, endives, fresh chervil leaves, garden cress, garden purslane, garland chrysanthemum, head lettuce, leaf lettuce, New Zealand spinach, orach leaves, parsley leaves, radicchio, spinach, upland cress, vine spinach, winter purslane radicchio (red chicory), spinach, New Zealand and vine spinach

[i] Amaranth (Chinese spinach), arugula (roquette), chervil, edible-leaved and garland chrysanthemum, corn salad, upland and garden cress, dandelion, dock (sorrel), endive (escarole), head and leaf lettuce, orach, parsley, garden and winter purslane, radicchio (red chicory), spinach, New Zealand and vine spinach

[j] Broccoli, Chinese Broccoli (gai lon), Brussels sprouts, cabbage, Chinese mustard cabbage (gai choy), cauliflower, cavolo broccolo, kohlrabi, mustard spinach, rape greens

[k] Broccoli and Chinese (gai lon) broccoli, Brussels sprouts, cabbage, Chinese mustard (gai choy) cabbage, cauliflower, cavalo broccolo, kohlrabi, mustard spinach, and rape greens

[l] Arracacha, arrowroot, cassava roots, chayote roots, Chinese artichokes, chufa, edible canna, ginger roots, Jerusalem artichokes, lerens, potatoes, sweet potato roots, tanier corms, taro corms, true yam tubers, turmeric roots, yam bean roots

[m] Arracacha, arrowroot, artichoke (Chinese, Jerusalem), artichoke (Jerusalem), canna (edible), cassava (bitter, sweet), chayote (root), chufa, dasheen, ginger, leren, potato, sweet potato, tanier, turmeric, yam (bean, true)

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

The Meeting received data from supervised residue trials conducted on strawberry, papaya, broccoli, cabbage, cucumber, melon, summer squash, peppers, tomato, sweet corn, head and leaf lettuce, spinach, mustard greens, beans with pods, pulses, cassava (manioc), potato, maize, popcorn, cotton, coffee, and tea.

The field trial reports included method validation data, as recoveries from spiked samples at levels reflecting those observed in the field trial samples; dates from critical events during the study, including application, harvest, storage, and analysis; as well as detailed information on the field site and treatment parameters. Analytical reports were sufficiently detailed and included example chromatograms and example calculations. Samples were analysed by the methods described above.

Unless otherwise noted in the tables below, harvested commodities were maintained whole in the field and not cut or homogenized until they reached the analytical laboratory. In the tables below, residues of spiromesifen-enol are reported in spiromesifen equivalents.

The field trial study designs included control plots. For most crops measured residues from control plots were < 0.01 mg/kg (i.e., < LOQ) and are not included in the summary tables in this evaluation. Cases where quantifiable residues were found in control matrices are noted in the tables below.

In the tables of crop field trial results below, residues for individual samples that were reported in the study volumes as finite values between the LOD and LOQ are shown as trace residues ('tr'). Residues reported as less than a specific LOD are shown as non-detected ('ND'). In determining combined residues of spiromesifen and Sp-enol, the following convention was used: > LOQ = finite residue, tr = LOQ (if both compounds are tr, then the combined is < 2× LOQ), and ND = 0 contribution (if both compounds are ND, then the combined is < LOQ). In determining mean values (in square brackets] from field trials, any value < 0.01 mg/kg was assumed to be 0.01 mg/kg. Mean values of spiromesifen + Sp-enol below the combined LOQ (0.02 mg/kg) are shown as < 0.02 mg/kg. Examples are shown below.

Spiromesifen, mg/kg	Sp-enol (as spiromesifen eq.), mg/kg	Spiromesifen + Sp-enol, mg/kg
0.12 (> LOQ)	0.041 (> LOQ)	0.16 (finite + finite)
0.003 (< LOQ, > LOD)	0.035 (> LOQ)	0.045 (finite + LOQ)
0.27(> LOQ)	< 0.0013 (< LOD)	0.27 (finite + 0)
0.004 (< LOQ, > LOD)	0.0081 (< LOQ, > LOD)	< 0.02 (< LOQ + < LOQ)
0.006 (< LOQ, > LOD)	< 0.0013 (< LOD)	< 0.01 (< LOQ + 0) [0.02]
< 0.0013 (< LOD)	< 0.0013 (< LOD)	< 0.01 (< LOD + < LOD) [0.02]

In the summary tables, values used for making maximum residue level recommendations are underlined and highest individual values for estimating dietary intake are bolded. Trial locations that the Meeting has determined are not independent are grouped by a heavy cell border in the tables (e.g., Table).

Supervised trials for spiromesifen:

Crop group	Commodity	Table No
Berries and other small fruits	Strawberry (FB 0725)	41
Trop. and sub-trop. Fruit—inedible peel	Papaya (FI 0350)	42
Brassica veg., head cabbage, flowerhead cabbage	Broccoli (VB 0400)	43
	Cabbage (VB 4175)	44
Fruiting veg., cucurbits	Cucumber (VC 0424)	45
	Melon (VC 0046)	46
	Summer squash (VC 0431)	47
Fruiting veg., other than cucurbits	Peppers (VO 0051)	48

Crop group	Commodity	Table No
	Tomato (VO 0448)	49
	Sweet corn (VO 0447)	50
Leafy veg., incl. Brassica leafy veg.	Lettuce (head) (VL 0482)	51
	Lettuce (leaf) (VL 0483)	51
	Spinach (VL 0502)	52
	Mustard greens (VL 0485)	53
Legume veg.	Bean with pod (VP 2060)	54
Pulses	Dry beans	55
Root and tuber veg.	Cassava (manioc) (VR 4555)	56
	Potato (VR 0589)	57
Cereal grains	Maize (GC 0645)	58
	Popcorn (GC 0656)	59
Oilseeds	Cotton (SO 0691)	60
Seed for beverages and sweets	Coffee (SB 0716)	61
Derived products of plant origin	Tea (DT 1114)	62
Straw, fodder, and forage of cereal grains	Maize (AF/AS 0645)	63
	Popcorn (AF/AS 0656)	63
	Sweet corn (AF/AS 0447)	63
Miscellaneous fodder and forage crops	Cotton gin trash (AB 1204)	64

Berries and other small fruits

Strawberry

Eight field trials were conducted in the USA during the 2000 and 2001 growing seasons (Lenz, 2002; Study 110315). Plants received three foliar applications of spiromesifen, on a 7 ± 2-day interval at ca. 0.280 kg ai/ha, with the final application occurring ca. 3 days before harvest. Samples were frozen within 4 hours of harvest and maintained frozen during transport and prior to analysis. Samples were homogenized in the presence of dry ice at the analytical laboratory and analysed for residues of spiromesifen and Sp-enol. Samples were stored for a maximum of 279 days prior to extraction and analysed within 2 days of extraction. Total residues of spiromesifen (parent + Sp-enol) were stable in fruits (cucumber, melon) for at least 727 days.

In addition, eight greenhouse trials were conducted in northern and southern Europe during the 1999 and 2000 growing seasons (Schöning, 2001; Studies RA-2086/99 and RA-2018/00). Strawberry plants received four applications of spiromesifen on a 9- to 11-day interval at ca. 144 g ai/ha. Strawberries were harvested 0 to 10 days after the last application. Harvested samples were frozen within 24 hours of collection and stored at –18 °C prior to analysis. Samples were held in frozen storage for approximately 5 months (RA-2018/00) or 18 months (RA-2086/99).

Overall, concurrent recovery samples for strawberry gave results ranging from 74 to 118% recovery, with relative standard deviations ranging from 1.8 to 11.9%.

Table 41 Results of spiromesifen residue trials in strawberry

Study No. Trial No. Country Location Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg) [a]		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
Field									
GAP Canada/USA/ Mexico	Low-growing berry (USA) Strawberry	3	0.280		–	3	–	–	–
110315 BS014-00D USA San Luis Obispo, California America, North 2000	Strawberry Sea-scape	3	0.285–0.290	0.0996–0.100	fruit	0	1.1, 1.4 [1.3]	0.088, 0.11 [0.099]	1.2, 1.5 [1.4]
						3	0.47, 0.49 [0.48]	0.090, 0.094 [0.092]	0.56, 0.58 [0.57]
						7	0.18, 0.40 [0.29]	0.090, 0.082 [0.086]	0.27, 0.48 [0.38]
						14	0.15, 0.12 [0.14]	0.12, 0.10 [0.11]	0.27, 0.23 [0.25]
						22	0.034, 0.026 [0.030]	0.073, 0.082 [0.077]	0.11, 0.11 [0.11]
						28	0.068, 0.095 [0.082]	0.059, 0.061 [0.060]	0.13, 0.16 [0.14]
110315 BS015-00H USA Corvallis, Oregon America, North 2000	Strawberry Selva	3	0.267–0.291	0.0953–0.103	fruit	3	0.43, 0.38 [0.40]	0.063, 0.069 [0.066]	0.49, 0.45 [0.47]
						9	0.15, 0.33 [0.24]	0.051, 0.090 [0.070]	0.20, 0.42 [0.31]
						14	0.26, 0.18 [0.22]	0.10, 0.083 [0.094]	0.36, 0.26 [0.31]
110315 BS016-00H USA Fresno, California America, North 2000	Strawberry Chandler	3	0.280–0.282	0.0996–0.1000	fruit	3	1.3, 1.5 [1.4]	0.15, 0.13 [0.14]	1.4, 1.6 [1.5]
						7	1.1, 0.81 [0.97]	0.15, 0.16 [0.16]	1.3, 0.97 [1.1]
						14	0.32, 0.35 [0.34]	0.14, 0.13 [0.13]	0.46, 0.48 [0.47]
110315 BS017-00H USA El Toro, California America, North 2000	Strawberry Camarosa	3	0.276–0.286	0.0741–0.0748	fruit	3	0.74, 0.49 [0.61]	0.087, 0.080 [0.083]	0.82, 0.57 [0.70]
						7	0.34, 0.37 [0.36]	0.077, 0.082 [0.080]	0.42, 0.45 [0.44]
						15	0.25, 0.18 [0.22]	0.080, 0.084 [0.082]	0.33, 0.27 [0.30]
110315 BS018-00H USA Circleville, Ohio	Strawberry Winona	3	0.278–0.288	0.178–0.201	fruit	4	0.24, 0.19 [0.21]	0.044, 0.040 [0.042]	0.28, 0.23 [0.26]

Study No. Trial No. Country Location Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg) [a]		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
America, North 2000									
						7	0.17, 0.24 [0.21]	0.043, 0.045 [0.044]	0.21, 0.29 [0.25]
						12	0.14, 0.27 [0.20]	0.038, 0.045 [0.041]	0.18, 0.31 [0.25]
110315 BS019-00H USA Seffner, Florida America, North 2001	Strawberry Sweet Charlie	3	0.282–0.284	0.0955–0.105	fruit	3	1.4, 1.5 [1.5]	0.11, 0.10 [0.11]	1.6, 1.6 [1.6]
						8	1.0, 0.76 [0.88]	0.13, 0.14 [0.13]	1.1, 0.91 [1.0]
						15	0.26, 0.33 [0.30]	0.089, 0.084 [0.087]	0.35, 0.41 [0.38]
110315 BS020-00H USA Jamesville, North Carolina America, North 2000	Strawberry Chandler	3	0.278–0.290	0.0631–0.0644	fruit	3	0.34, 0.44 [0.39]	0.078, 0.078 [0.078]	0.42, 0.52 [0.47]
						7	0.28, 0.17 [0.23]	0.12, 0.10 [0.11]	0.40, 0.27 [0.34]
						14	0.16, 0.21 [0.18]	0.12, 0.12 [0.12]	0.28, 0.33 [0.30]
110315 BS021-00H USA Frederick, Pennsylvania America, North 2000	Strawberry Earliglow	3	0.280–0.288	0.0748–0.0751	fruit	2	0.21, 0.17 [0.19]	0.078, 0.084 [0.081]	0.28, 0.25 [0.27]
						7	0.13, 0.18 [0.16]	0.096, 0.095 [0.095]	0.23, 0.28 [0.25]
						13	0.18, 0.19 [0.18]	0.12, 0.11 [0.11]	0.29, 0.30 [0.30]
Greenhouse									
GAP Europe (Greece, Italy)	Strawberry	4	0.144	0.014	–	3	–	–	–
RA-2086/99 R 1999 0285/9 0285-99 Spain E-08395 St. Pol de Mar Europe, South 1999	Strawberry Irvine	4	0.132–0.18	0.0144	fruit	0*	0.35	0.07	0.45
						0	0.43	0.06	0.51
						1	0.52	0.07	0.62
						3	0.43	0.07	0.53
						7	0.29	0.09	0.41
						10	0.26	0.08	0.37
RA-2086/99 R 1999 0286/7 0286-99 Italy	Strawberry Paiaro	4	0.144	0.0144	fruit	0	0.1	0.02	0.13

Study No. Trial No. Country Location Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg) [a]		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
I-75025 Policoro Europe, South 1999									
						3	0.07	0.02	0.1
						7	0.06	0.02	0.09
RA-2086/99 R 1999 0446/0 0446-99 Portugal P-2000 Santarem (Paco dos negros) Europe, South 1999	Strawberry Camerosa	4	0.144	0.0144	fruit	0*	0.03	0.01	0.04
						0	0.1	0.02	0.13
						1	0.08	0.02	0.11
						3	0.05	0.02	0.08
						7	0.04	0.02	0.07
						10	0.03	0.02	0.06
RA-2086/99 R 1999 0447/9 0447-99 France F-84200 Carpentras Europe, South 1999	Strawberry Pajaro	4	0.144	0.0144	fruit	0	0.11	0.06	0.19
						3	0.11	0.06	0.19
						7	0.08	0.05	0.15
RA-2018/00 R 2000 0103/7 0103-00 Belgium B-3770 Riemst Europe, North 2000	Strawberry Elsanta	4	0.144	0.0144	fruit	0*	0.13	0.04	0.18
						0	0.26	0.04	0.31
						1	0.22	0.03	0.26
						3	0.25	0.03	0.29
						7	0.19	0.02	0.22
						10	0.22	0.02	0.25
RA-2018/00 R 2000 0104/5 0104-00 Germany D-46397 Bocholt Europe, North 2000	Strawberry Elsanta	4	0.144	0.0144	fruit	0	0.12	0.07	0.22
						3	0.07	0.05	0.14
						6	0.04	0.04	0.09
RA-2018/00 R 2000 0109/6 0109-00 France F-27190 Glisolles Europe, North 2000	Strawberry Mara des Bois	4	0.144	0.0144	fruit	0*	0.13	0.04	0.18
						0	0.21	0.04	0.26

Study No. Trial No. Country Location Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg) [a]		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
						1	0.19	0.03	0.23
						3	0.15	0.03	0.19
						7	0.1	0.03	0.14
						10	0.09	0.03	0.13
RA-2018/00 R 2000 0162/2 0162-00 Netherlands Nl-4725-SG-25 Wouwse Plantage Europe, North 2000	Strawberry Elsanta	4	0.144–0.1512	0.0144	fruit	0	0.07	0.04	0.12
						3	0.08	0.03	0.12
						7	0.1	0.03	0.14

[a] Residues of spiromesifen-enol and total residues are reported in spiromesifen equivalents.

Assorted tropical and sub-tropical fruit—inedible peel

Papaya

Four trials were initiated on papaya in Ghana (Anon., 2006; ReportM-285076-01-1). Two were conducted during the November to December dry season (2005–2006) and two during the January to April rainy season (2006). Three treatments, each at 0.120 kg ai/ha, were scheduled with an application interval of 56 days. For the rainy season plots, samples were collected three days after the first and second application and at multiple times following the third application. An outbreak of Phytophthora disease interrupted the dry season trials at both locations, resulting in two of the three applications being made at the original location and a single application at each of two secondary locations. Papaya were harvested 3 days after the first and second applications and at multiple times after the application at the secondary trial locations. All harvested papaya fruits remained whole in the field. Samples were shipped, frozen, to the analytical laboratory.

Samples of papaya were analysed on a whole-fruit basis as well as for residues in peel and in pulp. Results were reported only for parent spiromesifen.

While the field portion of the papaya residue trials was not performed under GLP standards, the analytical portion was. Papaya samples were stored frozen for no more than 153 days prior to extraction and analysis. Concurrent recoveries reported for five samples ranged from 63 to 110%, with a mean and relative standard deviation of 93 ± 20%.

Table 42 Results of spiromesifen residue trials in papaya

Study No. Trial No. Country Location Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
GAP Ecuador	Papaya	2	0.12	–	–	7	–	–	–
GAP	Papaya	1	0.12	–	–	7	–	–	–

Study No. Trial No. Country Location Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
Colombia/Mexico									
GHA/PA/2005/01- 02-03-04 GHA/PA/2005/01 PGD-223-A1 Ghana Akwamu/Nsawam Africa, West 2005	Papaya Solo and Golden yellow	2	0.12	0.0139	fruit	–53	0.228	Not analysed	Not calc'd
						3	0.191		
GHA/PA/2005/01- 02-03-04 GHA/PA/2005/01 PGD-223-A2 Ghana Akwamu/Nsawam Africa, West 2006	Papaya Solo and Golden yellow	1	0.12	0.0139	fruit	0	0.288	Not analysed	Not calc'd
						14	0.093		
						28	0.025		
						42	< 0.01		
						56	< 0.01		
					peel	14	0.207		
					pulp	14	< 0.01		
GHA/PA/2005/01- 02-03-04 GHA/PA/2005/02 PGD-223-B1 Ghana Obom Africa, West 2005	Papaya Golden yellow	2	0.12	0.019	fruit	–53	0.081	Not analysed	Not calc'd
						3	0.231		
GHA/PA/2005/01- 02-03-04 GHA/PA/2005/02 PGD-223-B2 Ghana Obom Africa, West 2006	Papaya Golden yellow	1	0.12	0.019	fruit	0	0.367	Not analysed	Not calc'd
						14	0.134		
						28	0.054		
						42	0.014		
						56	< 0.01		
					peel	14	0.302		
					pulp	14	0.016		
GHA/PA/2005/01- 02-03-04 GHA/PA/2005/03 PGD-233-C1 Ghana Akwamu/Nsawam Africa, West 2006	Papaya Solo and Golden yellow	3	0.12	0.0166	fruit	–109	0.204	Not analysed	Not calc'd
						–53	0.208		
						0	0.213		

Study No. Trial No. Country Location Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
						3	0.093		
						7	0.116		
						14	0.025		
						28	< 0.01		
					peel	3	0.314		
						7	0.437		
					pulp	3	0.013		
						7	0.012		
GHA/PA/2005/01-02-03-04 GHA/PA/2500/04 PGD-233-C2 Ghana Obom Africa, West 2006	Papaya Golden yellow	3	0.12	0.0158	fruit	–109	0.179	Not analysed	Not calc'd
						–53	0.223		
						0	0.147		
						3	0.121		
						7	0.262		
						14	0.078		
						28	0.025		
					peel	3	0.393		
						7	0.513		
					pulp	3	0.010		
						7	0.016		

Brassica (cole or cabbage) vegetables, head cabbages, flowerhead cabbages

Broccoli

Seven broccoli trials were conducted in the USA during the 2000 and 2001 growing season (Duah, 2002; Report 200141). Three foliar applications of spiromesifen were made to each test plot on a 4- to 9-day interval, each at 0.146 to 0.159 kg ai/ha. Samples were harvested at maturity and frozen within 2 hours of collection. Samples remained frozen during transportation to the analytical facility, where they were maintained in frozen storage prior to analysis. Samples were homogenized in the presence of dry ice and analysed for residues of spiromesifen and Sp-enol using a modified version of Method 00631.

Samples were stored frozen for up to 263 days prior to extraction, and samples were analysed within 8 days of extraction. Total residues of spiromesifen (parent + Sp-enol) were stable in multiple crops for at least 318 days. Overall concurrent recovery results ranged from 71 to 112% recovery with relative standard deviations ranging from 1.6 to 11.7%.

Table 43 Results of spiromesifen residue trials in broccoli

Study No. Trial No. Country Location Region Year	Crop Variety	Application	Matrix	DALA (days)	Residues (mg/kg)

		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
GAP Canada/USA	Brassica leafy vegetables	3	0.144	–	–	7	–	–	–
200141 BS067-00H USA Porterville, California America, North 2000	Broccoli Marathon	3	0.15	0.0528–0.0530	head	8	0.085, 0.068 [0.077]	0.014, 0.015 [0.014]	0.099, 0.083 [0.091]
						14	0.018, 0.013 [0.016]	tr, tr [< 0.01]	0.028, 0.023 [0.026]
200141 BS068-00H USA Yuba City, California America, North 2000	Broccoli Laguna	3	0.150–0.151	0.0790–0.0802	head	7	0.67, 0.40 [0.54]	0.044, 0.037 [0.040]	0.71, 0.44 [0.58]
						14	0.30, 0.18 [0.24]	0.027, 0.024 [0.026]	0.33, 0.20 [0.27]
200141 BS069-00H USA Ventura, California America, North 2000	Broccoli Boskovich B1350	3	0.148–0.159	0.0532–0.0540	head	8	0.22, 0.15 [0.18]	0.029, 0.023 [0.026]	0.25, 0.17 [0.21]
						15	0.080, 0.13 [0.11]	0.011, 0.012 [0.012]	0.091, 0.14 [0.12]
200141 BS070-00HA BS070-00HA-A USA Hillsboro, Oregon America, North 2000	Broccoli Packman	3	0.150–0.156	0.0519–0.0541	head	7	0.13, 0.12 [0.12]	0.028, 0.025 [0.027]	0.16, 0.14 [0.15]
						14	tr, ND [< 0.01]	tr, tr [< 0.01]	< 0.02, < 0.01 [< 0.02]
200141 BS070-00HA BS070-00HA-B USA Hillsboro, Oregon America, North 2000	Broccoli Packman	3	0.150–0.156	0.0519–0.0541	head	7	0.067	< 0.01	0.077
200141 BS071-00H USA East Bernard, Texas America, North 2000	Broccoli Green Comet	3	0.151–0.154	0.0770–0.0817	head	9	0.011, tr [0.010]	tr, tr [< 0.01]	0.021, < 0.02 [0.020]
						15	ND, ND [ND]	tr, ND [< 0.01]	< 0.01, < 0.01 [< 0.02]
200141 BS072-00D USA Fresno, California America, North 2001	Broccoli Green Comet	3	0.146–0.150	0.0824–0.0854	head	0	0.64, 0.43 [0.54]	0.017, 0.013 [0.015]	0.66, 0.44 [0.55]
						5	0.17, 0.15	tr, tr [< 0.01]	0.18, 0.16

Study No. Trial No. Country Location Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
							[0.16]		[0.17]
						10	0.062, 0.051 [0.056]	ND, ND [ND]	0.062, 0.051 [0.056]
						16	0.018, 0.014 [0.016]	ND, ND [ND]	0.018, 0.014 [0.016]
						22	0.014, 0.010 [0.012]	ND, ND [ND]	0.014, 0.010 [0.012]

Cabbage

Six cabbage trials were conducted in the USA during the 2000 and 2001 growing season (Duah, 2002; Report 20141). Three foliar applications of spiromesifen were made to each test plot on a 4- to 9-day interval, each at 0.148 to 0.157 kg ai/ha. Samples were harvested at maturity and frozen within 2 hours of collection. Samples remained frozen during transportation to the analytical facility, where they were maintained in frozen storage prior to analysis. Samples were homogenized in the presence of dry ice and analysed for residues of spiromesifen and Sp-enol using a modified version of Method 00631.

Samples were stored frozen for up to 263 days prior to extraction, and samples were analysed within 8 days of extraction. Total residues of spiromesifen (parent + Sp-enol) were stable in multiple crops for at least 318 days. Overall concurrent recovery results ranged from 72 to 114% recovery with relative standard deviations ranging from 0.0 to 17%.

Table 44 Results of spiromesifen residue trials in cabbage

Study No. Trial No. Country Location Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
GAP Canada/USA	Brassica leafy vegetables	3	0.144	–	–	7	–	–	
200141 BS061-00H USA Germansville, Pennsylvania America, North 2000	Cabbage, white Market Prize	3	0.154– 0.157	0.0458– 0.0535	head	8	0.057, 0.18 [0.12]	0.024, 0.042 [0.033]	0.081, 0.22 [0.15]
						15	0.14, 0.15 [0.14]	0.033, 0.030 [0.031]	0.17, 0.18 [0.18]
					head, trimmed	8	ND	0.011	0.011
						15	ND	tr	< 0.01 [< 0.02]
200141 BS062-00H USA Richland, Iowa America, North 2000	Cabbage, white Emerald Cross	3	0.149– 0.156	0.111– 0.132	head	7	tr, tr [< 0.01]	tr, 0.011 [0.011]	< 0.02, 0.021 [< 0.02]

Study No. Trial No. Country Location Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
						13	tr, ND [< 0.01]	tr, tr [< 0.01]	< 0.02, < 0.01 [< 0.02]
					head, trimmed	7	ND	tr	< 0.01 [< 0.02]
						13	ND	tr	< 0.01 [< 0.02]
200141 BS063-00H USA East Bernard, Texas America, North 2001	Cabbage, white Early	3	0.148– 0.155	0.0576– 0.0601	head	9	0.31, 0.25 [0.28]	0.079, 0.092 [0.086]	0.39, 0.34 [0.37]
						14	0.15, 0.11 [0.13]	0.061, 0.040 [0.050]	0.21, 0.15 [0.18]
					head, trimmed	9	0.0094	0.0092	0.019
						14	0.0072	0.0056	0.013
200141 BS064-00H USA Vero Beach, Florida America, North 2001	Cabbage white Bravo	3	0.150– 0.157	0.102– 0.108	head	7	0.37, 0.47 [0.42]	0.017, 0.025 [0.021]	0.38, 0.50 [0.44]
						14	0.23, 0.29 [0.26]	0.014, 0.018 [0.016]	0.24, 0.31 [0.27]
					head, trimmed	7	0.014	ND	0.014
						14	0.013	ND	0.013
200141 BS065-00D USA Fresno California America, North 2000	Cabbage, white Copenhagen Market	3	0.149– 0.151	0.0836– 0.0879	head	0	0.43, 0.49 [0.46]	0.11, 0.14 [0.12]	0.54, 0.62 [0.58]
						6	0.43, 1.1 [0.77]	0.11, 0.090 [0.099]	0.54, 1.2 [0.87]
						13	1.4, 1.4 [1.4]	0.14, 0.17 [0.16]	1.5, 1.6 [1.6]
						21	1.1, 1.1 [1.1]	0.10, 0.13 [0.12]	1.2, 1.3 [1.2]
						28	0.71, 0.54 [0.62]	0.086, 0.085 [0.086]	0.80, 0.62 [0.71]
200141 BS066-00H USA Tifton, Georgia America, North 2000	Cabbage, white Blue Thunder	3	0.150– 0.150	0.0991– 0.104	head	7	1.6, 1.4 [1.5]	0.27, 0.28 [0.27]	1.9, 1.7 [1.8]
						14	0.79, 0.92 [0.86]	0.21, 0.21 [0.21]	1.0, 1.1 [1.1]
					head, trimmed	7	0.061	0.023	0.084
						14	0.037	0.024	0.061

Fruiting vegetables, cucurbits

Cucumber

Six field trials were conducted in the USA during the 2000 growing season (Fischer, 2002; Report 110341). Three foliar applications of spiromesifen were made to each test plot on a 5- to 9-day interval, each at 0.148 to 0.156 kg ai/ha. Samples were harvested at maturity and frozen within 3 hours of collection. Samples remained frozen during transportation to the analytical facility, where they were maintained in frozen storage prior to analysis. Samples were homogenized in the presence of dry ice and analysed for residues of spiromesifen and Sp-enol using a modified version of Method 00631.

Eight greenhouse trials were conducted in northern and southern Europe (Schöning and Elke, 2001; Report RA-2078/99 and RA-2079/99). For these trials, the application rate was adjusted to incorporate the plant height (0.108 kg ai/ha × foliage height; not to exceed 2 m), resulting in application rates ranging from 0.086 to 0.238 kg ai/ha. Four applications were made on 7- to 11-day intervals. Cucumbers were harvested 3 days after the last application and frozen within 24 hours of collection. Samples were maintained under frozen conditions during transportation to the analytical facility. Samples were homogenized in the presence of dry ice and returned to frozen storage prior to analysis.

Samples from the field trials were stored frozen for up to 326 days prior to extraction, and samples were analysed within 13 days of extraction. Samples from the greenhouse trials were stored frozen for up to 502 days prior to extraction. Total residues of spiromesifen (parent + Sp-enol) were stable in cucumbers and melon for at least 727 days. Overall concurrent recovery results ranged from 71 to 116% recovery with relative standard deviations ranging from 1.5 to 17%.

Table 45 Results of spiromesifen residue trials in cucumber

Study No. Trial No. Country Location Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
Field									
GAP Canada/USA	Fruiting vegetables, cucurbits	3	0.144	–	–	7	–	–	–
110341 BS097-00H USA Geneva, Minnesota America, North 2000	Cucumber Straight 8	3	0.148–0.150	0.0755–0.0771	fruit	7	tr, 0.011 [0.010]	0.014, 0.017 [0.016]	0.024, 0.028 [0.026]
						14	tr, tr [< 0.01]	0.010, 0.010 [0.010]	0.020, 0.020 [< 0.02]
110341 BS098-00H USA Brookshire, Texas America, North 2000	Cucumber Straight Eight	3	0.151–0.152	0.0356–0.0360	fruit	7	tr, tr [< 0.01]	0.011, 0.014 [0.012]	0.021, 0.024 [< 0.02]
						14	ND, ND [ND]	tr, ND [< 0.01]	< 0.01, < 0.01 [< 0.02]
110341 BS099-00D	Cucumber SMR58	3	0.150–0.152	0.100–0.102	fruit	0	0.032, 0.026 [0.029]	0.024, 0.024 [0.024]	0.056, 0.049 [0.053]

Study No. Trial No. Country Location Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
USA Oxford, Indiana America, North 2000									
						7	tr, tr [< 0.01]	0.027, 0.026 [0.026]	0.037, 0.036 [< 0.02]
						12	tr, ND [< 0.01]	0.015, 0.013 [0.014]	0.025, 0.013 [< 0.02]
						19	ND, ND [ND]	tr, tr [< 0.01]	< 0.01, < 0.01 [< 0.02]
						26	ND, ND [ND]	tr, tr [< 0.01]	< 0.01, < 0.01 [< 0.02]
110341 BS100-00H BS100-00H-A USA Vero Beach, Florida America, North 2000	Cucumber Dasher II	3	0.150– 0.156	0.0455– 0.0485	fruit	7	tr, tr [< 0.01]	0.012, tr [0.011]	0.022, < 0.02 [< 0.02]
						14	ND, ND [ND]	tr, ND [< 0.01]	< 0.01, < 0.01 [< 0.02]
110341 BS100-00H BS100-00H-B USA Vero Beach, Florida America, North 2000	Cucumber Dasher II	3	0.150– 0.156	0.0455– 0.0485	fruit	7	ND	< 0.01	< 0.01 [< 0.02]
110341 BS101-00H USA Tifton, Georgia America, North 2000	Cucumber Valas Pic	3	0.150– 0.150	0.103– 0.122	fruit	7	0.012, tr [0.011]	0.022, 0.023 [0.022]	0.034, 0.033 [0.034]
						14	ND, ND [ND]	tr, tr [< 0.01]	< 0.01, < 0.01 [< 0.02]
110341 BS102-00H USA Chula, Georgia America, North 2000	Cucumber Lightning	3	0.151– 0.152	0.0884– 0.0999	fruit	7	tr, tr [< 0.01]	0.024, 0.025 [0.025]	0.034, 0.035 [< 0.02]
						14	ND, ND [ND]	tr, tr [< 0.01]	< 0.01, < 0.01 [< 0.02]
Greenhouse									
GAP Europe (Italy)	Cucumber	4	0.216	0.014	–	3	–	–	–
RA-2078/99 R 1999 0281/6 0281-99	Cucumber Arcade	4	0.216	0.0144	fruit	0*	< 0.01	0.01	0.02

Study No. Trial No. Country Location Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
Belgium B-2860 St. Katelijne-Waver Europe, North 1999									
						0	0.07	0.03	0.11
						1	0.05	0.03	0.09
						3	0.02	0.02	0.05
						7	< 0.01	0.02	0.04
						10	< 0.01	0.01	0.02
RA-2078/99 R 1999 0282/4 0282-99 Netherlands NL-4691 HS Tholen Europe, North 1999	Cucumber Flamingo	4	0.216	0.0144	fruit	0	0.05	0.03	0.09
						3	0.03	0.02	0.06
						7	0.01	0.02	0.04
RA-2078/99 R 1999 0429/0 0429-99 Germany D-42799 Leichlingen Europe, North 1999	Cucumber Indira	4	0.162– 0.216	0.0144	fruit	0 [a]	0.02	< 0.01	0.03
						0	0.09	0.02	0.12
						1	0.05	0.02	0.08
						3	0.05	0.02	0.08
						7	0.03	0.02	0.06
						10	< 0.01	< 0.01	< 0.02
RA-2078/99 R 1999 0430/4 0430-99 Germany D-42799 Leichlingen Europe, North 1999	Cucumber Indira	4	0.108– 0.216	0.0144	fruit	0	0.04	0.02	0.07
						3	0.02	0.01	0.03
						7	0.02	0.01	0.03
RA-2079/99 R 1999 0283/2 0283-99 Spain E-08840 Viladecans Europe, South 1999	Cucumber Solverde	4	0.1212– 0.216	0.0144	fruit	0 [a]	< 0.01	< 0.01	< 0.02
						0	0.03	0.01	0.04
						1	0.03	0.01	0.04
						3	0.02	< 0.01	0.03
						7	0.01	0.01	0.02
						10	0.02	< 0.01	0.03
RA-2079/99	Cucumber	4	0.216–	0.0144	fruit	0	0.06	0.03	0.1

Study No. Trial No. Country Location Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
R 1999 0284/0 0284-99 Greece Gr- Vasilika Europe, South 1999	Mesa		0.2376						
						3	0.03	0.03	0.07
						7	< 0.01	0.02	0.04
RA-2079/99 R 1999 0431/2 0431-99 Italy I-97017 S. Croce di Camerina Europe, South 2000	Cucumber Jet	4	0.0864– 0.1512	0.0144	fruit	0 [a]	0.05	0.02	0.08
						0	0.13	0.02	0.16
						1	0.12	0.02	0.15
						3	0.11	0.02	0.14
						7	0.09	0.02	0.12
						10	0.05	0.01	0.06
RA-2079/99 R 1999 0432/0 0432-99 France F-84000 Avignon Europe, South 1999	Cucumber Gardon	4	0.1675– 0.216	0.0144	fruit	0	0.06	0.02	0.09
						3	0.03	0.01	0.04
						7	0.02	< 0.01	0.03

Melons

Six field trials were conducted in the USA during the 2000 growing season (Fischer, 2002; Report 110341). Three foliar applications of spiromesifen were made to each test plot on a 5- to 9-day interval, each at 0.139 to 0.158 kg ai/ha. Samples were harvested at maturity and frozen within 3 hours of collection. Samples remained frozen during transportation to the analytical facility, where they were maintained in frozen storage prior to analysis. Samples were homogenized in the presence of dry ice and analysed for residues of spiromesifen and Sp-enol using a modified version of Method 00631.

Eight greenhouse trials were conducted in northern and southern Europe (Schöning and Sur, 2001; Report RA-2080/99 and RA-2020/00). For these trials, application rates ranged from 0.565 to 0.587 kg ai/ha. Four applications were made on 10- to 11-day intervals. Melons were harvested 3 days after the last application and frozen within 24 hours of collection. Samples were maintained under frozen conditions during transportation to the analytical facility. Samples were homogenized in the presence of dry ice and returned to frozen storage prior to analysis.

Samples from the field trials were stored frozen for up to 326 days prior to extraction, and samples were analysed within 13 days of extraction. Samples from the greenhouse trials were stored frozen for up to 535 days prior to extraction. Total residues of spiromesifen (parent + Sp-enol) were stable in cucumbers and melon for at least 727 days. Overall concurrent recovery results ranged from 72 to 118% recovery with relative standard deviations ranging from 1.0 to 13%.

Table 46 Results of spiromesifen residue trials in melon

Study No. Trial No. Country Location Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
Field									
GAP– Canada/USA	Fruiting vegetables, cucurbits	3	0.144	–	–	7	–	–	–
110341 BS103-00H USA Eloy, Arizona America, North 2000	Melon, cantaloupe Gold Rush	3	0.139– 0.152	0.0546– 0.0550	fruit	7	0.017, 0.012 [0.015]	tr, tr [< 0.01]	0.027, 0.022 [0.025]
						14	tr, tr [< 0.01]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
110341 BS104-00H USA Los Banos, California America, North 2000	Melon, cantaloupe Durango	3	0.149– 0.150	0.0531– 0.0531	fruit	8	0.020, 0.029 [0.024]	tr, 0.010 [0.010]	0.030, 0.039 [0.035]
						14	0.026, 0.014 [0.020]	tr, tr [< 0.01]	0.036, 0.024 [0.030]
110341 BS105-00H USA Brookshire, Texas America, North 2000	Melon, cantaloupe Hale's Best 36	3	0.150– 0.151	0.0348– 0.0355	fruit	7	0.018, 0.016 [0.017]	tr, tr [< 0.01]	0.028, 0.026 [0.027]
						13	tr, tr [< 0.01]	tr, tr [< 0.01]	< 0.02, < 0.02 [< 0.02]
110341 BS106-00H BS106-00H-A USA Oxford, Indiana America, North 2000	Melon, cantaloupe Minnesota midget	3	0.151– 0.156	0.0998– 0.102	fruit	9	0.011, 0.013 [0.012]	tr, tr [< 0.01]	0.021, 0.023 [0.022]
						15	tr, 0.010 [0.010]	ND, ND [ND]	< 0.01, 0.010 [0.010]
110341 BS106-00H BS106-00H-B USA Oxford, Indiana America, North 2000	Melon, cantaloupe Minnesota midget	3	0.151– 0.156	0.0998– 0.102	fruit	9	tr, ND [< 0.01]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
					pulp	9	0.11, 0.085 [0.097]	0.014, 0.012 [0.013]	0.12, 0.097 [0.11]
110341 BS107-00D USA Fresno, California America, North 2000	Melon, cantaloupe Hale's Best Jumbo	3	0.147– 0.158	0.0835– 0.0845	fruit	0	0.062, 0.053 [0.057]	0.010, 0.010 [0.010]	0.072, 0.063 [0.068]

Study No. Trial No. Country Location Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
						7	0.032, 0.038 [0.035]	tr, 0.010 [0.010]	0.042, 0.048 [0.045]
						14	0.011, 0.024 [0.018]	tr, tr [< 0.01]	0.021, 0.034 [0.028]
						21	tr, 0.015 [0.012]	tr, tr [< 0.01]	< 0.02, 0.025 [0.022]
						23	0.024, 0.021 [0.022]	tr, tr [< 0.01]	0.034, 0.031 [0.032]
110341 BS108-00H USA Tifton, Georgia America, North 2000	Melon, cantaloupe Cordel	3	0.15	0.0997–0.123	fruit	7	tr, tr [< 0.01]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
						13	0.0090, 0.0038 [< 0.01]	< 0.003, < 0.003 [< 0.01]	< 0.01, < 0.01 [< 0.01]
Greenhouse									
GAP Europe (Greece, Italy)	Melon	4	0.144		–	3	–	–	–
RA-2080/99 R 1999 0279/4 0279-99 Italy I-48100 Ravenna Europe, South 1999	Melon Garbi	4	0.144	0.0144	fruit	0 [a]	< 0.01	< 0.01	< 0.01
						0	0.03	< 0.01	0.04
						1	0.02	< 0.01	0.03
						3	0.02	< 0.01	0.03
						7	0.01	< 0.01	0.02
						10	< 0.01	< 0.01	< 0.01
					pulp	3	< 0.01	< 0.01	< 0.01
						7	< 0.01	< 0.01	< 0.01
					peel	3	0.04	0.01	0.05
						7	0.02	< 0.01	0.03
RA-2080/99 R 1999 0280/8 0280-99 Spain E-04738 Vicar–El Fronton S.L Europe, South 1999	Melon Galia	4	0.144–0.1536	0.0144	fruit	0	0.07	0.01	0.08
						3	0.05	0.01	0.06
						7	0.05	0.01	0.06
					pulp	3	< 0.01	< 0.01	< 0.01
						3	< 0.01	< 0.01	< 0.01
					peel	3	0.31	0.04	0.36
						3	0.22	0.04	0.27
RA-2080/99 R 1999 0433/9 0433-99 Spain E-04007 La	Melon Galia	4	0.144	0.0144	fruit	0	0.1	0.02	0.13

Study No. Trial No. Country Location Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
Mojonera Europe, South 1999									
						1	0.06	0.01	0.07
						3	0.03	0.01	0.04
						7	0.04	0.01	0.05
						10	0.04	< 0.01	0.05
					pulp	3	< 0.01	< 0.01	< 0.01
						7	< 0.01	< 0.01	< 0.01
					peel	3	0.39	0.06	0.47
						7	0.22	0.05	0.29
RA-2080/99 R 1999 0434/7 0434-99 France F-82700 Montbartier Europe, South 1999	Melon Figaro	4	0.144	0.0144	fruit	0	0.14	0.03	0.18
						3	0.06	0.01	0.07
						7	0.03	< 0.01	0.04
					pulp	3	< 0.01	< 0.01	< 0.01
						3	< 0.01	< 0.01	< 0.01
					peel	3	0.19	0.05	0.26
						3	0.26	0.04	0.31
RA-2020/00 R 2000 0176/2 0176-00 Spain E-04738 Vicar–El Fronton S.L Europe, South 2000	Melon Galia	4	0.1332– 0.144	0.0144	fruit	0 [a]	0.06	< 0.01	0.07
						0	0.1	< 0.01	0.11
						1	0.09	< 0.01	0.10
						3	0.06	< 0.01	0.07
						7	0.07	0.01	0.08
						10	0.07	0.01	0.08
					pulp	3	< 0.01	< 0.01	< 0.01
						7	< 0.01	< 0.01	< 0.01
					peel	3	0.31	0.04	0.36
						7	0.35	0.05	0.42
RA-2020/00 R 2000 0177/0 0177-00 Italy I-97019 Vittoria Europe, South 2000	Melon Kiton	4	0.144	0.0144	fruit	0 [a]	< 0.01	< 0.01	0.02
						0	0.03	< 0.01	0.04
						1	0.02	< 0.01	0.03
						3	0.02	< 0.01	0.03
						7	0.02	< 0.01	0.03
						10	0.02	< 0.01	0.03
					pulp	3	< 0.01	< 0.01	< 0.01
						7	< 0.01	< 0.01	< 0.01

Study No. Trial No. Country Location Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
					peel	3	0.04	< 0.01	0.05
						7	0.05	< 0.01	0.06
RA-2020/00 R 2000 0178/9 0178-00 Spain E-04740 Roquetas de Mar Europe, South 2000	Melon Galia-Var. Eros	4	0.144	0.0144	fruit	0	0.09	0.01	0.1
						3	0.04	< 0.01	0.05
						7	0.02	< 0.01	0.03
RA-2020/00 R 2000 0179/7 0179-00 Italy I-97019 Vittoria Europe, South 2000	Melon Ramado	4	0.144	0.0144	fruit	0	0.03	< 0.01	0.04
						3	0.03	< 0.01	0.04
						7	0.02	< 0.01	0.03

Squash

Six summer squash trials were conducted in the USA during the 2000 growing season (Fischer, 2002; Report 110341). Three foliar applications of spiromesifen were made to each test plot on a 5- to 9-day interval, each at 0.145 to 0.155 kg ai/ha. Samples were harvested at maturity and frozen within 3 hours of collection. Samples remained frozen during transportation to the analytical facility, where they were maintained in frozen storage prior to analysis. Samples were homogenized in the presence of dry ice and analysed for residues of spiromesifen and Sp-enol using a modified version of Method 00631.

Samples were stored frozen for up to 263 days prior to extraction, and samples were analysed within 8 days of extraction. Total residues of spiromesifen (parent + Sp-enol) were stable in multiple crops for at least 318 days. Overall concurrent recovery results ranged from 71 to 101% recovery with relative standard deviations ranging from 1.6 to 13%.

Table 47 Results of spiromesifen residue trials in summer squash

Study No. Trial No. Country Location Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
GAP– Canada/USA	Fruiting vegetables, cucurbits	3	0.144	–	–	7	–	–	–
110341 BS109-00H USA Dundee, New York America, North	Squash, summer Zucchini Select	3	0.149–0.151	0.0532–0.0534	fruit	7	0.012, 0.011 [0.012]	tr, tr [< 0.01]	0.022, 0.021 [0.022]

Study No. Trial No. Country Location Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
2000									
						14	ND, ND [ND]	ND, tr [< 0.01]	< 0.01, < 0.01 [< 0.02]
110341 BS110-00HA BS110-00HA-A USA Vero Beach, Florida America, North 2000	Squash, summer Dixie	3	0.149–0.150	0.102–0.106	fruit	7	0.022, 0.017 [0.020]	0.026, 0.035 [0.031]	0.048, 0.052 [0.050]
						11	0.013, 0.017 [0.015]	0.010, 0.016 [0.013]	0.024, 0.033 [0.028]
110341 BS110-00HA BS110-00HA-B USA Vero Beach, Florida America, North 2000	Squash, summer Dixie	3	0.149–0.150	0.102–0.106	fruit	7	tr	0.021	0.031
110341 BS111-00H USA Oxford, Indiana America, North 2000	Squash, summer Yellow Straight-neck	3	0.150–0.152	0.100–0.102	fruit	7	tr, tr [< 0.01]	0.011, 0.010 [0.011]	0.021, 0.020 [< 0.02]
						12	ND, ND [ND]	tr, tr [< 0.01]	< 0.01, < 0.01 [< 0.02]
110341 BS112-00D USA Fresno, California America, North 2000	Squash, summer Black Beauty	3	0.150–0.155	0.0839–0.0854	fruit	0	0.14, 0.12 [0.13]	0.038, 0.031 [0.034]	0.18, 0.15 [0.16]
						7	0.011, tr [0.011]	tr, tr [< 0.01]	0.021, < 0.02 [0.021]
						14	ND, tr [< 0.01]	tr, tr [< 0.01]	< 0.01, < 0.02 [< 0.02]
						21	ND, ND [ND]	tr, tr [< 0.01]	< 0.01, < 0.01 [< 0.02]
						28	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
110341 BS113-00H USA Tifton, Georgia America, North 2000	Squash, summer Prelude 11	3	0.145–0.150	0.0581–0.101	fruit	8	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
						14	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]

Fruiting vegetables, other than Cucurbits

Peppers/Chilli pepper

In a study investigating spiromesifen residues in peppers, three field trials were conducted with chilli pepper in the USA during the 2005 and 2006 growing season (Dorschner, 2009; Report IR-4 PR 09361). Three foliar applications of spiromesifen were made at rates of 0.145 to 0.154 kg ai/ha on a 6- to 8- day interval. Peppers were harvested 1 and 3 or 4 days after the final application. Peppers were frozen within 3 hours of harvest (except for the 3-DALA sample from Ohio which was frozen ca. 7.5 hrs after harvest). Samples remained frozen during transportation to the analytical facility and prior to analysis. Pepper samples were homogenized in the presence of dry ice and analysed for residues of spiromesifen and Sp-enol using Method 00631/M001.

Samples were stored frozen for up to 1039 days prior to extraction, and extracts were stored for no more than one day prior to analysis. Total residues of spiromesifen (parent + Sp-enol) were stable in cucumber, melon, and bean for at least 727 days and in tomato for at least 316 days. Overall concurrent recovery results ranged from 74 to 95% recovery with relative standard deviations ranging from 5.2 to 8.9%.

In an additional field study, three trials with spiromesifen were conducted with chilli pepper (Lenz, 2002; Report 110966). Trials were conducted in the USA during the 2000 growing season. Pepper plants received three foliar applications of spiromesifen at rates of 0.146 to 0.186 kg ai/ha on 5- to 9-day interval. Peppers were harvested ca. 7 and 14 days after the final application. Peppers were frozen within six hours of harvest and remained frozen during transportation to the analytical facility and prior to analysis. Samples were homogenized in the presence of dry ice and analysed for residues of spiromesifen and Sp-enol using Method 00631.

Samples were stored frozen for up to 300 days prior to extraction, and extracts were stored for no more than six days prior to analysis. Total residues of spiromesifen (parent + Sp-enol) were stable in cucumber, melon, and bean for at least 727 days and in tomato for at least 316 days. Overall concurrent recovery results ranged from 71 to 117% recovery with relative standard deviations ranging from 4.3 to 15%.

Sweet pepper

The first study described above for chilli pepper (Dorschner, 2009; Report IR-4 PR 09361) included seven trials conducted with sweet pepper.

The second study described above for chilli pepper (Lenz, 2002; Report 110966) included six trials conducted with sweet pepper.

Nine residue trials were conducted with greenhouse-grown peppers in northern and southern Europe during the 2004, 2005, and 2006 growing seasons (Rosati and Eberhardt, 2005, Report RA-2069/04; Rosati and Gateaud, 2006, Report RA-2607/05; Schöning and Telscher, 2007, Report RA-2140/06). Four applications of spiromesifen were made at a rate of 0.108 kg ai/ha × height of the leafy surface, resulting in application rates ranging from 0.065 to 0.216 kg ai/ha. Applications were made on a 9- to 11-day interval. Peppers were harvested three days after the final application. Samples were frozen within 24 hours of collection and remained frozen during transportation to the analytical facility and prior to analysis. Samples were homogenized in the presence of dry ice and analysed for residues of spiromesifen and Sp-enol using Method 00631.

Table 48 Results of spiromesifen residue trials in peppers

Study No. Trial No. Country Location Region Year	Crop Variety	Application	Matrix	DALA (days)	Residues (mg/kg)

		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
Field									
GAP USA/ Mexico	Fruiting veg.	3	0.144	–	–	1	–	–	–
IR-4 PR No. 09361 09361.05-GA*16 USA Tifton America, North 2005	Chilli / non-bell pepper Sweet Banana	3	0.147	0.0324–0.0325	Chilli pepper/ fruit	1	0.27, 0.25 [0.26]	0.050, 0.030 [0.040]	0.32, 0.28 [0.30]
						3	0.18, 0.080 [0.13]	0.030, 0.010 [0.020]	0.21, 0.090 [0.15]
IR-4 PR No. 09361 09361.05-NM16 USA Dona Ana America, North 2005	Chilli / non-bell pepper Joe E. Parker	3	0.146–0.147	0.0620–0.0623	Chilli pepper/ fruit	1	0.020, 0.020 [0.020]	ND, ND [ND]	0.020, 0.020 [0.020]
						3	0.020, 0.020 [0.020]	ND, ND [ND]	0.020, 0.020 [0.020]
IR-4 PR No. 09361 09361.05-TX*32 USA Weslaco America, North 2005	Chilli / non-bell pepper Sonora Anaheim	3	0.147–0.148	0.0368–0.0397	Chilli pepper/ fruit	1	0.040, 0.040 [0.040]	0.010, 0.020 [0.015]	0.050, 0.060 [0.055]
						3	0.030, 0.040 [0.035]	ND, ND [ND]	0.030, 0.040 [0.035]
IR-4 PR No. 09361 09361.05-CA136 USA Holtville America, North 2006	Pepper, sweet Wizard	3	0.148	0.0583–0.0589	Pepper, sweet/ fruit	1	0.030, 0.050 [0.040]	ND, 0.010 [< 0.01]	0.030, 0.060 [0.045]
						3	0.020, 0.020 [0.020]	ND, ND [ND]	0.020, 0.020 [0.020]
IR-4 PR No. 09361 09361.05-CA137 USA Parlier America, North 2005	Pepper, chilli Indra	3	0.145–0.148	0.0376–0.0389	Pepper, sweet/ fruit	1	0.030, 0.020 [0.025]	ND, ND [ND]	0.030, 0.020 [0.025]
						3	0.030, 0.020 [0.025]	ND, ND [ND]	0.030, 0.020 [0.025]
IR-4 PR No. 09361 09361.05-FL46 USA Citra America, North 2006	Pepper, sweet Crusader	3	0.148–0.149	0.0517–0.0519	Pepper, sweet/ fruit	1	0.050, 0.080 [0.065]	0.010, 0.020 [0.015]	0.060, 0.10 [0.080]
						4	0.080, 0.040 [0.060]	0.010, 0.010 [0.010]	0.090, 0.050 [0.070]
IR-4 PR No. 09361 09361.05-GA*17 USA Tifton America, North 2006	Pepper, sweet California Wonder	3	0.147–0.148	0.0324–0.0325	Pepper, sweet/ fruit	1	0.13, 0.13 [0.13]	0.030, 0.030 [0.030]	0.16, 0.16 [0.16]
						3	0.080, 0.10 [0.090]	0.020, 0.020 [0.020]	0.10, 0.12 [0.11]
IR-4 PR No. 09361 09361.05-OH*17 USA Fremont	Pepper, sweet Queen	3	0.145–0.147	0.0182–0.0183	Pepper, sweet/ fruit	1	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]

Study No. Trial No. Country Location Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
America, North 2005									
						3	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
IR-4 PR No. 09361 09361.05-TX*33 USA Weslaco America, North 2006	Pepper, sweet Capistrano	3	0.147– 0.148	0.0367– 0.0398	Pepper, sweet/ fruit	1	0.040, 0.060 [0.050]	ND, 0.010 [< 0.01]	0.040, 0.070 [0.055]
						3	0.030, 0.030 [0.030]	ND, ND [ND]	0.030, 0.030 [0.030]
IR-4 PR No. 09361 09361.05-W124 USA Arlington America, North 2005	Pepper, sweet Bellboy	3	0.149– 0.154	0.0421– 0.0654	Pepper, sweet/ fruit	1	0.030, 0.030 [0.030]	ND, ND [ND]	0.030, 0.030 [0.030]
						3	0.020, 0.020 [0.020]	ND, ND [ND]	0.020, 0.020 [0.020]
110966 BS078-00H USA Maricopa, Arizona America, North 2000	Pepper, sweet Better Bell	3	0.146– 0.154	0.0530– 0.0539	Pepper, sweet/ fruit	7	0.016, 0.018 [0.017]	ND, ND [ND]	0.016, 0.018 [0.017]
						14	tr, tr [< 0.01]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
110966 BS079-00H USA East Bernard, Texas America, North 2000	Pepper, sweet Capistrano Bell	3	0.146– 0.155	0.0962– 0.105	Pepper, sweet/ fruit	7	tr, 0.013 [0.012]	ND, ND [ND]	< 0.01, 0.013 [0.012]
						14	tr, ND [< 0.01]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
110966 BS080-00H BS080-00H-A USA Stilwell, Kansas America, North 2000	Pepper, sweet California Wonder	3	0.150– 0.151	0.157– 0.160	Pepper, sweet/ fruit	7	0.014, 0.011 [0.013]	ND, ND [ND]	0.014, 0.011 [0.013]
						14	0.010, 0.011 [0.011]	ND, ND [ND]	0.010, 0.011 [0.011]
110966 BS080-00H BS080-00H-B USA Stilwell, Kansas America, North 2000	Pepper, sweet California Wonder	3	0.150– 0.151	0.157– 0.160	Pepper, sweet/ fruit	7	tr	ND	< 0.01 [< 0.02]
110966 BS081-00D	Pepper, sweet Enterprize	3	0.157– 0.161	0.107– 0.108	Pepper, sweet/	0	0.17, 0.26 [0.21]	0.011, 0.017 [0.014]	0.18, 0.27 [0.23]

Study No. Trial No. Country Location Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
USA Vero Beach, Florida America, North 2000					fruit				
						7	0.079, 0.058 [0.069]	tr, tr [< 0.01]	0.089, 0.068 [0.079]
						14	0.092, 0.040 [0.066]	tr, tr [< 0.01]	0.10, 0.050 [0.076]
						20	0.023, 0.041 [0.032]	ND, ND [ND]	0.023, 0.041 [0.032]
						27	tr, tr [< 0.01]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
110966 BS082-00H USA Tifton, Georgia America, North 2000	Pepper, sweet Camelot	3	0.150–0.150	0.100–0.112	Pepper, sweet/ fruit	7	0.041, 0.033 [0.037]	tr, tr [< 0.01]	0.051, 0.043 [0.047]
						14	tr, tr [< 0.01]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
110966 BS083-00D USA Fresno, California America, North 2000	Pepper, chilli Green Jalapeno	3	0.150–0.157	0.0839–0.0858	Pepper, chilli/ fruit	0	0.16, 0.14 [0.15]	0.060, 0.056 [0.058]	0.22, 0.19 [0.21]
						7	0.092, 0.12 [0.10]	0.015, 0.020 [0.018]	0.11, 0.14 [0.12]
						14	0.026, 0.013 [0.019]	tr, ND [< 0.01]	0.036, 0.013 [0.024]
						21	0.013, 0.017 [0.015]	ND, ND [ND]	0.013, 0.017 [0.015]
						28	0.028, 0.037 [0.032]	tr, tr [< 0.01]	0.038, 0.047 [0.042]
110966 BS189-00H USA Willcox, Arizona America, North 2000	Pepper, sweet Senora	3	0.152–0.186	0.0526–0.0625	Pepper, chilli/ fruit	7	0.032, 0.045 [0.039]	ND, tr [< 0.01]	0.032, 0.055 [0.044]
						14	0.040, 0.030 [0.035]	ND, ND [ND]	0.040, 0.030 [0.035]
110966 BS190-00H USA Levelland, Texas America, North 2000	Pepper, chilli Jalepeno M	3	0.153–0.161	0.0533–0.0534	Pepper, chilli/ fruit	9	tr, 0.028 [0.019]	ND, ND [ND]	< 0.01, 0.028 [0.019]
						16	0.015, ND [< 0.01]	ND, ND [ND]	0.015, < 0.01 [0.008]
110966 BS191-00H USA	Pepper, sweet Indra	3	0.149–0.154	0.0531–0.0549	Pepper, sweet/ fruit	7	0.051, 0.043 [0.047]	tr, tr [< 0.01]	0.061, 0.053 [0.057]

Study No. Trial No. Country Location Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
Fresno, California America, North 2000									
						14	0.034, 0.028 [0.031]	tr, ND [< 0.01]	0.045, 0.028 [0.036]
Greenhouse									
GAP Europe (Italy)	Pepper	4	0.216	–	–	3	–	–	–
RA-2140/06 R 2006 0817/9 0817-06 Italy I-71030 Zapponeta (Foggia) (Puglia) Europe, South 2006	Pepper, sweet Gemini F1	4	0.162	0.0144	fruit	0 [a]	0.08	< 0.01	0.09
						0	0.15	< 0.01	0.16
						1	0.15	< 0.01	0.16
						3	0.06	< 0.01	0.07
						7	0.06	< 0.01	0.07
						11	0.10	< 0.01	0.11
RA-2140/06 R 2006 0818/7* 0818-06 Spain E-12580 Benicarló (Comunidad Valenciana) Europe, South 2006	Pepper, sweet Almuden SG	4	0.203–0.216	0.0144	fruit	0 [a]	0.22	0.01	0.23
						0	0.24	0.01	0.25
						1	0.30	0.01	0.31
						3	0.21	0.01	0.22
						7	0.08	< 0.01	0.09
RA-2140/06 R 2006 0819/5 0819-06 France F-84210 Pernes les Fontaines (Provence-Cote D'azur) Europe, South 2006	Pepper, sweet Volga	4	0.1836 – 0.1944	0.0144	fruit	0 [a]	0.09	< 0.01	0.1
						0	0.14	0.01	0.15
						1	0.10	< 0.01	0.11
						3	0.11	< 0.01	0.12
						7	0.06	< 0.01	0.07
						10	0.06	< 0.01	0.07
RA-2140/06 R 2006 0820/9 0820-06 Germany D-88074 Meckenbeuren (Baden-Württemberg)	Pepper, sweet Confetti	4	0.1404 –0.216	0.0144	fruit	–1	0.21	< 0.01	0.22

Study No. Trial No. Country Location Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
Europe, North 2006									
						0	0.32	0.01	0.33
						1	0.36	0.01	0.37
						3	0.2	< 0.01	0.21
						7	0.21	0.01	0.22
						10	0.17	< 0.01	0.18
RA-2140/06 R 2006 0821/7 0821-06 Netherlands NL-1681 ND Zwaagdijk-Oost (Noord-Holland) Europe, North 2006	Pepper, sweet Sopra	4	0.1728 –0.216	0.0144	fruit	0 [a]	0.04	< 0.01	0.05
						0	0.16	0.01	0.17
						1	0.09	< 0.01	0.1
						3	0.08	< 0.01	0.09
						7	0.05	< 0.01	0.06
						10	0.06	< 0.01	0.07
RA-2140/06 R 2006 0822/5 0822-06 Spain E-04711 Almerimar (Andalucia) Europe, South 2006	Pepper, sweet Amadeo	4	0.2052 –0.216	0.0144	fruit	0 [a]	0.04	< 0.01	0.05
						0	0.24	< 0.01	0.25
						1	0.2	< 0.01	0.21
						3	0.18	< 0.01	0.19
						7	0.14	< 0.01	0.15
						10	0.11	< 0.01	0.12
RA-2069/04** R 2004 0483/2 0483-04 Italy I-70056 Molfetta (Puglia) Europe, South 2004	Pepper, sweet Sienor	4	0.162	0.0144	fruit	0 [a]	0.06	< 0.01	0.07
						0	0.09	< 0.01	0.1
						3	0.12	< 0.01	0.13
RA-2069/04 R 2004 0489/1 0489-04 Germany D-42799 Leichlingen (Hollweg) Europe, North 2004	Pepper, sweet Maratos	4	0.0648 – 0.0864	0.0144	fruit	0 [a]	0.08	< 0.01	0.09
						0	0.21	< 0.01	0.22
						1	0.17	< 0.01	0.18
						3	0.12	< 0.01	0.13

Study No. Trial No. Country Location Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
						7	0.12	< 0.01	0.13
						10	0.06	< 0.01	0.07
RA-2607/05 R 2005 1034/9 1034-05 France F-13960 Graveson (Cote D'azur) Europe, South 2005	Pepper, sweet Galileo	4	0.135–0.189	0.0144	fruit	0 [a]	0.017	< 0.01	0.031
						0	0.079	< 0.01	0.093
						3	0.059	< 0.01	0.073

[a] Prior to application

Tomato

The Meeting received data from 24 field trials conducted in the USA, three field trials conducted in Brazil, and 16 greenhouse trials conducted in Europe.

In the USA, twelve field trials were conducted during the 2000 growing season (Lenz, 2002; Report 110966) and another twelve field trials during the 2005 growing season (Gould and Harbin, 2007; Report RABSY014). For both sets of trials, three foliar applications of spiromesifen were made at 0.145 to 0.157 kg ai/ha on a 7 ± 2-day interval. Tomato fruits were frozen within six hours of harvest. Samples remained frozen during transportation to the analytical facility and prior to analysis. Fruits were homogenised in the presence of dry ice and analysed for residues of spiromesifen and Sp-enol using Method 00631.

Samples were stored frozen for up to 300 days prior to extraction, and extracts were stored for no more than six days prior to analysis. Total residues of spiromesifen (parent + Sp-enol) were stable in cucumber, melon, and bean for at least 727 days and in tomato for at least 316 days. Overall concurrent recovery results across both studies ranged from 72 to 119% recovery with relative standard deviations ranging from 0.6 to 17%.

In Brazil, three field residue trials were conducted during the 2002 growing season (Anon, 2003, Report UNSP RA-314/03; 2003, Report UNESP RA-315/03; 2013, Report UNESP RA-313/03). Four applications of spiromesifen were made to each of two plots at each location at either 0.144 kg ai/ha or 0.288 kg ai/ha. The reapplication interval was 7 days. Tomato fruits were stored for a maximum of 275 days; storage conditions were not reported. Tomato fruits were analysed for residues of spiromesifen and Sp-enol using Method 00631.

Sixteen residue trials were conducted with greenhouse-grown tomato in northern and southern Europe during the 1999 (eight trials on tomato; Schöning and Elke, 2001, Reports RA-2083/99 and RA-2084/99) and 2004 (eight trials on cherry tomato; Rosate and Eberhardt, 2005, Report RA-2068/04) growing seasons. Four applications of spiromesifen were made at a rate of 0.108 kg ai/ha × height of the leafy surface, resulting in application rates ranging from 0.108 to 0.235 kg ai/ha. Applications were made on a 9- to 11-day interval. Tomato fruits were harvested three days after the final application and at other times to assess residue decline. Samples were frozen within 24 hours of collection and remained frozen during transportation to the analytical facility and prior to analysis. Samples were homogenized in the presence of dry ice and analysed for residues of spiromesifen and Sp-enol using Method 00631.

Samples were stored frozen for up to 512 days prior to extraction. Storage durations for extracts were not provided. Total residues of spiromesifen (parent + Sp-enol) were stable in cucumber, melon, and bean for at least 727 days and in tomato for at least 316 days. Overall concurrent recovery results across all three studies ranged from 76 to 135% recovery with relative standard deviations ranging from 3.9 to 22%.

Table 49 Results of spiromesifen residue trials in tomato

Study No. Trial No. Country Location Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
Field									
GAP USA	Fruiting vegetables other than cucurbits	3	0.146	–	–	1	–	–	–
110966 BS085-00D USA Fresno, California America, North 2000	Tomato Ace 55	3	0.150–0.154	0.0829–0.0842	fruit	0	0.21, 0.19 [0.20]	0.024, 0.023 [0.023]	0.23, 0.22 [0.22]
						7	0.099, 0.051 [0.075]	tr, tr [< 0.01]	0.11, 0.061 [0.085]
						14	0.032, 0.035 [0.033]	tr, tr [< 0.01]	0.042, 0.045 [0.043]
						21	0.020, 0.020 [0.020]	tr, tr [< 0.01]	0.030, 0.030 [0.030]
						28	0.015, 0.010 [0.012]	tr, ND [< 0.01]	0.025, 0.010 [0.017]
110966 BS086-00D USA Vero Beach, Florida America, North 2000	Tomato Sunpar	3	0.152	0.0910–0.0964	fruit	0	0.13, 0.12 [0.12]	0.011, 0.015 [0.013]	0.14, 0.13 [0.14]
						7	0.034, 0.032 [0.033]	tr, tr [< 0.01]	0.044, 0.042 [0.043]
						14	tr, tr [< 0.01]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
						21	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
						28	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
110966 BS087-00H USA Hickman, California America, North 2000	Tomato Mt. Fresh	3	0.151–0.152	0.0801–0.0803	fruit	7	0.12, 0.11 [0.11]	0.010, tr [0.010]	0.13, 0.12 [0.12]
						14	0.082, 0.080 [0.081]	tr, tr [< 0.01]	0.092, 0.090 [0.091]
110966 BS088-00H USA Arroyo Grande, California America, North 2000	Tomato Shady Lady	3	0.145–0.151	0.0534–0.0537	fruit	7	0.011, 0.018 [0.014]	ND, ND [ND]	0.011, 0.018 [0.014]
						14	ND, tr [< 0.01]	ND, ND [ND]	< 0.01, < 0.01

Study No. Trial No. Country Location Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
									[< 0.02]
110966 BS089-00H USA Porterville California America, North 2000	Tomato VF 6203	3	0.150–0.152	0.0533–0.0547	fruit	7	0.055, 0.071 [0.063]	tr, tr [< 0.01]	0.065, 0.081 [0.073]
						14	0.055, 0.052 [0.053]	tr, tr [< 0.01]	0.065, 0.062 [0.063]
110966 BS090-00H USA Fresno, California America, North 2000	Tomato Cherry Tomato	3	0.147–0.150	0.0377–0.0399	fruit	7	0.24, 0.21 [0.23]	tr, tr [< 0.01]	0.25, 0.22 [0.24]
						14	0.16, 0.19 [0.18]	tr, tr [< 0.01]	0.17, 0.20 [0.19]
110966 BS091-00H USA Porterville California America, North 2000	Tomato ACE 55	3	0.152–0.156	0.0532–0.0537	fruit	7	0.057, 0.054 [0.056]	tr, tr [< 0.01]	0.067, 0.064 [0.066]
						14	0.048, 0.030 [0.039]	tr, tr [< 0.01]	0.058, 0.040 [0.049]
110966 BS092-00H USA San Marcos, California America, North 2000	Tomato La Roma Red	3	0.150–0.152	0.0528–0.0537	fruit	7	0.098, 0.057 [0.077]	tr, tr [< 0.01]	0.11, 0.067 [0.087]
						16	0.027, 0.017 [0.022]	tr, tr [< 0.01]	0.037, 0.027 [0.032]
110966 BS093-00H USA Tifton, Georgia America, North 2000	Tomato Sunny Hybrid	3	0.150–0.150	0.100–0.112	fruit	7	0.028, 0.018 [0.022]	tr, tr [< 0.01]	0.038, 0.028 [0.032]
						14	tr, tr [< 0.01]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
110966 BS094-00H USA Oxford, Indiana America, North 2000	Tomato Heinz 9423	3	0.150–0.151	0.0978–0.0986	fruit	7	0.047, 0.051 [0.049]	tr, tr [< 0.01]	0.057, 0.061 [0.059]
						14	0.037, 0.037 [0.037]	tr, tr [< 0.01]	0.047, 0.047 [0.047]
110966 BS095-00H USA Gretna, Florida America, North 2000	Tomato Florida 47	3	0.150–0.151	0.0821–0.0894	fruit	7	0.020, 0.014 [0.017]	tr, ND [< 0.01]	0.030, 0.014 [0.022]
						14	tr, 0.012 [0.011]	ND, ND [ND]	< 0.01, 0.012 [0.011]

Study No. Trial No. Country Location Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
110966 BS096-00H USA Dundee, New York America, North 2000	Tomato Mountain Pride	3	0.150–0.152	0.0535–0.0543	fruit	7	0.038, 0.041 [0.040]	tr, tr [< 0.01]	0.048, 0.051 [0.050]
						14	0.015, 0.021 [0.018]	0.010, tr [0.010]	0.025, 0.031 [0.028]
RABSY014 BS001-05H USA 18053 Germansville America, North 2005	Tomato Mountain Spring	3	0.152–0.155	0.0864–0.0866	fruit	1	0.11, 0.095 [0.10]	ND, ND [ND]	0.11, 0.095 [0.10]
						3	0.10, 0.11 [0.11]	ND, ND [ND]	0.10, 0.11 [0.11]
RABSY014 BS002-05H USA 31794 Tifton America, North 2005	Tomato Rutger	3	0.15	0.0888–0.0909	fruit	1	0.097, 0.10 [0.099]	ND, ND [ND]	0.097, 0.10 [0.099]
						3	0.047, 0.071 [0.059]	ND, ND [ND]	0.047, 0.071 [0.059]
RABSY014 BS003-05H USA 32577 Molino America, North 2005	Tomato Florida 47	3	0.148–0.157	0.117–0.125	fruit	1	0.061, 0.065 [0.063]	ND, ND [ND]	0.061, 0.065 [0.063]
						3	0.094, 0.029 [0.062]	ND, ND [ND]	0.094, 0.029 [0.062]
RABSY014 BS004-05H USA 32577 Molino America, North 2006	Tomato Amelia	3	0.146–0.150	0.103–0.114	fruit	1	0.028, 0.049 [0.038]	ND, ND [ND]	0.028, 0.049 [0.038]
						3	0.068, 0.026 [0.047]	ND, ND [ND]	0.068, 0.026 [0.047]
RABSY014 BS005-05H USA 68059 Springfield America, North 2005	Tomato Delicious	3	0.150–0.151	0.120–0.123	fruit	1	0.070, 0.060 [0.065]	ND, ND [ND]	0.070, 0.060 [0.065]
						3	0.051, 0.066 [0.058]	ND, ND [ND]	0.051, 0.066 [0.058]
RABSY014 BS006-05H USA 93706 Fresno America, North 2005	Tomato Rio Grande	3	0.148–0.149	0.0887–0.0909	fruit	1	0.14, 0.14 [0.14]	ND, ND [ND]	0.14, 0.14 [0.14]
						3	0.097, 0.13 [0.11]	ND, ND [ND]	0.097, 0.13 [0.11]
RABSY014	Tomato	3	0.150–	0.0888–	fruit	1	0.18, 0.15	ND, ND [ND]	0.18, 0.15

Study No. Trial No. Country Location Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
BS007-05H USA 93706 Fresno America, North 2005	PETO HYPEEL 303		0.151	0.0909			[0.17]		[0.17]
						3	0.11, 0.12 [0.12]	ND, ND [ND]	0.11, 0.12 [0.12]
RABSY014 BS008-05H USA Madera America, North 2005	Tomato ACE 55 VS	3	0.152–0.154	0.0805–0.0811	fruit	1	0.44, 0.23 [0.33]	0.013, ND [< 0.01]	0.45, 0.23 [0.34]
						3	0.13, 0.16 [0.15]	ND, ND [ND]	0.13, 0.16 [0.15]
RABSY014 BS009-05H USA 93657 Sanger America, North 2005	Tomato Quality 21	3	0.148–0.152	0.0955–0.1000	fruit	1	0.057, 0.055 [0.056]	ND, ND [ND]	0.057, 0.055 [0.056]
						3	0.040, 0.038 [0.039]	ND, ND [ND]	0.040, 0.038 [0.039]
RABSY014 BS010-05H USA Glenn America, North 2005	Tomato CXD 207	3	0.146–0.154	0.0844–0.0901	fruit	1	0.14, 0.11 [0.13]	ND, 0.014 [< 0.01]	0.14, 0.13 [0.14]
						3	0.17, 0.16 [0.16]	0.014, 0.019 [0.016]	0.18, 0.18 [0.18]
RABSY014 BS011-05H USA Maxwell America, North 2005	Tomato APT 410	3	0.150–0.151	0.107–0.108	fruit	1	0.042, 0.059 [0.050]	ND, ND [ND]	0.042, 0.059 [0.050]
						3	0.056, 0.074 [0.065]	ND, ND [ND]	0.056, 0.074 [0.065]
RABSY014 BS012-05H USA Hickman America, North 2005	Tomato Bobcat	3	0.152–0.153	0.108–0.108	fruit	1	0.098, 0.065 [0.082]	0.010, 0.015 [0.012]	0.11, 0.080 [0.094]
						3	0.080, 0.026 [0.053]	0.011, 0.013 [0.012]	0.091, 0.039 [0.065]
UNESP RA-313/03 BRA I-D1-624/02 BRA I-D1-624/02-S1-A Brazil Goiana–GO America, South 2002	Tomato Funny	4	0.14	0.014	fruit	3	< 0.01	n.a.	n.c.
UNESP RA-313/03 BRA I-D1-624/02 BRA I-D1-624/02-S1-B Brazil	Tomato Funny	4	0.29	0.029	fruit	3	< 0.01	n.a.	n.c.

Study No. Trial No. Country Location Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
Goiana–GO America, South 2002									
UNESP RA-314/03 BRA I-D1-624/02 BRA I-D1-624/02-S2-A Brazil Avare / SP America, South 2002	Tomato Santa Clara	4	0.14	0.014	fruit	3	< 0.01	n.a.	n.c.
UNESP RA-314/03 BRA I-D1-624/02 BRA I-D1-624/02-S2-B Brazil Avare / SP America, South 2002	Tomato Santa Clara	4	0.29	0.029	fruit	3	0.01	n.a.	n.c.
UNESP RA-315/03 BRA I-D1-624/02 BRA I-D1-624/02-C1-A Brazil Ouro Verde–GO America, South 2002	Tomato Funny	4	0.14	0.014	fruit	0	0.6	n.a.	n.c.
						1	0.14		
						3	0.01		
						5	< 0.01		
						7	n.d.		
UNESP RA-315/03 BRA I-D1-624/02 BRA I-D1-624/02-C1-B Brazil Ouro Verde–GO America, South 2002	Tomato Funny	4	0.29	0.029	fruit	3	0.02	n.a.	n.c.
Greenhouse									
GAP Europe (France/ Italy)	Tomato	4	0.216	–	–	3	–	–	–
RA-2083/99 R 1999 0291/3 0291-99 Germany D-42799 Leichlingen greenhouse Hollweg Europe, North 1999	Tomato Rogella	4	0.1728–0.2160	0.0144	fruit	0 [a]	0.05	< 0.01	0.06
						0	0.12	< 0.01	0.13
						1	0.09	< 0.01	0.10
						3	0.11	< 0.01	0.12
						6	0.06	< 0.01	0.07
						10	0.06	< 0.01	0.07
RA-2083/99 R 1999 0292/1 0292-99 Germany D-42799 Leichlingen, greenhouse Hollweg	Tomato Panovi	4	0.2160	0.0144	fruit	0	0.15	< 0.01	0.16

Study No. Trial No. Country Location Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
Europe, North 1999									
						3	0.15, 0.14 [0.15]	< 0.01, < 0.01 [< 0.01]	0.16, 0.15 [0.16]
						7	0.1	< 0.01	0.11
RA-2083/99 R 1999 0439/8 0439-99 Netherlands NL-4725 SG Wouwse Plantage Europe, North 1999	Tomato Ambiance	4	0.216	0.0144	fruit	0	0.33	< 0.01	0.34
						1	0.24	< 0.01	0.25
						3	0.21	< 0.01	0.22
						7	0.28	< 0.01	0.29
						10	0.18	< 0.01	0.19
RA-2083/99 R 1999 0440/1 0440-99 Belgium B-2860 St. Katelijne-Waver Europe, North 1999	Tomato Palmiro	4	0.216	0.0144	fruit	0	0.23	< 0.01	0.24
						3	0.19, 0.21 [0.20]	< 0.01, < 0.01 [< 0.01]	0.20, 0.22 [0.21]
						7	0.11	< 0.01	0.12
RA-2084/99 R 1999 0294/8 0294-99 Italy I-97017 S. Croce di Camerina Europe, South 1999	Tomato Felicia	4	0.1944–0.216	0.0144	fruit	0 [a]	0.14	< 0.01	0.15
						0	0.26	0.01	0.27
						1	0.25	0.01	0.26
						3	0.18	< 0.01	0.19
						7	0.17	< 0.01	0.18
						10	0.16	< 0.01	0.17
RA-2084/99 R 1999 0295/6 0295-99 Spain E-08358 Arenys de Munt Europe, South 1999	Tomato Bond	4	0.1836–0.2354	0.0144	fruit	0	0.08	< 0.01	0.09
						3	0.08	< 0.01	0.09
						7	0.06	< 0.01	0.07
RA-2084/99 R 1999 0442/8 0442-99 Portugal P-2000 Santarem (Quinta de S. Yorge)	Tomato Indalo	4	0.1080–0.1728	0.0144	fruit	0 [a]	0.05	< 0.01	0.06

Study No. Trial No. Country Location Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
Europe, South 1999									
						0	0.09	< 0.01	0.1
						1	0.09	< 0.01	0.1
						3	0.05	< 0.01	0.06
						7	0.06	< 0.01	0.07
						10	0.05	< 0.01	0.06
RA-2084/99 R 1999 0443/6 0443-99 France F-13550 Noves Europe, South 1999	Tomato Isabella	4	0.1944–0.2160	0.0144	fruit	0	0.2	0.01	0.21
						3	0.18	0.01	0.19
						7	0.17	< 0.01	0.18
RA-2068/04 R 2004 0474/3 0474-04 Belgium B-6280 Gerpinnes les Flaches (Brabant) Europe, North 2004	Tomato, cherry Bistrot	4	0.162	0.01344	fruit	0	0.17	< 0.01	0.18
						3	0.14	< 0.01	0.15
RA-2068/04 R 2004 0475/1 0475-04 Netherlands NL-2636 AR Schipluiden (Zuid-Holland) Europe, North 2004	Tomato, cherry Concita	4	0.216	0.0108	fruit	0 [a]	0.08	< 0.01	0.09
						0	0.14	< 0.01	0.15
						1	0.1	< 0.01	0.11
						3	0.09	< 0.01	0.10
						7	0.07	< 0.01	0.08
						10	0.07	< 0.01	0.08
RA-2068/04 R 2004 0477/8 0477-04 Spain E-18127 Jayena Granada (Andalucia) Europe, South 2004	Tomato, cherry Josefina	4	0.1836–0.216	0.01344	fruit	0	0.39	0.011	0.4
						3	0.23	< 0.01	0.24
RA-2068/04 R 2004 0478/6 0478-04 Italy I-97019 Vittoria (RG) (Sicilia) Europe, South 2004	Tomato, cherry Baby Tom	4	0.1783–0.216	0.0144	fruit	0	0.29	< 0.01	0.3
						3	0.16	< 0.01	0.17

Study No. Trial No. Country Location Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
RA-2068/04 R 2004 0479/4 0479-04 Greece GR-24400 Gargaliani Europe, South 2004	Tomato, cherry Sacura	4	0.162–0.216	0.012	fruit	0	0.52	0.015	0.54
						3	0.4	0.013	0.42
RA-2068/04 R 2004 0480/8 0480-04 Spain E-18127 Fornes Granada (Andalucia) Europe, South 2004	Tomato, cherry Josefina	4	0.1836–0.216	0.01344	fruit	0 [a]	0.1	0.01	0.11
						0	0.14	0.01	0.15
						1	0.07	< 0.01	0.08
						3	0.09	< 0.01	0.10
						7	0.1	0.01	0.11
						11	0.09	0.01	0.1
RA-2068/04 R 2004 0481/6 0481-04 Italy I-70054 Giovinazzo (Bari) (Puglia) Europe, South 2004	Tomato, cherry Naomy	4	0.216	0.0144	fruit	0 [a]	0.06	< 0.01	0.07
						0	0.11	< 0.01	0.12
						1	0.12	< 0.01	0.13
						3	0.08	< 0.01	0.09
						7	0.03	< 0.01	0.04
						10	0.03	< 0.01	0.04
RA-2068/04 R 2004 0482/4 0482-04 France F-13210 St Rémy de Provence (Provence-Cote D'azur) Europe, South 2004	Tomato, cherry Swit	4	0.216	0.0144	fruit	0 [a]	0.31	0.012	0.33
						0	0.48	0.014	0.5
						1	0.41	0.013	0.43
						3	0.48	0.014	0.50
						7	0.35	0.018	0.37
						10	0.25	0.011	0.26

[a] Prior to application

Sweet corn

Twelve residue field trials on sweet corn were conducted in the USA during the 2006 growing season (Dallstream and Gould, 2008; Report RABSP003). At each site, two treatment plots were established. To one plot, a single application was made at 0.28 kg ai/ha. To the other plot, two applications were

made on a 14-day retreatment interval, each at 0.15 kg ai/ha. Samples of sweet corn kernel (including cob, with husk removed) were frozen within 9 hours of harvest and remained frozen during transportation to the analytical facility and prior to analysis. Samples were homogenized in the presence of dry ice and analysed for residues of spiromesifen and Sp-enol using Method 00631/M001.

Samples were stored frozen for up to 452 days prior to extraction, and extracts were stored for no more than six days prior to analysis. Total residues of spiromesifen (parent + Sp-enol) were stable in corn forage, grain, and stover for at least 318 days and in wheat forage grain and hay, and sugar beet root for at least 679 days. Overall concurrent recovery results ranged from 71 to 112% recovery with relative standard deviations ranging from 4.1 to 10%.

Table 50 Results of spiromesifen residue trials in sweet corn

Study No. Trial No. Country Location Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
GAP USA	Sweet corn	1	0.28	–	–	5	–	–	–
		2	0.30	–	–	5	Do not exceed 0.30 kg ai/ha per season		
RABSP003 BS030-06HA BS030-06HA-A USA Germansville America, North 2006	Corn, sweet Butter & Sugarr	1	0.295	0.158	kernels and cob, husked	5	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
						10	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
		2	0.150–0.154	0.0846–0.0863	kernels and cob, husked	5	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
RABSP003 BS031-06HA USA North Rose America, North 2006	Corn, sweet Bold	1	Not Reported due to incomplete documentation						
		2	0.147–0.149	0.0871–0.0872	kernels and cob, husked	5	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
RABSP003 BS032-06HA BS032-06HA-A USA Tifton America, North 2006	Corn, sweet Sweet G-90	1	0.28	0.205	kernels and cob, husked	5	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
						10	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
		2	0.15	0.106–0.110	kernels and cob, husked	5	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
RABSP003 BS033-06HA BS033-06HA-A USA Molino America, North 2006	Corn, sweet Silver Queen	1	0.28	0.242	kernels and cob, husked	3	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
						9	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]

Study No. Trial No. Country Location Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
		2	0.145–0.147	0.126–0.130	kernels and cob, husked	3	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
RABSP003 BS034-06DA BS034-06DA-A USA Seymour America, North 2006	Corn, sweet BC 0805	1	0.292	0.228	kernels and cob, husked	0	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
						5	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
						9	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
						15	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
						19	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
		2	0.148–0.157	0.115–0.122	kernels and cob, husked	0	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
						5	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
						9	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
						15	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
						19	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
RABSP003 BS035-06HA BS035-06HA-A USA Springfield America, North 2006	Corn, sweet Serendipity	1	0.28	0.214	kernels and cob, husked	4	tr, ND [< 0.01]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
						9	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
		2	0.151–0.152	0.116–0.117	kernels and cob, husked	4	tr, tr [< 0.01]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
RABSP003 BS036-06HA BS036-06HA-A USA Earlham America, North 2006	Corn, sweet Mirai 131	1	0.284	0.221	kernels and cob, husked	5	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
						9	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
		2	0.148	0.123–0.126	kernels and cob, husked	5	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
RABSP003 BS037-06HA BS037-06HA-A USA	Corn, sweet 39H85 Pioneer	1	0.277	0.162	kernels and cob, husked	4	tr, ND [< 0.01]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]

Study No. Trial No. Country Location Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
Sabin America, North 2006									
						8	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
		2	0.145–0.148	0.0841–0.0873	kernels and cob, husked	5	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
RABSP003 BS038-06HA BS038-06HA-A USA Stilwell America, North 2006	Corn, sweet Peaches & Cream	1	0.279	0.173	kernels and cob, husked	4	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
						10	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
		2	0.147–0.148	0.0862–0.0930	kernels and cob, husked	4	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
RABSP003 BS039-06HA BS039-06HA-A USA Fresno America, North 2006	Corn, sweet Silver Queen	1	0.285	0.165	kernels and cob, husked	5	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
						10	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
		2	0.150–0.155	0.0889–0.0892	kernels and cob, husked	5	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
RABSP003 BS040-06HA BS040-06HA-A USA Rupert America, North 2006	Corn, sweet Northern Xtra	1	0.282	0.172	kernels and cob, husked	5	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
						8	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
		2	0.150–0.151	0.0924–0.17	kernels and cob, husked	5	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
RABSP003 BS041-06HA BS041-06HA-A USA Cornelius America, North 2006	Corn, sweet Serendipity	1	0.279	0.167	kernels and cob, husked	5	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
						11	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
		2	0.150–0.154	0.0907–0.0954	kernels and cob, husked	5	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]

Leafy vegetables (including Brassica leafy vegetables)

Lettuce (head lettuce and leaf lettuce)

Twelve field trials were conducted on lettuce (6 head + 6 leaf) in the USA during the 2000 growing season (Lenz, 2002; Report 200146). Three foliar applications ranging from 0.149 to 0.158 kg ai/ha were made on a 7 ± 2-day interval. Lettuce samples were harvested ca. 7 and 14 days after the last application. Samples were frozen within four hours of collection and remained frozen during transportation to the analytical facility and prior to analysis. Samples were homogenized in the presence of dry ice and analysed for residues of spiromesifen and Sp-enol using Method 00631.

Samples were stored frozen for up to 272 days prior to extraction, and extracts were stored for no more than nine days prior to analysis. Total residues of spiromesifen (parent + Sp-enol) were stable in mustard greens for at least 347 days. Overall concurrent recovery results ranged from 70 to 120% recovery with relative standard deviations ranging from 5.2 to 15%.

Table 51 Results of spiromesifen residue trials in lettuce

Study No. Trial No. Country Location Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
GAP Canada/USA	Leafy veg.	3	0.144	–	–	7	–	–	–
200146 BS049-00H USA North Rose, New York America, North 2000	Lettuce, head Ithaca	3	0.151–0.157	0.0648–0.0650	head	7	0.13, 0.13 [0.13]	0.022, 0.029 [0.026]	0.16, 0.16 [0.16]
						14	ND, 0.010 [< 0.01]	tr, tr [< 0.01]	< 0.01, 0.020 [0.015]
					head, trimmed	7	0.013	tr	0.023
						14	ND	ND	< 0.01 [< 0.02]
200146 BS050-00H USA High Springs, Florida America, North 2000	Lettuce, head Medallion	3	0.150–0.151	0.117–0.126	head	9	2.3, 2.1 [2.2]	0.16, 0.20 [0.18]	2.5, 2.3 [2.4]
						14	0.14, 0.39 [0.27]	0.14, 0.14 [0.14]	0.29, 0.52 [0.41]
					head, trimmed	9	0.99	0.1	1.1
						14	0.33	0.084	0.41
200146 BS051-00D USA Fresno, California America, North 2000	Lettuce, head Sharp Shooter	3	0.150–0.150	0.0694–0.0695	head	0	2.4, 2.0 [2.2]	0.15, 0.14 [0.15]	2.6, 2.1 [2.3]
						7	0.98, 0.75 [0.87]	0.086, 0.065 [0.076]	1.1, 0.82 [0.94]
						14	0.73, 0.51 [0.62]	0.069, 0.62 [0.34]	0.80, 1.1 [0.97]
						20	0.28, 0.34 [0.31]	0.027, 0.024 [0.026]	0.31, 0.36 [0.34]

Study No. Trial No. Country Location Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
						28	0.12, 0.28 [0.20]	tr, 0.020 [0.015]	0.13, 0.29 [0.21]
200146 BS052-00H USA San Marcos, California America, North 2000	Lettuce, head Salinas, MI	3	0.150–0.156	0.0538–0.0539	head	7	0.72, 0.58 [0.65]	0.099, 0.079 [0.089]	0.82, 0.66 [0.74]
						12	0.38, 0.39 [0.38]	0.049, 0.070 [0.059]	0.42, 0.46 [0.44]
					head, trimmed	7	0.026	tr	0.036
						12	0.088	0.02	0.11
200146 BS053-00H USA Stanfield, Arizona America, North 2000	Lettuce, head Diamond	3	0.150–0.150	0.0519–0.0536	head	7	4.2, 4.0 [4.1]	0.40, 0.31 [0.36]	4.6, 4.3 [4.5]
						14	2.7, 2.2 [2.5]	0.31, 0.36 [0.33]	3.0, 2.6 [2.8]
					head, trimmed	7	1.2	0.14	1.3
						14	0.2	0.04	0.24
200146 BS054-00H USA Guadalupe, California America, North 2000	Lettuce, head Cowboy	3	0.149–0.158	0.0535–0.0536	head	6	1.2, 1.3 [1.2]	0.15, 0.14 [0.15]	1.3, 1.4 [1.4]
						13	0.64, 0.60 [0.62]	0.071, 0.066 [0.069]	0.71, 0.67 [0.69]
					head, trimmed	6	0.014	ND	0.014
						13	ND	ND	< 0.01 [< 0.02]
200146 BS043-00H USA North Rose, New York America, North 2000	Lettuce, leaf Black Seeded Simpson	3	0.149–0.157	0.0650–0.0650	leaf	7	0.44, 0.46 [0.45]	0.062, 0.073 [0.068]	0.51, 0.53 [0.52]
						14	0.21, 0.18 [0.20]	0.039, 0.041 [0.040]	0.25, 0.22 [0.24]
200146 BS044-00H USA High Springs, Florida America, North 2000	Lettuce, leaf Green Vision	3	0.149–0.151	0.117–0.123	leaf	8	1.0, 0.82 [0.93]	0.086, 0.083 [0.085]	1.1, 0.91 [1.0]
						14	0.14, 0.13 [0.14]	0.027, 0.021 [0.024]	0.17, 0.16 [0.16]
200146 BS045-00D BS045-00D-A USA	Lettuce, leaf Waldmann's Green	3	0.149–0.158	0.0815–0.0852	leaf	0	4.8, 4.5 [4.7]	0.19, 0.20 [0.20]	5.0, 4.7 [4.9]

Study No. Trial No. Country Location Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
Fresno, California America, North 2000									
						7	2.4, 2.3 [2.4]	0.10, 0.091 [0.096]	2.6, 2.4 [2.5]
						14	1.1, 1.2 [1.2]	0.077, 0.069 [0.073]	1.2, 1.3 [1.2]
						21	0.90, 1.4 [1.1]	0.052, 0.059 [0.056]	0.95, 1.5 [1.2]
						28	0.67, 0.48 [0.57]	0.040, 0.046 [0.043]	0.71, 0.52 [0.62]
200146 BS045-00D BS045-00D-B USA Fresno, California America, North 2000	Lettuce, leaf Waldmann's Green	3	0.149–0.158	0.0815–0.0852	leaf	7	1.6	0.096	1.7
200146 BS046-00H USA Yuma, Arizona America, North 2000	Lettuce, leaf Shining Star	3	0.151–0.158	0.0525–0.0537	leaf	7	8.0, 9.5 [8.7]	0.50, 0.53 [0.52]	8.5, 10 [9.3]
						14	4.0, 3.3 [3.7]	0.32, 0.26 [0.29]	4.4, 3.5 [3.9]
200146 BS047-00H USA San Marcos, California America, North 2000	Lettuce, leaf Red salat bowl	3	0.151–0.155	0.0538–0.0539	leaf	7	0.84, 0.71 [0.77]	0.12, 0.14 [0.13]	0.95, 0.85 [0.90]
						13	0.071, 0.081 [0.076]	0.019, 0.022 [0.020]	0.090, 0.10 [0.097]
200146 BS048-00HA USA Spreckels, California America, North 2000	Lettuce, leaf Shining Star	3	0.152–0.154	0.0535–0.0536	leaf	7	1.4, 1.2 [1.3]	0.37, 0.36 [0.37]	1.7, 1.6 [1.7]
						14	0.020, 0.017 [0.019]	0.011, tr [0.010]	0.031, 0.027 [0.029]

Spinach

Six field trials were conducted on spinach in the USA during the 2000 growing season (Lenz, 2002; Report 200146). Parameters of the spinach trials are identical to those summarized above for lettuce. Overall concurrent recovery results ranged from 72 to 118% recovery with relative standard deviations ranging from 2.7 to 14%.

Table 52 Results of spiromesifen residue trials in spinach

Study No. Trial No. Country Location Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
GAP Canada/USA	Leafy veg.	3	0.144	–	–	7	–	–	–
200146 BS055-00H USA Germansville, Pennsylvania America, North 2000	Spinach Tyee	3	0.150–0.155	0.0516–0.0526	leaf	8	0.21, 0.14 [0.18]	0.095, 0.096 [0.095]	0.31, 0.24 [0.27]
						14	0.070, 0.092 [0.081]	0.041, 0.044 [0.042]	0.11, 0.14 [0.12]
200146 BS056-00H USA Wilcox, Arizona America, North 2001	Spinach Bolero F1	3	0.151–0.155	0.0795–0.0817	leaf	9	5.8, 8.4 [7.1]	0.25, 0.30 [0.27]	6.0, 8.6 [7.3]
						14	3.4, 4.6 [4.0]	0.17, 0.14 [0.16]	3.6, 4.8 [4.2]
200146 BS057-00H USA Brookshire, Texas America, North 2000	Spinach Bloomsdale	3	0.151–0.152	0.0354–0.0355	leaf	6	1.5, 1.4 [1.5]	0.34, 0.33 [0.34]	1.9, 1.8 [1.8]
						13	0.18, 0.17 [0.17]	0.050, 0.050 [0.050]	0.23, 0.22 [0.22]
200146 BS058-00D USA Fresno, California America, North 2000	Spinach Hybrid 424	3	0.149–0.150	0.0819–0.0861	leaf	0	7.3, 6.9 [7.1]	0.30, 0.27 [0.29]	7.6, 7.1 [7.4]
						7	4.4, 5.2 [4.8]	0.18, 0.22 [0.20]	4.6, 5.4 [5.0]
						14	0.75, 0.95 [0.85]	0.051, 0.022 [0.037]	0.80, 0.97 [0.89]
200146 BS059-00H USA Maricopa, Arizona America, North 2000	Spinach Bolero F1	3	0.156–0.161	0.0529–0.0542	leaf	7	5.2, 5.9 [5.5]	0.79, 0.91 [0.85]	6.0, 6.8 [6.4]
						14	4.9, 3.8 [4.4]	0.81, 0.64 [0.73]	5.7, 4.5 [5.1]
200146 BS060-00H BS060-00H-A USA Suffolk, Virginia America, North 2001	Spinach Tyee	3	0.152–0.157	0.0814–0.0876	leaf	7	2.2, 2.0 [2.1]	0.10, 0.094 [0.098]	2.3, 2.1 [2.2]
						14	0.12, 0.16 [0.14]	0.011, 0.011 [0.011]	0.13, 0.17 [0.15]
200146 BS060-00H	Spinach Tyee	3	0.152–0.157	0.0814–0.0876	leaf, un-washed	7	1.5	0.15	1.6

Study No. Trial No. Country Location Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
BS060-00H-B USA Suffolk, Virginia America, North 2001									

Mustard greens

Residue field trials were conducted in the USA during the 2000 (five trials; Duah, 2002, Report 200172) and 2004 (three trials; Duah and Harbin, 2005, Report RABSY011) growing seasons. Three foliar applications of spiromesifen were made at rates ranging from 0.148 to 0.155 kg ai/ha on a 5- to 10-day interval. Mustard greens samples were frozen within two hours of harvest and remained frozen during transportation to the analytical facility and prior to analysis. Samples were homogenized in the presence of dry ice and analysed for residues of spiromesifen and Sp-enol using Method 00631.

Samples were stored frozen for up to 314 days (200172) or 287 days (RABSY011) prior to extraction, and extracts were stored for no more than 5 days (200172) or 1 day (RABSY011) prior to analysis. Total residues of spiromesifen (parent + Sp-enol) were stable in mustard greens for at least 347 days. Overall concurrent recovery results across both studies ranged from 73 to 114% recovery with relative standard deviations ranging from 2.0 to 10%.

Table 53 Results of spiromesifen residue trials in mustard greens

Study No. Trial No. Country Location Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
GAP Canada/USA	Brassica leafy veg.	3	0.144	–	–	7	–	–	–
200172 BS073-00H USA Brookshire, Texas America, North 2000	Mustard, Indian Florida Broadleaf	3	0.149– 0.150	0.0352– 0.0356	leaf	6	0.45, 0.52 [0.49]	0.17, 0.17 [0.17]	0.63, 0.69 [0.66]
						13	0.045, 0.088 [0.066]	0.035, 0.037 [0.036]	0.080, 0.13 [0.10]
200172 BS074-00H BS074-00H-A USA Tifton, Georgia America, North 2000	Mustard, Indian Florida Broadleaf	3	0.150– 0.150	0.0967– 0.101	leaf	8	1.1, 1.0 [1.0]	0.25, 0.26 [0.25]	1.3, 1.3 [1.3]
						14	0.13, 0.50 [0.31]	0.045, 0.21 [0.13]	0.18, 0.70 [0.44]
200172 BS074-00H BS074-00H-B USA Tifton, Georgia America, North	Mustard, Indian Florida Broadleaf	3	0.150– 0.150	0.0967– 0.101	leaf	8	0.57	0.39	0.96

Study No. Trial No. Country Location Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
2000									
200172 BS075-00H USA Greenville Mississippi America, North 2000	Mustard, Indian Florida Broadleaf	3	0.149–0.150	0.123–0.128	leaf	6	2.6, 1.2 [1.9]	0.30, 0.20 [0.25]	2.9, 1.4 [2.1]
						14	0.38, 0.070 [0.23]	0.10, 0.033 [0.069]	0.48, 0.10 [0.29]
200172 BS076-00H USA Fresno, California America, North 2000	Mustard, Indian Florida Broadleaf	3	0.148–0.154	0.0814–0.0850	leaf	7	9.3, 9.6 [9.4]	0.61, 0.46 [0.53]	9.9, 10 [10]
						14	5.0, 5.6 [5.3]	0.20, 0.22 [0.21]	5.2, 5.8 [5.5]
200172 BS077-00D USA Oxford, Indiana America, North 2000	Mustard, Indian Southern giant	3	0.150–0.151	0.0992–0.102	leaf	0	3.3, 7.8 [5.5]	0.55, 0.66 [0.60]	3.8, 8.4 [6.1]
						8	1.2, 1.5 [1.3]	0.23, 0.27 [0.25]	1.4, 1.7 [1.6]
						14	0.50, 0.71 [0.61]	0.16, 0.20 [0.18]	0.66, 0.91 [0.79]
						21	0.16, 0.19 [0.18]	0.049, 0.055 [0.052]	0.21, 0.25 [0.23]
						28	0.081, 0.074 [0.077]	0.026, 0.026 [0.026]	0.11, 0.100 [0.10]
RABSY011 BS001-04H USA 31794 Tifton America, North 2004	Mustard, Indian Broadleaf Mustard	3	0.149	0.0876–0.0914	leaf	6	9.9, 9.2 [9.5]	0.38, 0.30 [0.34]	10, 9.5 [9.9]
RABSY011 BS002-04H USA 32577 Molino America, North 2004	Mustard, Indian Giant Southern Curled	3	0.148–0.151	0.0993–0.104	leaf	5	0.91, 1.0 [0.96]	0.12, 0.13 [0.12]	1.0, 1.1 [1.1]
RABSY011 BS003-04H USA 93706 Fresno America, North 2004	Mustard, Indian Florida Broadleaf	3	0.150–0.155	0.0872–0.0896	leaf	7	1.2, 1.3 [1.2]	0.23, 0.22 [0.22]	1.4, 1.6 [1.5]

Legume vegetables

Beans

Eight greenhouse trials were conducted in southern Europe with climbing French beans (Schöning and Elke, 2001; Reports RA-2085/99 and RA-2019/00). Four foliar applications were made at 8- to 13-day intervals, each at 0.072 kg ai/ha × foliage height (m). Total application rates ranged from 0.468 to 0.586 kg ai/ha. Bean (with pod) samples were frozen within two hours of harvest and remained frozen during transportation to the analytical facility and prior to analysis. Samples were homogenized in the presence of dry ice and analysed for residues of spiromesifen and Sp-enol using Method 00631.

Samples were stored frozen for up to 502 days prior to extraction. Total residues of spiromesifen (parent + Sp-enol) were stable in climbing French bean for at least 727 days. Overall concurrent recovery results across both studies ranged from 84 to 108% recovery with relative standard deviations ranging from 2.9 to 7.2%.

Table 54 Results of spiromesifen residue trials in beans with pods

Study No. Trial No. Country Location Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
GAP Europe (Greece, Italy)	Bean (fresh)	4	0.144	–	–	3	–	–	–
RA-2085/99 R 1999 0296/4 0296-99 Spain E-08358 Arenys de Munt Europe, South 1999	Bean, climbing French Iluro	4	0.1296–0.144	0.0144	bean with pod	0 [a]	0.02	0.01	0.03
						0	0.09	0.05	0.16
						1	0.06	0.04	0.11
						3	0.05	0.02	0.08
						7	0.04	0.02	0.07
						10	0.02	< 0.01	0.03
RA-2085/99 R 1999 0297/2 0297-99 Spain E-08840 Viladecans Europe, South 1999	Bean, climbing French Nuria	4	0.144	0.0144	bean with pod	0	0.09	0.02	0.12
						3	0.03	0.01	0.04
						6	0.02	< 0.01	0.03
RA-2085/99 R 1999 0444/4 0444-99 France F-13870 Rogonas Europe, South 1999	Bean, climbing French Helda	4	0.144	0.0144	bean with pod	0 [a]	0.02	0.02	0.05
						0	0.23	0.08	0.34
						1	0.17	0.07	0.27
						3	0.16	0.07	0.26
						7	0.1	0.06	0.18
						10	0.06	0.03	0.1

Study No. Trial No. Country Location Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
RA-2085/99 R 1999 0445/2 0445-99 Italy I-04100 Borgo Piave Europe, South 1999	Bean, climbing French Emerite	4	0.072–0.144	0.0144	bean with pod	0	0.09	0.04	0.14
						3	0.05	0.03	0.09
						7	0.03	0.02	0.06
RA-2019/00 R 2000 0171/1 0171-00 Portugal P-2580 Carmeiros Europe, South 2000	Bean, climbing French Festival	4	0.1368–0.144	0.0144	bean with pod	0 [a]	0.02	< 0.01	0.03
						0	0.1	0.01	0.11
						1	0.11	0.01	0.12
						3	0.05	< 0.01	0.06
						7	0.01	< 0.01	0.02
						10	0.02	< 0.01	0.03
RA-2019/00 R 2000 0173/8 0173-00 France F-13870 Rognonas Europe, South 2000	Bean, climbing French Helda	4	0.144	0.0144	bean with pod	0 [a]	0.15	0.01	0.16
						0	0.5	0.04	0.55
						1	0.58	0.04	0.63
						3	0.59	0.04	0.64
						7	0.46	0.03	0.5
						10	0.52	0.03	0.56
RA-2019/00 R 2000 0174/6 0174-00 Spain E-08840 Viladecans Europe, South 2000	Bean, climbing French Nuria	4	0.144–0.1546	0.0144	bean with pod	0	0.22	0.02	0.25
						3	0.13	0.01	0.14
						8	0.07	< 0.01	0.08
RA-2019/00 R 2000 0175/4 0175-00 Italy I-00050 Palidoro-Fiumicino Europe, South 2000	Bean, climbing French Emerite	4	0.0864–0.144	0.0144	bean with pod	0	0.12	0.02	0.15
						3	0.06	0.01	0.07
						7	0.02	< 0.01	0.03

[a] Before treatment

Pulses

Bean (dry seed)

Three field trials were conducted in Brazil during the 2002 growing season (Galhiane, and de Sousa, 2003, Report UNESP RA-310/03; Anon, 2003, Report UNESP RA-311/03; Anon, 2003, UNESP RA-312/03). Spiromesifen was applied to two plots at each location; one plot received three applications on a 7-day interval at 0.144 kg ai/ha and the second plot received three applications on a 7-day interval at 0.288 kg ai/ha. Bean seeds were harvested 21 days after the final application. Samples were stored for a maximum of 341 days; storage conditions were not reported. Bean seeds were analysed using Method 00631; only residues of spiromesifen were reported. Concurrent recovery samples gave recoveries ranging from 75 to 95%, with a maximum relative standard deviation of 8.9%.

Table 55 Results of spiromesifen trials on dry bean

Study No. Trial No. Country Location Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
GAP Brazil	Bean (dry seed)	3	0.144	–	–	21	–	–	–
UNESP RA-310/03 BRA I-D3-622/02 BRA I-D3-622/02-S1-A Brazil Porteirao–GO America, South 2002	Bean Perola	3	0.14	0.048	seed	21	0.01	n.a.	n.c.
UNESP RA-310/03 BRA I-D3-622/02 BRA I-D3-622/02-S1-B Brazil Porteirao–GO America, South 2002	Bean Perola	3	0.29	0.096	seed	21	0.02	n.a.	n.c.
UNESP RA-311/03 BRA I-D3-622/02 BRA I-D3-622/02-S2-A Brazil Avare / SP America, South 2002	Bean Carioca	3	0.14	0.048	seed	21	0.01	n.a.	n.c.
UNESP RA-311/03 BRA I-D3-622/02 BRA I-D3-622/02-S2-B Brazil Avare / SP	Bean Carioca	3	0.29	0.096	seed	21	0.03	n.a.	n.c.

Study No. Trial No. Country Location Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
America, South 2002									
UNESP RA-312/03 BRA I-D3-622/02 BRA I-D3-622/02-S3-A Brazil Santa Cruz do Sul / RS America, South 2002	Bean Minuano	3	0.14	0.048	seed	21	0.01	n.a.	n.c.
UNESP RA-312/03 BRA I-D3-622/02 BRA I-D3-622/02-S3-B Brazil Santa Cruz do Sul / RS America, South 2002	Bean Minuano	3	0.29	0.096	seed	21	0.02	n.a.	n.c.

n.a. = Not analysed

n.c. = Not calculated

Root and tuber vegetables

Cassava (manioc)

Five residue field trials were conducted in Brazil during the 2014 growing season (Silva, 2014; Report I14-032). Three applications of spiromesifen were made on a 5-day interval, each at between 0.135 and 0.156 kg ai/ha. At each trial, samples of cassava root were harvested 7 to 35 days after the last application. Residues of spiromesifen and Sp-enol were analysed using Method 00631. Samples were placed into frozen storage within one day of collection and were maintained frozen for, at most, 67 days prior to extraction and analysis. Concurrent recoveries ranged from 87 to 103%, with a range in relative standard deviation of 2.2 to 5.4%.

Table 56 Results of spiromesifen residue trials in cassava

Study No. Trial No., Country Location, Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
GAP US	Tuberous and corm veg.	2	0.28	–	–	7	–	–	–
Alternative GAP Canada	Tuberous and corm veg.	2	0.144	–	–	7	–	–	–
I14-032 I14-032-01	Manioc	3	0.135–0.153	0.0338–0.0383	root	7	< 0.01	< 0.01	< 0.02

Study No. Trial No., Country Location, Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
Brazil Paulinia America, South 2014									
						14	< 0.01	< 0.01	< 0.02
						21	< 0.01	< 0.01	< 0.02
						28	< 0.01	< 0.01	< 0.02
						35	< 0.01	< 0.01	< 0.02
I14-032 I14-032-02 Brazil Uberlandia America, South 2014	Manioc	3	0.142– 0.152	0.0355– 0.0380	root	7	< 0.01	< 0.01	< 0.02
						14	< 0.01	< 0.01	< 0.02
						21	< 0.01	< 0.01	< 0.02
						28	< 0.01	< 0.01	< 0.02
						33	< 0.01	< 0.01	< 0.02
I14-032 I14-032-03 Brazil Cravinhos America, South 2014	Manioc	3	0.144– 0.150	0.0360– 0.0375	root	7	< 0.01	< 0.01	< 0.02
						14	< 0.01	< 0.01	< 0.02
						21	< 0.01	< 0.01	< 0.02
						28	< 0.01	< 0.01	< 0.02
						35	< 0.01	< 0.01	< 0.02
I14-032 I14-032-04 Brazil Ribeirao Preto America, South 2014	Manioc	3	0.151– 0.156	0.0378– 0.0390	root	7	< 0.01	< 0.01	< 0.02
						14	< 0.01	< 0.01	< 0.02
						21	< 0.01	< 0.01	< 0.02
						28	< 0.01	< 0.01	< 0.02
						35	< 0.01	< 0.01	< 0.02
I14-032 I14-032-05 Brazil Trindade America, South 2014	Manioc	3	0.137– 0.142	0.0343– 0.0356	root	7	< 0.01	< 0.01	< 0.02
						14	< 0.01	< 0.01	< 0.02
						21	< 0.01	< 0.01	< 0.02
						28	< 0.01	< 0.01	< 0.02
						35	< 0.01	< 0.01	< 0.02

Potato

Sixteen field trials were conducted during the 2000 growing season in the USA (Fischer, 2002; Report 200182). Two foliar applications of spiromesifen were made at rates ranging from 0.270 to 0.297 kg ai/ha on a 7-day interval. Potato samples were harvested 7 days after the last application. Samples were frozen within four hours of harvest and remained frozen during transportation to the

analytical facility and prior to analysis. Samples were homogenized in the presence of dry ice and analysed for residues of spiromesifen and Sp-enol using Method 00631.

Samples were stored frozen for up to 258 days, and extracts were stored for no more than two day prior to analysis. Total residues of spiromesifen (parent + Sp-enol) were stable in potato for at least 320 days. Overall concurrent recovery results ranged from 76 to 111% recovery with relative standard deviations ranging from 6.7 to 10%.

Table 57 Results of spiromesifen residue trials in potato

Study No. Trial No., Country Location, Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
GAP USA	Tuberous and corm veg.	2	280	–	–	7	–	–	–
200182 BS172-00H USA Germansville, Pennsylvania America, North 2000	Potato Andover	2	0.287–0.289	0.0859–0.0866	tuber	7	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
200182 BS173-00H USA Dundee, New York America, North 2000	Potato Dark Red Norland	2	0.270–0.281	0.0992–0.100	tuber	7	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
200182 BS174-00H USA Tifton, Georgia America, North 2000	Potato Chieftain	2	0.277–0.280	0.192–0.240	tuber	8	ND, ND [ND]	tr, ND [< 0.01]	< 0.01, < 0.01 [< 0.02]
200182 BS175-00D USA Vero Beach, Florida America, North 2000	Potato Atlantic	2	0.282–0.291	0.190–0.190	tuber	0	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
						7	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
						14	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
						21	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
200182 BS176-00H USA Springfield, Nebraska America, North 2000	Potato Kennebec	2	0.277–0.278	0.175–0.178	tuber	7	ND, ND [ND]	tr, tr [< 0.01]	< 0.01, < 0.01 [< 0.02]
200182 BS177-00H USA Stilwell, Kansas America, North 2000	Potato Kennebec	2	0.281	0.295–0.298	tuber	7	ND, ND [ND]	tr, tr [< 0.01]	< 0.01, < 0.01 [< 0.02]
200182 BS178-00D USA Oxford, Indiana	Potato Kennebec	2	0.280–0.281	0.183–0.184	tuber	0	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]

Study No. Trial No., Country Location, Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
America, North 2000									
						6	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
						13	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
						20	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
200182 BS179-00H USA Britton, South Dakota America, North 2000	Potato Russet Burbank	2	0.273–0.281	0.150–0.150	tuber	7	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
200182 BS180-00H USA Smithfield, Utah America, North 2000	Potato Ranger Russet	2	0.277–0.281	0.100–0.101	tuber	7	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
200182 BS181-00H USA Kerman, California America, North 2000	Potato White Rose	2	0.285–0.288	0.101–0.101	tuber	7	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
200182 BS182-00H USA Pocatello, Idaho America, North 2000	Potato Ranger-G-3	2	0.284–0.297	0.142–0.148	tuber	7	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
200182 BS183-00H USA Jerome, Idaho America, North 2000	Potato Russett Burbank	2	0.279–0.294	0.090–0.0931	tuber	7	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
200182 BS184-00H USA Ephrata, Washington America, North 2000	Potato Russett Burbank	2	0.282–0.284	0.148–0.149	tuber	7	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
200182 BS185-00H USA Culver, Oregon America, North 2000	Potato Norchip	2	0.282–0.295	0.120–0.144	tuber	7	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
200182 BS186-00H USA Nampa, Idaho America, North 2000	Potato Ranger Russet	2	0.279–0.280	0.0994–0.100	tuber	7	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
200182 BS187-00H USA Hermiston, Oregon	Potato Russet Burbank	2	0.280–0.281	0.0964–0.0998	tuber	7	ND, ND [ND]	ND, tr [< 0.01]	< 0.01, < 0.01 [< 0.02]

Study No. Trial No., Country Location, Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
America, North 2000									

Cereal grains

Maize

Forty field trials were conducted on maize in the USA during the 2000 and 2001 (20 trials; Duah, 2002, Report 200178) or 2006 (20 trials; Dallstream, 2011; Report RABSP002-1) growing seasons. In the first study (200178), plots were treated twice, each at 0.140 to 0.156 kg ai/ha, on a ca. 14-day interval. In the second study, plots were treated once at 0.271 to 0.291 kg ai/ha. Corn grain was harvested 30 days after the last application. Samples were frozen within four hours of harvest and remained frozen during transportation to the analytical facility and prior to analysis. Samples were homogenized in the presence of dry ice and analysed for residues of spiromesifen and Sp-enol using Method 00631.

Samples were stored frozen for up to 239 days (200178) or 409 days (RABSP002-1), and extracts were stored for no more than nine days (200178) or three days (RABSP002-1) prior to analysis. Total residues of spiromesifen (parent + Sp-enol) were stable in corn grain for at least 318 days and in wheat grain for at least 679 days. Overall concurrent recovery results across both studies ranged from 72 to 119% recovery with relative standard deviations ranging from 8.8 to 18%.

Five residue field trials were conducted in Brazil during the 2013 growing season (Santiago, 2013; Report I12-057). Two applications of spiromesifen were made on a 7-day interval, each at between 0.144 and 0.157 kg ai/ha. At each trial, samples of corn grain were harvested 7 to 28 days after the last application. Residues of spiromesifen and Sp-enol were analysed using Method 00631. Samples were stored for a maximum of 179 days; storage conditions were not reported. Concurrent recoveries ranged from 84 to 103%, with a range in relative standard deviation of 2.2 to 6.1%.

Table 58 Results of spiromesifen residue trials in maize

Study No. Trial No., Country Location, Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
GAP USA	Maize (field corn)	1	0.28	–	–	30	–	–	–
		2	0.30	–	–	30	Do not exceed 0.30 kg ai/ha per season		
GAP Canada	Maize (field corn)	2	0.144	–	–	30	–	–	–
GAP Mexico	Maize	n.s.	0.192	–	–	10	–	–	–
200178 BS022-00HA USA Germansville, Pennsylvania America, North 2001	Maize/Corn 642XP	2	0.151– 0.156	0.0402– 0.0403	grain	28	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
200178 BS023-00DA USA Tifton, Georgia	Maize/Corn DK662RR	2	0.15	0.141– 0.151	grain	25	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]

Study No. Trial No., Country Location, Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
America, North 2001									
						32	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
						39	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
						42	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
200178 BS024-00H USA Carlyle, Illinois America, North 2000	Maize/Corn Pioneer 34B23	2	0.151– 0.154	0.0951– 0.0989	grain	28	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
200178 BS025-00D USA Louisville, Nebraska America, North 2000	Maize/Corn Cropland 666 RR	2	0.15	0.0928– 0.0973	grain	23	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
						30	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
						37	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
						42	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
200178 BS026-00H USA Stilwell, Kansas America, North 2000	Maize/Corn Asgrow	2	0.15	0.0791– 0.159	grain	29	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
200178 BS027-00H USA Oxford, Indiana America, North 2000	Maize/Corn Hoegemeyer 2551	2	0.149– 0.151	0.0721– 0.0763	grain	32	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
200178 BS028-00H USA Hedric, Iowa America, North 2000	Maize/Corn Golden Harvest H2552	2	0.149– 0.155	0.0469– 0.0480	grain	31	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
200178 BS029-00H USA Richland, Iowa America, North 2000	Maize/Corn Pioneer 33A14	2	0.151– 0.155	0.0452– 0.0493	grain	31	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
200178 BS030-00H USA Centerville, South	Maize/Corn Novartis N52- B2	2	0.15	0.0811– 0.0819	grain	30	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]

Study No. Trial No., Country Location, Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
Dakota America, North 2000									
200178 BS031-00H USA Lesterville, South Dakota America, North 2000	Maize/Corn Sucrosco SX109	2	0.150–0.154	0.0810–0.0825	grain	29	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [≤ 0.02]
200178 BS032-00H USA Kirklin, Indiana America, North 2000	Maize/Corn Pioneer 33A14	2	0.140–0.151	0.0960–0.0963	grain	30	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [≤ 0.02]
200178 BS033-00H USA Northwood, North Dakota America, North 2000	Maize/Corn Mycogen 2110	2	0.147–0.152	0.0523–0.0543	grain	31	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [≤ 0.02]
200178 BS034-00HA USA Berkley, Iowa America, North 2000	Maize/Corn 35R60	2	0.146–0.151	0.0834–0.0837	grain	30	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [≤ 0.02]
200178 BS035-00HA USA Perry, Iowa America, North 2000	Maize/Corn 35R60	2	0.148–0.149	0.0834–0.0837	grain	30	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [≤ 0.02]
200178 BS036-00HA USA Jefferson, Iowa America, North 2000	Maize/Corn 3489	2	0.149–0.151	0.0834–0.0843	grain	31	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [≤ 0.02]
200178 BS037-00HA USA Bagley, Iowa America, North 2000	Maize/Corn DK545RR	2	0.15	0.0723–0.0840	grain	30	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [≤ 0.02]
200178 BS038-00H USA Coon Rapids, Iowa America, North 2000	Maize/Corn 561SR	2	0.147–0.152	0.0717–0.0758	grain	31	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [≤ 0.02]
200178 BS039-00H USA Keysport, Illinois America, North 2000	Maize/Corn Buurus 671RR	2	0.149	0.0954–0.0990	grain	28	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [≤ 0.02]
200178	Maize/Corn	2	0.149–	0.0617–	grain	31	ND, ND [ND]	ND, ND [ND]	< 0.01,

Study No. Trial No., Country Location, Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
BS040-00H USA Wyoming, Illinois America, North 2000	Pioneer 34B23		0.152	0.0636					< 0.01 [≤ 0.02]
200178 BS041-00H USA Eakly, Oklahoma America, North 2000	Maize/Corn N7070BT	2	0.151– 0.154	0.0869– 0.0892	grain	29	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [≤ 0.02]
RABSP002-1 BS010-06HA USA North Rose America, North 2006	Maize/ Corn Garst 8917 CB/LL	1	0.29	0.167	grain	30	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [≤ 0.02]
RABSP002-1 BS011-06HA USA Tifton America, North 2006	Maize/ Corn 31N26	1	0.28	0.2	grain	30	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [≤ 0.02]
RABSP002-1 BS012-06DA USA Carlyle America, North 2006	Maize/ Corn B-T 6516RR2YG	1	0.285	0.169	grain	20	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
						25	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
						28	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [≤ 0.02]
						35	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
						40	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
RABSP002-1 BS013-06DA USA Richland America, North 2006	Maize/ Corn Pioneer 33P65	1	0.282	0.18	grain	20	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
						23	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
						29	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [≤ 0.02]
						34	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
						38	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]

Study No. Trial No., Country Location, Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
RABSP002-1 BS014-06HA USA Gardner America, North 2006	Maize/ Corn Garst 8881 RR	1	0.282	0.207	grain	28	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
RABSP002-1 BS015-06HA USA Springfield America, North 2006	Maize/ Corn NK38B4	1	0.282	0.209	grain	29	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
RABSP002-1 BS016-06HA USA Sabin America, North 2006	Maize/ Corn Pioneer 39H85	1	0.271	0.161	grain	29	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
RABSP002-1 BS017-06HA USA Seymour America, North 2006	Maize/ Corn Garst 8568 CB/LL	1	0.286	0.168	grain	28	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
RABSP002-1 BS018-06HA USA Earlham America, North 2006	Maize/ Corn 33N30 LL	1	0.277	0.243	grain	28	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
RABSP002-1 BS019-06HA USA Percival America, North 2006	Maize/ Corn NK 65C5	1	0.277	0.213	grain	28	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
RABSP002-1 BS020-06HA USA Freeman America, North 2006	Maize/ Corn Garst 8287 RR	1	0.282	0.209	grain	29	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
RABSP002-1 BS021-06HA USA New Holland America, North 2006	Maize/ Corn Crows 7R154	1	0.285	0.189	grain	30	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
RABSP002-1 BS022-06HA USA York America, North 2006	Maize/ Corn 34N45 RR/YG	1	0.279	0.152	grain	30	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
RABSP002-1 BS023-06HA USA Clarence America, North 2006	Maize/ Corn 34B20	1	0.28	0.156	grain	30	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]

Study No. Trial No., Country Location, Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
RABSP002-1 BS024-06HA USA Northwood America, North 2006	Maize/ Corn Pioneer 39D81	1	0.28	0.169	grain	31	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
RABSP002-1 BS025-06HA USA Gardner America, North 2006	Maize/ Corn DKC35-51	1	0.282	0.184	grain	30	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
RABSP002-1 BS026-06HA USA Sheridan America, North 2006	Maize/ Corn DKC 57-59 RR/plus	1	0.281	0.163	grain	30	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
RABSP002-1 BS027-06HA USA Arkansaw America, North 2006	Maize/ Corn 38B85	1	0.286	0.16	grain	30	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
RABSP002-1 BS028-06HA USA Geneva America, North 2006	Maize/ Corn Pioneer 38H66	1	0.279	0.185	grain	29	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
RABSP002-1 BS029-06HA USA Raymondville America, North 2006	Maize/ Corn Yellow Surecropper	1	0.291	0.197	grain	30	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
I12-057 I12-057-01 Brazil 2013	Maize/ Corn P30F35HR	2	0.144	0.072	corn cobs	7	< 0.01	< 0.01	< 0.02
						14	< 0.01	< 0.01	< 0.02
						21	< 0.01	< 0.01	< 0.02
						28	< 0.01	< 0.01	< 0.02
I12-057 I12-057-02 Brazil 2013	Maize/ Corn AG 7088 PRO	2	0.144– 0.14967	0.0720– 0.0748	corn cobs	7	< 0.01	< 0.01	< 0.02
						14	< 0.01	< 0.01	< 0.02
						21	< 0.01	< 0.01	< 0.02
						28	< 0.01	< 0.01	< 0.02
I12-057 I12-057-03 Brazil 2013	Maize/ Corn Formula TL	2	0.15167 – 0.15667	0.0758– 0.0783	corn cobs	7	< 0.01	< 0.01	< 0.02
						14	< 0.01	< 0.01	< 0.02
						21	< 0.01	< 0.01	< 0.02
						28	< 0.01	< 0.01	< 0.02
I12-057 I12-057-04	Maize/ Corn Bandei-rante	2	0.14532 –	0.0727– 0.0747	corn cobs	7	< 0.01	< 0.01	< 0.02

Study No. Trial No., Country Location, Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
Brazil 2013			0.14933						
						14	< 0.01	< 0.01	< 0.02
						21	< 0.01	< 0.01	< 0.02
						28	< 0.01	< 0.01	< 0.02
I12-057 I12-057-05 Brazil 2013	Maize/ Corn P30F50	2	0.14829 – 0.15384	0.0741– 0.0769	corn cobs	7	< 0.01	< 0.01	< 0.02
						14	< 0.01	< 0.01	< 0.02
						21	< 0.01	< 0.01	< 0.02
						28	< 0.01	< 0.01	< 0.02

Popcorn

Three residue trials were conducted on popcorn in the USA during the 2006 growing season (Dallstream, 2008; Report RABSP004). At each location spiromesifen was applied once at 0.275 to 0.278 kg ai/ha to one plot and twice, on a 12- to 14-day interval, at 0.149 to 0.152 kg ai/ha to a second plot. Popcorn grain was harvested ca. 30 days after the last application. Samples were frozen within four hours of harvest and remained frozen during transportation to the analytical facility and prior to analysis. Samples were homogenized in the presence of dry ice and analysed for residues of spiromesifen and Sp-enol using Method 00631 or Method 00631/M001.

Samples were stored frozen for up to 392 days, and extracts were stored for no more than one day prior to analysis. Total residues of spiromesifen (parent + Sp-enol) were stable in corn grain for at least 318 days and in wheat grain for at least 679 days. Overall concurrent recovery results ranged from 70 to 102% recovery. The relative standard deviation across all concurrent recovery samples was 19%.

Table 59 Results of spiromesifen field trials in popcorn

Study No. Trial No., Country Location, Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
GAP USA	Popcorn	1	0.28	–	–	30	–	–	–
		2	0.30	–	–	30	Do not exceed 0.30 kg ai/ha per season		
RABSP004 BS042-06HA BS042-06HA-A Earlham, Iowa America, North 2006	Popcorn HW214	1	0.275	0.224	grain	29	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
RABSP004 BS042-06HA BS042-06HA-B Earlham, Iowa America, North 2006	Popcorn HW214	2	0.149– 0.152	0.122– 0.142	grain	29	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
RABSP004 BS043-06HA BS043-06HA-A Stilwell, Kansas	Popcorn Yellow	1	0.276	0.200	grain	29	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]

Study No. Trial No., Country Location, Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
America, North 2006									
RABSP004 BS043-06HA BS043-06HA-B Stilwell, Kansas America, North 2006	Popcorn Yellow	2	0.150– 0.151	0.107– 0.108	grain	29	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
RABSP004 BS044-06HA BS044-06HA-A Springfield, Nebraska America, North 2006	Popcorn AP2501	1	0.278	0.212	grain	28	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
RABSP004 BS044-06HA BS044-06HA-B Springfield, Nebraska America, North 2006	Popcorn AP2501	2	0.151	0.115	grain	28	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]

Oilseeds

Cotton

Twelve field trials were conducted in the USA during the 2000 and 2001 growing seasons (Lemke, 2002; Report 200116). Two foliar applications of spiromesifen were made to cotton, on a 7-day interval, at 0.272 to 0.292 kg ai/ha. Cotton bolls were harvested ca. 30 days after the last application by manual methods as well as mechanical picker (three trials) and mechanical stripper (three trials). Samples were frozen within four hours of harvest (except for two samples which were frozen within 24 hours) and remained frozen during transportation to the analytical facility and prior to analysis, except for some samples which thawed (maximum temperature = 12 °C) due to a 48-hr power outage. Samples were ginned to produce undelinted cotton seed and cotton gin trash; each of which was homogenized in the presence of dry ice and analysed for residues of spiromesifen and Sp-enol using Method 00631 or Method 00631/M001.

Samples were stored frozen for up to 121 days, and extracts were stored for no more than three days prior to analysis. Total residues of spiromesifen (parent + Sp-enol) were stable in undelinted cotton seed for at least 318 days and in cotton gin trash for at least 322 days. Overall concurrent recovery results ranged from 77 to 91% recovery. The relative standard deviations ranged from 6.0 to 8.1%.

Three trials were conducted in Brazil during the 2002 growing season (Anon., 2003; Reports UNESP RA-307/03, UNESP RA-308/03, UNESP RA-309/03). Spiromesifen was applied two times to two treatment plots at each location with an interval of 10 days. The application rates were 0.144 kg ai/ha for one plot and 0.288 kg ai/ha for the second plot. Samples of cotton seed were harvested 21 days after the last application. Storage durations and conditions were not provided. Cotton seeds were analysed using Method 00631; only residues of spiromesifen were reported. Concurrent recovery samples gave recoveries ranging from 68 to 114%, with a maximum relative standard deviation of 17%.

Table 60 Results of spiromesifen residue trials in cotton

Study No. Trial No., Country Location, Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
GAP USA	Cotton	3	0.280	–	–	30	Do not exceed 0.56 kg ai/ha per season		
200116 BS001-00H USA Fresno, California America, North 2000	Cotton Riata C-176 (acala)	2	0.280	0.151–0.156	seed, undelinted	28	0.098, 0.17 [0.14]	0.034, 0.050 [0.042]	0.13, 0.22 [0.18]
200116 BS002-00D USA Benoit, Mississippi America, North 2000	Cotton ST 474	2	0.273–0.284	0.283–0.31	seed, undelinted	20	0.045	0.068	0.11
						28	0.034	0.076	0.11
						35	0.014	0.048	0.061
						46	0.010	0.055	0.066
200116 BS003-00H USA Poplar, California America, North 2000	Cotton Delta Pine 6102	2	0.280–0.284	0.0995–0.105	seed, undelinted	30	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
200116 BS004-00H USA Maricopa, Arizona America, North 2000	Cotton Nu-Cotton 33B	2	0.272–0.285	0.100–0.101	seed, undelinted	29	0.019, 0.014 [0.016]	ND, ND [ND]	0.019, 0.014 [< 0.02]
200116 BS005-00H USA Levelland, Texas America, North 2000	Cotton Paymaster 2200RR	2	0.285–0.292	0.100–0.101	seed, undelinted	28	0.012, 0.010 [0.011]	ND, ND [ND]	0.012, 0.010 [< 0.02]
200116 BS006-00H USA Groom, Texas America, North 2000	Cotton Delta Pine 2156	2	0.284–0.285	0.0827–0.0830	seed, undelinted	31	0.053, 0.063 [0.058]	0.061, 0.051 [0.056]	0.11, 0.11 [0.11]
200116 BS007-00H USA Colony, Oklahoma America, North 2000	Cotton PM 1218	2	0.280–0.281	0.127	seed, undelinted	35	0.090, 0.18 [0.13]	0.14, 0.16 [0.15]	0.23, 0.33 [0.28]
200116 BS008-00H USA Plainview, Texas America, North 2000	Cotton Paymaster 2200 RR	2	0.280–0.282	0.141–0.146	seed, undelinted	30	0.16, 0.18 [0.17]	0.14, 0.15 [0.15]	0.30, 0.33 [0.32]
200116 BS009-00HA USA Comanche, Oklahoma America, North	Cotton Paymaster 2326 BG/RR	2	0.273–0.277	0.125–0.131	seed, undelinted	32	tr, 0.019 [0.015]	0.010, 0.029 [0.020]	0.020, 0.048 [0.034]

Study No. Trial No., Country Location, Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
2001									
200116 BS010-00H USA Newport, Arkansas America, North 2000	Cotton Paymaster 1218 BG/RR	2	0.277–0.289	0.0996–0.0997	seed, undelinted	30	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [≤ 0.02]
200116 BS011-00H USA Oil Trough, Arkansas America, North 2000	Cotton Sure Grow SG 501 B/R	2	0.282–0.288	0.1	seed, undelinted	31	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [≤ 0.02]
200116 BS012-00H USA Tifton, Georgia America, North 2000	Cotton DPL-451	2	0.280	0.291–0.300	seed, undelinted	28	0.16, 0.24 [0.20]	0.16, 0.22 [0.19]	0.31, 0.46 [0.39]
UNESP RA-307/03 BRA I-K1-621/02 BRA I-K1-621/02-S1-A Brazil Goiania / GO America, South 2002	Cotton Ita 90	2	0.14	0.072	seed	21	0.01	n.a.	n.c.
UNESP RA-307/03 BRA I-K1-621/02 BRA I-K1-621/02-S1-B Brazil Goiania / GO America, South 2002	Cotton Ita 90	2	0.29	0.14	seed	21	0.02	n.a.	n.c.
UNESP RA-308/03 BRA I-K1-621/02 BRA I-K1-621/02-S2-A Brazil Avare / SP America, South 2002	Cotton Delta Opal	2	0.14	0.072	seed	21	0.01	n.a.	n.c.
UNESP RA-308/03 BRA I-K1-621/02 BRA I-K1-621/02-S2-B Brazil Avare / SP America, South 2002	Cotton Delta Opal	2	0.29	0.14	seed	21	0.03	n.a.	n.c.
UNESP RA-309/03 BRA I-K1-621/02 BRA I-K1-621/02-S3-A Brazil Bandeirantes–PR America, South 2002	Cotton IPR 95	2	0.14	0.072	seed	21	0.01	n.a.	n.c.
UNESP RA-309/03 BRA I-K1-621/02 BRA I-K1-621/02-S3-B Brazil Bandeirantes–PR America, South	Cotton IPR 95	2	0.29	0.14	seed	21	0.02	n.a.	n.c.

Study No. Trial No., Country Location, Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
2002									

Seed for beverages and sweets

Coffee

Five field trials were conducted in Brazil during the 2013 growing season (Santiago, 2014; Report I13-002). Two foliar applications of spiromesifen were made on a 7-day interval at rates ranging from 0.140 to 0.152 kg ai/ha. Samples of green coffee beans were harvested at post-treatment intervals ranging from 0 to 28 days. Residues of spiromesifen and Sp-enol were analysed using Method 00631. Details regarding sample storage conditions and durations were not provided in English. Concurrent recoveries ranged from 80 to 102%, with a range in relative standard deviation of 1.1 to 11%.

Table 61 Results of spiromesifen residue trials on coffee

Study No. Trial No., Country Location, Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
GAP Colombia	Coffee	1	0.120	–	–	35	–	–	–
GAP Brazil	Coffee	2	0.144			21			
I13-002 I13-002-01 Brazil Paulínia America, South 2013	Coffee Catuai Vermelho	2	0.144	0.036	bean, green	0	0.040, 0.030 [0.035]	ND, ND [ND]	0.040, 0.030 [0.035]
						7	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
						14	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
						21	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
						28	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
I13-002 I13-002-02 Brazil Araguari America, South 2013	Coffee Catuai Vermelho	2	0.1407– 0.14752	0.0352– 0.0369	bean, green	0	0.090, 0.10 [0.095]	0.010, 0.030 [0.020]	0.10, 0.13 [0.12]
						7	0.060, 0.060 [0.060]	0.010, 0.010 [0.010]	0.070, 0.070 [0.070]
						14	0.030, 0.030 [0.030]	ND, ND [ND]	0.030, 0.030 [0.030]
						21	0.020, 0.020 [0.020]	ND, ND [ND]	0.020, 0.020 [0.020]
						28	0.020, 0.020 [0.020]	0.010, 0.020 [0.015]	0.030, 0.040 [0.035]

Study No. Trial No., Country Location, Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
I13-002 I13-002-03 Brazil Ribeirão Preto America, South 2013	Coffee Mundo Novo	2	0.14135– 0.1524	0.0353– 0.0381	bean, green	0	0.020, 0.020 [0.020]	0.010, 0.020 [0.015]	0.030, 0.040 [0.035]
						7	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
						14	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
						21	ND, ND [ND]	0.12, 0.10 [0.11]	0.12, 0.10 [0.11]
						28	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
I13-002 I13-002-05 Brazil Pardinho America, South 2013	Coffee Catuai Vermelho	2	0.14016– 0.14647	0.0350– 0.0366	bean, green	0	0.11, 0.080 [0.095]	0.040, 0.040 [0.040]	0.15, 0.12 [0.14]
						7	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
						14	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
						21	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
						28	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
I13-002 I13-002-06 Brazil Cristais Paulistas America, South 2013	Coffee Catuai	2	0.14414– 0.147	0.0360– 0.0368	bean, green	0	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
						7	ND, ND [ND]	0.010, 0.010 [0.010]	0.010, 0.010 [< 0.02]
						14	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
						21	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]
						28	ND, ND [ND]	ND, ND [ND]	< 0.01, < 0.01 [< 0.02]

Processed foods of plant origin

Tea, green, black (black, fermented and dried)

Eight trials were conducted on tea; six GLP trials in India during the 2008 growing season (Sathya Moorthy, 2011; Report G5078) and two non-GLP trials in Japan (Anon., 2004; Reports JAP-GT 07.2004 and JAP-GT 10.2004). For both sets of trials, spiromesifen was applied once at a rate of 0.6 kg ai/ha and tea shoots were harvested 0 and 7 days after treatment. Samples from India were transported to the analytical facility within 24 hours where they were stored frozen prior to analysis of spiromesifen and Sp-enol using Method 01038. Samples from Japan were analysed per a non-GLP HPLC-MS method of Japan Plant Protection Association (M-387601-01-2).

Samples from India were stored frozen for up to 199 days. Storage time for extracts was not reported. Total residues of spiromesifen (parent + Sp-enol) were stable in various leafy crops for at least 318 days. Storage conditions and durations for the samples from Japan were not reported. Overall concurrent recovery results across three studies ranged from 72 to 116% recovery. The relative standard deviations ranged from 1.1 to 12%.

Table 62 Results of spiromesifen residue trials on tea

Study No. Trial No., Country Location, Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
GAP Japan	Tea	1	0.63	–	–	7	–	–	–
G5078 G5078-N1 India Jorhat, Assam Asia, Middle 2008	Tea Seed Jat	1	0.6	0.15	leaf, green	0	29, 28 [29]	15, 15 [15]	44, 44 [44]
						7	0.58, 0.53 [0.55]	0.11, 0.11 [0.11]	0.69, 0.64 [0.66]
G5078 G5078-N2 India Dikom, Assam Asia, Middle 2008	Tea	1	0.6	0.15	leaf, green	0	35, 35 [35]	9.3, 9.3 [9.3]	44, 44 [44]
						7	8.3, 8.4 [8.4]	1.6, 1.6 [1.6]	9.9, 10 [10]
G5078 G5078-N3 India Jalpaiguri, West Bengal Asia, Middle 2008	Tea TV 9	1	0.6	0.15	leaf, green	0	46, 45 [45]	11, 11 [11]	56, 56 [56]
						7	4.1, 4.0 [4.0]	0.28, 0.37 [0.32]	4.3, 4.4 [4.4]
G5078 G5078-S1 India Valparai, Tamil Nadu Asia, Middle 2008	Tea Mixture of clones (UPASI-6, UPASI-9, UPASI-3)	1	0.6	0.15	leaf, green	0	81, 78 [80]	1.4, 1.3 [1.3]	83, 79 [81]
						7	7.1, 6.7 [6.9]	0.18, 0.18 [0.18]	7.3, 6.9 [7.1]
					tea, green	7	14	2.3	17
					tea, black	7	17	5.7	23

Study No. Trial No., Country Location, Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
G5078 G5078-S2 India Coonoor, Tamil Nadu Asia, Middle 2008	Tea B/6/61 (UPASI-9)	1	0.6	0.15	leaf, green	0	44, 45 [44]	1.1, 1.1 [1.1]	46, 46 [46]
						7	7.2, 7.6 [7.4]	0.34, 0.32 [0.33]	7.5, 7.9 [7.7]
G5078 G5078-S3 India Munnar, Kerala Asia, Middle 2008	Tea UPASI-9	1	0.6	0.15	leaf, green	0	46, 58 [52]	0.80, 1.1 [0.93]	47, 59 [53]
						7	1.3, 1.3 [1.3]	0.16, 0.16 [0.16]	1.4, 1.4 [1.4]
JAP-GT 07.2004 JAP-GT 07.2004-A JAP-GT 07.2004-A1 Japan Saitama Asia, East 2004	Tea Yabukita	1	0.6	0.015	tea, green	3	66, 63 [65]	12, 11 [12]	78, 74 [76]
						7	15, 15 [15]	4.6, 4.6 [4.6]	19, 19 [19]
						14	3.4, 3.2 [3.3]	1.2, 1.2 [1.2]	4.6, 4.4 [4.5]
JAP-GT 07.2004 JAP-GT 07.2004-B JAP-GT 07.2004-B1 Japan Kagoshima Asia, East 2004	Tea Okumidori	1	0.6	0.015	tea, green	3	71, 70 [71]	23, 23 [23]	93, 93 [93]
						7	2.9, 2.8 [2.8]	2.6, 2.6 [2.6]	5.5, 5.4 [5.4]
						14	0.92, 0.92 [0.92]	1.8, 1.8 [1.8]	2.8, 2.7 [2.7]
JAP-GT 10.2004 JAP-GT 10.2004-A JAP-GT 10.2004-A1 Japan Saitama Asia, East 2004	Tea Yabukita	1	0.6	0.015	tea, green	3	55, 54 [55]	18, 18 [18]	73, 72 [73]
						7	14, 14 [14]	8.0, 7.7 [7.9]	22, 21 [21]
						14	2.9, 2.8 [2.9]	1.9, 1.8 [1.8]	4.8, 4.6 [4.7]
JAP-GT 10.2004 JAP-GT 10.2004-B JAP-GT 10.2004-B1 Japan Kagoshima Asia, East 2004	Tea Okumidori	1	0.6	0.015	tea, green	3	51, 50 [51]	34, 32 [33]	84, 82 [83]
						7	2.9, 2.6 [2.7]	3.8, 3.6 [3.7]	6.6, 6.2 [6.4]
						14	0.64, 0.62 [0.63]	2.9, 2.7 [2.8]	3.5, 3.4 [3.4]

Straw, fodder, and forage of cereal grains and grasses

Maize, popcorn, and sweetcorn

Residue trials for corn forage and fodder matrices are the same as those for corn grain described above. Concurrent recoveries over both studies ranged from 71 to 136% for maize forage and fodder, with relative standard deviations from 1.0 to 22%, from 72 to 101% for popcorn fodder, with an overall relative standard deviation of 15%, and from 76 to 115% for sweet corn forage and fodder, with relative standard deviations from 8.2 to 15%.

Table 63 Results from spiromesifen residue trials in corn forages and fodders.

Study No. Trial No., Country Location, Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
GAP USA	Maize (field corn)	1	0.28	–	–	5-Forage 30-Fodder	–	–	–
		2	0.30 (≤0.30/ year)	–	–	5-Forage 30-Fodder	–	–	–
GAP Canada	Maize (field corn)	2	0.144	–	–	5-Forage 30-Fodder	–	–	–
GAP Mexico	Maize	n.s.	0.192	–	–	10	–	–	–
200178 BS022-00HA USA Germansville, Pennsylvania America, North 2001	Maize/Corn 642XP	2	0.151–0.156	0.0402–0.0403	Forage	6	1.7, 2.0 [1.8]	0.089, 0.11 [0.099]	1.8, 2.1 [1.9]
					Fodder	28	0.49, 0.41 [0.45]	0.064, 0.063 [0.064]	0.56, 0.47 [0.51]
200178 BS023-00DA USA Tifton, Georgia America, North 2001	Maize/Corn DK662RR	2	0.15	0.141–0.151	Forage	7	2.6, 1.2 [1.9]	0.12, 0.072 [0.094]	2.7, 1.3 [2.0]
						14	0.32, 0.39 [0.35]	0.065, 0.079 [0.072]	0.38, 0.47 [0.43]
						21	0.15, 0.16 [0.16]	0.073, 0.058 [0.066]	0.23, 0.22 [0.22]
						28	0.11, 0.096 [0.11]	0.051, 0.054 [0.052]	0.16, 0.15 [0.16]
					Fodder	25	0.18, 0.15 [0.16]	0.073, 0.081 [0.077]	0.25, 0.23 [0.24]
						32	0.065, 0.068 [0.066]	0.070, 0.054 [0.062]	0.14, 0.12 [0.13]
						39	0.039, 0.090 [0.064]	0.047, 0.074 [0.061]	0.086, 0.16 [0.13]
						42	0.058, 0.038 [0.048]	0.073, 0.050 [0.062]	0.13, 0.088 [0.11]
200178 BS024-00H USA Carlyle, Illinois	Maize/Corn Pioneer 34B23	2	0.151–0.154	0.0951–0.0989	Forage	5	2.4, 1.5 [1.9]	0.46, 0.26 [0.36]	2.8, 1.7 [2.3]

Study No. Trial No., Country Location, Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
America, North 2000									
					Fodder	28	0.74, 0.78 [0.76]	0.39, 0.41 [0.40]	1.1, 1.2 [1.2]
200178 BS025-00D USA Louisville, Nebraska America, North 2000	Maize/Corn Cropland 666 RR	2	0.15	0.0928–0.0973	Forage	2	0.47, 0.22 [0.34]	0.36, 0.33 [0.34]	0.83, 0.55 [0.69]
						9	0.26, 0.11 [0.19]	0.54, 0.42 [0.48]	0.79, 0.53 [0.66]
						16	0.080, 0.075 [0.077]	0.40, 0.47 [0.44]	0.48, 0.55 [0.51]
						23	0.082, 0.13 [0.11]	0.37, 0.55 [0.46]	0.45, 0.68 [0.57]
					Fodder	23	0.16, 0.16 [0.16]	0.74, 0.71 [0.73]	0.90, 0.87 [0.88]
						30	0.12, 0.081 [0.10]	0.63, 0.45 [0.54]	0.76, 0.54 [0.65]
						37	0.097, 0.10 [0.10]	0.65, 0.71 [0.68]	0.75, 0.82 [0.78]
						42	0.089, 0.090 [0.089]	0.50, 0.50 [0.50]	0.59, 0.59 [0.59]
200178 BS026-00H USA Stilwell, Kansas America, North 2000	Maize/Corn Asgrow	2	0.15	0.0791–0.159	Forage	0	1.3, 1.5 [1.4]	0.13, 0.14 [0.13]	1.5, 1.6 [1.5]
					Fodder	29	0.14, 0.066 [0.10]	0.24, 0.26 [0.25]	0.38, 0.32 [0.35]
200178 BS027-00H USA Oxford, Indiana America, North 2000	Maize/Corn Hoegemeyer 2551	2	0.149–0.151	0.0721–0.0763	Forage	11	0.47, 0.71 [0.59]	0.14, 0.18 [0.16]	0.61, 0.89 [0.75]
					Fodder	32	0.41, 0.14 [0.28]	0.18, 0.100 [0.14]	0.60, 0.24 [0.42]
200178 BS028-00H USA Hedric, Iowa America, North 2000	Maize/Corn Golden Harvest H2552	2	0.149–0.155	0.0469–0.0480	Forage	7	0.84, 0.67 [0.76]	0.32, 0.42 [0.37]	1.2, 1.1 [1.1]
					Fodder	31	0.15, 0.17 [0.16]	0.22, 0.23 [0.22]	0.37, 0.40 [0.38]
200178 BS029-00H USA Richland, Iowa America, North 2000	Maize/Corn Pioneer 33A14	2	0.151–0.155	0.0452–0.0493	Forage	7	0.71, 0.70 [0.70]	0.16, 0.17 [0.17]	0.87, 0.88 [0.87]
					Fodder	31	0.41, 0.34 [0.38]	0.22, 0.18 [0.20]	0.64, 0.52 [0.58]
200178 BS030-00H USA	Maize/Corn Novartis N52-B2	2	0.15	0.0811–0.0819	Forage	12	0.081, 0.12 [0.10]	0.14, 0.17 [0.16]	0.23, 0.29 [0.26]

Study No. Trial No., Country Location, Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
Centerville, South Dakota America, North 2000									
					Fodder	30	0.060, 0.12 [0.087]	0.28, 0.44 [0.36]	0.33, 0.55 [0.44]
200178 BS031-00H USA Lesterville, South Dakota America, North 2000	Maize/Corn Sucrosco SX109	2	0.150–0.154	0.0810–0.0825	Forage	13	0.49, 0.43 [0.46]	0.14, 0.13 [0.13]	0.63, 0.55 [0.59]
					Fodder	29	0.66, 0.49 [0.57]	0.27, 0.29 [0.28]	0.93, 0.77 [0.85]
200178 BS032-00H USA Kirklin, Indiana America, North 2000	Maize/Corn Pioneer 33A14	2	0.140–0.151	0.0960–0.0963	Forage	7	0.88, 0.34 [0.61]	0.23, 0.086 [0.16]	1.1, 0.43 [0.77]
					Fodder	30	ND, 0.052 [0.026]	tr, 0.041 [0.025]	< 0.01, 0.093 [0.052]
200178 BS033-00H USA Northwood, North Dakota America, North 2000	Maize/Corn Mycogen 2110	2	0.147–0.152	0.0523–0.0543	Forage	5	1.0, 1.3 [1.2]	0.12, 0.16 [0.14]	1.2, 1.5 [1.3]
					Fodder	31	1.8, 1.1 [1.4]	0.31, 0.47 [0.39]	2.1, 1.6 [1.8]
200178 BS034-00HA USA Berkley, Iowa America, North 2000	Maize/Corn 35R60	2	0.146–0.151	0.0834–0.0837	Forage	7	1.2, 1.2 [1.2]	0.38, 0.38 [0.38]	1.6, 1.6 [1.6]
					Fodder	30	0.34, 0.55 [0.45]	0.93, 0.97 [0.95]	1.3, 1.5 [1.4]
200178 BS035-00HA USA Perry, Iowa America, North 2000	Maize/Corn 35R60	2	0.148–0.149	0.0834–0.0837	Forage	7	0.46, 0.75 [0.61]	0.61, 1.4 [0.99]	1.1, 2.1 [1.6]
					Fodder	30	0.24, 0.19 [0.21]	0.94, 1.2 [1.0]	1.2, 1.3 [1.3]
200178 BS036-00HA USA Jefferson, Iowa America, North 2000	Maize/Corn 3489	2	0.149–0.151	0.0834–0.0843	Forage	8	1.1, 1.2 [1.2]	0.89, 1.0 [0.95]	2.0, 2.2 [2.1]
					Fodder	31	0.46, 0.43 [0.45]	1.0, 1.0 [1.0]	1.5, 1.5 [1.5]
200178 BS037-00HA USA	Maize/Corn DK545RR	2	0.15	0.0723–0.0840	Forage	7	0.81, 0.91 [0.86]	0.33, 0.38 [0.35]	1.1, 1.3 [1.2]

Study No. Trial No., Country Location, Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
Bagley, Iowa America, North 2000									
					Fodder	30	0.78, 0.88 [0.83]	0.27, 0.30 [0.28]	1.1, 1.2 [1.1]
200178 BS038-00H USA Coon Rapids, Iowa America, North 2000	Maize/Corn 561SR	2	0.147–0.152	0.0717–0.0758	Forage	5	0.89, 1.1 [0.98]	0.43, 0.37 [0.40]	1.3, 1.4 [1.4]
					Fodder	31	0.098, 0.088 [0.093]	0.49, 0.47 [0.48]	0.59, 0.56 [0.57]
200178 BS039-00H USA Keysport, Illinois America, North 2000	Maize/Corn Buurus 671RR	2	0.149	0.0954–0.0990	Forage	5	1.6, 1.1 [1.4]	0.37, 0.42 [0.40]	2.0, 1.5 [1.8]
					Fodder	28	0.38, 0.43 [0.41]	0.41, 0.56 [0.49]	0.80, 0.99 [0.89]
200178 BS040-00H USA Wyoming, Illinois America, North 2000	Maize/Corn Pioneer 34B23	2	0.149–0.152	0.0617–0.0636	Forage	6	0.91, 1.0 [0.96]	0.40, 0.42 [0.41]	1.3, 1.4 [1.4]
					Fodder	31	0.68, 0.53 [0.60]	0.39, 0.42 [0.40]	1.1, 0.95 [1.0]
200178 BS041-00H USA Eakly, Oklahoma America, North 2000	Maize/Corn N7070BT	2	0.151–0.154	0.0869–0.0892	Forage	6	0.78, 0.52 [0.65]	0.12, 0.11 [0.11]	0.89, 0.63 [0.76]
					Fodder	29	0.074, 0.057 [0.065]	0.027, 0.028 [0.027]	0.10, 0.084 [0.093]
RABSP002-1 BS010-06HA USA North Rose America, North 2006	Maize/ Corn Garst 8917 CB/LL	1	0.29	0.167	Forage	5	1.4, 1.6 [1.5]	0.12, 0.16 [0.14]	1.5, 1.7 [1.6]
						10	1.4, 1.4 [1.4]	0.16, 0.14 [0.15]	1.6, 1.5 [1.5]
					Fodder	30	2.2, 2.1 [2.2]	0.42, 0.50 [0.46]	2.6, 2.6 [2.6]
RABSP002-1 BS011-06HA USA Tifton America, North 2006	Maize/ Corn 31N26	1	0.28	0.2	Forage	5	2.5, 2.4 [2.5]	0.64, 0.71 [0.68]	3.2, 3.1 [3.1]
						9	1.4, 1.4 [1.4]	0.16, 0.14 [0.15]	1.6, 1.5 [1.5]
					Fodder	30	0.41, 0.63 [0.52]	0.89, 1.2 [1.0]	1.3, 1.8 [1.6]
RABSP002-1 BS012-06DA	Maize/ Corn B-T	1	0.285	0.169	Forage	0	2.6, 2.2 [2.4]	0.23, 0.21 [0.22]	2.9, 2.4 [2.6]

Study No. Trial No., Country Location, Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
USA Carlyle America, North 2006	6516RR2YG								
						5	2.4, 2.0 [2.2]	0.30, 0.32 [0.31]	2.7, 2.3 [2.5]
						10	0.10, 0.076 [0.090]	0.024, 0.026 [0.025]	0.13, 0.10 [0.11]
						14	1.3, 0.64 [0.96]	0.30, 0.16 [0.23]	1.6, 0.80 [1.2]
						20	0.54, 0.73 [0.64]	0.18, 0.26 [0.22]	0.72, 1.00 [0.86]
					Fodder	20	1.4, 1.5 [1.4]	0.48, 0.43 [0.45]	1.8, 2.0 [1.9]
						25	1.6, 1.0 [1.3]	0.58, 0.51 [0.54]	2.1, 1.5 [1.8]
						28	0.52, 0.71 [0.61]	0.29, 0.38 [0.34]	0.81, 1.1 [0.95]
						35	0.38, 0.33 [0.36]	0.33, 0.28 [0.31]	0.72, 0.61 [0.66]
						40	0.71, 0.44 [0.57]	0.16, 0.11 [0.13]	0.87, 0.55 [0.71]
RABSP002-1 BS013-06DA USA Richland America, North 2006	Maize/ Corn Pioneer 33P65	1	0.282	0.18	Forage	0	3.1, 3.1 [3.1]	0.35, 0.34 [0.34]	3.5, 3.5 [3.5]
						3	3.1, 3.5 [3.3]	0.32, 0.37 [0.34]	3.4, 3.9 [3.6]
						8	2.2, 3.6 [2.9]	0.39, 0.42 [0.40]	2.6, 4.0 [3.3]
						14	1.8, 1.5 [1.7]	0.60, 0.45 [0.53]	2.4, 2.0 [2.2]
						20	0.54, 0.23 [0.38]	0.32, 0.13 [0.23]	0.86, 0.36 [0.61]
					Fodder	20	1.2, 1.9 [1.5]	0.73, 0.98 [0.86]	1.9, 2.9 [2.4]
						23	3.1, 2.2 [2.7]	1.2, 1.1 [1.2]	4.4, 3.4 [3.9]
						29	1.4, 3.7 [2.5]	1.2, 1.9 [1.5]	2.5, 5.7 [4.1]
						34	2.0, 1.4 [1.7]	1.5, 1.3 [1.4]	3.5, 2.7 [3.1]
						38	0.62, 2.1 [1.3]	0.86, 1.5 [1.2]	1.5, 3.6 [2.5]
RABSP002-1 BS014-06HA USA Gardner America, North 2006	Maize/ Corn Garst 8881 RR	1	0.282	0.207	Forage	5	3.3, 4.0 [3.6]	0.90, 0.73 [0.81]	4.2, 4.7 [4.4]
						10	1.4, 1.4 [1.4]	0.16, 0.14 [0.15]	1.6, 1.5 [1.5]
					Fodder	28	0.67, 0.87 [0.77]	0.53, 0.67 [0.60]	1.2, 1.5 [1.4]
RABSP002-1 BS015-06HA USA Springfield America, North 2006	Maize/ Corn NK38B4	1	0.282	0.209	Forage	4	0.59, 0.90 [0.75]	0.15, 0.16 [0.16]	0.74, 1.1 [0.91]
						9	0.49, 0.46	0.22, 0.17	0.71, 0.63

Study No. Trial No., Country Location, Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
							[0.48]	[0.19]	[0.67]
					Fodder	29	0.19, 0.13 [0.16]	0.21, 0.12 [0.16]	0.41, 0.25 [0.33]
RABSP002-1 BS016-06HA USA Sabin America, North 2006	Maize/ Corn Pioneer 39H85	1	0.271	0.161	Forage	5	0.70, 1.0 [0.86]	0.12, 0.16 [0.15]	0.82, 1.2 [1.0]
						8	0.84, 0.91 [0.87]	0.15, 0.16 [0.16]	0.98, 1.1 [1.0]
					Fodder	29	0.85, 0.45 [0.65]	0.43, 0.23 [0.33]	1.3, 0.68 [0.98]
RABSP002-1 BS017-06HA USA Seymour America, North 2006	Maize/ Corn Garst 8568 CB/LL	1	0.286	0.168	Forage	5	1.8, 3.4 [2.6]	0.27, 0.42 [0.35]	2.1, 3.8 [3.0]
						8	2.3, 2.0 [2.2]	0.56, 0.46 [0.51]	2.9, 2.5 [2.7]
					Fodder	28	1.1, 1.2 [1.1]	0.82, 0.75 [0.78]	1.9, 2.0 [1.9]
RABSP002-1 BS018-06HA USA Earlham America, North 2006	Maize/ Corn 33N30 LL	1	0.277	0.243	Forage	4	1.7, 2.1 [1.9]	0.16, 0.14 [0.15]	1.9, 2.2 [2.1]
						12	0.75, 1.0 [0.90]	0.13, 0.16 [0.15]	0.88, 1.2 [1.0]
					Fodder	28	0.45, 1.5 [0.98]	0.27, 0.38 [0.32]	0.72, 1.9 [1.3]
RABSP002-1 BS019-06HA USA Percival America, North 2006	Maize/ Corn NK 65C5	1	0.277	0.213	Forage	3	2.5, 2.4 [2.5]	0.34, 0.26 [0.30]	2.9, 2.7 [2.8]
						10	1.4, 1.2 [1.3]	0.43, 0.34 [0.39]	1.8, 1.5 [1.7]
					Fodder	28	0.99, 1.2 [1.1]	0.77, 0.66 [0.72]	1.8, 1.8 [1.8]
RABSP002-1 BS020-06HA USA Freeman America, North 2006	Maize/ Corn Garst 8287 RR	1	0.282	0.209	Forage	5	1.3, 1.4 [1.4]	0.36, 0.30 [0.33]	1.7, 1.7 [1.7]
						9	1.3, 1.3 [1.3]	0.67, 0.54 [0.61]	2.0, 1.8 [1.9]
					Fodder	29	0.65, 0.50 [0.57]	0.60, 0.69 [0.64]	1.2, 1.2 [1.2]
RABSP002-1 BS021-06HA USA New Holland America, North 2006	Maize/ Corn Crows 7R154	1	0.285	0.189	Forage	5	1.9, 1.6 [1.7]	0.12, 0.12 [0.12]	2.0, 1.7 [1.8]

Study No. Trial No., Country Location, Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
						10	1.2, 1.2 [1.2]	0.065, 0.058 [0.062]	1.3, 1.3 [1.3]
					Fodder	30	1.7, 0.89 [1.3]	0.27, 0.17 [0.22]	2.0, 1.1 [1.5]
RABSP002-1 BS022-06HA USA York America, North 2006	Maize/ Corn 34N45 RR/YG	1	0.279	0.152	Forage	3	0.26, 0.26 [0.26]	0.048, 0.060 [0.054]	0.31, 0.32 [0.32]
						12	0.091, 0.13 [0.11]	0.065, 0.073 [0.069]	0.16, 0.20 [0.18]
					Fodder	30	0.13, 0.12 [0.12]	0.17, 0.20 [0.19]	0.30, 0.33 [0.31]
RABSP002-1 BS023-06HA USA Clarence America, North 2006	Maize/ Corn 34B20	1	0.28	0.156	Forage	3	0.89, 1.2 [1.1]	0.065, 0.079 [0.072]	0.96, 1.3 [1.1]
						10	1.1, 0.80 [0.95]	0.13, 0.15 [0.14]	1.2, 0.96 [1.1]
					Fodder	30	1.2, 0.93 [1.0]	0.76, 1.0 [0.90]	1.9, 2.0 [2.0]
RABSP002-1 BS024-06HA USA Northwood America, North 2006	Maize/ Corn Pioneer 39D81	1	0.28	0.169	Forage	5	1.6, 1.9 [1.7]	0.11, 0.12 [0.12]	1.7, 2.0 [1.8]
						10	1.0, 1.7 [1.4]	0.097, 0.12 [0.11]	1.1, 1.8 [1.5]
					Fodder	31	0.59, 0.54 [0.57]	0.16, 0.17 [0.17]	0.75, 0.71 [0.73]
RABSP002-1 BS025-06HA USA Gardner America, North 2006	Maize/ Corn DKC35-51	1	0.282	0.184	Forage	4	2.1, 1.7 [1.9]	0.095, 0.063 [0.079]	2.2, 1.8 [2.0]
						10	2.0, 1.8 [1.9]	0.20, 0.19 [0.19]	2.2, 2.0 [2.1]
					Fodder	30	0.24, 0.42 [0.33]	0.065, 0.12 [0.090]	0.30, 0.54 [0.42]
RABSP002-1 BS026-06HA USA Sheridan America, North 2006	Maize/ Corn DKC 57-59 RR/plus	1	0.281	0.163	Forage	4	0.15, 0.31 [0.23]	0.029, 0.052 [0.040]	0.18, 0.36 [0.27]
						10	0.90, 0.010 [0.46]	0.65, ND [0.32]	1.6, 0.010 [0.78]
					Fodder	30	0.022, ND [0.011]	0.017, ND [< 0.01]	0.039, < 0.01 [0.020]
RABSP002-1 BS027-06HA USA Arkansaw America, North	Maize/ Corn 38B85	1	0.286	0.16	Forage	5	2.1, 2.5 [2.3]	0.20, 0.24 [0.22]	2.3, 2.7 [2.5]

Study No. Trial No., Country Location, Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
2006									
						10	1.5, 1.3 [1.4]	0.27, 0.26 [0.27]	1.8, 1.5 [1.7]
					Fodder	30	0.78, 0.65 [0.72]	0.36, 0.30 [0.33]	1.1, 0.95 [1.0]
RABSP002-1 BS028-06HA USA Geneva America, North 2006	Maize/ Corn Pioneer 38H66	1	0.279	0.185	Forage	4	2.7, 2.1 [2.4]	0.14, 0.12 [0.13]	2.8, 2.3 [2.5]
						9	1.5, 1.0 [1.3]	0.13, 0.081 [0.10]	1.7, 1.1 [1.4]
					Fodder	29	0.43, 0.84 [0.64]	0.25, 0.42 [0.34]	0.68, 1.3 [0.97]
RABSP002-1 BS029-06HA USA Raymondville America, North 2006	Maize/ Corn Yellow Surecropper	1	0.291	0.197	Forage	5	2.4, 3.3 [2.8]	0.34, 0.33 [0.34]	2.7, 3.6 [3.2]
						9	1.6, 1.4 [1.5]	0.61, 0.75 [0.68]	2.2, 2.1 [2.1]
					Fodder	30	0.32, 0.17 [0.24]	0.66, 0.60 [0.63]	0.98, 0.77 [0.88]
RABSP004 BS042-06HA BS042-06HA-A GLP: yes 2006	Popcorn HW214	1	0.275	0.224	Fodder	29	0.41, 0.38 [0.39]	0.11, 0.081 [0.098]	0.52, 0.46 [0.49]
		2	0.149–0.152	0.122–0.142	Fodder	29	0.14, 0.16 [0.15]	0.064, 0.051 [0.057]	0.21, 0.21 [0.21]
RABSP004 BS043-06HA BS043-06HA-A GLP: yes 2006	Popcorn Yellow	1	0.276	0.200	Fodder	29	0.52, 0.41 [0.46]	0.34, 0.40 [0.37]	0.86, 0.81 [0.84]
		2	0.150–0.151	0.107–0.108	Fodder	29	0.18, 0.14 [0.16]	0.19, 0.19 [0.19]	0.37, 0.33 [0.35]
RABSP004 BS044-06HA BS044-06HA-A GLP: yes 2006	Popcorn AP2501	1	0.278	0.212	Fodder	28	0.60, 0.68 [0.64]	0.22, 0.24 [0.23]	0.82, 0.92 [0.87]
		2	0.151	0.115	Fodder	28	0.13, 0.18 [0.16]	0.12, 0.22 [0.17]	0.26, 0.40 [0.33]
RABSP003 BS030-06HA BS030-06HA-A USA Germansville America, North 2006	Corn, sweet Butter & Sugar	1	0.295	0.158	Forage	5	2.8, 2.6 [2.7]	0.20, 0.17 [0.18]	3.0, 2.8 [2.9]
						10	3.2, 2.6 [2.9]	0.23, 0.18 [0.20]	3.4, 2.8 [3.1]
					Fodder	30	0.87, 0.96 [0.91]	0.14, 0.96 [0.55]	1.0, 1.9 [1.5]

Study No. Trial No., Country Location, Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
RABSP003 BS030-06HA BS030-06HA-B USA Germansville America, North 2006	Corn, sweet Butter & Sugar	2	0.150–0.154	0.0846–0.0863	Forage	5	1.4, 1.3 [1.4]	0.15, 0.15 [0.15]	1.6, 1.5 [1.5]
					Fodder	30	0.37, 0.36 [0.37]	0.14, 0.12 [0.13]	0.51, 0.48 [0.50]
RABSP003 BS031-06HA USA North Rose America, North 2006	Corn, sweet Bold	2	0.147–0.149	0.0871–0.0872	Forage	5	0.33, 0.36 [0.35]	0.071, 0.085 [0.078]	0.40, 0.45 [0.42]
					Fodder	30	0.13, 0.12 [0.12]	0.034, 0.028 [0.031]	0.16, 0.14 [0.15]
RABSP003 BS032-06HA BS032-06HA-A USA Tifton America, North 2006	Corn, sweet Sweet G-90	1	0.28	0.205	Forage	5	0.83, 0.90 [0.87]	0.18, 0.14 [0.16]	1.0, 1.0 [1.0]
						10	0.81, 0.72 [0.77]	0.24, 0.29 [0.26]	1.1, 1.0 [1.0]
					Fodder	29	0.074, 0.079 [0.076]	0.12, 0.11 [0.12]	0.20, 0.19 [0.19]
		2	0.15	0.106–0.110	Forage	5	0.90, 0.16 [0.53]	0.19, 0.054 [0.12]	1.1, 0.21 [0.65]
					Fodder	29	0.12, 0.095 [0.11]	0.14, 0.12 [0.13]	0.26, 0.21 [0.24]
RABSP003 BS033-06HA BS033-06HA-A USA Molino America, North 2006	Corn, sweet Silver Queen	1	0.28	0.242	Forage	3	1.8, 1.7 [1.8]	0.23, 0.21 [0.22]	2.0, 1.9 [2.0]
						9	0.50, 0.57 [0.54]	0.12, 0.12 [0.12]	0.62, 0.70 [0.66]
					Fodder	30	0.25, 0.28 [0.27]	0.43, 0.57 [0.50]	0.68, 0.86 [0.77]
		2	0.145–0.147	0.126–0.130	Forage	3	0.84, 0.77 [0.81]	0.15, 0.13 [0.14]	0.99, 0.90 [0.95]
					Fodder	30	0.10, 0.20 [0.15]	0.36, 0.37 [0.36]	0.46, 0.57 [0.51]
RABSP003 BS034-06DA BS034-06DA-A USA Seymour America, North 2006	Corn, sweet BC 0805	1	0.292	0.228	Forage	0	2.5, 2.5 [2.5]	0.28, 0.26 [0.27]	2.8, 2.8 [2.8]
						5	1.2, 1.4 [1.3]	0.16, 0.18 [0.17]	1.4, 1.5 [1.4]
						9	0.24, 0.34 [0.29]	0.056, 0.065 [0.060]	0.30, 0.41 [0.35]
						15	0.22, 0.16	0.098, 0.083	0.32, 0.24

Study No. Trial No., Country Location, Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
							[0.19]	[0.090]	[0.28]
						19	0.10, 0.21 [0.16]	0.078, 0.13 [0.10]	0.18, 0.34 [0.26]
					Fodder	19	0.33, 0.53 [0.43]	0.34, 0.42 [0.38]	0.67, 0.95 [0.81]
						25	0.10, 0.17 [0.14]	0.12, 0.17 [0.15]	0.23, 0.34 [0.28]
						28	0.18, 0.14 [0.16]	0.16, 0.10 [0.13]	0.34, 0.24 [0.29]
						34	0.079, 0.063 [0.071]	0.040, 0.031 [0.036]	0.12, 0.094 [0.11]
						40	0.069, 0.063 [0.066]	0.032, 0.026 [0.029]	0.10, 0.089 [0.095]
		2	0.148–0.157	0.115–0.122	Forage	0	1.6, 1.3 [1.4]	0.17, 0.14 [0.15]	1.7, 1.5 [1.6]
						5	0.85, 0.88 [0.87]	0.12, 0.14 [0.13]	0.96, 1.0 [0.99]
						9	0.16, 0.12 [0.14]	0.052, 0.045 [0.048]	0.22, 0.16 [0.19]
						15	0.082, 0.063 [0.073]	0.051, 0.048 [0.050]	0.13, 0.11 [0.12]
						19	0.061, 0.057 [0.059]	0.050, 0.047 [0.048]	0.11, 0.10 [0.11]
					Fodder	19	0.15, 0.12 [0.13]	0.13, 0.16 [0.15]	0.28, 0.28 [0.28]
						25	0.087, 0.067 [0.077]	0.068, 0.042 [0.055]	0.16, 0.11 [0.13]
						28	0.043, 0.043 [0.043]	0.036, 0.046 [0.041]	0.079, 0.089 [0.084]
						34	0.023, 0.039 [0.031]	tr, 0.010 [0.010]	0.033, 0.049 [0.041]
						40	0.015, 0.015 [0.015]	ND, 0.010 [< 0.01]	0.015, 0.025 [0.020]
RABSP003 BS035-06HA BS035-06HA-A USA Springfield America, North 2006	Corn, sweet Serendipity	1	0.28	0.214	Forage	4	4.0, 2.8 [3.4]	0.17, 0.15 [0.16]	4.1, 2.9 [3.5]
						9	0.92, 0.84 [0.88]	0.091, 0.095 [0.093]	1.0, 0.94 [0.98]
					Fodder	29	0.29, 0.20 [0.25]	0.11, 0.080 [0.094]	0.39, 0.28 [0.34]
		2	0.151–0.152	0.116–0.117	Forage	4	1.8, 1.6 [1.7]	0.13, 0.12 [0.12]	1.9, 1.7 [1.8]
					Fodder	29	0.093, 0.063 [0.078]	0.057, 0.058 [0.058]	0.15, 0.12 [0.14]
RABSP003 BS036-06HA BS036-06HA-A USA Earlham America, North 2006	Corn, sweet MIRAI 131	1	0.284	0.221	Forage	5	1.7, 1.5 [1.6]	0.15, 0.11 [0.13]	1.9, 1.6 [1.8]
						9	0.94, 0.34 [0.64]	0.061, 0.023 [0.042]	1.0, 0.37 [0.68]
					Fodder	30	0.14, 0.12 [0.13]	0.057, 0.069 [0.063]	0.20, 0.19 [0.19]

Study No. Trial No., Country Location, Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
		2	0.148	0.123–0.126	Forage	5	0.82, 0.56 [0.69]	0.053, 0.048 [0.050]	0.88, 0.61 [0.74]
					Fodder	29	0.27, 0.31 [0.29]	0.11, 0.15 [0.13]	0.38, 0.46 [0.42]
RABSP003 BS037-06HA BS037-06HA-A USA Sabin America, North 2006	Corn, sweet 39H85 Pioneer	1	0.277	0.162	Forage	4	1.8, 2.3 [2.1]	0.16, 0.22 [0.19]	1.9, 2.5 [2.2]
						8	1.3, 1.2 [1.2]	0.12, 0.10 [0.11]	1.4, 1.3 [1.3]
					Fodder	28	1.8, 1.3 [1.6]	0.94, 0.54 [0.74]	2.8, 1.8 [2.3]
		2	0.145–0.148	0.0841–0.0873	Forage	5	0.59, 0.79 [0.69]	0.10, 0.12 [0.11]	0.69, 0.91 [0.80]
					Fodder	29	0.83, 0.99 [0.91]	0.37, 0.43 [0.40]	1.2, 1.4 [1.3]
RABSP003 BS038-06HA BS038-06HA-A USA Stilwell America, North 2006	Corn, sweet Peaches & Cream	1	0.279	0.173	Forage	4	1.1, 1.2 [1.2]	0.10, 0.086 [0.094]	1.2, 1.3 [1.3]
						10	0.13, 0.090 [0.11]	0.033, 0.025 [0.029]	0.16, 0.12 [0.14]
					Fodder	30	0.051, 0.055 [0.053]	0.033, 0.021 [0.027]	0.084, 0.076 [0.080]
		2	0.147–0.148	0.0862–0.0930	Forage	4	1.3, 0.84 [1.1]	0.11, 0.074 [0.091]	1.4, 0.92 [1.2]
					Fodder	30	0.046, 0.032 [0.039]	0.019, 0.018 [0.018]	0.065, 0.050 [0.058]
RABSP003 BS039-06HA BS039-06HA-A USA Fresno America, North 2006	Corn, sweet Silver Queen	1	0.285	0.165	Forage	5	2.2, 2.1 [2.1]	0.093, 0.088 [0.090]	2.3, 2.2 [2.2]
						10	2.1, 1.7 [1.9]	0.086, 0.085 [0.085]	2.1, 1.8 [2.0]
					Fodder	28	2.5, 1.3 [1.9]	0.37, 0.22 [0.29]	2.9, 1.5 [2.2]
		2	0.150–0.155	0.0889–0.0892	Forage	5	0.76, 1.0 [0.89]	0.091, 0.11 [0.10]	0.86, 1.1 [0.99]
					Fodder	28	1.4, 0.81 [1.1]	0.36, 0.30 [0.33]	1.8, 1.1 [1.5]
RABSP003 BS040-06HA BS040-06HA-A USA Rupert America, North 2006	Corn, sweet Northern Extra	1	0.282	0.172	Forage	5	0.69, 0.73 [0.71]	0.080, 0.071 [0.076]	0.77, 0.80 [0.78]
						8	0.78, 0.73 [0.76]	0.061, 0.067 [0.064]	0.84, 0.80 [0.82]
					Fodder	30	0.48, 0.72	0.050, 0.071	0.53, 0.79

Study No. Trial No., Country Location, Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
							[0.60]	[0.060]	[0.66]
		2	0.150–0.151	0.0924–0.17	Forage	5	0.59, 0.50 [0.55]	0.064, 0.058 [0.061]	0.66, 0.56 [0.61]
					Fodder	30	0.14, 0.12 [0.13]	0.034, 0.027 [0.030]	0.17, 0.15 [0.16]
RABSP003 BS041-06HA BS041-06HA-A USA Cornelius America, North 2006	Corn, sweet Serendipity	1	0.279	0.167	Forage	5	4.6, 2.3 [3.5]	0.18, 0.086 [0.13]	4.8, 2.4 [3.6]
						11	1.8, 2.1 [2.0]	0.087, 0.073 [0.080]	1.9, 2.2 [2.1]
					Fodder	29	1.2, 1.2 [1.2]	0.049, 0.055 [0.052]	1.3, 1.3 [1.3]
		2	0.150–0.154	0.0907–0.0954	Forage	5	1.5, 2.5 [2.0]	0.075, 0.11 [0.094]	1.6, 2.6 [2.1]
					Fodder	29	0.38, 0.38 [0.38]	0.034, 0.023 [0.028]	0.42, 0.41 [0.41]

Miscellaneous fodder and forage crops

Cotton gin trash

Residue trials for cotton gin trash are the same as those for cotton seed described above. Concurrent recoveries for cotton gin trash ranged from 70 to 105% with relative standard deviations from 5.6 to 15%.

Table 64 Results of spiromesifen residue trials in cotton for cotton gin trash

Study No. Trial No., Country Location, Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
GAP USA	Cotton	3	0.280	–	–	30	Do not exceed 0.56 kg ai/ha per season		
200116 BS001-00H USA Fresno, California America, North 2000	Cotton Riata C-176 (acala)	2	0.28	0.151–0.156	gin trash	28	5.3, 4.6 [5.0]	1.0, 1.0 [1.0]	6.3, 5.6 [6.0]
200116 BS002-00D USA Benoit, Mississippi America, North 2000	Cotton ST 474	2	0.273–0.284	0.283–0.31	gin trash	20	4.1	1.7	5.8
						28	3.0	1.6	4.6
						35	1.4	1.1	2.5
						46	0.92	0.89	1.8
200116 BS007-00H USA Colony, Oklahoma	Cotton PM 1218	2	0.280–0.281	0.127	gin trash	35	0.94, 1.0 [0.99]	0.62, 0.59 [0.61]	1.6, 1.6 [1.6]

Study No. Trial No., Country Location, Region Year	Crop Variety	Application			Matrix	DALA (days)	Residues (mg/kg)		
		No.	kg/ha (ai)	kg/hL (ai)			Spiromesifen	Sp-enol	Total
America, North 2000									
200116 BS008-00H USA Plainview, Texas America, North 2000	Cotton Paymaster 2200 RR	2	0.280–0.282	0.141–0.146	gin trash	30	1.4, 1.4 [1.4]	1.3, 1.1 [1.2]	2.8, 2.6 [2.7]
200116 BS009-00HA USA Comanche, Oklahoma America, North 2001	Cotton Paymaster 2326 BG/RR	2	0.273–0.277	0.125–0.131	gin trash	30	0.16, 0.20 [0.18]	0.16, 0.32 [0.24]	0.31, 0.52 [0.41]
200116 BS012-00H USA Tifton, Georgia America, North 2000	Cotton DPL-451	2	0.280	0.291–0.300	gin trash	28	9.5, 7.3 [8.4]	2.8, 2.6 [2.7]	12, 10 [11]

FATE OF RESIDUES IN STORAGE AND PROCESSING

Nature of the residue during processing

High-temperature hydrolysis

High-temperature hydrolysis of spiromesifen was investigated by Babczinski (2001, MR-391/01). In the study, [dihydrofuranone-3-^{14}C]spiromesifen was spiked into buffered solutions, in duplicate, at a target concentration of 0.05 mg/L. The spiked solutions were put into conditions simulating pasteurisation (90 °C, pH 4, 20 min.); baking, brewing, boiling (100 °C, pH 5, 60 min); and sterilisation (120 °C, pH 6, 20 min.). Prior to and after processing, an aliquot from each sample was collected and analysed by LSC for total radioactivity and by radio-HPLC for determination of hydrolysis products. Mass balance of radioactivity after processing was 95, 103, and 98% for 90 °C/pH 4, 100 °C/pH 5, and 120 °C/pH 6, respectively.

The only residues identified in the high-temperature hydrolysis study were spiromesifen and the Sp-enol metabolite. The conversion of spiromesifen to Sp-enol was much less at pH 4 than under the higher pH conditions (Table).

Table 65 High-temperature hydrolysis radio-HPLC results for spiromesifen

	% of Radiolabel			
	Start		End	
Conditions	Spiromesifen	Sp-enol	Spiromesifen	Sp-enol
90 °C, 20 minutes, pH 4	95.7	Not detected	74.0	21.9
	96.0		75.3	22.1
100 °C, 60 minutes, pH 5	96.5		11.8	83.5
	97.5		14.3	78.3
120 °C, 20 minutes, pH 6	94.6		1.1	90.1
	97.3		0.7	92.7

Residues after processing

The Meeting received data depicting residues of spiromesifen in raw and processed commodities of strawberry, tomato, potato, sugar beet, cereal grains, cottonseed, and tea. In addition, studies showing the effects of washing and cooking on residue levels were provided for broccoli, cucumber, squash, peppers, lettuce, spinach, mustard greens, and beans.

Strawberry

The effects of washing and of processing strawberries into jam and preserves on residues of spiromesifen were investigated by Schöning and Sur (2001, Report RA-3018/00). Strawberries were harvested 3 DALA from greenhouse plots treated 4× 0.144 kg ai/ha. Washing and preparation of strawberry jam were done per household practices; preparation of strawberry preserves used simulated commercial practices. For all matrices, analysis of spiromesifen residues was by Method 00631.

Strawberries were washed in standing water; half of the washed fruits were then frozen, homogenized in the presence of dry ice, and returned to frozen storage prior to analysis. Aliquots of the wash water were also frozen and stored prior to analysis. The other half of the washed fruits were prepared into jam. Fruits were cut and/or minced in a blender. Sugar and gelling agent were added to the cut fruit and fruit pulp, and the mixture heated to 98–100 °C for ca. 3 minutes. The resulting jam was cooled and stored frozen prior to analysis.

Strawberry preserves were prepared by washing the fruit in lukewarm water, mixing the washed fruit with a sugar solution, transferring the mixture to preserving cans, and pasteurising (89–92 °C).

Samples of strawberry processed commodities and wash water were stored for a maximum of ca. five months. Total residues of spiromesifen (spiromesifen + Sp-enol) were stable in fruit-like commodities for at least 727 days (cucumber, melon peel) and 316 days (tomato). Concurrent recoveries across all matrices and fortification levels ranged from 90 to 108% with a maximum relative standard deviation of 5.7%.

Table 66 Effect of washing and processing on residues of spiromesifen in strawberry

Study Plot Year	Matrix	Sample No.	Spiromesifen, mg/kg	Sp-enol, mg/kg	Total, mg/kg	Processing factor (total)
RA-3018/00 103/7 2000	Fruit	7	0.245	0.038	0.283	–
	Fruit—washed	14	0.149, 0.166 [0.158]	0.042, 0.041 [0.041]	0.191, 0.207 [0.199]	0.70
		17	0.142, 0.154 [0.148]	0.046, 0.048 [0.047]	0.188, 0.202 [0.195]	0.69
	Washings	13	0.057, 0.055 [0.056]	< 0.01, < 0.01 [< 0.01]	0.067, 0.065 [0.066]	0.23
		20	0.048, 0.049 [0.049]	< 0.01, < 0.01 [< 0.01]	0.058, 0.059 [0.059]	0.21
	Jam	16	0.102, 0.098 [0.100]	0.033, 0.031 [0.032]	0.135, 0.129 [0.132]	0.47
		19	0.090, 0.090 [0.090]	0.034, 0.034 [0.034]	0.124, 0.124 [0.124]	0.44
	Preserve	15	0.063, 0.059 [0.061]	0.018, 0.019 [0.018]	0.081, 0.078 [0.079]	0.28
		18	0.072, 0.049 [0.061]	0.018, 0.018 [0.018]	0.090, 0.067 [0.078]	0.28
RA-3018/00 104/5 2000	Fruit	4	0.071	0.071	0.142	–
	Fruit—washed	10	0.046, 0.052 [0.049]	0.058, 0.060 [0.059]	0.104, 0.112 [0.108]	0.76
		14	0.034, 0.031 [0.033]	0.054, 0.053 [0.054]	0.088, 0.084 [0.086]	0.61
	Washings	9	0.012, < 0.01 [0.011]	< 0.01, < 0.01 [< 0.01]	0.022, < 0.01 [0.016]	0.11
		13	0.021, < 0.01 [0.016]	< 0.01, < 0.01 [< 0.01]	0.031, < 0.01 [0.021]	0.15

Study Plot Year	Matrix	Sample No.	Spiromesifen, mg/kg	Sp-enol, mg/kg	Total, mg/kg	Processing factor (total)
	Jam	12	0.025, 0.027 [0.026]	0.039, 0.041 [0.040]	0.064, 0.068 [0.066]	0.46
		16	0.034, 0.035 [0.035]	0.048, 0.048 [0.048]	0.082, 0.083 [0.082]	0.58
	Preserve	11	0.023, 0.024 [0.024]	0.024, 0.019 [0.022]	0.047, 0.043 [0.045]	0.32
		15	0.015, 0.015 [0.015]	0.024, 0.024 [0.024]	0.039, 0.039 [0.039]	0.27

Broccoli

A single sample of broccoli from the residue trials described above (Duah, 2002; Report 20141) was used to investigate the effect of washing and cooking on spiromesifen residues. To simulate domestic washing, a broccoli subsample was washed under lukewarm water for 30 seconds and allowed to dry on paper towels for ca. 2 minutes. The florets were then either halved or quartered, placed into plastic bags, and stored frozen prior to analysis. To investigate cooking, a broccoli subsample was placed into a steamer insert and cooked for 13 to 16 minutes. After cooling, the cooked broccoli was placed into a plastic bag and stored frozen prior to analysis. Washed and cooked broccoli samples were homogenized in the presence of dry ice and analysed for residues using Method 00631.

Total residues (spiromesifen + Sp-enol) in broccoli florets were reduced by a factor of 0.75 by washing (unprocessed mean residue = 0.08 mg/kg, washed mean residue = 0.06 mg/kg). Following cooking, mean total residues were 0.15 mg/kg, resulting in a concentration factor of 1.9.

Cucumber

A single sample of cucumbers from the residue trials described above (Fischer, 2002; Report 110341) was used to investigate the effect of washing and peeling on spiromesifen residues. The cucumber sample was washed under lukewarm-to-cool water for ca. 30 seconds, allowed to dry on a paper towel for 2 minutes, and then the ends were removed and discarded. The remaining cucumber was quartered, placed into a plastic bag, and frozen. Peeled cucumbers were prepared by peeling the outside layer using a potato peeler, removing the ends of the cucumber, halving or quartering the resulting peeled cucumber, and placing the sample in a plastic bag for frozen storage. Samples were homogenised in the presence of dry ice and analysed for residues using Method 00631.

Total residues (spiromesifen + Sp-enol) were < 0.01 mg/kg in all samples of unwashed, unpeeled cucumber as well as in all samples of peeled cucumber. Residues were slightly above the LOQ in washed, unpeeled cucumber (0.011, 0.013, and 0.014 mg/kg). No explanation was provided for why the residues were higher in washed fruit than in unwashed fruit.

Squash

A single sample of summer squash from the residue trials described above was used to investigate the effect of washing and cooking on spiromesifen residues. Squash samples were washed under lukewarm-to-cool water for ca. 30 seconds, allowed to dry on a paper towel for 2 minutes, and then the stems were removed and discarded. The remaining squash was halved or quartered, placed into a plastic bag, and frozen. Cooked squash were prepared by removing the end pieces of the squash, cutting the remainder into ca. 6 mm slices, and boiling the slices in salted water in a covered pan until they were “crisp-tender.” All samples were stored frozen, and were homogenised in the presence of dry ice prior to analysis for residues using Method 00631. Concurrent recoveries across all matrices and fortification levels ranged from 71 to 101% with a maximum relative standard deviation of 13%.

Table 67 Effect of washing and cooking on residues of spiromesifen in summer squash

Study Plot Year	Matrix	Spiromesifen, mg/kg	Sp-enol, mg/kg	Total, mg/kg [mean]	Processing factor (total)
110341 BS110-00HA 2000	Summer squash	0.0097, 0.0077, 0.0096	0.021, 0.020, 0.021	0.031, 0.028, 0.031 [0.030]	–

Study Plot Year	Matrix	Spiromesifen, mg/kg	Sp-enol, mg/kg	Total, mg/kg [mean]	Processing factor (total)
	Washed	< 0.004, < 0.004, < 0.004	0.013, 0.010, 0.011	0.013, 0.010, 0.011 [0.011]	0.37
	Cooked	< 0.004, < 0.004, < 0.004	0.020, 0.022, 0.020	0.020, 0.022, 0.020 [0.021]	0.70

Peppers

A single sample of sweet pepper from the residue trials described above was used to investigate the effect of washing on spiromesifen residues. Peppers were washed under lukewarm-to-cool water for ca. 30 seconds, allowed to dry on a paper towel for 2 minutes, and then the stems, core and seeds were removed and discarded. The inside of each pepper was washed again for ca. 30 seconds and allowed to dry for ca. 2 minutes. The peppers were quartered, placed into a plastic bag, and frozen. All samples were stored frozen, and were homogenised in the presence of dry ice prior to analysis for residues using Method 00631. Concurrent recoveries from pepper ranged from 71 to 117%, with a maximum relative standard deviation of 15%.

Residues of both spiromesifen and Sp-enol were below the limit of detection (0.005 mg/kg and 0.003 mg/kg, respectively) in both unwashed and washed pepper, and a processing factor could not be determined.

Tomato

Two studies depicting the effect of processing on residues of spiromesifen in tomatoes were submitted. In one, tomatoes from a field trial that received spiromesifen treatment at a three-fold exaggerated rate were processed into canned tomato, crushed tomato, dried tomato, juice, paste, and puree using simulated commercial processing practices (Lenz, 2002; Report 110991). In the second study, tomatoes grown under greenhouse conditions were processed into washed and peeled tomatoes using household practice, and into juice, preserves, and puree using simulated commercial practices (Schöning and Elke, 2001; Report RA-3083/99). For both studies, residues of spiromesifen and Sp-enol were determined using Method 00631.

Per Report 110991, tomato fruits were flume washed. Random tomatoes were selected at the end of the washing process and placed into plastic bags as washed tomato samples. Other washed tomatoes were crushed in a commercial grinder, pumped into a hot break system. The crush was heated to 98 °C. The hot crush was put through a finisher to remove peel and seeds (wet pomace), producing juice. The juice was canned in a retort machine (ca. 50 minutes at 117 °C). Juice was processed into puree and paste through evaporation. Samples for residue analysis were collected throughout the production process. Peeled tomatoes were produced by blanching tomato fruits in boiling water, cooling the tomatoes with cold water, and manually removing the skins. The resulting peeled tomatoes were cut into smaller segments, transferred to cans, and processed in a retort machine for ca. 50 minutes at 117 °C. Dried tomatoes were produced by taking washed fruits, cutting them into ca. 12 mm slices, dipping them in 55 ppm potassium metabisulphite, and drying in the sun for 3 days. Across the various matrices and analytes, concurrent recoveries ranged from 73 to 113%, with a maximum relative standard deviation of 12%.

For RA-3083/99, tomato fruits were hand washed and then cut with a knife into small pieces, which were collected, frozen, and homogenized in the presence of dry ice in preparation for residue analysis. Separate tomato fruits were washed in lukewarm water until the peel could be taken off. The resulting peeled tomatoes were canned in tomato juice to produce canned tomato (pasteurised at 91 to 98 °C), or cut with a knife into small pieces, which were collected, frozen, and homogenized in the presence of dry ice in preparation for residue analysis. Wash water from production of washed tomatoes and of peeled tomatoes (water + peel) was collected and stored frozen for residue analysis. Tomato juice was prepared by washing tomatoes in standing water, cutting them into small pieces, and then blanching them for 5 to 10 minutes at ca. 100 °C. The resulting tomato pulp was strained to

separate juice from wet pomace. Juice was pasteurised (90 to 92 °C). Tomato puree was obtained by centrifuging tomato juice followed by pasteurisation. Across the various matrices and analytes, concurrent recoveries ranged from 78 to 108%, with a maximum relative standard deviation of 8.1%.

Table 68 Effects of washing and processing on residues of spiromesifen in tomato

Study Plot Year	Matrix	Spiromesifen, mg/kg	Sp-enol, mg/kg	Total, mg/kg	Processing factor (total)
110991 BS084-00P 2000	Tomato fruit	0.246	0.011	0.257	–
	Washed	0.102	0.009	0.111	0.43
	Canned fruit	0.008	0.045	0.053	0.21
	Juice (raw)	0.210	0.018	0.228	0.89
	Juice (canned)	0.025	0.159	0.184	0.72
	Puree	0.115	0.086	0.201	0.78
	Paste	0.443	0.220	0.663	2.6
	Dried	1.17	0.118	1.29	5.0
RA-3083/99 0292-99(A) 1999	Tomato fruit	0.15	< 0.01	0.16	–
	Washed	0.07	< 0.01	0.08	0.50
	Wash water	< 0.01	< 0.01	< 0.01	< 0.06
	Peeled fruit	< 0.01	< 0.01	< 0.01	< 0.06
	Peeling water + peel	0.04	< 0.01	0.05	0.31
	Canned fruit	< 0.01	< 0.01	< 0.01	< 0.06
	Juice (raw)	0.01	< 0.01	0.02	0.12
	Juice (canned)	< 0.01	< 0.01	< 0.01	< 0.06
	Wet pomace	0.63	0.027	0.657	4.1
	Puree	0.06	0.054	0.114	0.72
RA-3083/99 0292-99(B) 1999	Tomato fruit	0.14	< 0.01	0.15	–
	Washed	0.12	< 0.01	0.13	0.87
	Wash water	< 0.01	< 0.01	< 0.01	< 0.07
	Peeled fruit	< 0.01	< 0.01	< 0.01	< 0.07
	Peeling water + peel	0.05	< 0.01	0.06	0.40
	Canned fruit	< 0.01	< 0.01	< 0.01	< 0.07
	Juice (raw)	0.02	< 0.01	0.03	0.20
	Juice (canned)	0.01	< 0.01	0.02	0.13
	Wet pomace	1.2	0.041	1.241	8.3
	Puree	0.10	0.081	0.181	1.2
RA-3083/99 0440-99(A) 1999	Tomato fruit	0.19	< 0.01	0.20	–
	Washed	0.12	< 0.01	0.13	0.65
	Wash water	0.02	< 0.01	0.03	0.15
	Peeled fruit	0.01	< 0.01	0.02	0.10
	Peeling water + peel	0.10	< 0.01	0.11	0.55
	Canned fruit	0.02	0.014	0.034	0.17
	Juice (raw)	0.06	< 0.01	0.070	0.35
	Juice (canned)	0.03	0.041	0.071	0.35
	Wet pomace	1.4	0.081	1.481	7.4
	Puree	0.25	0.204	0.454	2.3
RA-3083/99 0440-99(B) 1999	Tomato fruit	0.21	< 0.01	0.22	–
	Washed	0.13	< 0.01	0.14	0.64
	Wash water	0.02	< 0.01	0.03	0.14
	Peeled fruit	0.02	< 0.01	0.03	0.14

Study Plot Year	Matrix	Spiromesifen, mg/kg	Sp-enol, mg/kg	Total, mg/kg	Processing factor (total)
	Peeling water + peel	0.17	< 0.01	0.18	0.82
	Canned fruit	0.05	0.027	0.077	0.35
	Juice (raw)	0.05	0.027	0.07	0.35
	Juice (canned)	0.04	0.027	0.067	0.30
	Wet pomace	1.6	0.068	1.668	7.6
	Puree	0.23	0.204	0.434	2.0

Lettuce

A leaf lettuce sample from one trial performed in the USA as described above was washed in water using an automatic salad spinner (3×, water not changed). After washing, the sample was spun until dry. Residues of spiromesifen and Sp-enol were determined using Method 00631. Concurrent recoveries ranged from 77 to 120%, with a maximum relative standard deviation of 12%.

Table 69 Effects of washing on residues of spiromesifen in leaf lettuce

Study Plot Year	Matrix	Spiromesifen, mg/kg	Sp-enol, mg/kg	Total, mg/kg	Processing factor (total)
200146 BS045-00D 2000	Leaf lettuce	1.60	0.096	1.70	–
	Washed	0.958	0.080	1.04	0.61

Mustard greens

A mustard greens sample from one trial performed in the USA as described above was washed in water using an automatic salad spinner (3×, water not changed). After washing, the sample was spun until dry. The washed mustard greens were cooked for ca. 1 hour at 80 to 100°C. The cooked mustard greens were cooled and were then stored frozen prior to analysis of residues of spiromesifen and Sp-enol were determined using Method 00631. Concurrent recoveries ranged from 73 to 114%, with a maximum relative standard deviation of 10%.

Table 70 Effects of washing and cooking on residues of spiromesifen in mustard greens

Study Plot Year	Matrix	Spiromesifen, mg/kg	Sp-enol, mg/kg	Total, mg/kg	Processing factor (total)
200172 BS074-00H 2001	Mustard greens	0.565	0.386	0.951	–
	Washed	0.213	0.151	0.364	0.38
	Cooked	< 0.003	0.135	0.136	0.14

Spinach

A spinach sample from one trial performed in the USA as described above was prepared for cooking by removing and discarding the stems. The prepared spinach leaves were cooked for ca. 4 minutes at 95 °C. The cooked spinach was drained and stored frozen prior to analysis of residues of spiromesifen and Sp-enol were determined using Method 00631. Concurrent recoveries ranged from 77 to 120%, with a maximum relative standard deviation of 12%.

Table 71 Effects of cooking on residues of spiromesifen in spinach

Study Plot Year	Matrix	Spiromesifen, mg/kg	Sp-enol, mg/kg	Total, mg/kg	Processing factor (total)
200146 BS060-00H 2001	Spinach	1.46	0.152	1.61	–
	Cooked	1.37	2.09	3.46	2.1

Beans

Samples of Climbing French Beans from residue trials described above were washed in standing water, cut with a knife into small pieces, and then cooked for ca. 20 minutes in salted water. Processing and analysis are conducted and reported under study RA-3019/00 (Schoening and Elke, 2001). Residues of spiromesifen and Sp-enol were determined using Method 00631. Concurrent recoveries ranged from 84 to 105%, with a maximum relative standard deviation of 7.2%.

Table 72 Effects of washing and cooking on residues of spiromesifen in beans with pods

Study Plot Year	Matrix	Spiromesifen, mg/kg	Sp-enol, mg/kg	Total, mg/kg	Processing factor (total)
RA-3019/00 0173-00(A) 2000	Beans with pods	0.59	0.054	0.644	–
	Washed	0.38	0.014	0.394	0.61
	Washing water	0.09	0.010	0.100	0.16
	Cooked	0.06	0.177	0.237	0.37
	Cooking water	< 0.01	0.217	0.227	0.35
RA-3019/00 0173-00(B) 2000	Beans with pods	0.59	0.054	0.644	–
	Washed	0.38	0.014	0.394	0.61
	Washing water	0.09	0.010	0.100	0.16
	Cooked	0.08	0.109	0.189	0.29
	Cooking water	< 0.01	0.204	0.214	0.33
RA-3019/00 0174-00(A) 2000	Beans with pods	0.13	0.014	0.144	–
	Washed	0.09	0.010	0.100	0.70
	Washing water	0.02	0.010	0.030	0.21
	Cooked	0.01	0.041	0.051	0.35
	Cooking water	< 0.01	0.054	0.064	0.45
RA-3019/00 0174-00(B) 2000	Beans with pods	0.13	0.014	0.144	–
	Washed	0.09	0.010	0.100	0.70
	Washing water	0.02	0.010	0.030	0.21
	Cooked	0.02	0.041	0.061	0.42
	Cooking water	< 0.01	0.054	0.064	0.45

Potato

Samples from the residue trials in potatoes described above were processed into washed potato, peeled potato, wet potato peel, cooked potato, potato chips, and potato flakes using simulated commercial practices. Potatoes were washed using an automated brushwasher. The washed potatoes were then passed through an abrasion peeler to obtain peeled potatoes and a potato peel slurry; wet peels were obtained by decanting the peel slurry through a colander. Peeled potatoes were sliced and fried in vegetable oil at 179 to 202 °C to produce potato chips (crisps). Potato flakes were produced

from peeled potatoes which were diced, boiled in water until soft (cooked potato), mashed, and dried. Residues of spiromesifen and Sp-enol were determined using Method 00631. Concurrent recoveries across all matrices ranged from 80 to 115%, with a maximum relative standard deviation of 10%.

Residues of spiromesifen and Sp-enol in all matrices were below the respective LODs; therefore, processing factors could not be calculated.

Sugar beet

A sample of sugar beets was taken from the field rotational crop study described above and processed into dried pulp, molasses, and refined sugar using simulated commercial practices. Sugar beets were cleaned of heavy soil deposits, leaves, and foreign matter. The beets were then sliced into cossettes. Material was diffused from the cossettes using 90 °C water for 30–45 seconds followed by five separate baths in hot water (70 °C) for at least 9 minutes each. The resulting raw juice was screened. The diffused cossettes were pressed to remove water and the resulting beet pulp was dried in a forced-air oven. Raw juice was phosphatized by adjusting the pH to between 10.5 and 11.2 using calcium oxide or phosphoric acid. Precipitate was isolated by centrifugation. The supernatant was collected and, after heating to 80–85 °C the pH was adjusted to between 9.1 and 9.3 using phosphoric acid. Thin juice was obtained by centrifugation and vacuum filtration. The thin juice was evaporated to a thick juice consisting of 50 to 60% solids. The thick juice was filtered through cotton and further evaporated to a solids content of 80 to 85%. Crystallisation was initiated by seeding the thick juice with pulverized white sugar. Sugar and molasses were separated by centrifugation. Residues of spiromesifen and Sp-enol were determined using Method 00631. Concurrent recoveries across all matrices ranged from 73 to 102%, with a maximum relative standard deviation of 15%.

Residues of spiromesifen and Sp-enol were < 0.01 in all matrices except molasses, in which Sp-enol was 0.021 mg/kg (spiromesifen equivalents). Since residues in sugar beet root were not quantifiable, a finite concentration factor for sugar beet molasses could not be derived. Based on the limit of detection in sugar beet root, the processing factor is ≥ 2.1.

Maize

Residues of spiromesifen in corn grain, aspirated grain fractions, and processed commodities was investigated by Duah (2002; Report 200180). Spiromesifen was applied to maize as two foliar applications, at a 13-day interval, at a rate of 0.75 kg ai/ha, which is a five-fold exaggeration of the critical GAP rate. Corn grain was harvested 31 days after the last application and frozen within one hour of collection. Samples were shipped frozen to the analytical facility and remained frozen prior to analysis. Prior to analysis for residues of spiromesifen and Sp-enol by Method 00631, the corn grain samples were homogenized in the presence of dry ice. Concurrent recoveries ranged from 75 to 102% across both analytes.

Corn grain samples were stored for up to 318 days prior to analysis and all extracts were analysed within 4 days of extraction. Total residues of spiromesifen (spiromesifen + Sp-enol) were in corn grain stored frozen were stable for at least 318 days.

Residues of spiromesifen from three analyses were not detected (< 0.003 mg/kg, n = 2) or at trace levels (0.004 mg/kg), and residues of Sp-enol were not detected (< 0.003 mg/kg, n = 3). Total residues were, therefore, < 0.01 mg/kg. Due to residues being < LOQ and the 5-fold rate exaggeration in the study, aspirated grain fractions and processed corn products were not generated.

Sorghum

The effects of processing on residues of spiromesifen in sorghum was investigated by Lenz (2009; Report RABSP014). In a single field trial, sorghum was treated twice with spiromesifen on a 13-day interval at 0.743 and 0.748 kg ai/ha. The application represents a 5-fold exaggeration of the critical GAP. Sorghum grain was harvested 29 days after the last application and frozen within four hours of collection. The sample remained frozen during transportation to the processing facility a portion of the harvested grain was used to generate aspirated grain fractions.

Residues of spiromesifen and Sp-enol were analysed using a modified Method 00631/M001. Concurrent recoveries ranged from 80 to 96%, with a maximum relative standard deviation of 5.1%.

Table 73 Effects of formation of sorghum aspirated grain fractions on residues of spiromesifen

Study Plot Year	Matrix	Spiromesifen, mg/kg [mean]	Sp-enol, mg/kg [mean]	Total, mg/kg [mean]	Processing factor (total)
200146 BS050-08PA 2001	Grain	0.917, 0.778, 0.802 [0.832]	0.238, 0.213, 0.216 [0.222]	1.155, 0.991, 1.018 [1.055]	–
	Aspirated fractions	7.40, 7.21, 7.28 [7.30]	11.77, 12.09, 12.00 [11.95]	19.17, 19.30, 19.28 [19.25]	18

Wheat

The Meeting received two processing studies on wheat. In one, wheat was grown as a rotational crop and processed into bran, flour, shorts, middlings, and germ (Fischer, 2002; Report 200210). In the second study, wheat was treated as a primary crop using one application, of a suspension concentrate formulation, at a rate of 1.4 kg ai/ha (5-fold exaggerated rate). Wheat grain was harvested at normal maturity, 30 days after application. Subsamples of grain were used to produce aspirated grain fractions, bran, flour, shorts, middlings, and germ (Dallstream, 2009; Report RABSP012). Processing was by simulated commercial practices. In both studies, residues of spiromesifen and Sp-enol were determined using Method 00631. Concurrent recoveries across all analytes and matrices, except for Sp-enol in aspirated grain fractions, ranged from 82 to 116%, with a maximum relative standard deviation of 9.4%. Recoveries of Sp-enol in aspirated grain fractions ranged from 69 to 123% with a maximum relative standard deviation of 28%.

Table 74 Effects of processing on residues of spiromesifen in wheat

Study Plot Year	Matrix	Spiromesifen, mg/kg	Sp-enol, mg/kg	Total, mg/kg	Processing factor (total)
200210 BS146-00R 2000	Grain	< 0.001	< 0.001	< 0.01	–
	Bran	< 0.001	< 0.001	< 0.01	Not calculated
	Germ	< 0.002	< 0.002	< 0.01	Not calculated
	Flour	< 0.001	< 0.001	< 0.01	Not calculated
	Shorts	< 0.002	< 0.002	< 0.01	Not calculated
	Middlings	< 0.002	< 0.003	< 0.01	Not calculated
RABSP012 BS037-08PA 2008	Grain	0.389	0.210	0.599	–
	Bran	0.536	0.732	1.27	2.1
	Germ	0.457	0.544	1.00	1.7
	Flour	0.128	0.091	0.219	0.37
	Shorts	0.115	0.136	0.251	0.42
	Middlings	0.094	0.105	0.199	0.33
	Aspirated fractions	129	146	275	460

Cotton seed

The Meeting received data depicting the effects of processing on residues of spiromesifen on cottonseed commodities (Krolski, 2002; Report 200119). Cotton plants were treated with spiromesifen, two times on a 7-day interval, at 1.37 to 1.67 kg ai/ha. This reflects a 5-fold exaggeration of the critical GAP. Cotton was harvested 30 days after the last application and transported to the processing facility for ginning and processing. The seed cotton was dried (if necessary), ginned, and the delinted seed processed into meal and refined cottonseed oil using simulated commercial practices (which include solvent extraction, refining, bleaching, and

deodorization). After processing, samples were shipped, frozen, to the analytical lab where they were maintained frozen prior to analysis. Residues of spiromesifen and Sp-enol were determined using Method 00631. Concurrent recoveries for both analytes and across all matrices ranged from 70 to 114%, with a maximum relative standard deviation of 16%.

Table 75 Effects of processing on residues of spiromesifen in cotton

Study Plot Year	Matrix	Spiromesifen, mg/kg [mean]	Sp-enol, mg/kg [mean]	Total, mg/kg [mean]	Processing factor (total)
200119 BS013-00R 2000	Seed	0.315, 0.330, 0.432 [0.359]	0.293, 0.314, 0.335 [0.314]	0.608, 0.644, 0.767 [0.673]	–
	Meal	< 0.002, < 0.002, < 0.002 [< 0.01]	0.046, 0.059, 0.054 [0.052]	0.045, 0.061, 0.056 [0.054]	0.080
	Refined oil	0.024, 0.025, 0.029 [0.026]	< 0.003, < 0.003, < 0.003 [< 0.01]	0.027, 0.028, 0.032 [0.029]	0.043
200119 BS005-018P 2001	Seed	0.482, 0.435, 0.510 [0.476]	0.299, 0.284, 0.268 [0.284]	0.781, 0.719, 0.778 [0.759]	–
	Meal	0.0019, 0.0015, 0.0021, [< 0.01]	0.141, 0.169, 0.163 [0.158]	0.143, 0.170, 165 [0.160]	0.21
	Refined oil	0.010, 0.017, 0.023 [0.017]	< 0.003, < 0.003, < 0.003 [< 0.01]	0.013, 0.020, 0.026 [0.020]	0.026
	Hull	0.098, 0.098, 0.135 [0.110]	0.158, 0.159, 0.132 [0.150]	0.256, 0.257, 0.267 [0.260]	0.34

Tea

The Meeting received one GLP processing study for tea, conducted in India (Sathya Moorthy, 2008; Report G5078), and five non-GPL studies, conducted in Japan (Anon., 2004, Reports JAP-GT 07.2004; JAP-GT 10.2004; Kobayashi *et al*., 2004, Report 16P-3-110; Kurokouchi, 2004, Reports M-387437-0102 and M-387451-01-2).

In the study from India, tea from one of the field trial described above was processed into green tea, black tea, black tea infusion, and instant tea. For green tea, fresh leaves were dried following common practices of air drying in sun or shade. For black tea, the fresh leaves were withered for ca. 20 hours, rolled and crushed, fermented at ca. 93% humidity for an hour and then dried to form black tea. For the tea infusion, boiling water was added to black tea and steeped for 4 minutes. The aqueous extract was passed through a tea strainer to remove the leaf residue. Instant tea was made by extracting black tea with hot water for 1 hour, after which time, the tea leaves were removed using a filter press. The extract was clarified by centrifugation and the supernatant collected. Maltodextrin was added and the extract dried via rotary evaporation and spray drying. Residues of spiromesifen and Sp-enol were determined by Method 01038 for fresh leaves, green tea, and black tea, and by method 00631 for black tea infusion and instant tea. Across both analytes and all matrices, concurrent recoveries ranged from 70 to 121%, with a maximum relative standard deviation of 19%.

In the studies from Japan, tea leaves were harvested from the field trials described above. The fresh leaves were dried to obtain green tea. Green tea infusions were also prepared by adding boiling water to green tea leaves and steeping at room temperature for 5 minutes. The leaves and infusion were assayed for residues of spiromesifen and Sp-enol by a method developed by the Japan Plant Protection Association. The reported LOQ is 0.05 mg/kg. Concurrent recovery data were not provided.

Table 76 Effects of processing on residues of spiromesifen in tea

Study Plot Year	DALA	Matrix	Spiromesifen, mg/kg [mean]	Sp-enol, mg/kg [mean]	Total, mg/kg [mean]	Processing factor (total)
G5078 G5078-S1	7	Fresh leaf	7.26, 6.90, 6.72, 6.68 [6.89]	0.18, 0.18, 0.18, 0.18 [0.18]	7.44, 7.08, 6.90, 6.86 [7.07]	–

Study Plot Year	DALA	Matrix	Spiromesifen, mg/kg [mean]	Sp-enol, mg/kg [mean]	Total, mg/kg [mean]	Processing factor (total)
2008						
	7	Green tea	14.71, 13.91 [14.31]	2.30, 2.29 [2.30]	17.01, 16.20 [16.60]	2.3
	7	Black tea	17.44, 17.25 [17.35]	5.86, 5.51 [5.69]	23.30, 22.76 [23.03]	3.2
	7	Black tea infusion	< 0.01	0.24	0.24	0.034
	7	Instant tea	0.89	6.80	7.69	1.1
JAP-GT 07.2004 JAP-GT 07.2004-A 2004	3	Green tea	66.18, 63.30 [64.74]	12.00, 11.06 [11.53]	78.18, 74.36 [76.27]	–
	7		14.81, 14.68 [14.75]	4.60, 4.55 [4.58]	19.41, 19.23 [19.32]	–
	14		3.37, 3.25 [3.31]	1.21, 1.20 [1.21]	4.58, 4.45 [4.52]	–
JAP-GT 07.2004 JAP-GT 07.2004-B 2004	3	Green tea	70.57, 70.46 [70.52]	22.68, 22.62 [22.65]	93.25, 93.08 [93.17]	–
	7		2.86, 2.82 [2.84]	2.61, 2.57 [2.59]	5.47, 5.39 [5.43]	–
	14		0.92, 0.92 [0.92]	1.83, 1.81 [1.82]	2.75, 2.73 [2.74]	–
JAP-GT 10.2004 JAP-GT 10.2004-A 2004	3	Green tea	55.0, 54.3 [54.65]	18.4, 17.8 [18.10]	73.40, 72.10 [72.75]	–
		Green tea infusion	0.54, 0.51 [0.53]	22.2, 21.8 [22.00]	22.74, 22.31 [22.53]	0.31
	7	Green tea	13.7, 13.5 [13.60]	8.05, 7.70 [7.88]	21.75, 21.20 [21.48]	–
		Green tea infusion	0.13, 0.12 [0.13]	7.38, 7.11 [7.25]	7.51, 7.23 [7.37]	0.34
	14	Green tea	2.89, 2.83 [2.86]	1.89, 1.81 [1.85]	4.78, 4.64 [4.71]	–
		Green tea infusion	< 0.05, < 0.05 [< 0.05]	1.73, 1.71 [1.72]	1.73, 1.71 [1.72]	0.36
JAP-GT 10.2004 JAP-GT 10.2004-B 2004	3	Green tea	50.9, 50.3 [50.60]	33.5, 31.7 [32.60]	84.40, 82.00 [83.20]	–
		Green tea infusion	0.44, 0.42 [0.43]	40.1, 35.1 [37.60]	40.54, 35.52 [38.03]	0.46
	7	Green tea	2.89, 2.57 [2.73]	3.75, 3.59 [3.67]	6.64, 6.16 [6.40]	–
		Green tea infusion	< 0.05, < 0.05 [< 0.05]	3.03, 2.91 [2.97]	3.03, 2.91 [2.97]	0.46
	14	Green tea	0.64, 0.62 [0.63]	2.88, 2.73 [2.81]	3.52, 3.35 [3.44]	–
		Green tea infusion	< 0.05, < 0.05 [< 0.05]	2.19, 2.05 [2.12]	2.19, 2.05 [2.12]	0.62

Residues in animal commodities

The Meeting received a feeding study conducted with dairy cattle. A feeding study for poultry was not submitted based on the feeding level in the poultry metabolism study (10 mg/kg bw/day), the 5000-fold level of exaggeration relative to the dietary burden, and the low residues (≤ 0.0003 mg/kg) expected in edible poultry commodities.

In the cattle feeding study (De Haan and Woodard, 2002; Report 110318), lactating Holstein cows were dosed for 29 consecutive days via gelatine capsule using a balling gun at levels equivalent to ca. 5, 15, or 50 ppm in their feed (dry-weight basis). Three cows were used for each dose level. Milk was collected twice daily and composited (evening milking with the next morning milking). An extra milk sample was collected from the 50-ppm dose group and separated into cream and skim milk. All milk samples were held frozen prior to transport to the analytical facility.

After administration of the final dose, the animals were slaughtered and samples of liver, kidneys, fat (omental, renal, and subcutaneous), and muscle (loin, round, and flank) were collected and weighed. After collection, the liver, kidney, and muscle samples were cubed and frozen. Fat samples were cooled to a semi-frozen state prior to being cubed and fully frozen. Frozen tissue

samples were homogenized in the presence of dry ice and then shipped, frozen, along with the milk samples to the analytical facility.

Residues of spiromesifen, Sp-enol, and 4-hdyroxymethyl-Sp-enol were determined by Method 110878. Concurrent recoveries across all analytes and matrices ranged from 84 to 116% with a maximum relative standard deviation of 8.4%. All milk sample analyses were completed within 10 days of termination of the in-life phase of the study, and all tissue sample analyses were completed within 35 days of sample collection. Milk samples from the 5- and 15-ppm dose groups were not analysed due to the low residue levels observed in the 50-ppm group (Table).

Table 77 Residues of spiromesifen in cattle milk from the 50-ppm dose group during the 28-day cattle feeding study

Dosing day	Residues, mg/kg [mean]			
	Spiromesifen	Sp-enol	4-hydroxymethy-Sp-enol	Spiromesifen + Sp-enol
–1	< 0.005, < 0.005, < 0.005 [< 0.005]	< 0.005, < 0.005, < 0.005 [< 0.005]	< 0.005, < 0.005, < 0.005 [< 0.005]	< 0.005, < 0.005, < 0.005 [< 0.005]
0	< 0.005, < 0.005, < 0.005 [< 0.005]	< 0.005, < 0.005, < 0.005 [< 0.005]	< 0.005, < 0.005, < 0.005 [< 0.005]	< 0.005, < 0.005, < 0.005 [< 0.005]
4	< 0.005, 0.0114, 0.0095 [0.0086]	< 0.005, < 0.005, < 0.005 [< 0.005]	< 0.005, < 0.005, < 0.005 [< 0.005]	< 0.005, 0.0164, 0.0145 [0.012]
8	< 0.005, 0.0065, 0.0063 [0.0059]	< 0.005, < 0.005, < 0.005 [< 0.005]	< 0.005, < 0.005, < 0.005 [< 0.005]	< 0.005, 0.0115, 0.0113 [0.0093]
12	0.0051, 0.0063, 0.0082 [0.0065]	< 0.005, < 0.005, < 0.005 [< 0.005]	< 0.005, < 0.005, < 0.005 [< 0.005]	0.0101, 0.0113, 0.0132 [0.012]
16	0.0050, 0.0076, 0.0078 [0.0068]	< 0.005, < 0.005, < 0.005 [< 0.005]	< 0.005, < 0.005, < 0.005 [< 0.005]	0.01, 0.0126, 0.0128 [0.012]
18	0.0052, 0.0088, 0.0082 [0.0074]	< 0.005, < 0.005, < 0.005 [< 0.005]	< 0.005, < 0.005, < 0.005 [< 0.005]	0.0102, 0.0138, 0.0132 [0.012]
20	< 0.005, 0.0095, 0.0064 [0.0070]	< 0.005, < 0.005, < 0.005 [< 0.005]	< 0.005, < 0.005, < 0.005 [< 0.005]	< 0.005, 0.0145, 0.0114 [0.010]
22	< 0.005, 0.0074, < 0.005 [0.0058]	< 0.005, < 0.005, < 0.005 [< 0.005]	< 0.005, < 0.005, < 0.005 [< 0.005]	< 0.005, 0.0124, < 0.005 [0.0075]
24	< 0.005, 0.0083, 0.0051 [0.0061]	< 0.005, < 0.005, < 0.005 [< 0.005]	< 0.005, < 0.005, < 0.005 [< 0.005]	< 0.005, 0.0133, 0.0101 [0.0095]
26	< 0.005, 0.0115, 0.0077 [0.0081]	< 0.005, < 0.005, < 0.005 [< 0.005]	< 0.005, < 0.005, < 0.005 [< 0.005]	< 0.005, 0.0165, 0.0127 [0.011]
28	< 0.005, 0.0143, 0.0052 [0.0082]	< 0.005, < 0.005, < 0.005 [< 0.005]	< 0.005, < 0.005, < 0.005 [< 0.005]	< 0.005, 0.0193, 0.0102 [0.012]
26 Skim	*	< 0.005, < 0.005, < 0.005 [< 0.005]	< 0.005, < 0.005, < 0.005 [< 0.005]	< 0.005, < 0.005, < 0.005 [< 0.005]
26 Cream	*	0.0208, 0.0355, 0.0234 [0.027]	< 0.005, < 0.005, < 0.005 [< 0.005]	0.0208, 0.0355, 0.0234 [0.027]

[a] The hydrolysis step used in the analysis of skim milk and cream samples quantitatively converts spiromesifen to Sp-enol.

Table 78 Residues in cattle tissues following 28 days of dosing with spiromesifen

Tissue	Feeding level, ppm	Residues, mg/kg [mean]		
		Spiromesifen + Sp-enol [a]	4-hydroxymethy-Sp-enol	Total
Fat	5	< 0.01, < 0.01, < 0.01 [< 0.01]	< 0.01, < 0.01, < 0.01 [< 0.01]	< 0.01, < 0.01, < 0.01 [< 0.01]
	15	0.0216, 0.0229, 0.0454 [0.030]	< 0.01, < 0.01, < 0.01 [< 0.01]	0.0216, 0.0229, 0.0454 [0.030]
	50	0.0523, 0.1175, 0.1097 [0.093]	< 0.01, < 0.01, < 0.01 [< 0.01]	0.0523, 0.1175, 0.1097 [0.093]
Kidney	5	< 0.05, < 0.05, < 0.05 [< 0.05]	< 0.05, < 0.05, < 0.05 [< 0.05]	< 0.05, < 0.05, < 0.05 [< 0.05]
	15	< 0.05, 0.0955, < 0.05 [0.065]	< 0.05, < 0.05, < 0.05 [< 0.05]	< 0.05, 0.0955, < 0.05 [0.065]
	50	0.1074, 0.2457, 0.0971 [0.15]	< 0.05, < 0.05, < 0.05 [< 0.05]	0.1074, 0.2457, 0.0971 [0.15]
Liver	5	< 0.05, < 0.05, < 0.05 [< 0.05]	< 0.05, < 0.05, < 0.05 [< 0.05]	< 0.05, < 0.05, < 0.05 [< 0.05]

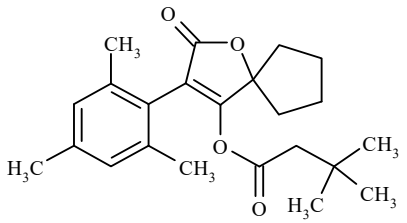
Tissue	Feeding level, ppm	Residues, mg/kg [mean]		
		Spiromesifen + Sp-enol [a]	4-hydroxymethy-Sp-enol	Total
	15	< 0.05, < 0.05, < 0.05 [< 0.05]	< 0.05, < 0.05, < 0.05 [< 0.05]	< 0.05, < 0.05, < 0.05 [< 0.05]
	50	< 0.05, 0.0590, < 0.05 [0.053]	< 0.05, < 0.05, < 0.05 [< 0.05]	< 0.05, 0.0590, < 0.05 [0.053]
Muscle	5	< 0.01, < 0.01, < 0.01 [< 0.01]	< 0.01, < 0.01, < 0.01 [< 0.01]	< 0.01, < 0.01, < 0.01 [< 0.01]
	15	< 0.01, < 0.01, < 0.01 [< 0.01]	< 0.01, < 0.01, < 0.01 [< 0.01]	< 0.01, < 0.01, < 0.01 [< 0.01]
	50	< 0.01, < 0.01, < 0.01 [< 0.01]	< 0.01, < 0.01, < 0.01 [< 0.01]	< 0.01, < 0.01, < 0.01 [< 0.01]

[a] The hydrolysis step used in the analysis quantitatively converts spiromesifen to Sp-enol.

APPRAISAL

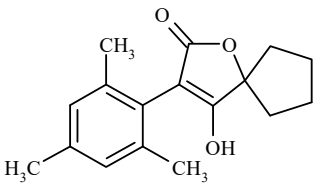
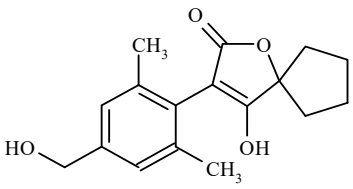
Residue and analytical aspects of spiromesifen were considered for the first time by the present Meeting. The residue evaluation was scheduled for the 2016 JMPR by the 47th Session of the CCPR.

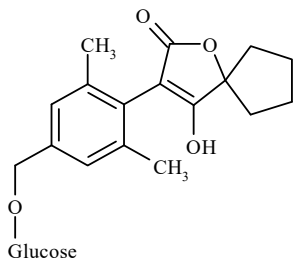
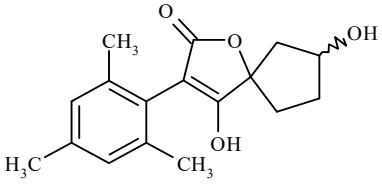
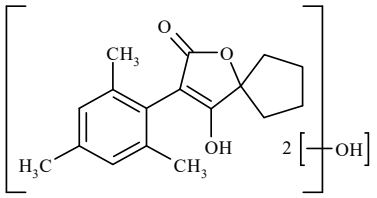
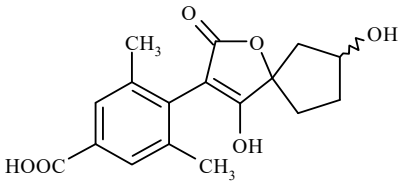
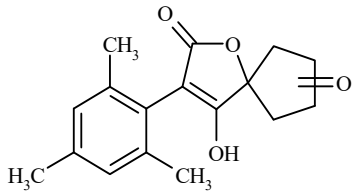
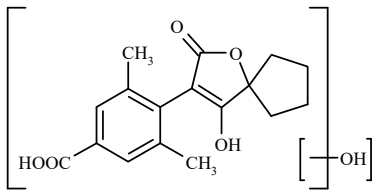
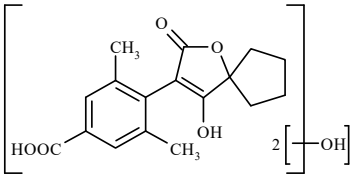
Spiromesifen is a contact insecticide-acaricide belonging to the titronic acid class of compounds. The pesticidal mode of action is inhibition of lipid biosynthesis, especially triglycerides and free fatty acids. The product is mixed with water and applied as a foliar spray using ground, aerial, or chemigation equipment. The Meeting received information on identity, animal and plant metabolism, environmental fate in soil, rotational crops, analytical methods, storage stability, use pattern, supervised trials, dairy cattle feeding studies, and fate of residues in processing.



Butanoic acid, 3,3-dimethyl-, 2 oxo-3-(2,4,6-trimethylphenyl)-1-oxaspiro[4.4]non-3-en-4-yl ester

In this appraisal, the following abbreviated names were used for metabolites.

Identifier	Chemical Structure
Spiromesifen-enol Sp-enol	
4-Hydroxymethyl-Sp-enol	

Identifier	Chemical Structure
4-Hydroxymethyl-glucoside-Sp-enol	
3-Pentanol-Sp-enol	
Dihydroxy-Sp-enol	
4-Carboxy-3-hydroxy-Sp-enol	
Oxo-cyclopentyl-Sp-enol	
4-Carboxy-hydroxy-Sp-enol	
4-Carboxy-dihydroxy-Sp-enol	

Plant metabolism

The Meeting received studies conducted with spiromesifen radiolabelled at the 3-hydrofuranone carbon depicting metabolism of spiromesifen in tomato, lettuce, and cotton.

In the tomato metabolism study, plants growing in a plastic tunnel received two applications of spiromesifen at ca. 400 g ai/ha. The interval between applications was 24 days, and the second application was seven days prior to harvest. Additional treatments were made to plants with protected fruits and to fruits directly, to evaluate translocation. Application to the fruits was equivalent to ca. a three-fold exaggerated application rate.

The translocation portion of the study indicates that residues did not translocate following foliar application. This finding is supported by the wash data showing that ca. 80% of the total radioactive residue (TRR) was associated with the surface wash fraction of both unripe (0.36 mg eq/kg) and ripe fruits (0.67 mg eq/kg). Solvent extraction (acetonitrile followed by acetonitrile:H_2O) removed ca. 0.13 mg eq/kg from unripe and ripe fruits, accounting for 25% and 17% of the whole fruit TRR, respectively. Surface washing and extraction, combined, accounted for ca. 97% of the whole fruit TRR. Spiromesifen was the only major residue (≥ 10% TRR and ≥ 0.01 mg/kg) in both surface washes and in extracts, accounting for 86–87% of the total residue in unripe (0.43 mg/kg) and ripe fruits (0.73 mg/kg). The next most abundant residue was the glucoside conjugate of 4-hydroxymethyl-Sp-enol. This metabolite was observed in extracts only (not surface washes), and made up 5.4 to 7.0% of the TRR (0.035 to 0.046 mg/kg). Treatment of the unripe fruit extracts with ß-glucosidase resulted in a decrease in 4-hydroxymethyl-glucoside-Sp-enol and a nearly quantitative increase in the non-conjugated 4-hydroxymethyl-Sp-enol.

In the lettuce metabolism study, lettuce plants grown in a plastic tunnel were treated with radiolabelled spiromesifen, at 300 g ai/ha, 26 days after planting and 1 week prior to harvest.

Of the radioactivity in the lettuce leaves, 0.41 mg eq/kg (98%) was extracted (acetonitrile + acetonitrile:H_2O). Spiromesifen accounted for 58% of the TRR (0.24 mg/kg) in the extract. The only other major residue was 4-hydroxymethyl-glucoside-Sp-enol (12% TRR, 0.049 mg/kg).

In the cotton metabolism study, cotton plants were treated three times, on a 7-day interval during boll-set to boll-split growth stages. Cotton was harvested 21 days after the last application (DALA). Two treatment regimes were established, one at 300 g ai/ha/application and the other at 1000 g ai/ha/application. Some bolls were protected during treatment to assess translocation. Cotton was harvested and separated into gin trash and undelinted seed. The undelinted seed was processed into delinted seed and seed lint.

Total radioactive residue in treated boll components was approximately an order of magnitude greater than that in protected bolls. Solvent extraction (acetonitrile followed by acetonitrile:H_2O) removed 94 and ca. 100% of the radioactivity from undelinted seed and cotton gin trash, respectively. For cotton seed, radioactivity was associated more with seed lint (73% TRR, 0.37 mg eq/kg) than with delinted seed (21% TRR, 0.011 mg eq/kg). In seed lint and delinted seed, spiromesifen and Sp-enol, combined, accounted for ca. 100% of the TRR [seed lint: 52% TRR (0.020 mg/kg) spiromesifen + 48% TRR (0.018 mg/kg) Sp-enol; delinted seed: 82% TRR (0.009 mg/kg) spiromesifen + 18% TRR (0.002 mg/kg) Sp-enol]. As with cotton seed, the only major residues in cotton gin trash were spiromesifen (26% TRR, 1.7 mg/kg) and Sp-enol (49% TRR, 3.1 mg/kg); however, in cotton gin trash, a higher proportion of the residue was the Sp-enol metabolite. Enzymatic treatment did not result in substantial changes in the residue profile.

In the confined rotational crop study, radiolabelled spiromesifen was applied to bare soil at 800 g ai/ha and rotational crops of spring wheat, spinach, and turnip were planted into the treated soil 30, 120–187, and 365 days after application.

Total radioactive residues ranged from 0.027 mg eq/kg (wheat grain) to 1.1 mg eq/kg (wheat straw) at the 30-day plant-back interval (PBI), and decreased with increasing PBI for all matrices except wheat grain. Wheat grain showed a peak TRR (0.18 mg eq/kg) at the 187-day PBI, which declined to 0.082 mg eq/kg at the 365-day PBI. Unlike primary crops, spiromesifen was not a major

residue in any rotational crop sample from any PBI. Identified residues in wheat grain were < 0.01 mg eq/kg at all PBIs. The principal residue, especially at earlier PBIs, in all matrices was 4-hydroxymethyl-Sp-enol. With the exception of wheat grain, 4-hydroxymethyl-Sp-enol (including the glucoside conjugate) ranged from 50–65% TRR (0.042–0.61 mg/kg) at the 30-day PBI to < 9–21% TRR (< 0.004–0.067 mg/kg) at the 365-day PBI. Of that, up to 83% was made up of the glucoside conjugate. The other major residues in confined rotational crops were 3-pentanol-Sp-enol (spinach: up to 29% TRR and 0.057 mg/kg; turnip leaves: up to 18% TRR and 0.025 mg/kg; turnip roots: up to 13% TRR and 0.01 mg/kg; and wheat straw: up to 11% TRR and 0.08 mg/kg) and dihydroxy-Sp-enol (spinach: up to 19% TRR and 0.038 mg/kg; wheat forage: up to 11% TRR and 0.071 mg/kg, wheat hay: up to 11% TRR and 0.032 mg/kg; and wheat straw: up to 13% TRR and 0.094 mg/kg).

Field accumulation in rotational crops

Field rotational crop studies were conducted with bulb onion, green onion, sugar beet, barley, wheat, sugar cane, and alfalfa. In addition, greenhouse rotational crop studies were conducted with carrot, lettuce, and tomato. For all of the field studies, spiromesifen was applied to bare soil at a total rate of ca. 840 g ai/ha (as one application for sugar cane or as three applications for other crops). The maximum seasonal rate from any of the registered uses is 864 g ai/ha (as four applications, each at 216 g ai/ha). Rotational crops were planted into the treated soil ca. 30 days after the last application (14 days for sugar cane). In the greenhouse trials, tomato was treated as a primary crop, receiving four applications at 216 g ai/ha. Approximately 30 days after the last application, treated tomato plants were removed and rotational carrot, lettuce, or tomato were planted. In the case of rotational tomato, additional plantings were made at PBIs of 45 days and 127 or 140 days. With the exception of tomato, rotational crops were analysed for spiromesifen, Sp-enol, and 4-hydroxymethyl-Sp-enol; tomato samples were assayed for 4-hydroxymethyl-Sp-enol only. For all crops except sugar cane, the analytical method included a hydrolysis step; therefore, reported concentrations of 4-hydroxymethyl-Sp-enol include the residue contribution from the glucoside metabolite.

Residues of spiromesifen, *per se*, were non-quantifiable in all field rotational crop matrices. Residues of Sp-enol were as follows < 0.01 mg/kg in all matrices except for one sample of bulb onion (0.033 mg/kg) and one sample of green onion (0.039 mg/kg). Residues of 4-hydroxymethyl-Sp-enol in rotational crops were as follows: bulb onion (n = 5), < 0.01 (5) mg/kg; green onion (n = 3), < 0.01 (2), and 0.032 mg/kg, sugar beet roots (n = 11), < 0.01 (11) mg/kg; sugar beet tops (n = 11), < 0.01 (10), and 0.16 mg/kg; barley hay (n = 12), 0.02 (2), 0.04 (3), 0.06 (3), 0.11, 0.12, 0.15, and 0.18 mg/kg; barley straw (n = 12), < 0.01, 0.01, 0.02 (2), 0.03 (3), 0.04, 0.06 (2), 0.09, and 0.11 mg/kg; wheat forage (n = 20), < 0.01 (6), 0.01 (4), 0.02, 0.03 (4), 0.04, 0.08 (2), 0.09, and 0.14 mg/kg; wheat hay (n = 20), < 0.01 (3), 0.01, 0.02 (5), 0.03 (2), 0.04, 0.05, 0.06, 0.07 (2), 0.09, and 0.10 (3) mg/kg; wheat straw (n = 20), 0.01 (6), 0.02 (3), 0.03 (2), 0.04 (3), 0.05, 0.06, 0.07, 0.09, 0.11, and 0.21 mg/kg; sugar cane stalks (n = 6), < 0.01 (6) mg/kg; alfalfa forage (n = 12), < 0.01 (3), 0.02, 0.05, 0.09, 0.14, 0.22, 0.23, 0.33, 0.36, and 0.85 mg/kg; alfalfa hay (n = 12); 0.01 (3), 0.03, 0.12, 0.22, 0.39, 0.64, 0.75, 0.80, 1.0, and 2.2 mg/kg; carrot (greenhouse; n = 4), < 0.02 (4) mg/kg; lettuce (greenhouse; n = 4), < 0.02 (2), 0.03 (2) mg/kg; and tomato (greenhouse; n = 4): < 0.01 (4) mg/kg.

Overall, the residue profile in primary crops and rotational crops differ, with spiromesifen and Sp-enol being the principal residues in primary crops and 4-hydroxy-Sp-enol generally being the principal residue in rotational crops. Both primary and rotational crop residues include the glucose-conjugated 4-hydroxymethyl-Sp-enol metabolite.

Animal metabolism

The Meeting received animal metabolism studies with spiromesifen in rats, lactating goat and laying hens. The metabolism and distribution of spiromesifen in animals were investigated using test material radiolabelled at the 3-dihydrofuranone moiety.

The metabolism of spiromesifen in rats was evaluated by the WHO Core Assessment Group of the 2016 JMPR.

A single lactating goat was dosed with [^{14}C]spiromesifen at a dose equivalent to approximately 344 ppm in the diet daily for three consecutive days. Approximately 50% of the applied dose (AD) was recovered: 33% in urine, 15% in faeces, 1% in cage wash, and < 1% in milk and tissues.

Total radioactive residues (TRR) in the milk accounted for 0.02% AD. Residues in milk may have plateaued; however, the duration of dosing was not sufficient to make a definitive conclusion. In tissues residue levels were highest in kidney (8.4 mg eq/kg), followed by liver (3.8 mg eq/kg), fat (ca. 0.5 mg eq/kg) and muscle ca. 0.23 mg eq/kg.

The principal residue in all matrices was Sp-enol, ranging from 29% TRR (1.0 mg/kg) in liver to 77% TRR (6.9 mg/kg) in kidney. Spiromesifen was not a major residue in tissues. In milk spiromesifen and Sp-enol were reported as combined residues and represented 33% TRR (0.03 mg/kg). Analysis of milk and tissue samples at ca. 1–2 months after sacrifice and ca. 11 months after sacrifice indicated some degradation of spiromesifen and a concomitant increase in Sp-enol in fat and milk; spiromesifen was generally stable in other matrices. 4-hydroxymethyl-Sp-enol was also a significant residue in milk (24% TRR, 0.02 mg/kg). The only other major residue that was reported was a glucuronide conjugate of Sp-enol at 21% TRR (0.77 mg/kg) in liver.

Laying hens were orally dosed with [^{14}C]spiromesifen at a dose equivalent to 190 ppm in the diet daily for three consecutive days. Sixty-three percent of the AD was recovered. The majority of the dose was eliminated in the excreta (58%) and cage wash (4%). Retained residues were highest in liver (1.7 mg eq/kg), lower in skin (0.32 mg eq/kg), fat (0.09 mg eq/kg), muscle (0.067 mg eq/kg), and eggs (0.026 mg eq/kg). Total ^{14}C residues in eggs did not plateau during the dosing period and were 0.032 mg eq/kg on the third day of dosing.

Spiromesifen was the only major residue in fat (51% TRR, 0.046 mg/kg) and a large percentage of the TRR in egg (28% TRR, 0.007 mg/kg). In liver, the only major residue was Sp-enol (38% TRR, 0.026 mg/kg), which was also a major residue in skin (18% TRR, 0.06 mg/kg), liver (18% TRR, 0.30 mg/kg), and egg (44% TRR, 0.011 mg/kg). In skin and liver, 4-carboxy-hydroxy-Sp-enol was a major residue, occurring at 12% TRR (0.04 mg/kg) and 16% TRR (0.26 mg/kg, respectively. The 4-carboxy-3-hydroxy-Sp-enol metabolite was a major residue in liver (20% TRR, 0.34 mg/kg). In other matrices, the 4-carboxy-3-hydroxy-Sp-enol metabolite could not be analytically resolved from oxo-cyclopentyl-Sp-enol. The unresolved residues were major only in skin (42% TRR, 0.13 mg/kg).

Metabolism of spiromesifen in animals is generally similar, with desterification leading to spiromesifen-enol, followed by multiple oxidations to the alkane portions of the molecule. Some differences, however, are noted. While both goats and hens form glucuronide conjugates of metabolized spiromesifen, in the hen, conjugation was to spiromesifen-enol whereas in goats it was to the more oxidized 4-hyroxymethyl-Sp-enol; glucuronide conjugates were not identified in the rat.

Spiromesifen plus spiromesifen-enol (free and glucuronide conjugate) made up the majority of residues in all matrices except poultry skin and liver. In those matrices, the principal residues were comprised of carboxylated and hydroxylated forms of spiromesifen-enol.

Environmental fate in soil and water

The Meeting received information on the environmental fate of spiromesifen in laboratory and field soil systems and on aqueous hydrolysis and photolysis dissipation pathways.

In an aqueous hydrolysis study, spiromesifen was quantitatively hydrolysed to Sp-enol, with hydrolysis occurring more rapidly under higher pH conditions. Estimated hydrolysis half-lives at 20 °C were 4.8 days (pH 9), 45 days (pH 7), and 107 days (pH 4). Spiromesifen is labile to photolysis. In two studies, spiromesifen had a photolysis half-life of 2 to 11 days. A photolysis study with Sp-enol indicated that it is relatively stable to photolysis (93% of the test substance remained after photo radiation equivalent to 31 days at 33° latitude).

In aerobic laboratory soil dissipation studies using four soils, major residues were spiromesifen (81–88% TRR at Day 0; < 5% TRR by Day 30 or 120), Sp-enol (30–60% TRR, Day 7

or Day 14), 4-carboxy-Sp-enol (3–11% TRR, Day 14 or Day 30), and CO_2 (58–70% TRR, Day 120). Other residues, categorized as unknown, were ≤ 2% TRR throughout the study period. At the 120-day time point, unextracted residues accounted for 16 to 21% of the radioactivity in the system. Field dissipation studies resulted in half-life estimates of 2–6 days for spiromesifen, 6–21 days for Sp-enol, and 7–15 days for total residues (spiromesifen, Sp-enol, 4-carboxy-Sp-enol, and photo isomers of spiromesifen and Sp-enol).

The results of the environmental fate studies, including rotational crop studies, indicate that spiromesifen and its major metabolites (Sp-enol and its carboxylated and/or hydroxylated degradation products) are not likely to be persistent in the environment, and mineralisation to CO_2 is likely to be significant.

Methods of analysis

The Meeting received description and validation data for analytical methods for residues of spiromesifen, Sp-enol, 4-hydroxymethyl-Sp-enol, and 4-hydroxymethyl-Sp-enol glucoside in plant and livestock matrices.

For most data gathering methods, residues are extracted with acetonitrile:H_2O (4:1, v/v). Methods designed to include analysis of 4-hydroxymethyl-Sp-enol generally included a hydrolysis step to cleave the glucose conjugate from the molecule. Extracted residues undergo clean-up by solid-phase extraction (SPE). Residue separation and analysis is by HPLC-MS/MS using deuterated internal standards or matrix-matched standards. For most matrices, the limits of quantification (LOQs) for each analyte, defined as the lowest limit of method validation, is 0.01 mg/kg. Papaya and tea have LOQs of 0.02 mg/kg for each analyte.

The method for livestock matrices specifies extraction with acetonitrile:H_2O (4:1, v/v) at elevated temperature and pressure (70 °C, 10300 KPa) and includes both acid and alkaline hydrolysis steps in order to assay free and conjugated 4-hydroxymethyl-Sp-enol. The reported LOQs for each analyte in livestock matrices are 0.005 mg/kg for milk, 0.01 mg/kg for fat and muscle, and 0.05 mg/kg for kidney and liver.

All of the submitted methods are adequate for the direct analysis of residues of spiromesifen and Sp-enol. 4-hydroxymethyl-Sp-enol and its conjugates are also supported with adequate methods, provided that a hydrolysis step is included in order to assay both free and conjugated forms of the hydroxymethyl metabolite.

Stability of residues in stored analytical samples

The Meeting received data on the stability of residues of spiromesifen and Sp-enol in multiple crop commodities, and the stability of 4-hydroxymethyl-Sp-enol in turnip root and wheat commodities. In the case of spiromesifen and Sp-enol, both compounds were added to homogenized test matrix. Samples were placed into frozen storage and analysed by the method(s) used in the supervised residue trial. For 4-hydroxymethyl-Sp-enol, separate samples were prepared and fortified with the test substance. During the storage periods, the percent remaining of spiromesifen decreased in most commodities, with corresponding increases in the percent remaining of Sp-enol, indicating that the parent compound was degrading in storage and forming the enol metabolite, which remained stable.

Spiromesifen was stable for less than or up to ca. 160 days in mustard greens, maize stover, potato tuber, undelinted cotton seed, and cotton gin trash; for up to or at least 316 to 376 days in cucumber, tomato fruit and processed commodities, maize grain and forage, and potato processed commodities; and at least 679 to 727 days in melon peel, wheat grain, forage, and hay, and turnip root.

The sum of spiromesifen and Sp-enol amounted to ca. 100% of the applied material remaining throughout the storage periods for the various storage stability samples, indicating that in total, those residues are stable for at least ca. 365 days in tomato (fruit and processed commodities), mustard greens, maize (grain and forage), potato (tuber and processed commodities), and cotton (seed

and gin trash); and at least ca. 700 days in cucumber, melon peel, French beans, wheat grain, and turnip root.

Residues of 4-hydroxymethyl-Sp-enol were stable during frozen storage for at least ca 450 days in wheat and sugar beet raw commodities.

Analyses from livestock feeding studies were completed within 35 days of sample collection; therefore, supporting storage stability data for residues of spiromesifen in livestock commodities are not required. The Meeting noted, however, that analyses of livestock matrices in the goat metabolism study at 1–2 months after slaughter and again ca. 11 months after slaughter indicated that spiromesifen likely degraded to Sp-enol in fat and milk.

Definition of the residue

Plants

In metabolism studies with tomato, lettuce, and cotton as primary crops, spiromesifen, Sp-enol, and 4-hydroxymethyl-Sp-enol (free and conjugated) consistently accounted for 75 to 100 percent of the total radioactive residues in the harvested commodities at maturity. Spiromesifen was the predominant residue in tomato fruit (86% TRR), lettuce leaves (58% TRR), and cotton seed (82% TRR in delinted seed). Sp-enol was the predominant residue in cotton gin trash (49% TRR), followed by spiromesifen (26% TRR). 4-Hydroxymethyl-Sp-enol was > 10% TRR only in lettuce leaves. Spiromesifen concentrations were at quantifiable levels (0.24–1.7 mg/kg) in all matrices except cotton seed, where the TRR was low (ca. 0.05 mg eq/kg). In crop field trials, spiromesifen was also consistently observed to occur at levels greater than Sp-enol; although in multiple cases, the proportion of Sp-enol increased relative to spiromesifen at longer time periods between final application and harvest. Residues of 4-hydroxymethyl-Sp-enol were not measured in crop field trials.

In confined and field rotational crop studies, spiromesifen and Sp-enol were < LOQ in all crop samples, and 4-hydroxymethyl-Sp-enol (free and conjugated) was the predominant residue (ca. 50% TRR; up to 0.16 mg/kg in food crops and 2.2 mg/kg in feed crops at a 30-day PBI). Residues of 4-hydroxymethyl-Sp-enol in wheat grain were still the predominant residues, but were < 0.005 mg/kg at all PBIs. Two additional metabolites occurred as major residues in most commodities in the confined rotational crop study: 3-pentanol-Sp-enol and dihydroxy-Sp-enol. Maximum residues for both compounds were observed at the 30-day PBI, and ranged across all matrices from 0.004 to 0.09 mg/kg each. Levels were generally lower at longer PBIs and were observed at 10 to 40% of the levels of free and conjugated 4-hydroymethyl-Sp-enol. These compounds were not assayed in the field rotation studies.

In the high-temperature hydrolysis study, spiromesifen was converted to Sp-enol. The conversion was ca 25% under pasteurisation conditions, more extensive (ca. 85%) under baking, boiling, and brewing conditions, and essentially complete under conditions mimicking sterilisation. No other residues were identified in the study. In many crop matrices, spiromesifen was not demonstrated to be stable during storage. Data indicate that the parent compound degrades to the Sp-enol metabolite, which is stable during storage. As a result, when considered together, residues of spiromesifen and Sp-enol are stable during frozen storage.

The analytical method for plant matrices is able to determine residues of spiromesifen, Sp-enol, and free 4-hydroxymethyl-Sp-enol, and with the addition of a hydrolysis step, glucose-conjugated 4-hydroxymethyl-Sp-enol.

Although spiromesifen was the most predominant residue in primary crops and would be a suitable marker for compliance purposes, its breakdown during storage to Sp-enol necessitates that residues of the Sp-enol metabolite be taken into account in stored analytical samples. The Meeting agreed that combined residues of spiromesifen and spiromesifen-enol {4-hydroxy-3-(2,4,6-trimethylphenyl)-1-oxaspiro[4.4]non-3-en-2-one}, expressed as parent spiromesifen, are suitable for enforcement purposes in plant commodities.

In considering residues for dietary risk assessment, crop field trials reported residues of spiromesifen and Sp-enol. However, the trials did not include analysis of free and conjugated 4-hydroxymethyl-Sp-enol. Based on the data from the lettuce metabolism study, residues of 4-hydroxymethyl-Sp-enol (free + conjugated) are expected to be at one fourth the concentration of spiromesifen and Sp-enol (combined) in leafy crops (significant residues of 4-hydroxymethyl-Sp-enol are not expected in other crops). A comparison of dietary exposure estimates with and without residues of 4-hydroxymethyl-Sp-enol in leafy crops indicates that exposure to that compound is not negligible. The Meeting determined that Sp-enol and 4-hydroxymethyl-Sp-enol are no more toxic than spiromesifen, and that for risk assessment, dietary exposure is adequately covered by the ADI for spiromesifen. Therefore, the Meeting determined that combined residues of spiromesifen, Sp-enol, and free and conjugated 4-hydroxymethyl-Sp-enol {4-hydroxy-3-[4-(hydroxymethyl)-2,6-dimethylphenyl]-1-oxaspiro[4.4]non-3-en-2-one} expressed as spiromesifen, are appropriate for assessing dietary risk from residues in plant commodities.

Livestock

In the lactating goat metabolism study (dose = 344 ppm in the feed), TRR were higher in kidney (8.9 mg eq/kg) and liver (3.6 mg eq/kg) than in fat (0.47 mg eq/kg), muscle (0.26 mg eq/kg), and milk (0.11 mg eq/kg). The principal residue in all goat matrices was Sp-enol, making up 29 to 77% of the total residue. The glucose conjugate of Sp-enol was also a major residue in liver (21% TRR), and 4-hydroxymethyl-Sp-enol was a major residue in milk (24% TRR). All other residues were < 10% TRR. In the feeding study with lactating cattle (dosing up to 50 ppm in the feed), residues of spiromesifen + Sp-enol were found at quantifiable levels in samples of milk, fat, kidney, and liver from the highest dose group, and in fat and kidney samples from the middle dose group; residues of spiromesifen + Sp-enol were < LOQ in samples of muscle from all dose groups and in all other matrices at the lowest dosing level. Residues of 4-hydroxymethyl-Sp-enol (free and conjugated) were < LOQ in all samples from the feeding study.

In the laying hen metabolism study, TRR were highest in liver (1.7 mg eq/kg) and skin (0.32 mg eq/kg), with lower residues in muscle (0.067 mg eq/kg), fat (0.09 mg eq/kg), and egg (0.026 mg eq/kg). Spiromesifen was the only major residue in fat (51% TRR) and a major residue in egg (28% TRR). The Sp-enol metabolite was a major residue in all matrices except fat, ranging from 18% TRR in skin to 44% TRR in egg. The only other major residues were 4-carboxy-3-hydroxy-Sp-enol in liver (20% TRR), unresolved 4-carboxy-3-hydroxy-Sp-enol + oxo-cyclopentyl-Sp-enol (42% TRR) in skin, and 4-carboxy-hydroxy-Sp-enol in liver (16% TRR) and skin (12% TRR).

Analytical methods for animal matrices are available for the analysis of spiromesifen, Sp-enol, and free and glucuronide-conjugated 4-hydroxymethyl-Sp-enol. Some degradation of spiromesifen to Sp-enol was noted in samples from the lactating goat metabolism study.

The Meeting agreed that combined residues of spiromesifen and Sp-enol are suitable markers for compliance with MRLs in livestock commodities.

In the feeding study, total residues of spiromesifen and spiromesifen-enol were ca. 11 fold greater in fat than in muscle and ca. 22 fold greater in cream than in skim milk. On that basis, the Meeting concluded that the residue is fat soluble.

For assessing dietary risk, the Meeting noted that residues of 4-hydroxymethyl-Sp-enol were not detected in any sample from the cattle feeding study. Therefore, it was excluded from the residue definition for dietary risk assessment. 4-carboxy-3-hydroxy-Sp-enol was as significant residue only in liver and skin (both of which are modelled as edible offal for dietary risk assessment). The Meeting determined that 4-carboxy-3-hydroxy-Sp-enol is not more toxic than spiromesifen and that dietary risk assessment to that metabolite is adequately covered by the ADI for spiromesifen. A similar conclusion could not be made for the related, non-specific hydroxyl analogues (4-carboxy-hydroxy-Sp-enol). For the specific 4-carboxy-3-hydroxy-Sp-enol metabolite, a comparison of long-term dietary exposure estimates for spiromesifen with and without inclusion of 4-carboxy-3-hydroxy-Sp-enol residues are indistinguishable, leading to the conclusion that relative exposure to the metabolite is negligible and that it can be excluded from the residue definition for dietary risk assessment. The

Meeting determined that the residue definition for assessing dietary risk from livestock commodities is the sum of residues of spiromesifen and Sp-enol, expressed as spiromesifen.

The non-specific hydroxyl analogues observed in the poultry metabolism study were 4-carboxy-hydroxy-Sp-enol and 4-carboxy-dihydroxy-Sp-enol. The Meeting decided to use the TTC approach to evaluate dietary risk from exposure to these compounds (as combined residues). The ratio of the hydroxy analogues in liver (0.33 mg/kg) to spiromesifen + Sp-enol (0.31 mg/kg) is 1.1. The estimated residue for evaluating both one-day and long-term dietary exposure is 0.055 mg/kg. The IESTI resulted in one-day exposure estimates of ≤ 0.36 µg/kg bw/day. The long-term dietary exposure estimates were ≤ 0.005 µg/kg bw/day. As the Cramer Class III TTC thresholds are 5 µg/kg bw/day for one-day intake and 1.5 µg/kg bw/day for long-term intake, public-health concerns were considered unlikely for 4-carboxy-hydroxy-Sp-enol and 4-carboxy-dihydroxy-Sp-enol based on the uses evaluated by this Meeting.

Definition of the residue for plant and animal commodities (for compliance with the MRL): *sum of spiromesifen and 4-hydroxy-3-(2,4,6-trimethylphenyl)-1-oxaspiro[4.4]non-3-en-2-one (spiromesifen-enol), expressed as spiromesifen.*

Definition of the residue for plant commodities (for dietary risk assessment): *sum of spiromesifen, 4-hydroxy-3-(2,4,6-trimethylphenyl)-1-oxaspiro[4.4]non-3-en-2-one (spiromesifen-enol), and 4-hydroxy-3-[4-(hydroxymethyl)-2,6-dimethylphenyl]-1-oxaspiro[4.4]non-3-en-2-one (4-hydroxymethyl-spiromesifen-enol) (free and conjugated), all expressed as spiromesifen.*

Definition of the residue for livestock commodities (for dietary risk assessment): *sum of spiromesifen and 4-hydroxy-3-(2,4,6-trimethylphenyl)-1-oxaspiro[4.4]non-3-en-2-one (spiromesifen-enol), expressed as spiromesifen.*

The Meeting concluded that the residue is fat soluble.

Results of supervised residue trials on crops

The Meeting received supervised trial data for the foliar application of spiromesifen on strawberry, papaya, broccoli, cabbage, cucumber, melon, summer squash, peppers, tomato, sweet corn, head and leaf lettuce, spinach, mustard greens, common bean (pods and/or immature seeds), dry bean, cassava, potato, maize, popcorn, cotton, coffee, and tea.

Labels for end-use products containing spiromesifen were available from Belgium, Brazil, Canada, Central America, Colombia, Ecuador, France, Greece, India, Italy, Japan, Kenya, Mexico, the Netherlands, New Zealand, Peru, Spain, and the United States describing the registered uses of spiromesifen.

For all trials, residues were determined by a method involving extraction with acetonitrile:H_2O and analysis by HPLC-MS/MS with either deuterated internal standards or matrix-matched standards. For most crops, the extracts underwent clean-up by solid-phase extraction.

For all trials, combined residues of spiromesifen and Sp-enol (i.e., total spiromesifen) are supported by adequate storage stability data.

In determining combined residues of spiromesifen and Sp-enol, the following convention was used: residue > LOQ = finite residue, residue between LOQ and LOD = LOQ (if both compounds are in this category, then combined is < 2× LOQ), and residue < LOD = 0 contribution (if both compounds < LOD, then combined is < LOQ).

Based on the ratio of free and conjugated 4-hydroxymethyl-Sp-enol to spiromesifen + Sp-enol from the lettuce metabolism study (0.25), residue estimates of spiromesifen + Sp-enol have been multiplied by 1.25 to derive residue estimates for assessing dietary intake from leafy vegetables and Brassica leafy vegetables. An adjustment is not necessary for other crops.

Berries and other small fruits

Spiromesifen is registered in the US for outdoor use on low-growing berry, which includes bearberry, bilberry, blueberry (lowbush), cloudberry, cranberry, lingonberry, muntries, partridgeberry, and

strawberry. The US GAP is for up to three applications, on a 7-day interval, each at 0.28 kg ai/ha. The PHI is 3 days. Spiromesifen is also registered for greenhouse use on strawberry in the Netherlands, with an unspecified number of applications at 0.12 kg ai/ha and a 1-day PHI.

Strawberry

Eight supervised trials were conducted in the US according to the US GAP. The trials resulted in the following independent residue values (n = 8): 0.26, 0.27, 0.47 (2), 0.57, 0.70, 1.5, and 1.6 mg/kg.

Four trials were conducted in Europe according to the Netherlands GAP. The trials resulted in the following independent residue values (n = 4): 0.11, 0.23, 0.26, and 0.62 mg/kg.

The residue data show the US GAP to be the critical GAP. The Meeting estimated a maximum residue level and STMR for spiromesifen residues in strawberries of 3 mg/kg and 0.52 mg/kg, respectively.

As the berries covered by the registered use correspond to the Codex subgroups for low-growing berries (FB 2009), the Meeting recommends extrapolating the estimates from strawberry to subgroup FB 2009.

Assorted tropical and sub-tropical fruit—inedible peel

Papaya

The GAP for papaya is from registrations in Ecuador, with two applications at 0.12 kg ai/ha and a 7-day PHI. An alternative GAP exists from registrations in Colombia and Mexico, consisting of a single application at 0.12 kg ai/ha and a 7-day PHI. Six residue trials were conducted in Ghana wherein 1 to 3 applications were made at 0.12 kg ai/ha. Fruits were harvested 0 to 56 DALA, with harvest 7 DALA for only two trials. Residues were reported for spiromesifen only.

Two trials were conducted matching the Ecuador GAP and two trials were conducted matching the Columbia and Mexico GAP. The number of trials reflecting either GAP is insufficient. Furthermore, the residues that were reported do not address the residue definition for either enforcement or dietary intake. For these reasons, the Meeting is not making a recommendation for residues of spiromesifen in papaya.

Brassica vegetables, head cabbage, flowerhead cabbage

Spiromesifen is registered in Canada and the US for use on Brassica leafy vegetables—broccoli and Chinese (gai lon) broccoli, Brussels sprouts, cabbage, Chinese mustard (gai choy) cabbage, cauliflower, cavalo broccolo, kohlrabi, mustard spinach, and rape greens. The GAP is three applications, each at 0.144 kg ai/ha and a 7-day PHI.

Broccoli

Seven supervised trials were conducted in the US at GAP. The two trials at Hillsboro, Oregon are not considered to be independent. The trials resulted in the following independent residue values (n = 6): 0.02, 0.056, 0.091, 0.15, 0.21, and 0.58 mg/kg.

Cabbage

Six supervised trials were conducted in the US, five of which were at GAP. The trials resulted in the following independent residue values (n = 5): < 0.02, 0.18 0.44, 1.6, and 1.8 mg/kg.

As the registered use and available data correspond to the Codex group for Brassica (cole or cabbage) vegetables, head cabbages, flowerhead Brassicas (VB 0040); the median residues from the broccoli and cabbage trials are within 5-fold of each other; and analysis by the Kruskal-Wallis test showed no evidence of a difference between the residue populations, the Meeting decided to make a recommendation for the group based on the following combined broccoli and cabbage data (n = 11): < 0.02, 0.02, 0.056, 0.091, 0.15, 0.18, 0.21, 0.44, 0.58, 1.6, and 1.8 mg/kg.

The Meeting estimated a maximum residue level STMR, and highest residue for spiromesifen residues in Brassica (cole or cabbage) vegetables, head cabbages, flowerhead Brassicas of 3 mg/kg, 0.18 mg/kg, and 1.8 mg/kg, respectively.

Fruiting vegetables, Cucurbits

Spiromesifen has registration for use on field-grown cucurbit vegetables as a group, as well as greenhouse-grown cucumber and melon as individual crops. The field and greenhouse uses are considered to be different GAPs. As such the Meeting has evaluated the greenhouse data against the individual-crop greenhouse GAPs and considered the field data against the crop group field GAP.

Cucumber

Spiromesifen is registered in Greece for use on greenhouse-grown cucumbers, with four applications, each at 0.216 kg ai/ha and a 3-day PHI. In addition, spiromesifen is registered for use on field-grown cucurbit vegetables in Canada and the US, with three applications at 0.144 kg ai/ha and a 7-day PHI.

Seven supervised greenhouse trials were conducted in Europe according to the Grecian GAP. The trials resulted in the following independent residue values (n = 7): 0.03 (2), 0.04, 0.05, 0.06, 0.07, and 0.08 mg/kg.

Six supervised field trials were conducted according to the Canadian/US GAP. The trials resulted in the following independent residue values (n = 6): < 0.02 (4), 0.026, and 0.034 mg/kg.

The residue data show the greenhouse use to be the critical GAP. The Meeting estimated a maximum residue level and STMR for spiromesifen residues in cucumber of 0.15 mg/kg and 0.05 mg/kg, respectively.

Melon

Spiromesifen is registered in Greece for use on greenhouse-grown melons, with four applications, each at 0.216 kg ai/ha and a 3-day PHI. In addition, spiromesifen is registered for use on field-grown cucurbit vegetables in Canada and the US, consisting of three applications at 0.144 kg ai/ha and a 7-day PHI.

Eight supervised greenhouse trials were conducted in Europe with application rates of ca. 0.6× the greenhouse GAP. The trials resulted in the following independent residue values (n = 8): 0.03 (2), 0.04, 0.05 (2), 0.06, and 0.07 (2) mg/kg.

The Meeting noted that the supervised greenhouse trials available did not correspond to the submitted GAP and decided to apply the proportionality approach. After scaling residues to an application rate of 0.216 kg ai/ha by using a proportionality factor of approximately 1.5 (0.216 kg ai/ha ÷ 0.144 kg ai/ha; individual trial results were scaled based on actual application rates used in the field trials), the following data set resulted (n = 8): 0.045 (2), 0.06, 0.075 (2), 0.088, and 0.11 (2) mg/kg.

In five trials also analysing the pulp, no residues of total spiromesifen above the LOQ of 0.01 mg/kg were found. However, since the proportionality approach was applied by upscaling the results, the LOQ information cannot be used for the refined estimation of the dietary intake. Therefore, the Meeting based its estimation of STMR and HR values on whole melons instead.

Six supervised field trials were conducted according to the Canadian/US GAP. The trials resulted in the following independent residue values (n = 6): < 0.02, 0.025, 0.027, 0.035, and 0.045 mg/kg.

The residue data show the greenhouse use to be the critical GAP. The Meeting estimated a maximum residue level and STMR for spiromesifen residues in melon, except watermelon of 0.3 mg/kg and 0.075 mg/kg, respectively.

Summer squash

The GAP for summer squash is from registrations in Canada and the US for cucurbit vegetables, with three applications, each at 0.144 kg ai/ha and a 7-day PHI.

Six supervised trials were conducted in the US at GAP. The two trials conducted in Vero Beach, Florida are not considered to be independent. The trials resulted in the following independent residue values (n = 5): < 0.02 (2), 0.021, 0.022, and 0.05 mg/kg.

As the registrations in Canada and the US correspond to the "fruiting vegetables, cucurbit" Codex group; the median residue values from the trials conducted according to the Canadian/US GAP with cucumber, melon, and summer squash do not differ by more than 5-fold; and analysis by the Kruskal-Wallis test showed no evidence of a difference between the residue populations, the available data support a group recommendation. The combined field residues from cucumber, melon, and summer squash are (n = 16): < 0.02 (7), 0.021, 0.022, 0.025, 0.026, 0.027, 0.034, 0.035, 0.045, and 0.05 mg/kg.

The Meeting noted the individual recommendations for cucumber and melon from the greenhouse GAPS, and estimated for fruiting vegetables, cucurbit except cucumber and melon, maximum residue level and STMR for spiromesifen residues of 0.09 mg/kg and 0.021 mg/kg, respectively.

Fruiting vegetables, other than Cucurbits

Spiromesifen has registrations for use on greenhouse-grown cucumber and melon as individual crops, as well as field-grown fruiting vegetables, other than cucurbits, as a group. The field and greenhouse uses are considered to be different GAPs. As such the Meeting has evaluated the greenhouse data against the individual-crop greenhouse GAPs and considered the field data against the crop group GAP.

Tomato

Spiromesifen is registered in France and Italy for use on greenhouse-grown tomato, with four applications, each at 0.216 kg ai/ha and a 3-day PHI. In addition, there are registrations in Canada, Mexico, and the US for use on field-grown crops in the NAFTA crop group fruiting vegetables, which corresponds to the Codex group fruiting vegetables other than cucurbits—except sweet corn and mushrooms. Under the field GAP, three applications are allowed, each at 0.144 kg ai/ha, with a 1-day PHI.

Sixteen supervised greenhouse trials were conducted in Europe according to the greenhouse GAP. The trials resulted in the following independent residue values (n = 16): 0.07, 0.09 (2), 0.10, 0.11, 0.12, 0.15, 0.16, 0.17, 0.19 (2), 0.21, 0.24, 0.29, 0.42, and 0.50 mg/kg.

Twelve supervised field trials were conducted in the US according to the field GAP. The trials resulted in the following independent residue values (n = 12): 0.047, 0.056, 0.063, 0.065 (2), 0.094, 0.099, 0.11, 0.14, 0.17, 0.18, and 0.34 mg/kg.

The residue data show the greenhouse use to be the critical GAP for tomato. The Meeting estimated a maximum residue level and STMR for spiromesifen residues in tomato of 0.7 mg/kg and 0.165 mg/kg, respectively. Noting the registration for use of spiromesifen in Greece on greenhouse-grown eggplant, the Meeting decided to extrapolate these estimates to greenhouse-grown eggplant.

Peppers

Spiromesifen is registered in Greece and Italy for use on greenhouse-grown sweet peppers, with four applications, each at 0.216 kg ai/ha, and a 3-day PHI. In addition, there are registrations in Canada, Mexico, and the US for use on the NAFTA crop group fruiting vegetables, which corresponds to the Codex group fruiting vegetables other than cucurbits—except sweet corn and mushrooms. Under the Canada, Mexico, and US GAPs, three applications are allowed, each at 0.144 kg ai/ha, with a 1-day PHI.

Nine supervised greenhouse trials were conducted on sweet peppers in Europe according to the greenhouse GAP. The trials resulted in the following independent residue values (n = 9): 0.07, 0.09, 0.11, 0.12, 0.13 (2), 0.19, and 0.22 (2) mg/kg.

Twenty supervised field trials were conducted on peppers (including chilli peppers) in the US; however only ten matched the GAP with respect to the PHI. The trials resulted in the following independent residue values (n = 10): < 0.02, 0.030, 0.035, 0.040, 0.050, 0.060 (2), 0.085, 0.17, and 0.32 mg/kg.

The residue data show the field use to be the critical GAP for peppers. Given that, and noting the GAPs for use of fruiting vegetables other than cucurbits in Canada, Mexico, and the US, the Meeting decided to explore using combined field data to evaluate residues in fruiting vegetables, other than cucurbits—except sweet corn and mushroom (excluding tomato and eggplant, as they are addressed by the greenhouse use discussed above). The residue data from field-grown peppers and tomato have median values that do not differ by more than five-fold; however, the Kruskal-Wallis test indicates that the residues are from separate populations.

As the residues in field-grown tomato are, overall, greater than those in field-grown pepper, the Meeting used the data from field-grown tomato and estimated maximum residue levels and STMRs for spiromesifen residues in peppers, okra, and pepino of 0.5 mg/kg and 0.097 mg/kg, respectively.

Of the available data from peppers, only three trials were conducted with chilli pepper varieties, which is insufficient for making a recommendation for dried chilli pepper based on chilli-pepper-specific data. Therefore, the Meeting used the generic pepper data and a default processing factor of 10 to estimate a maximum residue level and STMR for spiromesifen residues in chilli pepper (dried) of 5 mg/kg and 0.55 mg/kg, respectively.

Sweet corn

The GAP for sweet corn is from a registration in the US, with two applications, each at up to 0.3 kg ai/ha (not to exceed 0.3 kg ai/ha/season) and a 5-day PHI for sweet corn for fresh consumption.

Twelve supervised trials were conducted in the US at GAP. The trials resulted in the following independent residue values (n = 12): < 0.02 (12) mg/kg.

Noting that residues were below the limit of detection (0.0016 mg/kg) in all samples, the Meeting estimated a maximum residue level, STMR, and highest residue for spiromesifen residues in sweet corn (corn-on-the-cob) of 0.02* mg/kg, 0 mg/kg, and 0 mg/kg, respectively.

Leafy vegetables, including Brassica leafy vegetables

The GAP is from registrations in Canada and the US for leafy vegetables and Brassica leafy vegetables, with three applications, each at 0.144 kg ai/ha and a 7-day PHI.

Six supervised trials were conducted on head lettuce in the US at GAP. The trials resulted in the following independent residue values (n = 6): 0.16, 0.74, 0.97, 1.4, 2.4, and 4.5 mg/kg.

Seven supervised trials were conducted on leaf lettuce in the US at GAP. The two trials in Fresno, CA are not considered to be independent. The trials resulted in the following independent residue values (n = 6): 0.52, 0.90, 1.0, 1.7, 2.5, and 9.3 mg/kg.

Seven supervised trials were conducted on spinach in the US at GAP. The two trials in Suffolk, VA are not considered to be independent. The trials resulted in the following independent residue values (n = 6): 0.27, 1.8, 2.2, 5.0, 6.4, and 7.3 mg/kg.

Eight supervised trials were conducted on mustard greens in the US at GAP. The trials resulted in the following independent residue values (n = 8): 0.66, 1.1, 1.3, 1.5, 1.6, 2.1, 9.9, and 10 mg/kg.

The Meeting noted that the GAP in the US covers the Codex group of leafy vegetables including Brassica leafy vegetables and decided to explore the possibility of estimating a group

maximum residue level for spiromesifen. As median residues in head lettuce, leaf lettuce, spinach, and mustard greens differed by less than 5-fold and the residue populations were not significantly different by the Kruskal-Wallis test, the Meeting decided to make a recommendation for leafy vegetables, including Brassica leafy vegetables based on the following combined data set (n = 26): 0.16, 0.27, 0.52, 0.66, 0.74, 0.90, 0.97, 1.0, 1.1, 1.3, 1.4, 1.5, 1.6, 1.7, 1.8, 2.1, 2.2, 2.4, 2.5, 4.5, 5.0, 6.4, 7.3, 9.3, 9.9, and 10 mg/kg.

From those combined data, the Meeting estimated a maximum residue level for spiromesifen residues in leafy vegetables and in Brassica leafy vegetables of 15 mg/kg. The median and highest residues are 1.65 mg/kg and 10 mg/kg, respectively. Correcting these values to account for residues of 4-hydroxymethyl-Sp-enol (factor of 1.25) results in STMR and highest residue estimates of 2.06 mg/kg and 12.5 mg/kg, respectively.

Legume vegetables

Beans (Phaseolus spp.) (green pods and/or immature seeds)

The GAP in the Netherlands allows for an unspecified number of applications to greenhouse-grown beans, each at up to 0.12 kg ai/ha, with a 1-day PHI.

Four supervised greenhouse trials were conducted in southern Europe approximating the Netherlands GAP were (n = 4): 0.11, 0.12, 0.27, and 0.64 mg/kg.

As four trials are not adequate for making robust residue estimates for beans, and no other trials matching the Netherlands GAP were available, the Meeting considered an alternate greenhouse GAP from Greece (up to four applications, each up to 0.144 kg ai/ha, and a 3-day PHI).

Eight supervised greenhouse trials were conducted in southern Europe matching the Greece GAP. The trials resulted in the following independent residue values (n = 8): 0.04, 0.06, 0.07, 0.08, 0.09, 0.14, 0.26, and 0.64 mg/kg.

The Meeting estimated a maximum residue level and STMR for spiromesifen residues in beans (*Phaseolus)* (green pods and/or immature seeds) of 1 mg/kg and 0.085 mg/kg, respectively.

Pulses

Dry beans

The GAP for bean (dry seed) is from a registration in Brazil, with three applications, each at 0.144 kg ai/ha and a 21-day PHI.

Three supervised trials were conducted in Brazil at GAP and at an application rate of 2× GAP. The trials reported residues of parent spiromesifen only.

Three trials are insufficient for making robust estimates of expected residues. Furthermore, the residues that were reported do not address the residue definition for either enforcement or dietary intake. For these reasons, the Meeting is not making a recommendation for residues of spiromesifen in bean (dry seed).

Root and tuber vegetables

The GAP for is from a registration in the US for tuberous and corm vegetables, with two applications, each at 0.280 kg ai/ha and a 7-day PHI. An alternate GAP exists from registration in Canada for use on tuberous and corm vegetables, with two applications, each at 0.144 kg ai/ha and a 7-day PHI.

Cassava (manioc)

Five supervised trials on cassava (manioc) were conducted in Brazil using three applications, each at ca. 0.14 kg ai/ha and included harvest 7 DALA. The trials resulted in the following independent residue values (n = 5): < 0.02 (5) mg/kg.

Potato

Sixteen supervised trials on potatoes were conducted in the US at GAP. The trials resulted in the following independent residue values (n = 16): < 0.02 (16) mg/kg.

Based on the registrations on tuberous and corm vegetables, and the lack of quantifiable residues in cassava and the lack of detected residues in potato, the Meeting extrapolated the results to include sweet potato, and estimated a maximum residue level, STMR, and highest residue for spiromesifen residues in cassava, potato, and sweet potato of 0.02* mg/kg, 0.01 mg/kg, and 0.01 mg/kg, respectively.

Cereal grains

Maize

The GAP for maize is from a registration in the US, with two applications, each at up to 0.3 kg ai/ha (not to exceed 0.3 kg ai/ha/season) and a 30-day PHI.

Forty supervised trials were conducted in the US at GAP. The trials resulted in the following independent residue values (n = 40): < 0.02 (40) mg/kg. In addition, spiromesifen + Sp-enol residues in corn grain were < 0.02 mg/kg from three trials conducted at a 5-fold exaggerated rate.

The Meeting estimated a maximum residue level and STMR for spiromesifen residues in maize of 0.02* mg/kg and 0 mg/kg, respectively.

Popcorn

The GAP for popcorn is from a registration in the US, with two applications, each at up to 0.3 kg ai/ha (not to exceed 0.3 kg ai/ha/season) and a 30-day PHI.

Three supervised trials were conducted in the US at GAP. The trials resulted in the following independent residue values (n = 3): < 0.02 (3) mg/kg.

Three trials are insufficient for making robust estimates of expected residues; however, based on the data from maize showing residues below the LOD in all trials, including exaggerated rate trials, the Meeting estimated a maximum residue level and STMR for spiromesifen residues in popcorn of 0.02* mg/kg and 0 mg/kg, respectively.

Oilseeds

Cotton

The GAP for cotton is from a registration in the US, with applications up to 0.28 kg ai/ha each (not to exceed three sprays or 0.56 kg ai/ha per season) and a 30-day PHI.

Twelve supervised trials were conducted in the US compliant with the US GAP. The trials resulted in the following independent residue values (n = 12): < 0.02 (4), 0.034, 0.11 (2), 0.18, 0.28, 0.32, 0.34, and 0.39 mg/kg.

The Meeting determined that the trials are suitable and estimated a maximum residue level and STMR for spiromesifen residues in cotton seed of 0.7 mg/kg and 0.11 mg/kg, respectively.

Seed for beverages and sweets

Coffee

The GAP for coffee is from a registration in Brazil, with two applications, each at 0.144 kg ai/ha and a 21-day PHI.

Five supervised trials were conducted in Brazil matching the Brazil GAP. The trials resulted in the following independent residue values (n = 5): < 0.02 (4), 0.035 and 0.11 mg/kg.

The Meeting estimated a maximum residue level and STMR for spiromesifen residues in coffee beans of 0.2 mg/kg and 0.02 mg/kg, respectively.

Derived products of plant origin

Tea

The GAP for tea is from a registration in Japan, with one application of a spray concentration of 0.015 kg ai/hL and a 7-day PHI. The label recommends spray rates of 2000 to 4000 L/ha.

Two trials were conducted in Japan according to the Japanese GAP; however, only summary reports were provided and they could not be adequately assessed. No other trials were available matching the Japanese GAP.

Six supervised trials were conducted in India with an application rate of 0.63 kg ai/ha and a 7-day PHI. The Meeting noted that the spray concentration used in the trials was exaggerated approximately 10-fold relative to the Japanese GAP. The Meeting used the spray concentration and maximum spray rate from the Japanese label to derive an application rate on a kg a.i./ha basis. As the application rate (0.63 kg ai/ha) from the India trials corresponds to the estimated maximum per-hectare rate from the Japanese label, the Meeting considered the residues resulting from the India trials approximating the Japanese GAP.

In trials conducted in India approximating the Japanese GAP, residues in fresh tea leaves were (n = 6): 0.66, 1.4, 4.4, 7.1, 7.7, and 10 mg/kg.

Sample of fresh tea (spiromesifen + Sp-enol = 7.1 mg/kg) from one trial in India were dried to form green tea (16 mg/kg) or fermented to form black tea (23 mg/kg). Applying the ratios of the residues for green tea (2.3) and black tea (3.2) results in residues as follows: green tea (n = 6), 1.5, 3.2, 10, 16, 18, and 23 mg/kg; and black tea (n = 6), 2.1, 4.5, 14, 23, 25, and 32 mg/kg.

Using the anticipated residues in black tea, the Meeting estimated a maximum residue level and STMR for tea green and black (black fermented and dried) of 70 mg/kg and 18.5 mg/kg, respectively.

Straw, fodder, and forage of cereal grains

Maize (including popcorn and sweet corn)

The GAP for maize is from a registration in the US, with two applications, each at up to 0.3 kg ai/ha (not to exceed 0.3 kg ai/ha/season) and PHIs of 5 days for forage and 30 days for fodder.

Forage

Fifty-six supervised trials were conducted with maize, popcorn, and sweet corn in the US at GAP. The trials resulted in the following independent residue values for forage (n = 27): 0.27, 0.42, 0.78, 1.0 (2), 1.3, 1.4, 1.5, 1.6, 1.7, 1.8 (3), 2.0, 2.1, 2.2 (2), 2.5 (3), 2.9, 3.0, 3.1, 3.2, 3.5, 3.6, and 4.4 mg/kg.

The Meeting estimated a median and highest residue for spiromesifen residues in maize forage (fresh) of 2 mg/kg and 4.4 mg/kg, respectively.

Fodder

The trials resulted in the following independent residue values for fodder (n = 34): 0.02, 0.08, 0.19 (2), 0.29, 0.31, 0.33, 0.34, 0.42, 0.49, 0.66, 0.73, 0.77, 0.84, 0.87, 0.88, 0.95, 0.97, 0.98, 1.0, 1.2, 1.3 (2), 1.4, 1.5, 1.6, 1.8, 1.9, 2.0, 2.2, 2.3, 2.6, and 4.1 mg/kg.

Based on a dry matter content of 83%, the Meeting estimated a maximum residue level for spiromesifen residues in maize fodder (dry) of 6 mg/kg.

The Meeting estimated median, and highest residues for spiromesifen residues in maize fodder (as received) of 0.96 mg/kg, and 4.1 mg/kg, respectively.

Miscellaneous fodder and forage crops

Cotton gin trash

The GAP for cotton is from a registration in the US, with three applications, each at maximum of 0.28 kg ai/ha (not to exceed 0.56 kg ai/ha/season) and a 30-day PHI.

Six supervised trials were conducted in the US at GAP. The trials resulted in the following independent residue values (n = 6): 0.41, 1.6, 2.7, 4.6, 6.0, and 11 mg/kg.

The Meeting estimated a median for spiromesifen residues in cotton gin trash of 3.65 mg/kg and a highest residue (from a single sample) of 12 mg/kg.

Fate of residues during processing

Under high-temperature hydrolysis conditions meant to mimic pasteurisation (90 °C, pH 4, 20 min.); baking, brewing, boiling (100 °C, pH 5, 60 min); and sterilisation (120 °C, pH 6, 20 min.), spiromesifen was converted to its Sp-enol metabolite. No other residues were identified. Conversion was less under the pH-4 conditions (ca. 20% formation of Sp-enol) and essentially quantitative under the pH-6 conditions.

The Meeting received data depicting the effects of processing and preparation on residue levels in strawberry, cucumber, summer squash, peppers, tomato, lettuce, spinach, beans, potato, sugar beet, maize, sorghum, wheat, cotton seed, and tea. Residue information, processing factors, and recommendations of STMR-P, HR-P, and MRLs relevant to the current evaluation are shown in the table, below.

Summary of spiromesifen residues (spiromesifen + Sp-enol) in processed commodities

	RAC residues, mg/kg				Processed residues, mg/kg	
RAC	MRL	STMR	Processed commodity	Processing factors [Median/Best estimate]	MRL	STMR-P
Strawberry	3	0.52	Jam	0.44, 0.46, 0.47, 0.58 [0.46]	–	0.24
			Preserve	0.28, 0.28, 0.32, 0.27 [0.28]	–	0.15
Broccoli	3	0.15	Cooked	1.9 [1.9]	–	0.28
Summer squash	0.09	0.02	Cooked	0.70 [0.7]	–	0.014
Tomato	0.7	0.165	Canned	0.21, < 0.06, < 0.07, 0.17, 0.35 [0.21]	–	0.035
			Juice (canned)	0.72, < 0.06, 0.13, 0.35, 0.3 [0.35]	–	0.058
			Puree	0.78, 0.72, 1.2, 2.3, 2 [1.2]	–	0.20
			Paste	2.6 [2.6]	2	0.43
			Wet pomace	4.1, 8.3, 7.4, 7.6 [7.5]	–	1.2
			Dried	5 [5]	4	0.82
Mustard greens	20	1.55	Cooked	0.14 [0.14]	–	0.22
Spinach	15	2.06	Cooked	2.1 [2.1]	–	4.3
Cotton seed	0.7	0.11	Refined oil	0.043, 0.026 [0.034]	–	0.0037
			Meal	0.08, 0.21 [0.14]	–	0.015
			Hulls	0.34 (0.34)		0.04
Tea, green, black (black, fermented and dried)	70	18.5	Black tea infusion	0.034 [0.034]	–	0.63

Residue in animal commodities

Farm animal dietary burden

The Meeting estimated the dietary burden of spiromesifen in farm animals on the basis of the diets listed in Appendix IX of the FAO Manual 2009. Calculation from highest residue, STMR (some bulk commodities) and STMR-P values provides levels in feed suitable for estimating MRLs, while calculation from STMR and STMR-P values for feed is suitable for estimating STMR values for animal commodities.

Estimated maximum and mean dietary burdens of farm animals

Dietary burden calculations for beef cattle, dairy cattle, broilers and laying poultry are provided in Appendix IX of the FAO manual. The calculations were made according to the animal diets from US-Canada, EU, Australia and Japan in the Table (Appendix IX of the FAO manual). The diets are based on residues in kale, and cotton, corn, and potato livestock feed commodities.

Livestock dietary burden for spiromesifen, ppm of dry matter diet								
	US-Canada		EU		Australia		Japan	
	Max	Mean	Max	Mean	Max	Mean	Max	Mean
Beef cattle	2.3	0.97	25	6.7	9.4	4.6	–	0.015
Dairy cattle	5.0	2.3	23	5.8	**40**	**8.6**	5.5	2.5
Poultry—broiler	–	–	0.01	0.01	–	–	–	–
Poultry—layer	–	–	**5.3**	**1.2**	–	–	–	–

The bold values, above, reflect the highest burdens for both MRL estimation (maximum diet) and STMR estimation (mean diet). Burdens from dairy cattle and layer hens are being used for beef cattle and broiler poultry, respectively.

Farm animal feeding studies

The Meeting received lactating dairy cow feeding studies, which provided information on likely residues resulting in animal commodities and milk from spiromesifen residues in the animal diet.

Lactating dairy cows

Lactating dairy cows were dosed with spiromesifen for 29 days at the equivalent of 5, 15 or 50 ppm in the diet. Analysis was for residues of spiromesifen, Sp-enol, and 4-hydroxymethyl-Sp-enol. Residues for all analyses and tissues were < 0.01 for control animals.

Residues of spiromesifen in milk reached a plateau beginning on Day 4. From Day 4 through Day 28 median and maximum residues were both 0.012 mg/kg at the 50-ppm dose level (other levels not analysed). In tissues, mean (and maximum) residues at the 5, 15, and 50-ppm feeding levels, respectively were: fat < 0.01 (< 0.01), 0.03 (0.045), and 0.093 (0.12) mg/kg; kidney < 0.05 (< 0.05), 0.065 (0.096), and 0.15 (0.26) mg/kg; liver < 0.05 (< 0.05), < 0.05 (< 0.05), and 0.053 (0.059) mg/kg; and muscle < 0.01 (< 0.010), < 0.01 (0.01), and < 0.01 (< 0.01) mg/kg.

Laying hens

The Meeting did not receive a feeding study for poultry. In the metabolism study conducted with laying hens, daily dosing at a rate of ca. 190 ppm resulted in combined residues of spiromesifen and Sp-enol of 0.018 mg/kg in egg (may be less than plateau level), 0.049 mg/kg in fat, 0.3 mg/kg in liver, 0.028 mg/kg in muscle, and 0.07 mg/kg in skin.

Animal commodities maximum residue levels

For MRL estimation in animal commodities, the residue definition is the combined residues of spiromesifen and spiromesifen-enol, expressed as spiromesifen

Estimated residues in tissues and milk at the dietary burden summarized above are shown in the table below.

Spiromesifen feeding study	Feed level (ppm) for milk residues	Residues (mg/kg) in milk	Feed level (ppm) for tissue residues	Residues (mg/kg)			
				Muscle	Liver	Kidney	Fat
MRL beef or dairy cattle							

Spiromesifen feeding study	Feed level (ppm) for milk residues	Residues (mg/kg) in milk	Feed level (ppm) for tissue residues	Residues (mg/kg)			
Feeding study [a]	50	0.012	15	< 0.01	< 0.05	0.096	0.045
			50	< 0.01	0.059	0.26	0.12
Dietary burden and high residue	40	0.0096	40	< 0.01	0.056	0.21	0.099
STMR beef or dairy cattle							
Feeding study [b]	50	0.012	5	< 0.01	< 0.05	< 0.05	< 0.01
			15	< 0.01	< 0.05	0.065	0.03
Dietary burden and residue estimate	8.6	0.0021	8.6	< 0.01	< 0.05	0.055	0.017

[a] Highest residues for tissues and mean residues for milk

[b] Mean residues for tissues and mean residues for milk

The Meeting estimated the following maximum residue levels: Milks = 0.015 mg/kg; mammalian fats except milk fats = 0.15 mg/kg; meat (from mammals other than marine mammals) = 0.15 (F) mg/kg; and edible offal (mammalian) 0.3 mg/kg.

The Meeting estimated the following STMR levels: Milks = 0.0021 mg/kg; mammalian fats except milk fats = 0.017 mg/kg; meat (from mammals other than marine mammals) = 0.017 (F) mg/kg, 0.01 mg/kg (meat); and edible offal (mammalian) 0.055 mg/kg.

For poultry, a comparison of the feeding level in the metabolism study (190 ppm) with the maximum dietary burden (5.3 ppm) indicates that residues of spiromesifen plus Sp-enol would not be expected to exceed 0.0073 mg/kg in any poultry commodity. For poultry meat, egg, and fat, the Meeting estimated maximum residue levels of 0.02 mg/kg and STMRs of 0.01 mg/kg. For poultry edible offal, the Meeting estimated a maximum residue level of 0.05* mg/kg, and STMR and highest residue of 0.05 mg/kg, each.

RECOMMENDATIONS

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

Definition of the residue for plant and animal commodities (for compliance with the MRL): *sum of spiromesifen and 4-hydroxy-3-(2,4,6-trimethylphenyl)-1-oxaspiro[4.4]non-3-en-2-one, expressed as spiromesifen.*

Definition of the residue for plant commodities (for dietary risk assessment): *sum of spiromesifen, 4-hydroxy-3-(2,4,6-trimethylphenyl)-1-oxaspiro[4.4]non-3-en-2-one, and 4-hydroxy-3-[4-(hydroxymethyl)-2,6-dimethylphenyl]-1-oxaspiro[4.4]non-3-en-2-one (free and conjugated), all expressed as spiromesifen.*

Definition of the residue for livestock commodities (for dietary risk assessment): *sum of spiromesifen and 4-hydroxy-3-(2,4,6-trimethylphenyl)-1-oxaspiro[4.4]non-3-en-2-one, expressed as spiromesifen.*

The residue is fat-soluble.

		MRL, mg/kg			
CCN	Crop/Commodity	New	Previous	STMR	Highest residue
FB 2009	Low-growing berries	3		0.52	–
VB 0040	Brassica (cole or cabbage) vegetables, head cabbages, flowerhead Brassicas	3		0.21	1.8
VC 0045	Fruiting vegetables, cucurbits, except melon and cucumber	0.09		0.021	–
VC 0046	Melon, except watermelon	0.3		0.075	–
VC 0424	Cucumbers	0.15		0.05	–
VO 0051	Peppers	0.5		0.097	–
VO 0440	Eggplants	0.7		0.165	–

		MRL, mg/kg			
CCN	Crop/Commodity	New	Previous	STMR	Highest residue
VO 0442	Okra	0.5		0.097	–
VO 0443	Pepino	0.5		0.097	–
VO 0447	Sweet corn (corn-on-the-cob)	0.02*		0	–
VO 0448	Tomato	0.7		0.165	–
VL 0053	Leafy vegetables	15		2.06	–
VL 0054	Brassica leafy vegetables	15		2.06	12.5
VP 0526	Beans (*Phaseolus spp.*) (pods and/or immature seeds)	1		0.085	–
VR 0463	Cassava	0.02*		0.01	0.01
VR 0508	Sweet potato	0.02*		0.01	–
VR 0589	Potato	0.02*		0.01	0.01
GC 0645	Maize	0.02*		0	–
GC 0656	Popcorn	0.02*		0	–
SO 0691	Cotton seed	0.7		0.11	–
SB 0716	Coffee beans	0.2		0.02	
DT 1114	Tea, Green, Black (black, fermented and dried)	70		18.5	
AS 0645	Maize fodder (dry)	6		0.96 (as rec'd)	4.1 (as rec'd)
VW 0448	Tomato paste	2		0.43	–
DV 0448	Tomato, dried	4		0.82	–
HS 0444	Peppers chilli, dried	5		0.55	–
MO 0105	Edible offal (Mammalian)	0.3		0.055	–
MF 0100	Mammalian fats (except milk fats)	0.15		0.017	–
MM 0095	Meat (from mammals other than marine mammals)	0.15 (F)		0.01 (muscle) 0.017 (fat)	–
ML 0106	Milks	0.015		0.0021	–
PE 0112	Eggs	0.02		0.01	–
PF 0111	Poultry fats	0.02		0.01	–
PM 0110	Poultry meat	0.02		0.01	–
PO 0111	Poultry, edible offal of	0.05		0.05	0.05

For dietary burden/intake only

		MRL, mg/kg			
CCN	Crop/Commodity	New	Previous	Median	Highest residue
	Tomato (puree)			0.20	
	Tomato (wet pomace)			1.2	
	Maize forage			2	4.4
	Cotton gin trash			3.65	12
	Cotton seed (hull)			0.04	
	Cotton seed (meal)			0.02	
	Tea (green and black infusion)			0.63	

DIETARY RISK ASSESSMENT

Long-term intake

The International Estimated Daily Intakes (IEDIs) of spiromesifen were calculated for the 17 GEMS/Food cluster diets using STMRs/STMR-Ps estimated by the current Meeting. The ADI is 0–0.03 mg/kg bw and the calculated IEDIs were 2–20% of the maximum ADI (0.03 mg/kg bw). The Meeting concluded that the long-term intakes of residues of spiromesifen, resulting from the uses considered by the current JMPR, are unlikely to present a public health concern.

Short-term intake

The Meeting determined that an ARfD is not necessary for spiromesifen. The Meeting therefore concluded that the short-term intake of residues of spiromesifen resulting from uses that have been considered by the JMPR is unlikely to present a public health concern.

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