OXATHIAPIPROLIN (291)

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EXPLANATION

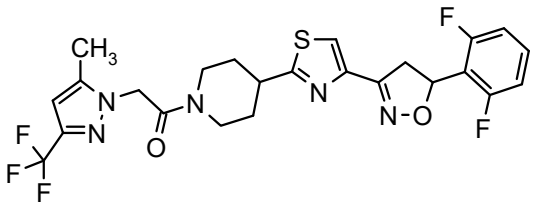
Oxathiapiprolin is systemic piperidinyl thiazole isoxazoline fungicide effective against oomycete pathogens, acting by inhibiting mycelial growth and zoospore release, encystment and mobility. It exhibits translaminar efficacy and gives systemic disease control following soil applications.

Authorisations exist for the use of oxathiapiprolin as foliar treatments or as soil treatments at planting (band/in-furrow or in transplant water) or via drip irrigation in North America, Canada, a number of countries in the Pacific, Asia, Central and South Americas and are pending in Europe.

Oxathiapiprolin was scheduled by the 47th Session of the CCPR as a new compound for consideration by the 2016 JMPR. Residue and analytical aspects were considered for the first time by the present meeting. The manufacturer submitted studies on metabolism, analytical methods, supervised field trials, processing, freezer storage stability and environmental fate in soil.

In this evaluation, the values presented in the tables are as reported in the various studies, but in the accompanying text, they have generally been rounded to two significant digits.

IDENTITY

ISO common name:	oxathiapiprolin
Code number	DPX-QGU42
IUPAC name:	1-(4-{4-[(5RS)-5-(2,6-difluorophenyl)-4,5-dihydro-1,2-oxazol-3-yl]-1,3-thiazol-2-yl}-1-piperidyl)-2-[5-methyl-3-(trifluoromethyl)-1H-pyrazol-1-yl]ethanone
Chemical Abstracts name:	1-[4-[4-[5-(2,6-Difluorophenyl)-4,5-dihydro-3-isoxazolyl]-2-thiazolyl]-1-piperidinyl]-2-[5-methyl-3-(trifluoromethyl)-1H-pyrazol-1-yl]-ethanone
CAS number	1003318-67-9
Molecular mass:	539.53
Molecular formula	$C_{24}H_{22}F_5N_5O_2S$
Structural formula:	

Oxathiapiprolin consists of a pair of enantiomers, both of which are fungicidally active. The technical active ingredient is manufactured under non-stereospecific conditions giving a 1:1 racemic mixture of (R)-oxathiapiprolin (IN-Q7N24) and (S)-oxathiapiprolin (IN-Q7N25) enantiomers.

Physical and chemical properties

Pure active ingredient

A detailed chemical and physical characterisation of the active ingredient is given in Table 1.

Table 1 Physical and chemical data of oxathiapiprolin (active ingredient and technical grade material)

Test or Study & Annex point	Test material purity and specification	Findings and comments	Reference
Melting point	Pure ai (98.9%) Technical (95.8%)	146.4 °C 138.7 °C	DP-32686 DP-32687
Boiling point	Pure ai (98.9%) Technical (95.8%)	Not measured- decomposition at *ca.* 290 °C Not measured- decomposition at *ca.* 283 °C	DP-32686 DP-32687
Relative density	Pure ai (98.9%) Technical (95.8%)	1.4645 (20 °C) 1.4684 (20 °C)	DP-32487 DP-32475
Vapour pressure	Pure ai (98.9%)	1.141×10^{-6} Pa at 20 °C (extrapolated) 1.405×10^{-6} Pa at 25 °C 2.359×10^{-6} Pa at 35 °C 3.28×10^{-6} Pa at 45 °C	DP-31751
pH	Pure ai (98.9%) Technical (95.8%)	6.24 (1 min), 6.37 (10 min) in 1% aqueous suspension 5.04 (1 min), 5.41 (10 min) in 1% aqueous suspension	DP-32487 DP-32475
Henry's law constant	calculated	K_H = 3.52 Pa m³/mol (20 °C)	DP-34110
Appearance	Pure ai (98.9%) Technical (95.8%)	Off-white odourless crystalline solid Off-white odourless crystalline solid	DP-32487 DP-32475
Solubility in water (20 °C) (mg/L)	Pure ai (98.9%)	0.1749 mg/L in distilled water 0.2111 mg/L in pH 4 buffer 0.1844 mg/L in pH 7 buffer 0.206 mg/L in pH 9 buffer	DP-29277
	Metabolite IN-E8S72 (mg/L) IN-RAB06 (mg/L) IN-QPS10 (mg/L) IN-RTD31 (mg/L)	Dist. Water / pH 4 / pH 7 / pH 9 28240 / 31860 / 27670 / 27100 32 / 50 / 2237 / 3738 804 1.04	 DP-37866 DP-37865 DP-37863 DP-37864
Solubility in organic solvents (g/L, 20 °C)	Pure ai (98.9) Technical (95.8%)	Solvent / Pure ai / Technical Dichloromethane: 347 / 353 Acetone: 147 / 163 Acetonitrile: 111 / 130 Ethyl acetate: 31.7 / 33.9 Methanol: 13 / 13.5 o-Xylene: 5.7 / 5.8 n-Octanol: 0.04 / 0.03 Hexane: 0.01 / 0.01	DP-38201 DP-32486
Octanol/water partition coefficient (20 °C)	Pure ai (98.9%)	Oxathiapiprolin Log P_{OW} 3.62-3.67 at pH 4-9 IN-SXS67 Log P_{OW} -1.71 (mean) IN-E8S72 Log P_{OW} -0.52 (mean) IN-Q7H09 Log P_{OW} 2.86 (mean IN-RDG40 Log P_{OW} 2.73 (mean) IN-RAB06 Log P_{OW} -0.05 (mean) IN-QPS10 Log P_{OW} -2.02 (mean) IN-RTD31 Log P_{OW} 2.95 (mean) IN-Q7D41 Log P_{OW} 4.31 (mean)	DP-29274 DP-33852
Hydrolysis (sterile buffer in the dark)	^{14}C labelled pure ai (>99%)	Stable to hydrolysis at pH 4-9. DT_{50} >1 year	DP-28424
Photolysis characteristics	^{14}C labelled pure ai (> 99%)	DT_{50} (continuous light, sterile buffer, pH 7, 25 °C): 15.4 days DT_{50} (continuous light, natural water, 25 °C): 20.2 days Quantum yield: $\Phi = 3.179 \times 10^{-6}$ molecules degraded/photon (pH 7)	DP020874

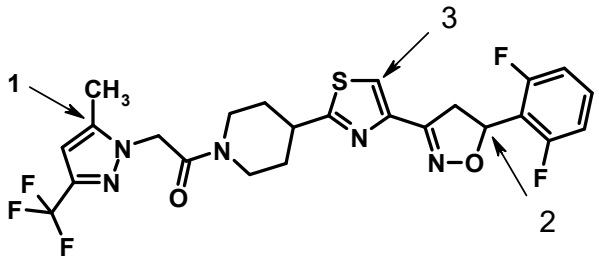
Formulations

Formulations of oxathiapiprolin are available for use as foliar applications, in-furrow/banded soil applications or applied in transplant water or through drip irrigation systems.

Formulation type	Oxathiapiprolin content	
OD (Oil dispersion)	100 g/kg	Foliar or soil applications
SC (Suspension concentrate)	200 g/kg	Soil applications

METABOLISM AND ENVIRONMENTAL FATE

The Meeting received oxathiapiprolin metabolism studies on plants (potato, grape, lettuce, courgette), animals (rats, lactating goats, laying hens), soil and rotational crops (lettuce, turnip and wheat).

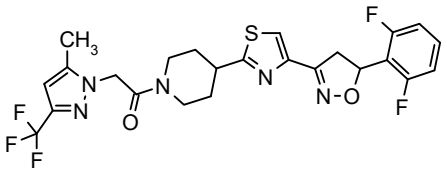
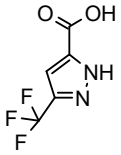
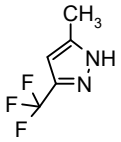
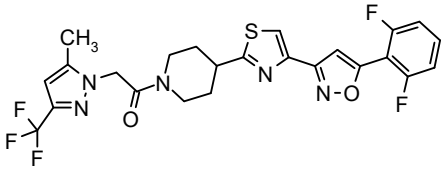
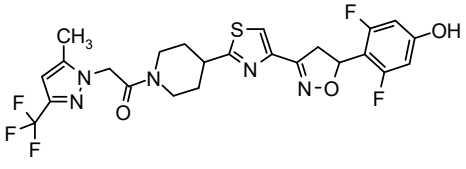


1 [Pyrazole-^{14}C]-oxathiapiprolin
2 [Isoxazoline-^{14}C]-oxathiapiprolin
3 [Thiazole-^{14}C]-oxathiapiprolin

Figure 1 Oxathiapiprolin radiolabelled in the 5 position on the pyrazole, thiazole or the isoxazoline rings were used in these studies. The label positions (*) are shown above

Major metabolites identified in these studies and discussed in this evaluation are listed below.

Table 2 Major oxathiapiprolin metabolites identified in plant, animal and soil matrices

Compound/Code	Name and Matrix	Structure
Oxathiapiprolin (DPX-QGU42)	1-[4-[4-[5-(2,6-Difluorophenyl)-4,5-dihydro-3-isoxazolyl]-2-thiazolyl]-1-piperidinyl]-2-[5-methyl-3-(trifluoromethyl)-1*H*-pyrazol-1-yl]-ethanone MW: 539 Rat, goat, hen, plants, soil	
IN-E8S72	5-(Trifluoromethyl)-1*H*-pyrazole-3-carboxylic acid MW: 180 Rat, plants, goat, soil	
IN-KJ552	3-Methyl-5-(trifluoromethyl)-1*H*-pyrazole MW: 150 Rat, plants	
IN-Q7D41	1-[4-[4-[5-(2,6-Difluorophenyl)-3-isoxazolyl]-2-thiazolyl]-1-piperidinyl]-2-[5-methyl-3-(trifluoromethyl)-1*H*-pyrazol-1-yl]ethanone MW: 537 Rat (transitory), goat, hen, soil, plants	
IN-Q7H09	1-[4-[4-[5-(2,6-Difluoro-4-hydroxyphenyl)-4,5-dihydro-3-isoxazolyl]-2-thiazolyl]-1-piperidinyl]-2-[5-methyl-3-(trifluoromethyl)-1*H*-pyrazol-1-yl]ethanone MW: 556 Rat, plants, hen, goat	

Compound/Code	Name and Matrix	Structure
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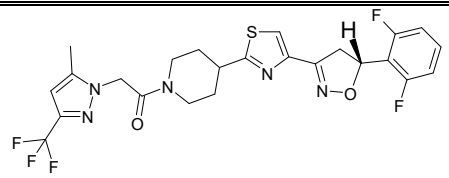
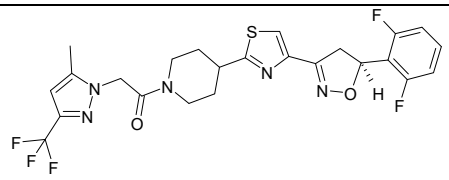
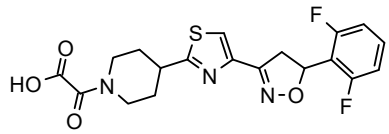
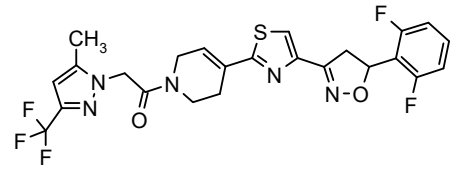
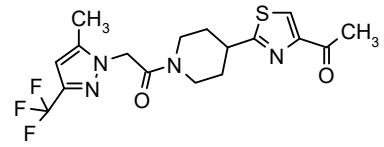
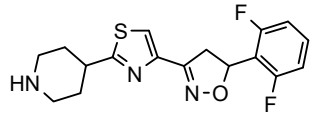
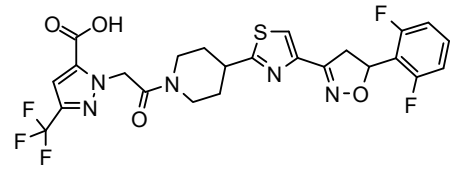
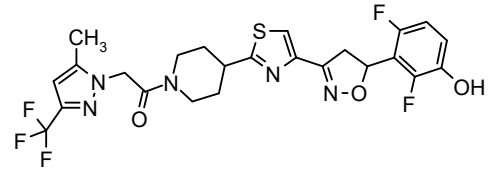
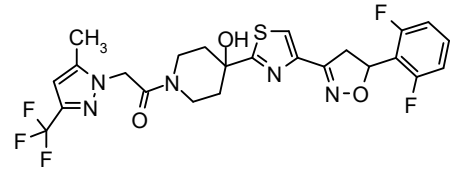
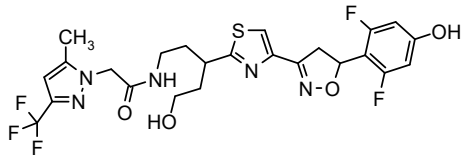
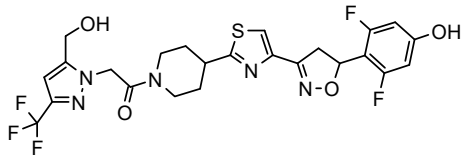
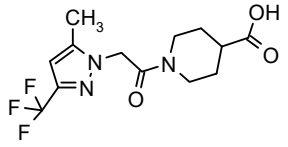
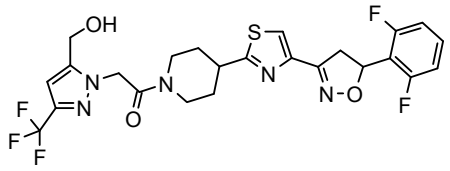
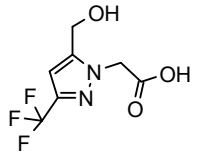
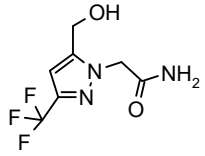
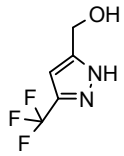
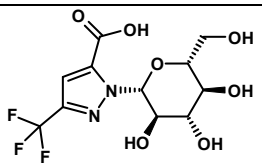
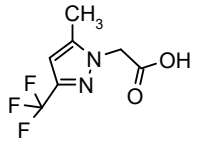
IN-Q7N24 ((*R*)-oxathiapiprolin)	1-[4-[4-[(5R)-5-(2,6-Difluorophenyl)-4,5-dihydro-3-isoxazolyl]-2-thiazolyl]-1-piperidinyl]-2-[5-methyl-3-(trifluoromethyl)-1*H*-pyrazol-1-yl]-ethanone MW: 539 Rat, goat, hen, plants, soil	
IN-Q7N25 ((*S*)-oxathiapiprolin)	1-[4-[4-[(5S)-5-(2,6-Difluorophenyl)-4,5-dihydro-3-isoxazolyl]-2-thiazolyl]-1-piperidinyl]-2-[5-methyl-3-(trifluoromethyl)-1*H*-pyrazol-1-yl]-ethanone MW: 539 Rat, goat, hen, plants, soil	
IN-Q9L80	4-[4-[5-(2,6-Difluorophenyl)-4,5-dihydro-3-isoxazolyl)-2-thiazolyl]-α-oxo-1-piperidineactetic acid MW: 421 Rat, plants	
IN-Q9R70	1-[4-[4-[5-(2,6-Difluorophenyl)-4,5-dihydro-3-isoxazolyl]-2-thiazolyl]-3,6-dihydro-1(2*H*)-pyridinyl]-2-[5-methyl-3-(trifluoromethyl)-1*H*-pyrazol-1-y1]ethanone MW: 537 Rat (IN-Q7D41 isomer), plants	
IN-QFD61	1-[4-(4-Acetyl-2-thiazolyl)-1-piperindyl]-2-[5-methyl-3-(trifluoromethyl)-1*H*-pyrazol-1-yl]ethanone MW: 400 Goat, hen	
IN-QPS10	4-[4-[5-(2,6-Difluorophenyl)-4,5-dihydro-3-isoxazolyl)-2-thiazolyl]piperidine MW: 349 Plants, soil	
IN-RAB06	1-[2-[4-[4-[5-(2,6-Difluorophenyl)-4,5-dihydro-3-isoxazolyl]-2-thiazolyl]-1-piperidinyl]-2-oxoethyl]-3-(trifluoromethyl)-1*H*-pyrazole-5-carboxylic acid MW: 569 Rat, goat, hen, soil	
IN-RDG40	1-[4-[4-[5-(2,6-Difluoro-3-hydroxyphenyl)-4,5-dihydro-3-isoxazolyl]-2-thiazolyl]-1-piperidinyl]-2-[5-methyl-3-(trifluoromethyl)-1*H*-pyrazol-1-yl]ethanone MW: 556 Rat, plants, hen, goat	
IN-RDT31	1-[4-[4-[5-(2,6-Difluorophenyl)-4,5-dihydro-3-isoxazolyl]-2-thiazolyl]-4-hydroxy-1-piperidinyl]-2-[5-methyl-3-(trifluoromethyl)-1*H*-pyrazol-1-yl]ethanone MW: 556 Rat, soil	

Compound/Code	Name and Matrix	Structure
IN-RLB26	N-[3-[4-[5-(2,6-Difluoro-4-hydroxyphenyl)-4,5-dihydro-3-isoxazolyl]-2-thiazolyl]-5-hydroxypentyl]-5-methyl-3-(trifluoromethyl)-1*H*-pyrazole-1-acetamide MW: 574 Rat, hen	
IN-RLB67	1-[4-[4-[5-(2,6-Difluoro-4-hydrophenyl)-4,5-dihydro-3-isoxazolyl]-2-thiazolyl]-1-piperidinyl]-2-[5-(hydroxymethyl)-3-(trifluoromethyl)-1*H*-pyrazol-1-yl]ethanone MW: 572 Rat, goat	
IN-RLD51	1-[2-[5-Methyl-3-(trifluoromethyl)-1*H*-pyrazol-1-yl]acetyl]- 4-piperidinecarboxylic acid MW: 319 Rat, soil, photolysis, hen	
IN-RPD37	1-[4-[4-[5-(2,6-Difluorophenyl)-4,5-dihydro-3-isoxazolyl]-2-thiazolyl]-1-piperidinyl]-2-[5-hydroxymethyl)-3-(trifluoromethyl)-1*H*-pyrazol-1-yl]ethanone MW: 556 Rat (transient), goat, hen	
IN-RZB20	5-(Hydroxymethyl)-3-(trifluoromethyl)-1*H*-pyrazole-1-acetic acid MW: 224 Rat, plants	
IN-RZB21	5-(Hydroxymethyl)-3-(trifluoromethyl)-1*H*-pyrazole-1-acetamide MW: 223 Plants	
IN-RZD74	3-(Trifluoromethyl)-1*H*-Pyrazole-5-methanol MW: 166 Plants, transient in goat, hen, rat	
IN-SXS67	1-ß-D-Glucopyranosyl-3-(-(trifluoromethyl)-1*H*-pyrazole-5-carboxylic acid MW: 342 Plants	
IN-WR791	5-Methyl-3-(trifluoromethyl)-1*H*-pyrazole-1-acetic acid MW: 208 Plants, goat, rat, soil	

Plant metabolism

The Meeting received plant metabolism studies on potato, grape and lettuce following foliar applications of [^{14}C]-oxathiapiprolin and on courgette, lettuce and potato grown in [^{14}C]-oxathiapiprolin-treated soil.

Grape – foliar applications

In a study reported by Chapleo and Hobbs, 2011 [Ref: DP-28070], outdoor grape vines grown in plastic containers were treated with three foliar applications of [pyrazole-^{14}C]-oxathiapiprolin or [thiazole^{14}C]-oxathiapiprolin (OD formulations) at a rate equivalent to 0.07 kg ai/ha/treatment to give a total application rate of 0.21 kg ai/ha. The applications were 14 days apart at flowering (BBCH 63–65), BBCH 73 (cereal grain sized berries), and BBCH 79 (pre-bunch-closure - adjacent berries on bunch beginning to touch).

Foliage samples were taken at immediately after each treatment, 14 days after treatments 2 and 3 and at grape maturity (76 DAT3). Berry (grape) samples were taken 14 days after the second treatment, immediately after the third treatment, 14 days later and at grape maturity.

Selected samples (14 DAT2 onwards) were surface rinsed in acetonitrile prior to further analysis and the surface residues determined. Samples were also extracted with acetonitrile and re-extracted twice with acetonitrile:water (3:1, v/v). Some samples were also subjected to further extractions using water, acetone, α-amylase, combined amyloglucosidase and cellulase, NaOH (0.1 N), and HCl (1.0 N). The 0 DAT1 samples were extracted in the same manner with methanol replacing acetonitrile. The total radioactive residues (TRR) in each sample were determined by summing radioactivity in the surface wash, extracts, and post extraction solids and were expressed as mg/kg equivalents of the parent oxathiapiprolin.

The majority of the radioactive residue in the berries and foliage was removed by surface washing and/or initial extraction (69–99% TRR). The total residue levels in berries increased from 0.25 and 0.34 mg eq/kg prior to the third application to 0.46 and 0.47 mg eq/kg after application (0DAT3) in the [thiazole-^{14}C] and [pyrazole-^{14}C] labels, respectively, and declined to 0.30–0.32 mg eq/kg at mature berries (76DAT3). The total residue levels measured in foliage at the point of fruit maturity (76DAT3) were 1.1 mg eq/kg (thiazole-label) and 1.4 mg eq/kg (pyrazole-label).

More extensive extractions were able to remove further 7–20% TRR from the [pyrazole-^{14}C] treated foliage samples, 3.4-7.2% TRR from the [thiazole-^{14}C] treated foliage samples, a further 4.1–4.8% TRR from the immature berries and a further 15.8% TRR (0.051 mg eq/kg) from the [thiazole-^{14}C] treated mature grapes.

Unextracted residues were about 10-16% TRR (0.14-0.18 mg eq/kg) in foliage, 6.4–7.5% TRR (0.03–0.04 mg eq/kg) in immature berries and about 15% TRR (0.048 mg eq/kg) in mature berries

Table 3 Total radioactive residues in grape berries and foliage following three foliar applications of 0.07 kg ai/ha [^{14}C]-oxathiapiprolin – a total of 0.21 kg ai/ha

Harvest and sample	Site of label	Surface wash		Extracted		Unextracted		Total
		% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	mg eq/kg
0DAT1 Foliage	Pyrazole	NA	NA	99.3	14.865	0.7	0.105	14.969
	Thiazole	NA	NA	98.8	15.243	1.3	0.201	15.428
0DAT2 Foliage	Pyrazole	NA	NA	98.8	32.064	1.2	0.389	32.453
	Thiazole	NA	NA	99.0	22.801	1.0	0.230	23.031
14DAT2 Foliage	Pyrazole	45.7	3.299	38.6	2.786	15.7	1.133	7.218
	Thiazole	74.2	12.108	20.3	3.312	5.5	0.897	16.318
0DAT3 Foliage	Pyrazole	81.7	30.667	14.7	5.518	3.6	1.351	37.536
	Thiazole	87.5	25.040	10.4	2.976	2.1	0.601	28.617
14DAT3 Foliage	Pyrazole	51.2	5.586	37.9	4.135	10.9 (3.9)[a]	1.189 (0.426)[a]	10.911
	Thiazole	70.5	6.027	22.5	1.924	7.0 (3.6)[a]	0.598 (0.308)[a]	8.549
76DAT3 Foliage	Pyrazole	24.2	0.334	45.6	0.630	30.2 (10.3)[a]	0.417 (0.142)[a]	1.381
	Thiazole	35.9	0.401	41.0	0.458	23.1 (16)[a]	0.258 (0.179)[a]	1.116
14DAT2 Berries	Pyrazole	NA	NA	NA	NA	NA	NA	0.340
	Thiazole	NA	NA	NA	NA	NA	NA	0.248

Harvest and sample	Site of label	Surface wash		Extracted		Unextracted		Total
		% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	mg eq/kg
0DAT3 Berries	Pyrazole	NA	NA	NA	NA	NA	NA	0.468
	Thiazole	NA	NA	NA	NA	NA	NA	0.463
14DAT3 Berries	Pyrazole	10.5	0.048	78.3	0.361	11.2 (6.4)[a]	0.052 (0.03)[a]	0.461
	Thiazole	30.0	0.164	58.4	0.319	11.6 (7.5)[a]	0.063 (0.041)[a]	0.545
76DAT3 Berries	Pyrazole	3.9	0.012	86.9	0.264	9.2	0.028	0.304
	Thiazole	15.4	0.049	53.8	0.172	30.8 (15.1)[a]	0.098 (0.048)[a]	0.318

[a] After further extraction with water (overnight, ambient), acetone (*ca.* 40 °C, 2 × 30 min, ultrasonication), α-amylase in phosphate buffer (*ca.* 50 °C, pH 7.2, 2 × 72 h), amyloglucosidase plus cellulase in acetate buffer (*ca.* 50 °C, pH 5.0, 2 × 48 h), 0.1 N sodium hydroxide (*ca*. 60 °C, 2 × 6 h), and 1.0 N hydrochloric acid (*ca.* 60 °C, 2 × 6 h)

Extracts containing significant radioactivity (> 0.01 mg eq/kg) were analysed by high performance liquid chromatography (HPLC) and identification of the principal ^{14}C-residues was accomplished by HPLC using liquid chromatography mass spectrometry (LC-MS), with reference to authenticated reference standards.

Oxathiapiprolin was the major component identified in immature berries (36–74% TRR, 0.165–0.41 mg/kg) and accounted for 41% TRR (0.131 mg/kg) at maturity in [thiazole-^{14}C]-treated berries and 10% TRR (0.03 mg/kg) in the [pyrazole-^{14}C]-treated berries.

In [pyrazole-^{14}C]-treated berries at maturity, the polar pyrazole-specific metabolites IN-WR791 (19% TRR, 0.06 mg eq/kg) and IN-E8S72 (14% TRR, 0.04 mg eq/kg) were predominant residues in fruit.

Minor metabolites detected in berries included IN-RDG40, IN-RAB06, IN-Q7H09, IN-KJ552, IN-Q7D41, IN-QPS10, IN-Q9R70, IN-Q9L80, IN-SXS67, IN-RZB20 and up to 23 unidentified metabolites. None of these were found at levels above 6.2% TRR.

Oxathiapiprolin was the predominant component identified in all foliage samples from both labels with initial levels of 14-15 mg eq/kg (92-98% TRR) immediately after the first application declining to 32–60% TRR (0.441–0.672 mg eq/kg) at fruit maturity. Metabolites detected in foliage included IN-E8S72, IN-RDG40, IN-WR791, IN-RAB06, IN-Q7H09, IN-KJ552, IN-Q7D41, IN-QPS10, IN-Q9L80, IN-SXS67, and IN-RZB20 and up to 44 unidentified metabolites. None of these were found at levels above 6.3% TRR (1.5% TRR in the [thiazole-^{14}C]-treated leaves.

Table 4 Characterisation and identification of residues in grape foliage following three foliar applications of 0.07 kg ai/ha [^{14}C]-oxathiapiprolin

Radioactive residues in grape foliage								
	0DAT1		14DAT2		14DAT3		76DAT3	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
[Pyrazole-14C]-label								
Identified components								
Oxathiapiprolin	98.4	14.731	63.5	4.576	66.3	7.242	32.0	0.441
IN-RAB06	ND	ND	< 0.1	0.003	1.1	0.118	ND	ND
IN-RDG40	ND	ND	0.8	0.056	1.7	0.190	1.9	0.025
IN-Q7H09	ND	ND	5.0	0.360	1.6	0.178	1.0	0.013
IN-Q7D41	ND	ND	0.6	0.045	2.1	0.228	0.4	0.006
IN-E8S72	ND	ND	ND	ND	ND	ND	2.3	0.032
IN-WR791	ND	ND	1.0	0.072	0.4	0.045	1.0	0.014
IN-KJ552	ND	ND	0.1	0.006	0.6	0.064	0.9	0.012
IN-SXS67	ND	ND	ND	ND	ND	ND	4.2	0.058
IN-RZB20	ND	ND	ND	ND	ND	ND	6.3	0.087
Unidentified metabolites								
Number	2	-	44	-	37	-	24	-
Highest	0.5	0.074	4.0	0.290	3.4	0.373	2.8	0.038

Radioactive residues in grape foliage								
	0DAT1		14DAT2		14DAT3		76DAT3	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Total	0.9	0.133	12.0	0.904	21.6	2.359	25.0	0.343
Unextracted	0.7	0.105	15.7	1.133	3.9	0.426	10.3	0.142
[Thiazole-14C]-label								
Identified components								
Oxathiapiprolin	92.1	14.206	92.0	15.015	82.0	7.013	60.1	0.672
IN-Q9L80	ND	ND	ND	ND	0.3	0.028	ND	ND
IN-QPS10	ND	ND	ND	ND	0.2	0.015	1.1	0.012
IN-RAB06	0.2	0.030	ND	ND	0.5	0.045	2.3	0.025
IN-RDG40	0.5	0.076	ND	ND	0.7	0.060	1.5	0.017
IN-Q7H09	ND	ND	ND	ND	0.2	0.015	0.9	0.011
IN-Q7D41	0.3	0.046	0.1	0.024	0.3	0.026	0.2	0.002
Unidentified metabolites								
Number	12	-	8	-	23	-	32	-
Highest	1.4	0.213	1.2	0.195	1.3	0.112	1.7	0.018
Total	5.6	0.853	2.1	0.365	13.0	1.086	12.4	0.136
Unextracted	1.3	0.201	5.5	0.897	3.6	0.308	16.0	0.179

Table 5 Characterisation and identification of residues in grape berries following three foliar applications of 0.07 kg ai/ha [^{14}C]-oxathiapiprolin

Berries	[Pyrazole-^{14}C]-label				[Thiazole-^{14}C]-label			
	14DAT3		76DAT3		14DAT3		76DAT3	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Identified components								
Oxathiapiprolin	35.9	0.165	9.9	0.030	74.2	0.406	41.0	0.131
IN-RAB06	1.6	0.007	ND	ND	0.5	0.003	ND	ND
IN-RDG40	0.2	0.001	0.5	0.002	2.9	0.016	0.1	< 0.001
IN-Q7H09	1.5	0.007	ND	ND	0.6	0.003	0.2	0.001
IN-Q7D41	0.1	0.001	ND	ND	ND	ND	0.1	< 0.001
IN-E8S72	13.3	0.062	14.4	0.044	NA	NA	NA	NA
IN-WR791	15.1	0.069	18.6	0.057	NA	NA	NA	NA
IN-KJ552	1.1	0.005	ND	ND	NA	NA	NA	NA
IN-SXS67	ND	ND	4.2	0.013	NA	NA	NA	NA
IN-RZB20	ND	ND	6.2	0.019	NA	NA	NA	NA
IN-Q9L80	ND	ND	ND	ND	0.8	0.004	ND	ND
IN-QPS10	NA	NA	NA	NA	0.3	0.001	ND	ND
IN-Q9R70	ND	ND	ND	ND	ND	ND	0.3	0.001
Unidentified metabolites								
Number	23	-	19	-	17	-	7	-
Highest	6.0	0.028	3.5	0.011	1.7	0.009	6.2	0.020
Total	24.0	0.111	33.2	0.099	10.1	0.055	20.3	0.063
Unextracted	6.4	0.030	9.2	0.028	7.5	0.041	15.1	0.048

The ratio of oxathiapiprolin enantiomers was assessed by chromatographic analysis of the peak assigned to oxathiapiprolin using a chiral HPLC method developed to separate the two enantiomers (IN-Q7N24 and IN-Q7N25). The non-radiolabeled oxathiapiprolin reference standard, each radiolabeled form of oxathiapiprolin, and an isolate of the aqueous acetonitrile berry extract (14DAT3, [pyrazole-^{14}C]-label) were analysed, in each case confirming that a 1:1 enantiomeric ratio and an overall net retention of stereochemistry was maintained throughout the study.

The major metabolic pathways in grape vines were primarily through hydroxylation of the phenyl ring (forming IN-Q7H09 and IN-RDG40) and the isoxazoline ring (leading to the formation of IN-Q7D41), together with cleavage between the piperidine and pyrazole rings to form IN-Q9L80 and IN-QPS10 (thiazole) and the more polar (pyrazole) metabolites IN-KJ552, IN-RZB20, IN-WR791 and IN-E8S72 (and its glucose conjugate IN-SXS67).

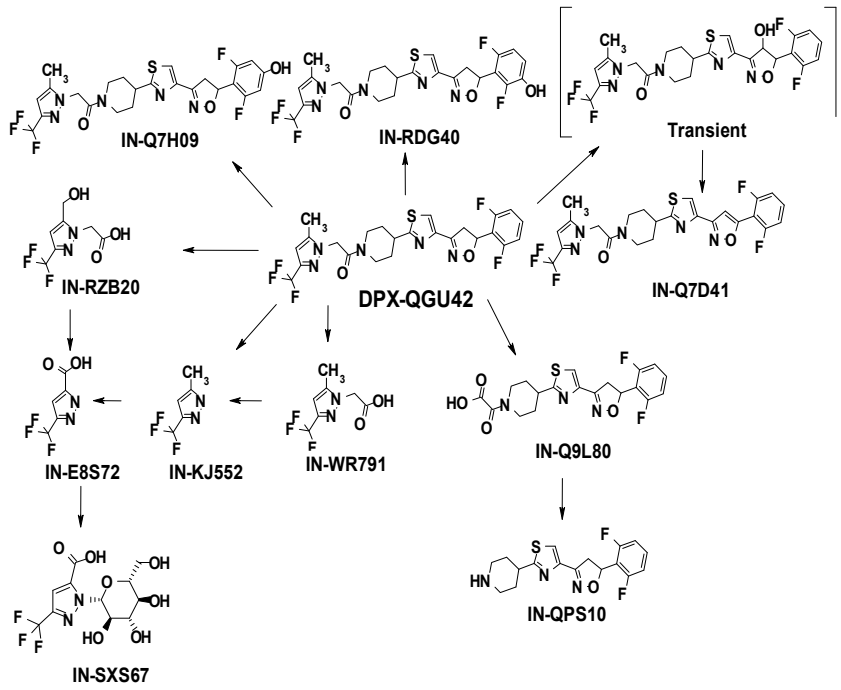


Figure 2 Proposed metabolic pathways for oxathiapiprolin (DPX-QGU42) in grape vines

Lettuce – foliar applications

In a study reported by Doig, 2011 [Ref: DP-28069], outdoor lettuce plants were treated with three foliar applications of 0.07 kg ai/ha [pyrazole-^{14}C]-oxathiapiprolin or [thiazole-^{14}C]-oxathiapiprolin (OD formulations) at 10 day intervals from BBCH 15 (5-leaf stage) to BBCH 19 (9-leaf stage). Lettuce foliage was sampled immediately after each application, 10 days after the first and second applications and 3, 7 and 14 days after the last application.

Selected samples were rinsed with acetonitrile to measure surface residues and all samples were homogenised and extracted twice with acetonitrile and reextracted twice with acetonitrile:water (3:1, v/v). Levels of radioactivity were determined in each extract by LSC and in post extraction solid samples by oxidative combustion followed by LSC.

Further sequential extractions were conducted (on selected samples of PES) with water, acetone (ca. 40 °C, with ultrasonication), α-amylase in phosphate buffer (ca. 50 °C, pH 7.0), amyloglucosidase plus cellulase in acetate buffer (ca. 50 °C, pH 5.0), 0.1 N sodium hydroxide (ca. 60 °C), and 1.0 N hydrochloric acid (ca. 60 °C).

Extracts containing significant radioactivity (> 0.01 mg eq/kg) were analysed by high performance liquid chromatography (HPLC) and identification of the principal ^{14}C-residues was accomplished by HPLC using liquid chromatography mass spectrometry (LC-MS), with reference to authenticated reference standards (oxathiapiprolin, IN-Q7D41, IN-Q7D13, and IN-Q7H09).

The majority of the radioactive residue in the foliage was removed by surface washing and/or initial extraction (83–99.7% TRR). The total radioactive residues were shown to decline post application with levels of 0.52 and 0.47 mg eq/kg in the mature (14 DAT3) [pyrazole-^{14}C] and [thiazole-^{14}C] samples, respectively.

The acetonitrile surface wash in samples taken immediately after the last application contained 70-80% TRR (3.2–3.7 mg eq/kg) and were 18–23% TRR (0.09–0.12 mg eq/kg) in the surface wash from the samples taken 14 days later. Unextracted residues in samples taken 7–14 days after treatment ranged from 7–14% TRR in the [pyrazole-^{14C}]-treated plants and from 14–17% TRR in the [thiazole-^{14C}]-treated plants.

Table 6 Total radioactive residues in lettuce following three foliar applications of 0.07 kg ai/ha [^{14}C]-oxathiapiprolin – a total of 0.21 kg ai/ha

Sampling interval	Site of label	Surface wash		Extracted		Unextracted		Total	Oxathiapiprolin	
		%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	mg eq/kg	%TRR	mg eq/kg
0DAT1	Pyrazole			99.7	5.376	0.4	0.022	5.392	89.8	4.842
	Thiazole			99.7	11.252	0.4	0.045	11.286	97.6	11.015
10DAT1	Pyrazole			88.2	0.635	11.8	0.085	0.719	64.6	0.464
	Thiazole			89.9	0.466	10.1	0.052	0.518	79.1	0.41
0DAT2	Pyrazole			97.6	5.381	2.4	0.132	5.514	82.9	4.571
	Thiazole			98.4	5.688	1.6	0.092	5.780	96.7	5.589
10DAT2	Pyrazole	14.1	0.069	71.9	0.351	14.0	0.068	0.488	48.9	0.239
	Thiazole	24.9	0.231	61.3	0.568	13.7	0.127	0.927	61.9	0.573
0DAT3	Pyrazole	80.1	3.788	18.5	0.875	1.3	0.061	4.729	97.3	4.601
	Thiazole	69.3	3.176	28.2	1.293	2.5	0.115	4.583	92.2	4.225
3DAT3	Pyrazole	49.2	0.626	46.4	0.590	4.3	0.055	1.272	78.8	1.002
	Thiazole	46.8	1.229	48.1	1.263	5.1	0.134	2.627	85.1	2.235
7DAT3	Pyrazole	33.6	0.21	58.7	0.367	7.7	0.048	0.626	77.3	0.484
	Thiazole	19.7	0.132	66.4	0.444	13.9	0.093	0.669	75.1	0.503
14DAT3	Pyrazole	22.5	0.117	65.4	0.341	12.1	0.063	0.520	64.9	0.337
	Thiazole	18.2	0.086	64.5	0.305	17.2	0.081	0.473	56.9	0.269

Oxathiapiprolin was the major residue in both the immature and mature lettuce foliage, making up 79% TRR (1.0 mg/kg) and 65% TRR (0.34 mg/kg) respectively in the [pyrazole-^{14}C]-treated plants and 85% TRR (2.24 mg/kg) and 57% TRR (0.27 mg/kg) respectively in the [thiazole-^{14}C]-treated plants.

Metabolites detected in lettuce foliage included IN-Q7H09, IN-Q7D41 and numerous other unidentified metabolites including multiple hydroxylated oxathiapiprolin compounds, all present individually at ≤ 6% TRR.

Table 7 Characterisation and identification of residues in lettuce foliage following three foliar applications of 0.07 kg ai/ha [^{14}C]-oxathiapiprolin – a total of 0.21 kg ai/ha

	[Pyrazole-^{14}C]-label						[Thiazole-^{14}C]-label					
	3DAT3		7DAT3		14DAT3		3DAT3		7DAT3		14DAT3	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Identified components												
Oxathiapiprolin	78.8	1.002	77.3	0.484	64.9	0.337	85.1	2.235	75.1	0.503	56.9	0.269
IN-Q7H09	1.2	0.015	5.1	0.032	1.0	0.005	0.6	0.016	ND	ND	1.2	0.006
IN-Q7D13	ND	ND	ND	ND	ND	ND	0.4	0.011	ND	ND	ND	ND
IN-Q7D41	0.9	0.011	0.8	0.005	1.0	0.005	0.2	0.005	ND	ND	1.1	0.005
Multiple hydroxylated components	3.4	0.043	ND	ND	4.2	0.022	2.7	0.071	6.3	0.042	5.7	0.027
Unidentified metabolites												
Number	11		8		7		3		7		18	
Highest	1.1	0.014	1.5	0.009	3.0	0.016	1.0	0.026	1.3	0.009	2.0	0.009
Total	5.5	0.071	7.1	0.044	11.8	0.061	1.4	0.037	6.4	0.043	13.3	0.061
Unextracted	4.3	0.055	7.7	0.048	12.1	0.063	5.1	0.134	13.9	0.093	17.2	0.081

The ratio of oxathiapiprolin enantiomers was assessed by chromatographic analysis of the peak assigned to oxathiapiprolin using a chiral HPLC method developed to separate the enantiomers (IN-Q7N24 and IN-Q7N25). The non-radiolabeled oxathiapiprolin reference standard, each radiolabeled form of oxathiapiprolin used for the applications to lettuce, and an isolate from the

mature lettuce [pyrazole-^{14}C]-label foliage extract sample were analysed and determined that the enantiomeric ratio (1:1) of the test substance remained unchanged, suggesting that the metabolism of oxathiapiprolin in lettuce was not stereoselective.

The primary metabolic pathway of oxathiapiprolin in lettuce following foliar treatment involves hydroxylation of the phenyl ring (forming IN-Q7H09) and the isoxazoline ring (leading to the formation of IN-Q7D41), generally consistent with those of other plants and animals. No cleavage products were identified.

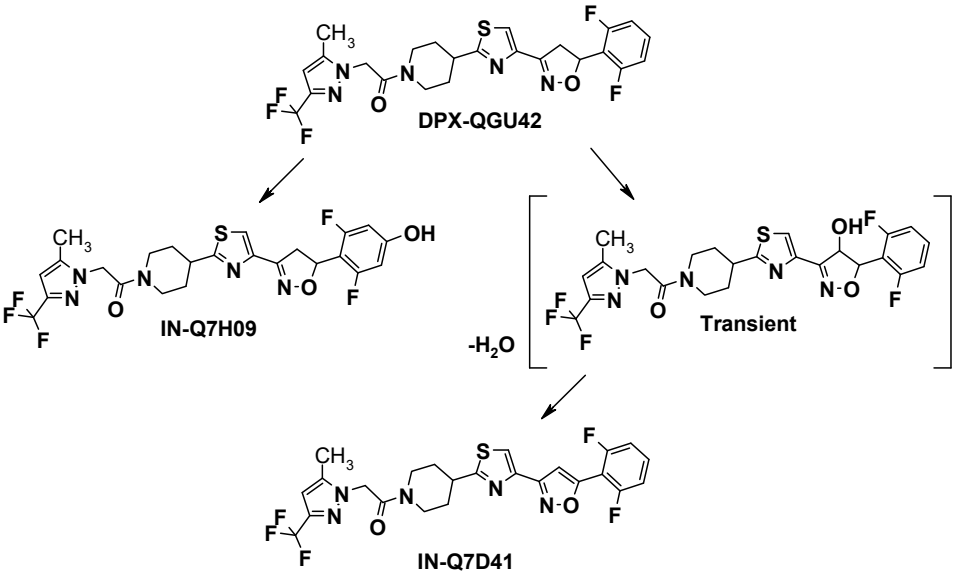


Figure 3 Proposed metabolic pathways for oxathiapiprolin (DPX-QGU42) in foliar treated lettuce

Potato – foliar applications

In a study reported by Doig, 2013 [Ref: DP-28068], three foliar applications of [pyrazole-^{14}C]-oxathiapiprolin or [thiazole^{14}C]-oxathiapiprolin were applied to potato plants in outdoor 1 m^2 plots at a rate equivalent to 0.07 kg ai/ha/treatment to give a total application rate of 0.21 kg ai/ha. Applications were made just before flowering (BBCH 53), at first flowering (BBCH 59) and 14 days later, at the end of first flowering (BBCH 69).

Immature potato foliage samples (whole plants) were taken immediately after the first application, before and after the second and third (final) applications, and fourteen days after the final application. The final (maturity) harvest was taken 28 days after the final application. Potato tuber samples were taken before the third (final) treatment, 14 days after the final treatment (14 DAT3) and at final harvest (28 DAT3). All samples were stored frozen for up to 4 weeks before extraction and initial chromatography.

Foliage samples were rinsed with acetonitrile prior to further analysis and the surface residues were determined. At selected sampling points, portions of pulverised samples were extracted using acetonitrile followed by acetonitrile: water (3:1, v/v). Selected post extracted solid (PES) samples were subjected to exhaustive extractions using water, acetone, α-amylase, combined amyloglucosidase and cellulase, NaOH (0.1 N), and HCl (1.0 N).

In mature tubers (28DAT3), TRRs were 0.012 mg eq/kg and 0.005 mg eq/kg from the [pyrazole-^{14}C] and [thiazole-^{14}C] label plots, respectively, with 99.8% TRR (0.012 mg eq/kg) of the pyrazole-label and 37% TRR (0.002 mg eq/kg) of the thiazole-label being extracted. Since each of the partitioned fractions contained less than 0.01 mg eq/kg, no further analysis was conducted. Unextracted tuber residues were less than 0.01 mg eq/kg for both radiolabels.

The majority of the radioactive residue in the immature foliage was removed by surface washing and/or initial extraction (75–96% TRR). Extracted radioactive residues in mature foliage

samples ranged from 0.57 to 0.73 mg eq/kg in the [pyrazole-^{14}C] treated samples and 0.75 to 1.16 mg eq/kg in the [thiazole-^{14}C] samples.

Representative post extracted solids (PES) of foliage from both radiolabels were subjected to the more aggressive extractions to characterise unextracted residues and a further 8–11% TRR (0.055–0.1 mg eq/kg) was characterized from 14DAT1 and 14DAT3 [pyrazole-^{14}C] labeled foliage and a further 7.9–12%TRR (0.07–0.12 mg eq/kg) was characterised from the corresponding [thiazole-^{14}C] labeled samples.

In mature foliage, the majority of the radioactive residue was removed by surface washing and initial extraction (75.5–78.3% TRR). The extractable radioactive residues, including surface wash at maturity, were 0.127 and 0.193 mg eq/kg in the [pyrazole-^{14}C] and [thiazole-^{14}C] samples, respectively.

Table 8 Total radioactive residues in potato foliage and tubers following three foliar applications of 0.07 kg ai/ha [^{14}C]-oxathiapiprolin – a total of 0.21 kg ai/ha

Harvest	Sample	Site of label	Surface wash		Extracted		Unextracted		Total
			% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	mg eq/kg
14DAT1	Foliage	Pyrazole	20.0	0.139	61.5	0.426	18.5[(b)]	0.128	0.694
		Thiazole	25.9	0.232	58.0	0.519	16.1[(b)]	0.144	0.894
0DAT2	Foliage	Pyrazole	-	-	95.4	1.655	4.6	0.080	1.735
		Thiazole	-	-	95.8	5.689	4.3	0.255	5.938
14DAT2	Foliage	Pyrazole	17.4	0.143	65.9	0.540	16.6	0.136	0.819
		Thiazole	35.4	0.466	52.9	0.697	11.6	0.153	1.317
14DAT3	Foliage	Pyrazole	21.2	0.195	58.6	0.538	20.2[(b)]	0.185	0.918
		Thiazole	15.6	0.155	60.0	0.596	24.4[(b)]	0.242	0.993
28DAT3	Foliage	Pyrazole	9.1	0.015	69.2	0.112	21.7	0.035	0.162
		Thiazole	9.8	0.025	65.7	0.168	24.6	0.063	0.255
28DAT3	Tuber	Pyrazole	-	-	99.8	0.012	0.2	< 0.001	0.012
		Thiazole	-	-	37.1	0.002	62.9	0.003	0.005

Extracts containing significant radioactivity (≥ 0.01 mg eq/kg) were analysed by HPLC and identified by LC-MS, with reference to authenticated reference standards and the enantiomer ratio of oxathiapiprolin isolates was determined by chiral column chromatography.

In the immature foliage samples, oxathiapiprolin was the major residue identified in samples, making up about 40-59% TRR. Multiple metabolites (each less than 10% TRR) were detected in immature foliage samples including IN-Q7H09, IN-Q7D41, a glucose conjugate of IN-RPD37, a hydroxymethyl-pyrazole glucoside of oxathiapiprolin-diol, and multiple hydroxylated oxathiapiprolin metabolites. Numerous unidentified metabolites were detected at levels up to 4.5% TRR (0.037 mg eq/kg).

In mature foliage, unchanged oxathiapiprolin was also the major component, accounting for about 25% TRR (0.04 mg/kg) and 42% TRR (0.11 mg/kg) in the [pyrazole-^{14}C] and [thiazole-^{14}C] samples, respectively. Other components detected in samples from both labels included IN-Q7H09, the glucose conjugate of IN-RPD37, the hydroxymethyl-pyrazole glucoside of oxathiapiprolin-diol and multiple hydroxylated oxathiapiprolin metabolites, individually present at no greater than 6.7% TRR. Several unknown metabolites were detected none greater than 5.9% TRR.

Table 9 Characterisation and identification of residues in potato foliage following three foliar applications of 0.07 kg ai/ha [^{14}C]-oxathiapiprolin

	14DAT1 foliage		14DAT2 foliage		14DAT3 foliage		Maturity foliage	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
[Pyrazole-^{14}C]-label								
Identified components								
Oxathiapiprolin	54.3	0.377	39.8	0.326	39.6	0.364	24.6	0.04
IN-RDG40	ND	ND	0.9	0.007	0.4	0.004	ND	ND
IN-Q7H09	0.9	0.006	0.4	0.003	1.3	0.012	0.3	< 0.001
IN-Q7D41	1.1	0.008	2.0	0.016	ND	ND	ND	ND
Glucose conjugate of IN-RPD37	2.6	0.018	ND	ND	1.9	0.017	6.5	0.01
Hydroxymethyl-pyrazole glucoside of oxathiapiprolindiol	1.2	0.008	9.9	0.081	3.5	0.032	3.6	0.006
Multiple hydroxylated components	7.2	0.05	5.8	0.048	4.2	0.038	0.5	< 0.002
Unidentified metabolites								
Number	5		13		16		12	
Highest	2.8	0.019	4.5	0.037	3.4	0.031	5.9	0.010
Total	9.2	0.063	23.0	0.188	25.4	0.232	40.0	0.066
Terminal Unextracted	4.8	0.033	16.6	0.136	2.7	0.025	21.7	0.035
[Thiazole-^{14}C]-label								
Identified components								
Oxathiapiprolin	47.8	0.427	58.9	0.775	43.2	0.429	42.4	0.108
IN-RDG40	0.4	0.004	0.5	0.007	0.3	0.003	0.4	0.001
IN-Q7H09	0.9	0.008	1.4	0.018	1.7	0.017	ND	ND
IN-Q7D41	1.5	0.013	1.0	0.013	ND	ND	ND	ND
Glucose conjugate of IN-RPD37	4.0	0.036	7.9	0.104	4.0	0.04	6.7	0.017
Hydroxymethyl-pyrazole glucoside of oxathiapiprolindiol	2.4	0.021	ND	ND	4.6	0.045	5.2	0.013
Multiple hydroxylated components	5.1	0.045	5.6	0.074	3.2	0.032	4.6	0.012
Unidentified metabolites								
Number	10		8		11		5	
Highest	2.5	0.022	2.4	0.032	1.9	0.019	3.6	0.009
Total	15.1	0.135	11.4	0.150	12.3	0.123	7.7	0.019
Terminal Unextracted	10.0	0.089	11.6	0.153	7.8	0.077	24.6	0.063

Chiral chromatographic analysis of the 28DAT3 (final harvest) [pyrazole-^{14}C] label foliage extract confirmed that the enantiomeric ratio of IN-Q7N24 and IN-Q7N25 for oxathiapiprolin remained unchanged throughout the study at 1:1.

The metabolic pathways of oxathiapiprolin in potato plants (foliage) were in general consistent with that of other plants and animals, primarily involving hydroxylation of the phenyl ring (forming IN-Q7H09 and IN-RDG40) and the isoxazoline ring (leading to the formation of IN-Q7D41). Several glucose conjugates of IN-RPD37 were also tentatively identified.

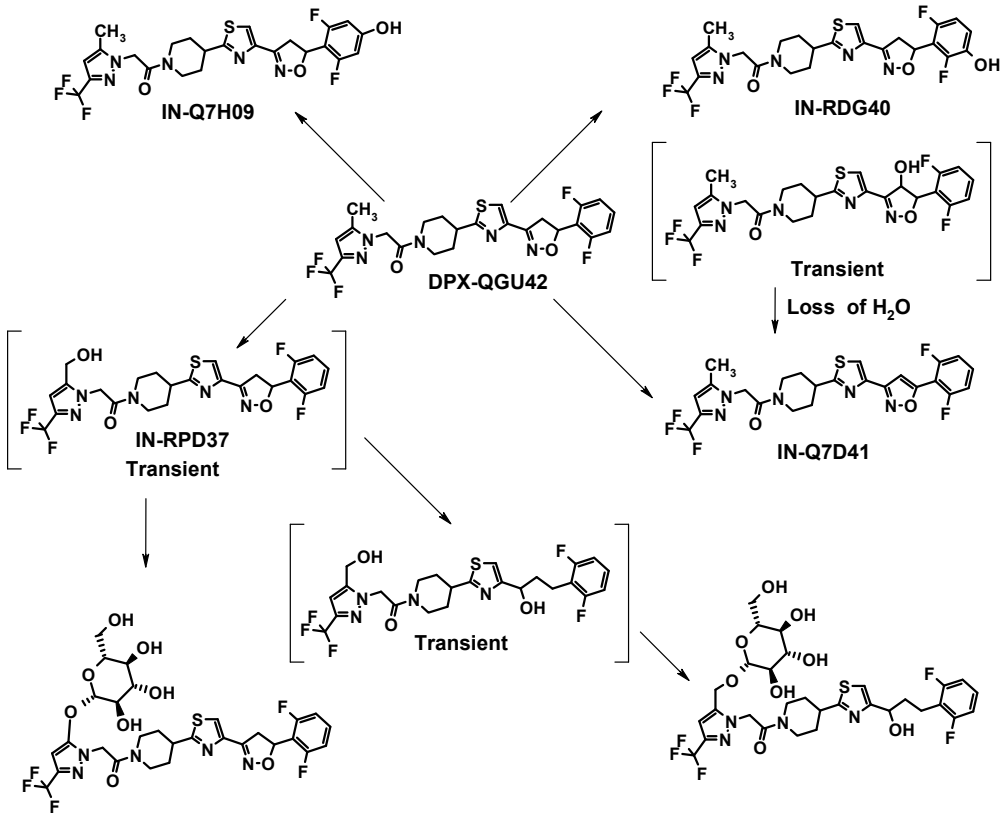


Figure 4 Proposed metabolic pathways for oxathiapiprolin (DPX-QGU42) in foliar-treated potatoes

Potato–soil applications

In a greenhouse study to examine the metabolic fate of [^{14}C]-oxathiapiprolin in potatoes following soil application, reported by Doig, 2012 [Ref: DP-31742], seed potatoes were sown into a loam soil (3.1% organic matter, pH 5.3) immediately after a single soil application of either [pyrazole-^{14}C]-oxathiapiprolin or [isoxazoline-^{14}C]-oxathiapiprolin (SC formulation) at a rate equivalent to 0.6 kg ai/ha.

Whole potato plants, separated into foliage and tubers, were sampled 37 days after treatment (BBCH 65) and at maturity (BBCH 91), 72 days after treatment. Foliage and tuber samples were extracted twice with acetonitrile followed by acetonitrile:water (3:1, v/v). The unextracted radioactivity in the post extraction solids was determined by combustion analysis. The total radioactive residues (TRR) in each sample were determined by summing radioactivity in the extracts and unextracted residues and were expressed as mg/kg equivalents of the parent oxathiapiprolin.

The total radioactive residues increased from 0.026 mg eq/kg (37DAT) to 0.11 mg eq/kg (72DAT) in foliage and decreased from 0.023 mg eq/kg (37DAT) to 0.013 mg eq/kg (72DAT) in the tubers following soil treatment with [pyrazole-^{14}C]-oxathiapiprolin. Total radioactive residues in the corresponding samples following [isoxazoline-^{14}C]-oxathiapiprolin soil treatment increased over time from 0.021 mg eq/kg (37DAT) to 0.056 mg eq/kg (72DAT) in foliage and decreased from 0.013 to 0.006 mg eq/kg in tubers.

The majority of the radioactive residue in the immature tuber samples was able to be extracted with acetonitrile, ranging from 77% TRR – 0.01 mg eq/kg (isoxazoline-label) to 85% TRR – 0.02 mg eq/kg (pyrazole-label). Unextracted residues accounted for 0.003 mg eq/kg in both samples. In mature tubers from the [pyrazole-^{14}C]-oxathiapiprolin treated soil, about 81% TRR (0.01 mg eq/kg) was extracted with acetonitrile, with unextracted residues being 0.003 mg eq/kg (19.3% TRR). TRR

levels in extracts from both the mature and immature tubers samples grown in the [isoxazoline-^{14}C]-oxathiapiprolin treated soil were low (≤ 0.01mg eq/kg) and were not investigated further.

The majority of the radioactive residue in foliage was extracted in the initial acetonitrile extract (about 80–91% TRR). Unextracted residues accounted for 0.003–0.004 mg eq/kg in immature foliage and 0.008–0.01 mg eq/kg in mature foliage.

Table 10 Total radioactive residues in potato foliage and tubers following a soil application of 0.6 kg ai/ha [^{14}C]-oxathiapiprolin

Sampling interval	Site of label	Sample	TRR (mg eq/kg)	Extracted ^{14}C		Oxathiapiprolin mg/kg		Unextracted ^{14}C	
				%TRR	mg eq/kg	%TRR	mg/kg	%TRR	mg eq/kg
Immature (37 DAT)	Pyrazole	Foliage	0.026	89.1	0.023		< 0.001	10.9	0.003
		Tubers	0.023	85.2	0.020	6.9	0.002	14.8	0.003
	Isoxazoline	Foliage	0.021	79.6	0.017		< 0.001	20.4	0.004
		Tubers	0.013	77.2	0.010	NA		22.8	0.003
Mature (72 DAT)	Pyrazole	Foliage	0.108	90.8	0.098	4.2	0.005	9.2	0.010
		Tubers	0.013	80.7	0.01		< 0.001	19.3	0.003
	Isoxazoline	Foliage	0.056	85.0	0.048	9.2	0.005	15.0	0.008
		Tubers	0.006	Not extracted					

Extracts containing significant radioactivity (≥ 0.01 mg eq/kg) were analysed by high performance liquid chromatography (HPLC) and identification of ^{14}C residues was accomplished by HPLC using liquid chromatography mass spectrometry (LC-MS), with reference to authenticated reference standards. Metabolites IN-RZD74 and IN-RZB20 were not confirmed by LC-MS, however the retention times were consistent with that of the standards. The reference standards of IN-RZB21 and IN-RZD74 eluted from the column with insufficient separation to enable definitive identification of the two metabolites by HPLC. In the instances where a radioactive residue was observed in this region the peak has been denoted IN-RZB21/IN-RZD74 indicating the possibility of a mixture of these metabolites unless otherwise stated.

Oxathiapiprolin was only identified in immature tuber samples at 6.9% TRR, 0.002 mg/kg. IN-WR791 was the major component identified in both immature and mature potato tubers following soil treatment with [pyrazole-^{14}C]-oxathiapiprolin (0.003 mg/kg). Minor tuber metabolites included IN-E8S72, IN-RZB20, IN-KJ552, IN-SXS67, IN-RZB21/IN-RZD74 (unresolved), and numerous low level unidentified metabolites following soil treatment with [pyrazole-^{14}C]-oxathiapiprolin.

Oxathiapiprolin was only identified at low levels (0.005 mg/kg–9.2% TRR) in foliage from the [isoxazoline-^{14}C]-treatment and 0.005 mg/kg (4.2% TRR) in the foliage collected from [pyrazole-^{14}C]-treated soil.

IN-RZB21/IN-RZD74 (unresolved) was the principal component identified in mature potato foliage following soil treatment with [pyrazole-^{14}C]-oxathiapiprolin at 0.014 mg eq/kg. Minor foliage metabolites included IN-WR791, IN-RZB20, IN-KJ552, IN-E8S72, and IN-SXS67 (only found in immature foliage) and numerous low level unidentified metabolites.

Table 11 Characterisation and identification of residues in potato foliage and tubers grown in soil treated with 0.07 kg ai/ha [^{14}C]-oxathiapiprolin

	Immature foliage		Immature tubers		Mature foliage		Mature tubers	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
[Pyrazole-^{14}C]-label								
Identified components								
Oxathiapiprolin	ND	ND	6.9	0.002	4.2	0.005	ND	ND
IN-E8S72	11.5	0.003	5.8	0.001	5.1	0.006	13.9	0.002
IN-WR791	13.3	0.003	14.3	0.003	7.3	0.008	25.3	0.003
IN-SXS67	6.2	0.002	3.7	0.001	4.2	0.005	7.1	0.001
IN-RZB20	13.1	0.003	12.0	0.003	11.5	0.012	12.2	0.001
IN-RZB21/IN-RZD74	18.8	0.005	2.7	0.001	13.1	0.014	5.5	0.001

	Immature foliage		Immature tubers		Mature foliage		Mature tubers	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
IN-KJ552	4.1	0.001	7.3	0.002	4.4	0.005	6.5	0.001
26.3 min	ND	ND	ND	ND	ND	ND	ND	ND
29.5 min	ND	ND	ND	ND	ND	ND	ND	ND
33.5 min	ND	ND	ND	ND	ND	ND	ND	ND
Solvent front	ND	ND	ND	ND	ND	ND	ND	ND
Unidentified metabolites								
Number	6		5		13		2	
Highest	4.7	0.001	2.6	0.001	4.5	0.005	4.5	0.001
Total	14.1	0.003	11.2	0.003	26.9	0.028	4.9	0.001
Unextracted	10.9	0.003	14.8	0.003	9.2	0.010	19.3	0.003
[Isoxazoline-^{14}C]-label								
Identified components								
Oxathiapiprolin	ND	ND			9.2	0.005		
IN-E8S72	ND	ND			ND	ND		
IN-WR791	ND	ND			ND	ND		
IN-SXS67	ND	ND			ND	ND		
IN-RZB20	ND	ND			ND	ND		
IN-RZB21/IN-RZD74	ND	ND			ND	ND		
IN-KJ552	ND	ND			ND	ND		
26.3 min	9.6	0.002			ND	ND		
29.5 min	11.3	0.002			ND	ND		
33.5 min	8.0	0.002			ND	ND		
Solvent front	ND	ND			2.1	0.001		
Unidentified metabolites								
Number	10				18			
Highest	6.9	0.001			8.5	0.005		
Total	40.6	0.009			66.6	0.038		
Unextracted	20.4	0.004			15.0	0.008		

Oxathiapiprolin metabolites found in potato foliage following soil application are predominantly those containing the pyrazole moiety (possibly reflecting preferential root uptake of the more polar pyrazole cleavage products). Cleavage of the parent compound results in the formation of IN-RZB20, IN-WR791, IN-RZB21, IN-KJ552 and IN-E8S72 (which undergoes *N*-glucose conjugation to form IN-SXS67.

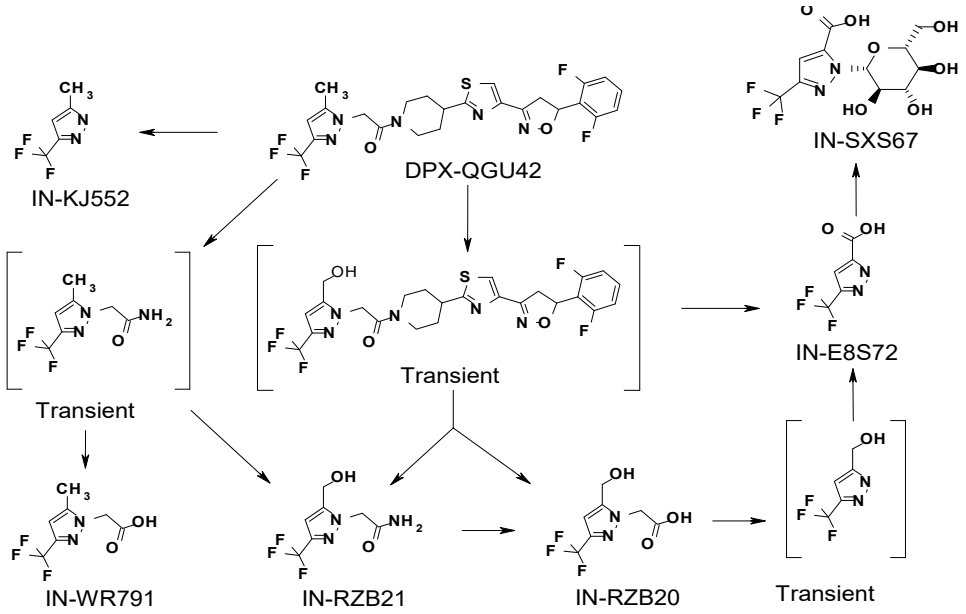


Figure 5 Proposed metabolic pathways for oxathiapiprolin (DPX-QGU42) in soil treated potato plants

Lettuce–soil applications

In a greenhouse study to examine the metabolic fate of [^{14}C]-oxathiapiprolin in lettuce following soil application, reported by Doig & Inns, 2012 [Ref: DP-31741], lettuce seeds were sown into a loam soil (3.1% organic matter, pH 5.3) immediately after a single soil application of either [pyrazole-^{14}C]-oxathiapiprolin or [isoxazoline-^{14}C]-oxathiapiprolin (SC formulation) at a rate equivalent to 0.6 kg ai/ha.

Leaf samples were taken 44 days after treatment (BBCH 45) and at maturity, 57 days after treatment. Samples were homogenised and extracted twice with acetonitrile and reextracted twice with acetonitrile:water (3:1, v/v). Levels of radioactivity were determined in each extract by LSC and in post extraction solid samples by oxidative combustion followed by LSC.

Extracts containing significant radioactivity (>0.01 mg eq/kg) were analysed by high performance liquid chromatography (HPLC) and identification of the principal ^{14}C-residues was accomplished by HPLC using liquid chromatography mass spectrometry (LC-MS), with reference to authenticated reference standards.

The total radioactive residues in lettuce samples grown in [isoxazoline-^{14}C]-oxathiapiprolin treated soil were less than the limit of quantification (0.010 mg eq/kg) and were not investigated further.

TRR in lettuce samples grown in [pyrazole-^{14}C]-treated soil decreased from 0.019 mg eq/kg (44DAT) to 0.014 mg eq/kg at maturity (57DAT) with 88–91% TRR being removed in the initial acetonitrile extract (0.012–0.017 mg eq/kg). Unextracted residues accounted for 9.5–12% TRR (0.002 mg eq/kg).

Oxathiapiprolin was not detected in any of the samples. Metabolites making up more than 10% TRR in immature and mature foliage samples were IN-WR791 (23% TRR and 30% TRR respectively), IN-E8S72 (19-21%TRR) and unresolved IN-RZB21/IN-RZD74 (19–21% TRR), but these were present at levels of 0.004 mg eq/kg or less.

Minor metabolites were IN-RZB20 (≤ 6.5% TRR or 0.001 mg eq/kg), IN-SXS67 and IN-KJ552 (each below 4% TRR).

Table 12 Characterisation and identification of residues in foliage of lettuce grown in soil treated with one application of 0.6 kg ai/ha [^{14}C]-oxathiapiprolin

	[Pyrazole-^{14}C]-label			
	Immature (44 days after treatment)		Mature (57 days after treatment)	
	% TRR	mg eq/kg	% TRR	mg eq/kg
TRR		0.019		0.014
Extracted	90.5	0.017	88.3	0.012
Identified components				
Oxathiapiprolin	ND	ND	ND	ND
IN-SXS67	1.9	< 0.001	3.5	< 0.001
IN-RZB20	5.1	0.001	6.5	0.001
IN-RZB21/IN-RZD74 (unresolved)	21.4	0.004	19.0	0.002
IN-E8S72	18.9	0.004	21.2	0.003
IN-WR791	22.7	0.004	29.5	0.004
IN-KJ552	ND	ND	3.1	< 0.001
Unidentified metabolites				
Number	0		1	
Highest	ND	ND	1.2	< 0.001
Total	ND	ND	1.2	< 0.001
Unextracted	9.5	0.002	11.7	0.002

Oxathiapiprolin metabolites found in lettuce foliage following soil application are predominantly those containing the pyrazole moiety (possibly reflecting preferential root uptake of the more polar pyrazole cleavage products). Cleavage between the piperidine and pyrazole rings form the pyrazole-containing polar metabolites IN-WR791, IN-E8S72, IN-KJ552, IN-RZB20, IN-RZB21, and/or IN-RZD74 with subsequent glucose conjugation of IN-E8S72 forming IN-SXS67.

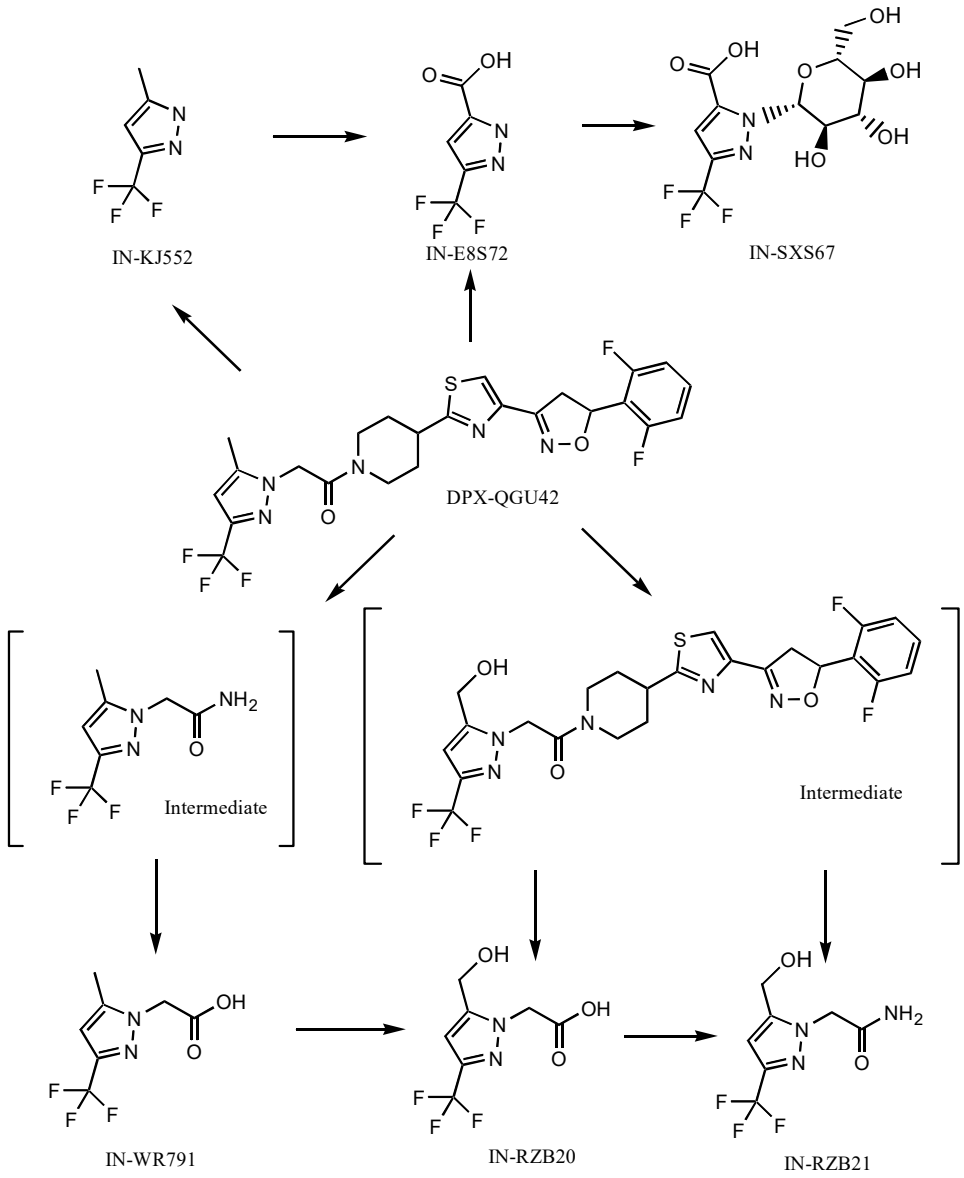


Figure 6 Proposed metabolic pathways for oxathiapiprolin (DPX-QGU42) in lettuce following soil application

Courgettes–soil applications

In a greenhouse study to examine the metabolic fate of [^{14}C]-oxathiapiprolin in courgettes following soil application, reported by Hobbs, 2012 [Ref: DP-32237], courgette seeds were sown into a loam soil (3.1% organic matter, pH 5.3) immediately after a single soil application of either [pyrazole-^{14}C]-oxathiapiprolin or [Isoxazoline-^{14}C]-oxathiapiprolin (SC formulations) at a rate equivalent to 0.6 kg ai/ha.

Leaf and fruit samples were taken 44 days after treatment (BBCH 71) and at maturity, 79 days after treatment. Samples were homogenised and extracted twice with acetonitrile and reextracted twice with acetonitrile:water (3:1, v/v). Levels of radioactivity were determined in each extract by LSC and in post extraction solid samples by oxidative combustion followed by LSC.

The total radioactive residues (TRR) increased over time from 0.045 mg eq/kg (44DAT) to 0.170 mg eq/kg (79DAT) in foliage and from 0.013 to 0.023 mg eq/kg in the corresponding fruit samples from [pyrazole-^{14}C]-treated soil. Conversely, the TRR in the foliage from [isoxazoline-^{14}C]-treated soil decreased over time from 0.028 mg eq/kg (44DAT) to 0.008 mg eq/kg (79DAT). Total radioactive residues in fruit from [isoxazoline-^{14}C]-treated soil were below the limit of detection throughout the study (< 0.006 mg eq/kg).

The total radioactive residues in fruit and mature foliage samples from plants grown in [isoxazoline-^{14}C]-oxathiapiprolin treated soil were less than the limit of quantification (0.01 mg eq/kg) and were not investigated further.

The majority of the radioactive residue in the foliage was extracted in the initial acetonitrile extract, 77% TRR in the [isoxazoline-^{14}C]-treatment immature foliage samples and 91–94% TRR in the [pyrazole-^{14}C]-treatment samples. Unextracted residues accounted for 0.006 mg eq/kg (23% TRR) and 0.004–0.01 mg eq/kg (6–9.3% TRR), respectively.

The majority of the radioactive residue in immature and mature fruit from [pyrazole-^{14}C]-oxathiapiprolin treated soil was extracted in the initial acetonitrile extract (94–97% TRR, 0.012–0.022 mg eq/kg), with unextracted residues accounting for 0.001 mg eq/kg (3.2–6.3% TRR)

Extracts containing significant radioactivity (> 0.01 mg eq/kg) were analysed by high performance liquid chromatography (HPLC) and identification of the principal ^{14}C-residues was accomplished by HPLC using liquid chromatography mass spectrometry (LC-MS), with reference to authenticated reference standards. IN-RZD74, IN-RZB20, and IN-RZB21 were not confirmed by LC-MS however, the retention times were consistent with that of the standards. Residues tentatively identified as IN-RZD74, IN-RZB20, and IN-RZB21 were found at low concentrations (up to 0.021 mg eq/kg) and co-eluting with a large quantity of endogenous material making positive identification by LC-MS difficult.

Oxathiapiprolin was detected in the fruit from [pyrazole-^{14}C] treated soil at trace levels (0.5% TRR, < 0.001 mg/kg) but not in mature fruit.

IN-WR791 was the major component identified in fruit, found at 0.008 mg eq/kg (57% TRR) in immature fruit and 0.016 mg eq/kg (74% TRR) in mature fruit. Other metabolites (each ≤ 0.001 mg eq/kg, ≤ 4.5% TRR) included IN-E8S72, IN-SXS67, IN-RZB20, IN-KJ552, and IN-RZB21/IN-RZD74. The reference standards of IN-RZB21 and IN-RZD74 eluted from the column with insufficient separation to enable definitive identification of the two metabolites. Unidentified components each accounted for less than 0.001 mg eq/kg (≤ 1.3% TRR).

In foliage, oxathiapiprolin was the major component (24% TRR, 0.007 mg/kg) identified in the immature foliage grown in [isoxazoline-^{14}C]-treated soil but not detected in the samples grown in [pyrazole-^{14}C]-treated soil. In mature foliage from the [pyrazole-^{14}C]-treated soil, oxathiapiprolin was detected at low levels (4.6% TRR, 0.008 mg/kg).

Major metabolites in foliage were IN-WR791 (24–27.5% TRR and 0.011 mg eq/kg in immature foliage and 0.047 mg eq/kg in mature foliage) and IN-E8S72 (21–24% TRR and 0.011 mg eq/kg in immature foliage and 0.036 mg eq/kg in mature foliage). IN-RZB20 was found in mature foliage at 12% TRR – 0.021 mg eq/kg but was less than 0.008 mg eq/kg in immature foliage.

IN-Q7H09 (18.5% TRR, 0.005 mg eq/kg), an unidentified polar metabolite (13% TRR, 0.004 mg eq/kg), and a highly polar region associated with the solvent front (9.4% TRR, 0.003 mg eq/kg) were also identified in immature foliage with other minor metabolites included IN-SXS67, IN-KJ552 and IN-RZB21/IN-RZD74, present at up to 0.008 mg eq/kg (17% TRR). Unidentified components did not exceed 2–3% TRR.

Unresolved IN-RZB21/IN-RZD74 was present in mature foliage at 11% TRR (0.018 mg eq/kg) with other identified metabolites (IN-SXS67, IN-Q7H09, and IN-KJ552) found at up to 0.01 mg eq/kg (6% TRR). Unidentified components accounted for less than 0.004 mg eq/kg (2.7% TRR).

Table 13 Characterisation and identification of residues in courgettes (fruit and foliage) grown in soil treated with one application of 0.07 kg ai/ha [^{14}C]-oxathiapiprolin

	[Pyrazole-^{14}C]-label								[Isoxazoline-^{14}C]-label	
	Immature fruit		Immature foliage		Mature fruit		Mature foliage		Immature foliage	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
TRR		0.013		0.045		0.023		0.17		0.028
Extracted	93.7	0.012	90.7	0.041	96.8	0.22	94	0.16	76.9	0.022

	[Pyrazole-^{14}C]-label								[Isoxazoline-^{14}C]-label	
	Immature fruit		Immature foliage		Mature fruit		Mature foliage		Immature foliage	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Identified components										
Oxathiapiprolin	0.5	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	4.6	0.008	24.4	0.007
IN-E8S72	4.5	0.001	23.5	0.011	4.3	0.001	21.1	0.036	ND	< 0.001
IN-WR791	56.7	0.008	23.7	0.011	73.7	0.016	27.5	0.047	ND	< 0.001
IN-SXS67	4.0	0.001	7.2	0.003	1.3	< 0.001	6.0	0.010	ND	< 0.001
IN-RZB20	2.2	< 0.001	16.8	0.008	3.3	0.001	12.4	0.021	ND	< 0.001
IN-RZB21+ IN-RZD74	4.3	0.001	12.7	0.006	4.3	0.001	10.9	0.018	ND	< 0.001
IN-KJ552	2.6	< 0.001	3.4	0.002	2.0	< 0.001	1.5	0.002	ND	< 0.001
IN-Q7H09	ND	ND	ND	ND	ND	ND	1.7	0.003	18.5	0.005
Solvent front	0.0	< 0.001	1.8	0.001	ND	ND	1.0	0.002	9.4	0.003
Unidentified metabolites										
Number	3		5		2		4		1	
Highest	1.3	< 0.001	1.7	0.001	2.5	0.001	>2.7	0.004	13.2	0.004
Total	3.3	< 0.001	6.2	0.003	5.0	0.002	6.7	0.010	13.2	0.004
Unextracted	6.3	0.001	9.3	0.004	3.2	0.001	6.0	0.010	23.1	0.006
ND = Not detected										

Oxathiapiprolin metabolites found in courgettes following soil application are predominantly those containing the pyrazole moiety (possibly reflecting preferential root uptake of the more polar pyrazole cleavage products). These pyrazole-containing cleavage products include IN-WR791, IN-E8S72, IN-KJ552, IN-RZB21, IN-RZD74, and IN-RZB20. Hydroxylation of the parent compound (phenyl ring) forms IN-Q7H09.

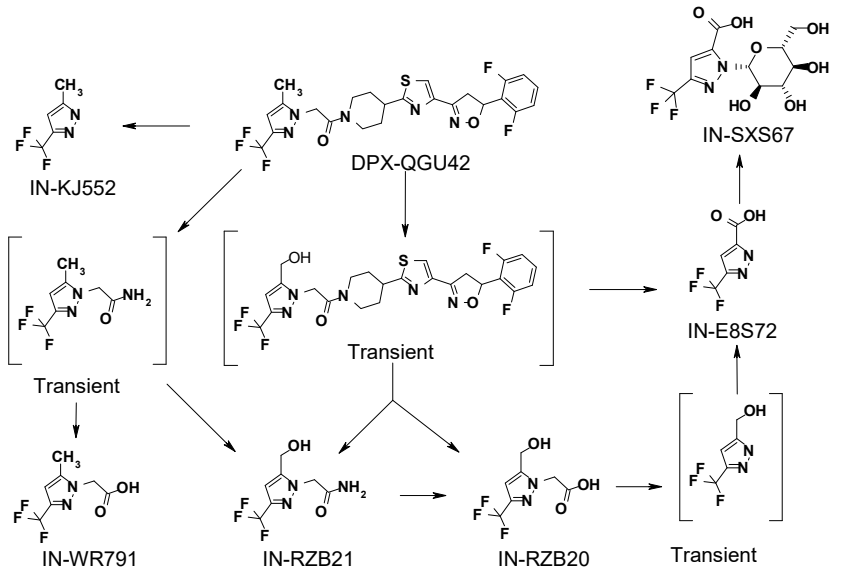


Figure 7 Proposed metabolic pathways for oxathiapiprolin (DPX-QGU42) in courgette plants

In summary, when oxathiapiprolin was applied as a foliar treatment, parent compound was the major residue in lettuce, potato, grape leaves and berries, making up 25–85% TRR. Significant metabolites were the cleavage products IN-E8S72 and IN-WR791 in grape berries, present at 14% TRR (0.044 mg eq/kg) and 19% TRR (0.057 mg eq/kg) respectively. Many other metabolites were identified but all at low levels, each less than 7% TRR.

Following soil applications, oxathiapiprolin is a minor component of the total residue, with the major residues (above 10% TRR) being those containing the pyrazole moiety (IN-WR791, IN-E8S72, IN-RZB21/IN-RZD74 (unresolved) and RZB20) and present at concentrations less than 0.05 mg eq/kg. These findings are generally consistent with the results of the confined rotational crop

studies where these metabolites (together with IN-SXS67 – the glucose conjugate of IN-E8S72) were also found at levels above 10% TRR.

Animal metabolism

The Meeting received animal metabolism studies on rats, lactating goats and laying hens where animals were dosed with oxathiapiprolin radiolabelled in the pyrazole ring, the thiazole ring or the isoxazoline ring (rat) and in lactating goats dosed with pyrazole-labelled IN-E8S72, a glucoside plant metabolite of oxathiapiprolin.

Rats

The metabolism of oxathiapiprolin in rats was reviewed in the framework of the toxicological evaluation by the WHO Core Assessment Group of the 2016 JMPR.

Lactating goats

In a study reported by Melville & Vance, 2013 [Ref: DP-28213], two 4-year old lactating goats (Toggenburg breed) were dosed orally by capsule for 7 consecutive days with either [pyrazole-^{14}C]- or [thiazole-^{14}C]-oxathiapiprolin at doses equivalent to 14.2 and 14.3 ppm in the diet (21.36 and 17.66 mg/goat/day based on an average feed consumption of 1.25–1.58 kg/day and body weights of 55.5–60.5 kg).

The goats were dosed once a day, in the morning. During the dosing period, milk production was normal (*ca*. 1 kg/day) and there were no adverse clinical findings during dosing. Urine and faeces were collected daily from each goat, and cage wash samples (1:1 water/acetonitrile) were obtained at sacrifice. Milk was collected twice daily from each goat and tissue samples were taken at sacrifice, 12 hours after the final dose. All samples were stored frozen (-20 °C) and all primary extracts (initial solvent/buffer extractions) were analysed by HPLC within 80 days of sacrifice.

Tissue and fat samples were homogenised in dry ice and analysed by combustion\LSC or solubilised for LSC. Equal amounts of faeces, urine, and milk from Days 1–7 were pooled by dose group. Subsamples (30–100 g) of tissues, milk, and faeces were extracted with dichloromethane, acetonitrile, and acetonitrile/water (4:1, v/v), concentrated, and analysed by LSC and HPLC. The liver post extracted solids (PES) from both goats were further treated with protease (37 °C; 120 h) to release unextracted residues.

Radioactive residues were analysed by HPLC, using a C18 column with a mobile phase gradient of 20 mM ammonium acetate and acetonitrile. Radioactive residues of the collected HPLC fractions were quantified by LSC. Residues were initially identified by comparison of their retention times with the retention times for reference standards. The initial acetonitrile/water extracts from excreta, liver, and kidney samples were used for further confirmation of metabolite identities and analysed by HPLC/LC-MS, then compared to the reference standards. Chiral column analysis was used to determine the enantiomer ratio of the oxathiapiprolin isolates from goat faeces and liver extract.

The total recovery was 94–99.6% of the administered radioactivity (AR) for both goats. For both [^{14}C]-labels, 84-86% of the dose was recovered from the urine, faeces, and cage wash, 8–12% was found in the gastrointestinal tract, 0.1–0.2% AR in milk and 0.7–1.1% AR was recovered in edible tissues. Radioactivity plateaued in milk within 5 days at about 0.03 mg eq/kg.

Total radioactive residue levels in milk were 0.02–0.01 mg eq/kg and in edible tissues were 0.97–0.75 mg eq/kg in liver, 0.084–0.065 mg eq/kg in kidney, 0.009–0.013 mg eq/kg in muscle and 0.025–0.03 in fats from the [pyrazole-^{14}C] and [thiazole-^{14}C]-dosed goats respectively.

Table 14 Percent administered dose recovered in milk, tissues, and excreta from lactating goats following 7 consecutive daily oral doses of 14.2 ppm [^{14}C]-oxathiapiprolin in the diet

Goat	[Pyrazole-14C]-label	[Thiazole-14C]-label
Sample	% Dose	% Dose
Faeces	81.8	79.4
Urine	3.8	4.0
Cage wash	0.8	0.9
Milk (Total Day 1–7)	0.2	0.1
Liver	0.5	0.7
Kidney	< 0.1	< 0.1
Muscle [a]	0.1	0.2
Omental fat [a]	< 0.1	0.1
Renal fat [a]	< 0.1	< 0.1
Subcutaneous fat [a]	0.1	0.1
GI contents	12.3	8.4
Total	99.6	94.0

[a] Total muscle mass was assumed to be approximately 25% of body weight, total fat approximately 15% of body weight. Each fat type accounted for the following percentages of total bodyweight, renal fat ca. 0.9%, omental fat ca. 4.1% and subcutaneous fat ca. 9.4%

Initial solvent extractions with acetonitrile/water released 52–100% TRR from tissues and milk and subsequent enzymatic hydrolysis released another 26–29% TRR from liver. The remaining solids accounted for up to 12% TRR. The majority of radioactive residues (52–100% TRR) were identified and/or characterised in milk and tissues.

Oxathiapiprolin accounted for 8.7–11% TRR (0.002 mg/kg) in milk, 6.4–12% TRR (0.05–0.11 mg/kg) in liver, 13–14% TRR (0.01 mg/kg) in kidney, 27–43% TRR (0.004 mg/kg) in muscle, and 36–58% TRR (up to 0.016 mg/kg) in the fat fractions.

In milk and tissues, the major pyrazole-derived metabolite was IN-E8S72 (maximum 24% TRR in kidney). A number of unresolved mono-hydroxy metabolites (including IN-RDG40 and IN-Q7H09) were measured at combined levels above 0.01 mg eq/kg in liver (up to 0.13 mg eq/kg – 13% TRR) and kidney (0.013 mg eq/kg – up to 21% TRR). Other metabolites found at more than 10% TRR were IN-Q7D41 (up to 15% TRR, 0.004–0.008 mg eq/kg in fat) and IN-RAB06 (up to 11% TRR, 0.002 mg eq/kg in milk.

Minor metabolites (< 10% TRR) included IN-QFD61 (up to 0.004 mg eq/kg in milk), IN-RLB67 (up to 0.05 mg eq/kg in liver. Several minor unidentified metabolites were also detected, none of which individually were greater than 6% TRR or 0.01 mg eq/kg.

Oxathiapiprolin and metabolites IN-E8S72, IN-RAB06, IN-RLB67, IN-QFD61 IN-RDG40, IN-Q7H09, and IN-Q7D41 were confirmed in liver or kidney extracts by LC-MS/MS or by accurate mass measurement.

Table 15 Characterisation and identification of radioactivity in milk and tissues from lactating goats dosed with [^{14}C]-oxathiapiprolin

Fraction	Radioactivity recovered (% TRR and mg eq/kg)									
	Composite milk		Liver		Kidney		Muscle		Fats	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
[Pyrazole-^{14}C]-label										
Oxathiapiprolin	10.8	0.002	11.8	0.114	12.6	0.011	42.6	0.004	45.6 (o) 36.4 (r) 57.6 (s)	0.011 0.01 0.013
IN-E8S72	ND	ND	ND	ND	24.4	0.021	ND	ND	ND	ND
IN-QFD61	7.6	0.002	ND	ND	ND	ND	ND	ND	ND	ND
IN-Q7D41	2.9	0.001	0.9	0.008	ND	ND	ND	ND	15.3 (o) 11.7 (r) 10.9 (s)	0.004 0.003 0.003
IN-RAB06	11.1	0.002	1.6	0.016	3.0	0.003	ND	ND	ND	ND

Fraction	Radioactivity recovered (% TRR and mg eq/kg)									
	Composite milk		Liver		Kidney		Muscle		Fats	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
IN-RLB67	ND	ND	5.2	0.051	ND	ND	ND	ND	ND	ND
Hydroxy metabolites[a] IN-RDG40/IN-Q7H09	4.0	0.001	13.3	0.129	15.6	0.013	30.1	0.003	25.1 (o) 25.7 (r) 15.9 (s)	0.006 0.007 0.004
Peak at 22.4 min	ND	ND	4.0	0.039	ND	ND	ND	ND	ND	ND
Peak at 28.2 min	ND	ND	2.3	0.023	ND	ND	ND	ND	ND	ND
Peak at 31.1 min	17.9	0.004	ND	ND	ND	ND	ND	ND	ND	ND
Peak at 34.5 min	ND	ND	1.4	0.014	ND	ND	ND	ND	ND	ND
Peak at 36.5 min	13.1	0.003	ND	ND	ND	ND	ND	ND	ND	ND
Peak at 53.1 min	ND	ND	1.5	0.014	ND	ND	ND	ND	ND	ND
Peak at 54.5 min	ND	ND	2.8	0.027	ND	ND	ND	ND	ND	ND
Peak at 57.4 min	ND	ND	1.3	0.012	ND	ND	ND	ND	ND	ND
Minor unidentified metabolites	15.6	0.004	13.2	0.127	19.1	0.017	12.8	0.001	14.0 (o) 9.4 (r) 15.6 (s)	0.003 0.002 0.005
Total characterised by HPLC	83.0	0.019	59.3	0.574	74.7	0.065	85.5	0.008	100 (o) 83.2 (r) 100 (s)	0.024 0.022 0.025
Solvent extracted	96.7	0.021	60.1	0.58	91.3	0.077	100.0	0.009	100 (o) 99.8 (r) 100 (s)	0.025 0.026 0.023
Enzyme extracted	NA	NA	25.7[b]	0.248[b]	NA	NA	NA	NA	NA	NA
Unextracted	3.3	0.001	11.7	0.113	8.8	0.007	< 0.1	< 0.001	< 0.1 (o) 0.2 (r) < 0.1 (s)	< 0.001 < 0.001 < 0.001
Total	100.0	0.022	97.5	0.941	100.0	0.084	100.0	0.009	100 (o) 100 (r) 100 (s)	0.025 0.026 0.025
[Thiazole-^{14}C]-label										
Oxathiapiprolin	8.7	0.002	6.4	0.048	13.8	0.009	26.8	0.004	54.1 (o) 49.3 (r) 48.3 (s)	0.016 0.014 0.013
IN-QFD61	8.6	0.002	0.6	0.004	ND	ND	ND	ND	ND	ND
IN-Q7D41	2.1	< 0.001	0.6	0.005	ND	ND	5.5	0.001	10.2 (o) 14.6 (r) 9 (s)	0.003 0.004 0.002
IN-RAB06	10.7	0.002	1.8	0.014	4.8	0.003	ND	ND	ND	ND
IN-RLB67	ND	ND	4.9	0.037	ND	ND	ND	ND	ND	ND
Hydroxy metabolites [a] IN-RDG40/IN-Q7H09	2.9	0.001	10.6	0.078	20.6	0.013	27.6	0.004	14.2 (o) 24.3 (r) 12.6 (s)	0.004 0.007 0.003
Peak at 15.5 min	ND	ND	2.7	0.020	ND	ND	ND	ND	ND	ND
Peak at 22.2 min	ND	ND	2.3	0.017	ND	ND	ND	ND	ND	ND
Peak at 30.2 min	12.7	0.003	1.4	0.010	ND	ND	ND	ND	ND	ND
Peak at 36.4 min	9.7	0.002	ND	ND	ND	ND	ND	ND	ND	ND
Peak at 54.5 min	ND	ND	1.6	0.012	ND	ND	ND	ND	ND	ND
Minor unidentified metabolites	18.0	0.004	18.7	0.138	39.4	0.026	5.0	0.001	9.8 (o) 11.1 (r) 13.3 (s)	0.004 0.003 0.004
Total characterised by HPLC	73.4	0.016	51.6	0.383	78.6	0.051	64.9	0.010	88.3 (o) 99.3 (r) 83.2 (s)	0.027 0.028 0.022
Solvent extracted	88.1	0.02	52.4	0.392	88.9	0.058	97.2	0.013	99.9 (o) 99.3 (r) 99 (s)	0.03 0.029 0.026
Enzyme extracted	NA	NA	29.2[c]	0.218[c]	NA	NA	NA	NA	NA	NA
Unextracted	11.9	0.003	16.4	0.123	11.0	0.007	2.9	< 0.001	0.2 (o) 0.6 (r) 0.9 (s)	< 0.001 < 0.001 < 0.001

Fraction	Radioactivity recovered (% TRR and mg eq/kg)									
	Composite milk		Liver		Kidney		Muscle		Fats	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Total	100.0	0.023	98	0.773	100.0	0.065	100.0	0.013	100 (o) 100 (r) 100 (s)	0.03 0.029 0.026

(o) Omental fat, (r) renal fat, (s) subcutaneous fat

[a] Reference standards, IN-RDG40 and IN-Q7H09 were not resolved in the routine HPLC method. A modified method was used during LC-MS analysis to resolve these components in liver but not other tissues due to low residue levels. 2 minor unidentified hydroxy components were also present at similar retention time to IN-RDG40 and IN-Q7H09

[b] Extract profiled by HPLC and shown to contain 4 clear peaks which were a peak at Rt 32.3 min (1.7% TRR; 0.016 mg eq/kg), a peak at Rt 36.5 min (0.4% TRR; 0.004 mg eq/kg), IN-RAB06 (0.8% TRR; 0.008 mg eq/kg) and IN-QFD61 (1.4% TRR; 0.013 mg eq/kg) as well as an elevated region of unresolved radioactivity throughout the chromatogram which was postulated to contain numerous low level components

[c] Extract profiled by HPLC and shown to contain 4 clear peaks which were a peak at Rt 31.5 min (1.4% TRR; 0.014 mg eq/kg) and IN-QFD61 (1.0% TRR; 0.009 mg eq/kg) as well as an elevated region of unresolved radioactivity throughout the chromatogram which was postulated to contain numerous low level components

The ratio of oxathiapiprolin enantiomers (IN-Q7N24 and IN-Q7N25) was assessed by chromatographic analysis of the peak assigned to oxathiapiprolin using a chiral HPLC system. The non-radiolabeled oxathiapiprolin reference standard, each radiolabeled form of oxathiapiprolin and extracts were analysed. Analysis of the composite faeces extract from the goat dosed with pyrazole label and the liver extract from the goat dosed with thiazole label confirmed the ratio of enantiomers of the parent compound remained unchanged in the goat tissue and faeces.

The proposed metabolic pathway of oxathiapiprolin in the goat occurs primarily through hydroxylation of the pyrazole methyl moiety and subsequent oxidation to form a carboxylic acid. Hydroxylation in the 3- and 4- positions of phenyl ring carbons resulted in IN-Q7H09 and IN-RDG40. Hydroxylation in the isoxazoline-5 carbon followed by dehydration resulted in IN-Q7D41. Hydroxylation at the piperidine-2 carbon followed by ring opening liberates an amide metabolite, IN-WR791. IN-E8S72 was formed either by cleavage of IN-RAB06 at the bond between the bridge methylene and pyrazole moiety or by loss of $-CH_2CO_2H$ from IN-WR791 followed by hydroxylation of pyrazole methyl carbon and further oxidation to acid.

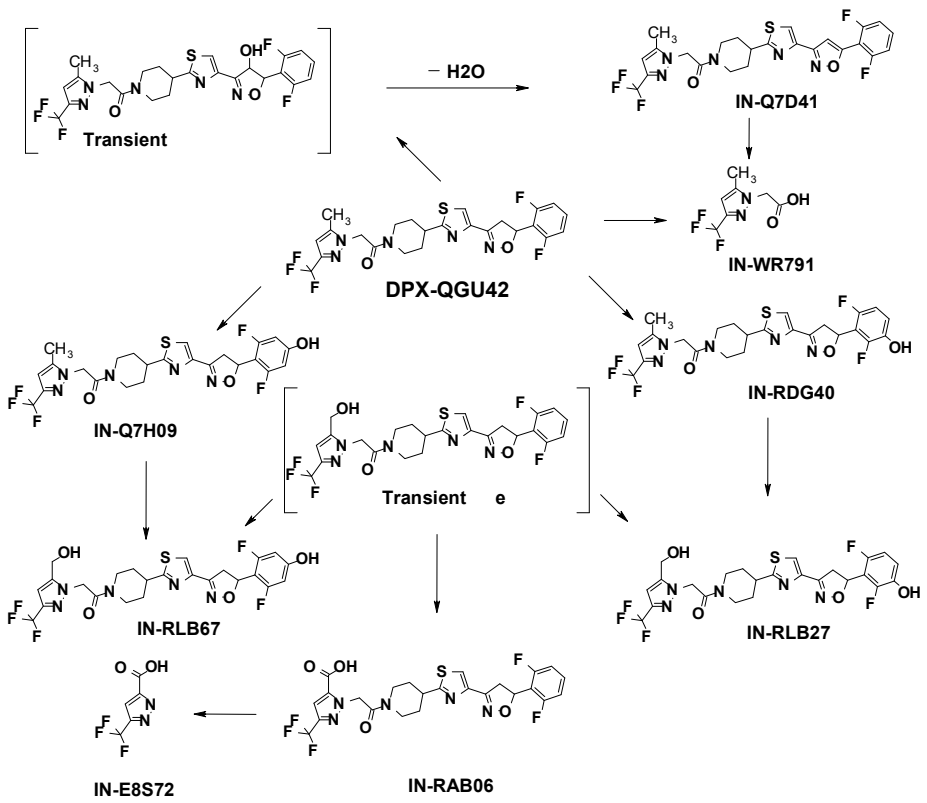


Figure 8 Proposed metabolic pathways for oxathiapiprolin (DPX-QGU42) in lactating goat

Lactating goats - IN-SXS67

A study examining the fate of IN-SXS67 (the IN-E8S72 glucose conjugate found in plants) in a lactating goat was reported by Dohn & Woodbury, 2013 [Ref: DP-34913]. In this study, one goat was dosed orally with [pyrazole-^{14}C]-IN-SXS67 for 7 consecutive days with single daily doses of 18.95 ppm in the diet (based on an average feed consumption of 1.37 kg dry matter/day and a dose rate of 26 mg/animal/day) and the animal was sacrificed 6 hours after administration of the last dose. The goat was in good health throughout the acclimatisation and dosing periods and there was no significant loss in the body weight during the study period (35.9 kg at dose initiation to 33.6 kg at termination).

Tissue samples were homogenised in dry ice, mixed with 2 mL of tissue solubiliser and incubated at 50 °C for about 18 hours before analysis by LSC. Triplicate aliquots of milk, urine, and cage wash samples were analysed for total radioactivity by LSC. Tissue samples were also extracted with 1:1, v/v acetonitrile:water (2 × 100 mL) followed by acetonitrile (100 mL) and analysed for radioactivity by LSC. Radioactivity remaining in the pellets was determined by combustion analysis following the final extraction. The liver extracts were purified by C18 solid phase extraction prior to LC/MS analysis.

Administered radioactivity (AR) was rapidly eliminated via urine (26% of the dose) and feces (59% of the dose). The GI tract contained 12.5% of the dose at sacrifice. About 0.3% AR was found in milk and edible tissues. Total recovery of administered radiocarbon was 97.7%.

The TRR in milk plateaued within 2 days after the first dose and remained at low concentrations during the entire dosing period (0.002 to 0.004 mg eq/kg). Residues in muscle (0.005 mg eq/kg) and fat (0.002 to 0.006 mg eq/kg) were also very low indicting no potential for IN-SXS67 and its metabolite to accumulate in these tissues. A composite milk sample was separated into milk fat and skim milk, and both fractions had equivalent residues (0.003 mg eq/kg). Residues in liver (0.038 mg eq/kg) and kidney (0.48 mg eq/kg) were higher and were extracted and analysed by HPLC and HPLC/MS within 57 days of sacrifice.

Aqueous acetonitrile extraction was able to release the majority of the radioactivity in liver (92% TRR – 0.035 mg eq/kg) and kidney (96% TRR – 0.46 mg eq/kg). Residues in the post-extracted solids (PES) of liver and kidney were 0.003 mg eq/kg (7.9% TRR) and 0.019 mg eq/kg (3.9% TRR), respectively and were not investigated further.

HPLC analysis of liver and kidney extracts identified two components, unchanged IN-SXS67 and its aglycone metabolite, IN-E8S72. IN-SXS67 made up 77% TRR (0.029 mg eq/kg) in liver and 58% TRR in kidney (0.28 mg eq/kg) while IN-E8S72 accounted for 16% TRR (0.006 mg eq/kg) in liver and 39% TRR (0.19 mg eq/kg) in kidney.

Table 16 Characterisation and identification of radioactivity in liver and kidney from a lactating goat dosed with 19 ppm [pyrazole-^{14}C]-IN-SXS67 in the diet

	Liver		Kidney	
	mg eq/kg	% TRR	mg eq/kg	% TRR
IN-SXS67	0.029	77.3	0.278	57.6
IN-E8S72	0.006	15.8	0.186	38.5
Subtotal in acetonitrile:water extracts	0.035	92.1	0.464	96.1
PES	0.003	7.9	0.019	3.9
Total	0.038	100	0.483	100

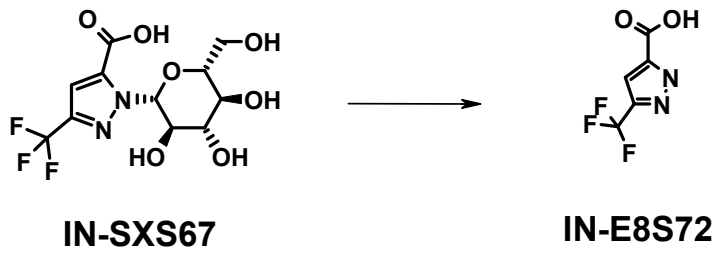


Figure 9 Proposed metabolic pathway for IN-SXS67 in the lactating goat

Laying hens

In a study reported by Lowrie et al, 2013 [Ref: DP-28244], two groups (5 hens/group) of laying hens were dosed orally by capsule once a day (in the morning) for 14 consecutive days with either [pyrazole-^{14}C] or [thiazole-^{14}C]-oxathiapiprolin at doses averaging 2.42 and 2.39 mg/hen/day, respectively. These dose levels are equivalent to 17.4 and 17.8 mg/kg of oxathiapiprolin in the diet, based on the average feed consumption of 138–143 g/day during the dosing period and body weights of 1.34–1.92 kg).

Egg production was normal, with generally 1 egg produced per day, and there were no adverse clinical findings during dosing. Excreta were collected daily from each animal, and cage wash samples (1:1 water/acetonitrile) were obtained at sacrifice. Eggs were collected twice daily from each hen. Approximately 6 hours after the final dose, the hens were sacrificed. The entire liver was collected from each hen, and equal portions of composite fat (skin with fat and abdominal fat) and muscle (thigh and breast) were collected from each hen. The remaining carcass of each hen was kept frozen until the completion of the study. All tissue samples were chopped, flash-frozen, and stored at -20°C for up to 6 months before extraction and analysis.

Tissue samples were extracted with acetonitrile (4:1, v/v) and acetonitrile/water (1:1, v/v). Whole egg and fat samples were extracted with dichloromethane, acetonitrile, and acetonitrile/water (4:1 v/v). The resulting extracts were concentrated, and analysed by LSC and HPLC. The liver post extracted solids (PES) were further treated with protease (37 °C; 120 h) to release unextracted residues and residues extracted with acetonitrile and analysed by LSC and HPLC.

Radioactive residues in the extracts were analysed by HPLC, using a C18 column with a mobile phase gradient of 10 mM ammonium acetate and acetonitrile. Radioactive residues were

quantified by LSC of the collected HPLC fractions. Residues were initially identified by comparison of their retention times with the retention times for reference standards. The initial acetonitrile/water extracts from excreta and liver samples were used for further isolation and confirmation of metabolite identities and analysed by HPLC/LC-MS, then compared to the reference standards. Chiral column analysis was used to determine the enantiomer ratio of oxathiapiprolin isolates from hen faeces and liver extract.

The radioactivity recovery was 95–100% of the total administered dose with 95-100% AR being recovered from the excreta and cage wash. About 0.02% of the dose was recovered in edible tissues and another 0.01 - 0.02% in the eggs from the [pyrazole-^{14}C]- and the [thiazole-^{14}C]- dose groups respectively. Radioactivity plateaued in whole eggs within 6 days from the start of dosing.

TRRs in edible tissues and eggs from both the radiolabels were low (less than 0.03 mg eq/kg) except for the liver which had higher residues (0.1mg eq/kg).

Table 17 Total radioactive residues (TRR) in excreta, eggs, cage wash, and tissues following daily oral administration of 17.6 ppm (diet) [^{14}C]-oxathiapiprolin to laying hens for 14 consecutive days

Sample, composite	[Pyrazole-^{14}C]-label		[Thiazole-^{14}C]-label	
	TRR (mg eq/kg)	% Administered dose	TRR (mg eq/kg)	% Administered dose
Excreta	N/A	97.94	N/A	91.89
Cagewash	N/A	2.42	N/A	2.93
Whole egg	0.012	0.02	0.008	0.01
Partially formed eggs	0.031	0.01	0.020	< 0.01
Liver	0.096	0.02	0.103	0.02
Muscle [a]	0.003	< 0.01	0.003	< 0.01
Skin with fat [a]	0.016	< 0.01	0.011	< 0.01
Abdominal fat [a]	0.030	< 0.01	0.024	< 0.01
Total recovery	N/A	100.41	N/A	94.85

[a] Total muscle mass was assumed to be approximately 25% of body weight, total fat approximately 12% of body weight

The initial dichloromethane, acetonitrile, acetonitrile/water extractions released more than 79% TRR from skin+fat, abdominal fat and [pyrazole-^{14}C]-label eggs and 46–56% TRR from liver and [thiazole-^{14}C]-label eggs. Subsequent protease treatments released another 21–54% TRR from liver and from [thiazole-^{14}C]-label eggs. The post extracted solids contained 21% TRR (0.003–0.005 mg eq/kg) in eggs and up to 13% TRR in tissues (0.002 mg eq/kg).

The majority of radioactive residues (37.5–77% pyrazole-derived TRR and 20–100% thiazole-derived TRR) was identified and/or characterised in tissues and eggs.

IN-RAB06 was the only identified component present at more than 0.01 mg eq/kg, detected only in liver at 0.007–0.014 mg eq/kg (7.7–13.5% TRR).

While oxathiapiprolin was found in all tissues and eggs, levels were all not more than 0.01 mg/kg, making up 10–22% TRR (0.002–0.003 mg/kg) in eggs, 4.0–8.2% TRR (0.004–0.008) mg/kg in liver, 28–66% TRR (0.01 mg/kg) in abdominal fat, and 22–37% TRR (0.003–0.004) mg/kg in skin with fat.

The hydroxy metabolites IN-RDG40 and IN-Q7H09 together accounted for 25–33% TRR (0.003–0.004 mg eq/kg) in skin+fat, 15% TRR (0.005 mg eq/kg) in abdominal fat, up to 7.6% TRR (0.007 mg eq/kg) in liver and 3–5% TRR in eggs. IN-Q7D41 was detected in eggs and fats accounting for 5–10% TRR (0.001-0.002 mg eq/kg) but was not observed in liver. Metabolite IN-QFD61 was reported as a significant component of the liver protease enzyme extract (34–38% TRR – 0.03–0.04 mg eq/kg).

Minor unidentified metabolites accounted for a combined total of 1.5–20% TRR (0.001–0.022 mg eq/kg) in eggs and tissues, with no individual metabolite greater than 8.6% TRR.

Table 18 Characterisation and identification of radioactivity in eggs and tissues from laying hens dosed with [^{14}C]-oxathiapiprolin

Sample	Whole egg		Liver		Abdominal fat		Skin with fat	
Fraction	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
[Pyrazole-^{14}C]-label								
Oxathiapiprolin	21.9	0.003	8.2	0.008	27.5	0.009	21.9	0.003
IN-RAB06	ND	ND	7.7	0.007	ND	ND	ND	ND
IN-Q7D41	7.1	0.001	ND	ND	4.9	0.002	7.4	0.001
Hydroxy metabolites [a] IN-RDG40/IN-Q7H09	4.8	0.001	7.6	0.007	15.4	0.005	33.2	0.004
Unidentified metabolites [b]	3.8	0.001	9.8	0.010	10.7	0.004	11.8	0.001
Total characterised	37.6	0.006	76.6	0.073	58.5	0.020	74.3	0.009
Organo-soluble fraction [c]	41.6	0.007	2.9	0.003	28.3	0.009	13.8	0.002
ACN/water extracts	78.9	0.013	45.9	0.044	94.2	0.03	88.1	0.012
Protease enzyme extract [d]	NA	NA	54.1 [e]	0.052 [e]	NA	NA	NA	NA
Unextracted	21.1	0.003	< 0.1	< 0.001	5.8	0.002	11.9	0.002
Total	100.0	0.016	100.0	0.096	100.0	0.032	100.0	0.013
[Thiazole-^{14}C]-label								
Oxathiapiprolin	10.1	0.002	4.0	0.004	66.2	0.010	36.9	0.004
IN-RAB06	ND	ND	13.5	0.014	ND	ND	ND	ND
IN-QFD61	ND	ND	9.8	0.010	ND	ND	ND	ND
IN-Q7D41	5.6	0.001	ND	ND	ND	ND	10.1	0.001
Hydroxy metabolites [a] IN-RDG40/IN-Q7H09	3.0	0.001	4.2	0.004	ND	ND	25.3	0.003
Unidentified metabolites [b]	1.5	< 0.001	20.3	0.022	ND	ND	ND	ND
Total characterised	20.2	0.004	91.0	0.093	66.2	0.010	79.9	0.008
Organo-soluble fraction [c]	23.5	0.006	1.8	0.002	22.0	0.003	6.9	0.001
ACN/water extracts	56.0	0.015	53.9	0.055	88.2	0.013	86.7	0.008
Protease enzyme extract [d]	20.6	0.005	46.1 [e]	0.047 [e]	NA	NA	NA	NA
Unextracted	20.5	0.005	< 0.1	< 0.001	11.8	0.002	13.2	0.001
Total	97.1	0.025	100.0	0.102	100.0	0.015	100.0	0.009

[a] Reference standards IN-RDG40 and IN-Q7H09 were not fully resolved by HPLC in the most tissue extracts

[b] Sum of all other minor (< 0.010 mg eq/kg) unidentified radioactive regions in chromatograms

[c] Radioactivity partitioned into hexane prior to and following concentration which could not be further characterized

[d] Tissue debris remaining following initial solvent extraction was treated with protease enzyme and further extracted

[e] Liver extract profiled by HPLC and shown to contain a single significant radiolabeled component, which was assigned as IN-QFD61 (33.5-38.3% TRR; 0.032-0.039 mg eq/kg)

The ratio of oxathiapiprolin enantiomers (IN-Q7N24 and IN-Q7N25) was assessed by chromatographic analysis of the peak assigned to oxathiapiprolin using a chiral HPLC system. The non-radiolabeled oxathiapiprolin reference standard, each radiolabeled form of oxathiapiprolin and extracts were analysed. Analysis of the liver extract from hens dosed with pyrazole-label and the excreta extract from hens dosed with thiazole-label confirmed the ratio of enantiomers of oxathiapiprolin remained unchanged in the hen tissue and excreta.

Biotransformation of oxathiapiprolin in the hen occurred primarily through hydroxylation of the phenyl moiety to form metabolites IN-Q7H09 and IN-RDG40. Hydroxylation at piperidine-2-carbon in the IN-Q7H09 followed by ring opening resulted in the formation of IN-RLB26. Hydroxylation and subsequent oxidation of the pyrazole methyl carbon of oxathiapiprolin gave the corresponding carboxylic acid metabolite, IN-RAB06. Hydroxylation at isoxazoline-5-carbon of oxathiapiprolin followed by dehydration within the isoxazoline ring resulted in the formation of isoxazole in the metabolite, IN-Q7D41.

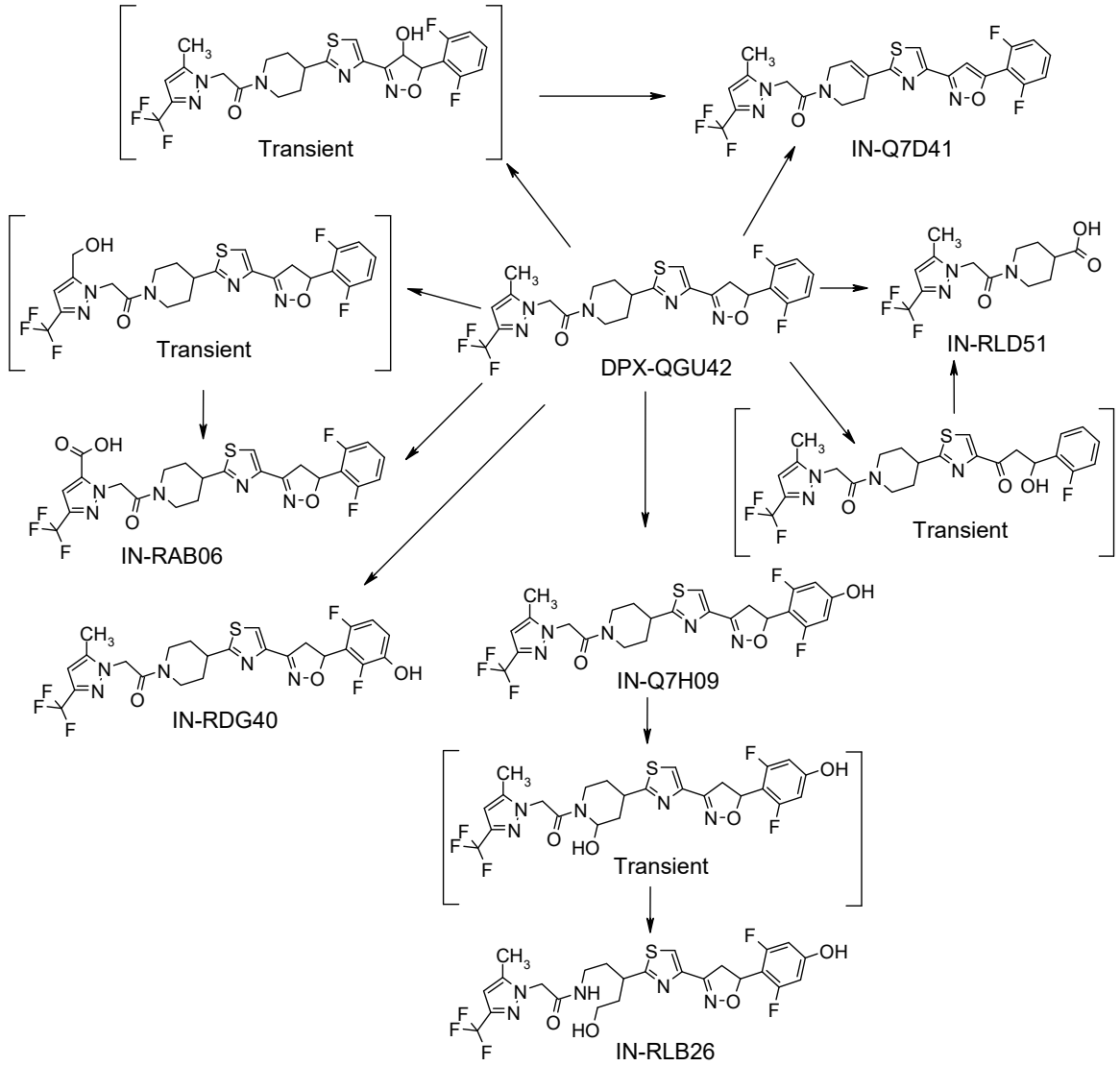


Figure 10 Proposed metabolic pathways for oxathiapiprolin (DPX-QGU42) in laying hen

In summary, about 1.2% of the applied dose remained in goat tissues and about 0.02% remained in hen tissues, with 0.1–0.2% of the dose being eliminated in eggs and milk respectively.

Residues of oxathiapiprolin were present in most tissues including milk and eggs, highest levels (about 0.01 mg/kg) being found in liver, kidney and fat.

Metabolites found at greater than 10% TRR and more than 0.01 mg eq/kg in the various goat tissues and/or milk were the hydroxy metabolites IN-RDG40 and IN-Q7H09 in liver (totalling up to 13% TRR – 0.13 mg eq/kg) and kidney (totalling up to 21% TRR – 0.01 mg eq/kg) and IN-E8S72 in kidney (up to 24% TRR – 0.02 mg eq/kg).

In poultry tissues, the only significant metabolites were the combined hydroxy metabolites (IN-RDG40 and IN-Q7H09) in skin and fat (15–33% TRR - 0.003–0.005 mg eq/kg) and IN-RAB06 in liver, found at up to 13.5% TRR (0.014 mg eq/kg).

Environmental fate

The Meeting received information the environmental fate and behaviour of oxathiapiprolin, including hydrolytic stability, photochemical degradation in soils and aerobic metabolism studies.

Hydrolysis

The hydrolytic degradation of oxathiapiprolin was investigated at pH 4, 7 and 9 using either [thiazole-^{14}C]-oxathiapiprolin or [pyrazole-^{14}C]-oxathiapiprolin and reported by Anand, 2010 [Ref: DP-28424].

Radiolabeled oxathiapiprolin (0.1 mg/litre) was incubated in the dark in sterile aqueous buffered solutions at pH 4, 7, and 9 for 5 days at 50 °C. Dimethyl formamide (1%) was used as a co-solvent. Samples were taken at 0 and 5 days and analysed for total radioactivity by LSC. HPLC was used to determine the levels of unchanged oxathiapiprolin.

No significant decline of oxathiapiprolin was observed at the end of the 5-day incubation period. Oxathiapiprolin residues were 94–102% AR in the pH 4 samples, 94.5–98% AR in the pH 7 samples and 91–92% AR in the pH 9 samples. No major transformation products were observed.

Hydrolytic degradation of oxathiapiprolin in pH 4, 7 and 9 buffer solutions was less than 10% after 5 days of incubation at 50ºC. Oxathiapiprolin can be considered hydrolytically stable.

Photochemical degradation in soil

Artificial sunlight photodegradation of [pyrazole-^{14}C]-oxathiapiprolin and [isoxazoline-^{14}C]-oxathiapiprolin in a 'Sassafras' sandy loam soil (pH 5.3) was investigated in a study reported by Cleland, 2013 [Ref: DP-28075].

Thin layers of soil, *ca.* 2 mm under moist (6.75–9% moisture content) and dry conditions, were treated with oxathiapiprolin to obtain a soil concentration of 0.2 mgkg. The soils were continuously irradiated for up to 15 days with simulated sunlight (xenon arc), maintained at 20 ± 2 °C and sampled at intervals up to 15 days after application. Controls were incubated at 20 ± 2 °C in the dark simultaneously. Soil samples were sequentially extracted twice with acetonitrile:water (90:10), then acetonitrile:0.1 M ammonium carbonate (70:30), and acetonitrile:0.1% formic acid (70:30). Residues in the combined extracts were measured by reversed-phase HPLC with radiochemical detection. Identification of unchanged oxathiapiprolin was by co-chromatography using reversed-phase HPLC and LC/MS-MS. Non-extractable residues were quantified by combustion analysis. The average mass balance was greater than 95% applied radiolabel.

At the end of the 15-day study period, about 87–88% AR was extracted from the moist irradiated soil samples and about 84–97% AR from the dry irradiated soil samples. No quantifiable levels of evolved $^{14}CO_2$ or non-specific volatile organic components were found.

HPLC analysis of the 15-day moist non-irradiated soil extracts showed that [^{14}C]-oxathiapiprolin declined to a mean value of about 99% AR. In the irradiated soil, [^{14}C]-oxathiapiprolin levels has declined to about 80% in the dry soil extracts and to about 70% AR in the moist soil extracts at the end of the study period.

Three metabolites were found at levels above 5% AR, IN-RDT31 (max 5.7% AR in moist soil) and IN-E8S72 (max 6.4% AR in moist soil), IN-RAB06 (max 5.2% TRR in dry soil) with at least 15 minor transformation products also being detected, all below 5% AR at two consecutive sampling intervals or 10% AR at a single sampling interval.

Degradation of oxathiapiprolin under irradiated conditions was faster than in the non-irradiated soils. The degradation was similar under both moist and dry conditions. Calculated DT_{50} values (SFO model) for oxathiapiprolin in the irradiated soil were 28.2 days (moist soil) and 36.3 days (dry soil) and the DT_{90} values were 93.5 days and 120.7 days respectively.

Aerobic soil metabolism

The biotransformation of [^{14C}]-oxathiapiprolin was studied in a loamy sand soil (Sassafras) under aerobic conditions in two studies reported by Cleland, 2013 [Ref: DP-28071] and McCorquodale, 2013 [Ref: DP-29443]. Soil samples were mixed with [pyrazole-^{14}C]-oxathiapiprolin, [isoxazoline-^{14}C]-oxathiapiprolin or [thiazole-^{14}C]-oxathiapiprolin at a rate of 0.2 mg/kg and incubated for 120–134 days in darkness at 20 ± 2 °C.

A similar study with 4 different soils treated with [pyrazole-^{14}C]-oxathiapiprolin or [thiazole-^{14}C]-oxathiapiprolin at a rate of 0.2 mg/kg and incubated under similar conditions for 120 days was reported by Manjunatha, 2011 [Ref: DP-28072].

Table 19 Characteristics of the soils used in the oxathiapiprolin aerobic soil metabolism studies.

Soil characteristics	Sassafras	Nambsheim	Tama	Lleida	Speyer 2.2
Soil type	Loamy sand	Sandy Loam	Silty clay loam	Clay loam	Sand
Sand (%)	80	66	17	26	89
Silt (%)	17	23	52	35	10
Clay (%)	3	11	31	39	1
Organic carbon (%)	0.81	1.6	2.5	1.2	1.9
CEC (meq/100 g)	5.4	10.1	22.1	27.2	8
pH (H_2O)	5.3	7.6	6.8	7.9	6.1
Moisture content (%)	10.7%	26.3	39.6	32.3	24.3
Microbial biomass (day 0)	90.1	1078	685	1394	651
Microbial biomass (day 120)	69.5	1106	706	1418	673
Bulk density (g/cm^3)	1.29	1.13	1.01	1.07	1.19

The test systems consisted of separate sets of glass incubation vessels for each radiolabel containing a sample of soil (50 g oven dry equivalent) in individual flow-through systems with traps for the collection of CO_2 and volatile organic compounds. A single test vessel from each radiolabel (total two samples per sampling interval) was removed for analysis at multiple sampling intervals. The soil samples were extracted with a multi-step extraction procedure consisting of two acetonitrile: water (90:10) extractions, one of acetonitrile:0.1 M ammonium carbonate (70:30), and one acetonitrile:0.1% formic acid (aq) (70:30). The extracts were combined and the composition of radioactivity was determined by reversed-phase HPLC with radiochemical detection. Identification of metabolites was performed by co-chromatography with standard on reversed-phase HPLC and LC/MS-MS. Non-extractable residues were quantified by combustion analysis.

In the five soils tested the material balances were quantitative in the range of 90–110% of applied radioactivity (% AR). Extractable radioactivity recovered from soil as oxathiapiprolin was quantitative at zero time, and decreased with time with 7–38% AR being unextracted at the end of the study period. Volatile organics were not produced in significant amounts. The amount of $^{14}CO_2$ evolved was significant (ranging from <LOQ to 11.8% AR), demonstrating that oxathiapiprolin is ultimately mineralized in soil. Chiral HPLC analysis in one of these studies showed no change in the isomer ratio over the study period.

Oxathiapiprolin residues remaining after 120 days ranged from 17–77% AR in the five soils with at least 15 extractable degradates being observed. The major transformation product (up to 13% AR) was IN-RAB06. Other degradates included IN-RDT31 and IN-QPS10 (up to 9% AR) and IN-E8S72 (up to 7% AR). The remaining unidentified components individually did not exceed 10% AR at any sampling interval or 5% AR at two consecutive sampling intervals and were not increasing at the end of the study. A summary of the range of parent and identified degradate levels found in the five different soils is presented below.

Table 20 Aerobic degradation of [^{14}C]-oxathiapiprolin incubated in five soils at 20 °C

Component	% Applied radioactivity (range)							
	Day 0	Day 3	Day 7	Day 14-15	Day 28-30	Day 60	Day 90	Day 120
[Pyrazole-^{14}C]-label								
Oxathiapiprolin	95-103	94-95	62-88	56-87	50-83	46-64	36-54	20-50
IN-RDT31	ND-3.3	1.7	<LOQ-6.2	1.9-4.7	2.1-5.0	3.8-7.0	4.2-9.1	4.2-9.4
IN-RAB06	ND-1.6	ND-1.3	2.6-4.9	1.5-5.0	2.4-5.2	4.1-11	5.4-13	3.8-10
IN-Q7D41	ND-0.8	ND	<LOQ	ND-1.5	ND-0.7	<LOQ-1.5	<LOQ-1.6	1.2-1.6
IN-RLD51	<LOQ		<LOQ	<LOQ-2.3	<LOQ-2.0	<LOQ-3.6	1.2-3.2	<LOQ-3.3
IN-E8S72	ND-<LOQ	ND	<LOQ-4.8	ND-3.4	ND-4.9	2.6-6.3	1.3-6.2	1.9-6.7
Unidentified	ND-2.2	1.9-6.0	9.7-18	1.6-17	5.5-22	7.7-23	8.1-27	10-30
Unextracted	<LOQ	<LOQ-1.2	2.6-8.2	1.4-11	2.6-7.6	6.0-14	8.6-22	9.9-34

Component	% Applied radioactivity (range)							
	Day 0	Day 3	Day 7	Day 14-15	Day 28-30	Day 60	Day 90	Day 120
CO_2		<LOQ		<LOQ-0.39	<LOQ-1.2	<LOQ-3.3	<LOQ-5.1	<LOQ-6.8
Recovery	99-105	99-104	95-104	95-104	94-102	92-104	91-103	92-103
[Thiazole-^{14}C]-label								
Oxathiapiprolin	94-95	88	75-82	41-90	31-79	19-80	17-69	17-68
IN-RDT31	ND-3.2	1.4	3.5-8.0	2.3-5.3	2.6-5.5	2.3-4.8	<LOQ-5.3	3.8-6.7
IN-RAB06	<LOQ-3.1	1.5	3.4-5.1	1.6-5.8	3.2-7.0	3.8-6.4	2.0-6.8	2.3-7.7
IN-Q7D41	ND-<LOQ	ND	<LOQ	ND-<LOQ	<LOQ-1.2	<LOQ-1.5	<LOQ-2.5	<LOQ-2.5
IN-QPS10	ND-<LOQ	ND	ND-3.1	ND-4.0	ND-6.8	<LOQ-8.7	<LOQ-7.2	<LOQ-6.3
Unidentified	<LOQ-1.6	4.3	<LOQ-7.1	2.0-16	8.7-19	2.6-23	13-28	9.4-21
Unextracted	<LOQ	1.1	1.8-11	2.8-25	2.2-32	4.2-43	8.8-43	6.7-38
CO_2		0.29	<LOQ	<LOQ-3.6	<LOQ-7.3	<LOQ-8.7	<LOQ-10	<LOQ-12
Recovery	94-97	97	93-106	93-101	92-108	92-99	83-103	92-96
[Isoxazoline-^{14}C]-label								
Oxathiapiprolin	108	95		89	82	82	59	76 (77)[a]
IN-RDT31	ND	ND		1.1	2.9	3.4	8.8	7.1 (6.0)[a]
IN-RAB06	ND	ND		2.4	4.8	4.4	7.6	1.6 (0.37)[a]
IN-Q7D41	ND	ND		ND	ND	0.67	1.4	1.6 (1.4)[a]
IN-QPS10	ND	ND		ND	ND	0.69	2.9	0.99 (1.2)[a]
Unidentified	ND	ND		1.6	5.6	ND	4.9	1.7 (6.2)[a]
Total extracted	108	95		94	95	91	85	89 (92)[a]
Unextracted	<LOQ	<LOQ		1.9	2.9	4.6	9.4	8.2 (8.1)[a]
CO_2		<LOQ		0.88	1.7	2.5	3.3	4.0 (5.0)[a]
Recovery	108	95		97	100	99	97	101 (105)[a]

[a] = %AR levels in soil extracts after 134 days incubation

The degradation of oxathiapiprolin in soil proceeded along multiple primarily biotic pathways including:

- Cleavage of the pyrazole ring to form IN-QPS10 and IN-E8S72.
- Cleavage of the thiazole ring, generating CO_2 and multiple minor degradates including IN-RLD51 which undergoes rapid oxidation to form IN-WR791 and subsequent hydroxylation and multiple oxidations to form IN-E8S72.
- Hydroxylation of the methyl group on the pyrazole ring followed by oxidation to IN-RAB06.
- Hydroxylation of the piperidine ring (at the 4-position) to form IN-RDT31, with subsequent cleavage to form IN-WR791 which then degrades to IN-E8S72.

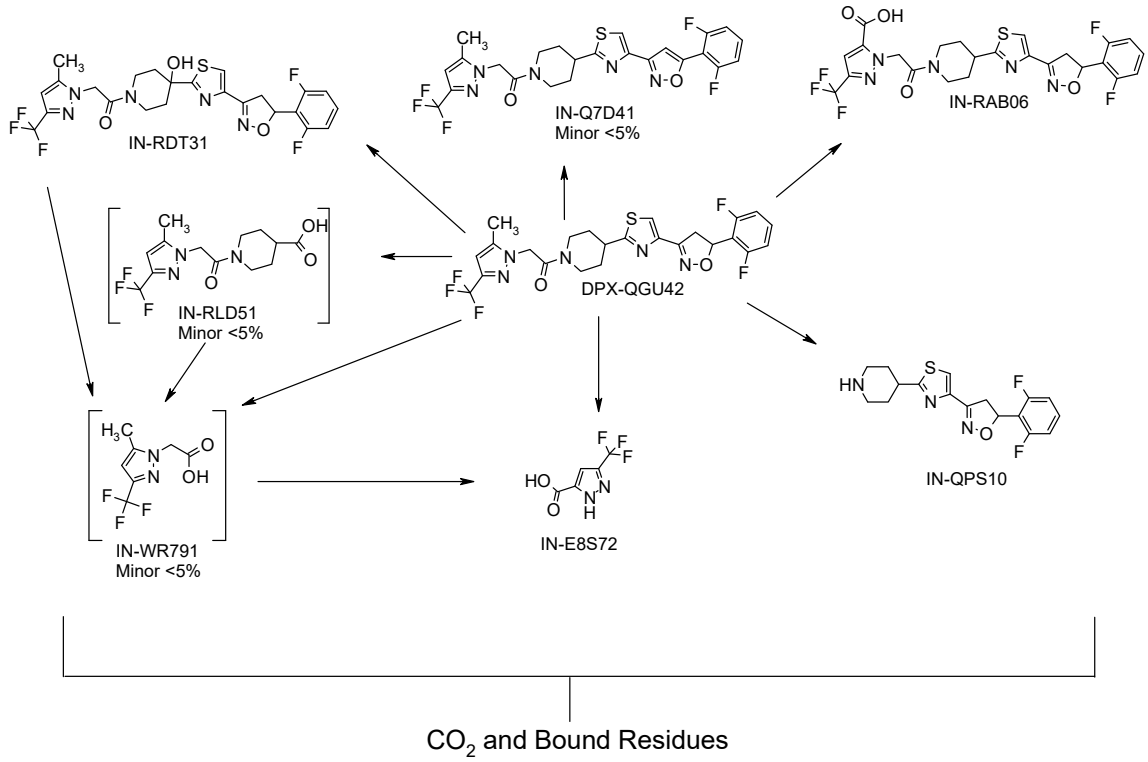


Figure 11 Degradation pathways for aerobic degradation of oxathiapiprolin (DPX-QGU42) in soil

Studies on the degradation rate of [^{14}C]-oxathiapiprolin in the above five aerobic soils in the dark at 20 °C were reported by Khanijo *et al*, 2015 [Ref: DP-31761]. Soil samples were adjusted to 50% maximum water holding capacity at 0 Bar and treated with thiazole-, pyrazole- or isoxazoline-labelled oxathiapiprolin at a rate equivalent to 0.2 mg/kg soil dry weight. Calculated degradation rates are summarized below:

Table 21 Aerobic degradation rates of [^{14}C]-oxathiapiprolin incubated in five soils at 20 °C.

Radiolabel position	Soil	DT_{50} (days)	DT_{90} (days)	pH	Model
Thiazole and Pyrazole	Sassafras	89	626	5.3	DFOP
Pyrazole and Isoxazoline	Sassafras	131	434	5.3	SFO
Thiazole and Pyrazole	Lleida	59	197	7.9	SFO
Thiazole and Pyrazole	Nambsheim	134	639	7.6	HS
Thiazole and Pyrazole	Speyer 2.2	116	386	6.1	SFO
Thiazole and Pyrazole	Tama	18	1224	6.8	FOMC
	Geometric mean	76	503		

Degradation studies were also conducted with the four degradates where residues were either above 10% AR (IN-RAB06) or exceeded 5% AR at two or more consecutive sampling points (IN-RDT31, IN-QPS10 and IN-E8S72). Four to five soils were used in these studies, with treated soils incubated in the dark at 20 ºC. The results of these studies were summarized by Khanijo *et al*, 2015 [Ref: DP-31761] and the calculated degradation rates are summarized below:

Table 22 Aerobic degradation rates of [^{14}C]-oxathiapiprolin degradates incubated in five soils at 20 °C

Compound	Soil	Texture	pH	% OC	DT_{50} (days)	DT_{90} (days)	Reference
IN-E8S72	Speyer 2.2	Loamy sand	5.3	2.9	477	1586	DP-32061
	Lleida	Clay	7.8	3.2	272	903	
	Tama	Silty clay loam	6.6	3.8	216	718	

Compound	Soil	Texture	pH	% OC	DT_{50} (days)	DT_{90} (days)	Reference
	Nambsheim	Sandy loam	7.8	2.6	328	1091	
	Sassafras	Sandy loam	5.4	2.0	380	1261	
	Geometric mean				323	1072	
IN-QPS10	Nambsheim	Sandy loam	7.6	2.9	3	171	DP-32065
	Lleida	Clay	7.5	3.5	19	193	
	Speyer	Loamy sand	5.5	3.2	300	1723	
	Sassafras	Sandy loam	5.4	5.4	310	2267	
	Geometric mean				48	599	
IN-RAB06	Speyer 2.2	Loamy sand	5.4	2.8	84	347	DP-35533
	Lleida	Silty clay loam	6.2	3.9	52	258	
	Tama	Clay	7.7	3.3	75	248	
	Nambsheim	Sandy loam	7.6	2.8	39	214	
	Cajon	Sandy loam	7.6	1.2	67	226	
	Tama	Silty clay loam	6.3	3.3	10	167	DP-32060
	Lleida	Clay	7.7	4.1	9	64	
	Sassafras	Loam	5.5	2.6	101	604	
	Nambsheim	Sandy loam	7.7	2.9	4	59	
	Speyer	Loamy sand	5.5	5.9	172	679	
	Geometric mean				37	221	
IN-RDT31	Nambsheim	Sandy loam	7.6	2.9	46	235	DP-32062
	Tama	Silty clay loam	5.9	3.3	152	979	
	Lleida	Clay	7.5	3.5	50	223	
	Speyer	Loamy sand	5.5	3.2	736	3652	
	Sassafras	Sandy loam	5.4	5.4	216	1160	
	Geometric mean				141	737	

Ten field-based soil degradation studies (four in Europe and six in North America were conducted with oxathiapiprolin (OD formulation) and summarized by Khanijo *et al*., 2015 [Ref: DP-31761]. Bare soils were treated with either single applications of 0.2 kg ai/ha (European studies) or 1-2 applications of 0.5–0.77 kg ai/ha total (North American studies) and stratified soil samples were taken down to 90 cm at intervals up to 24 months. Samples were extracted with formic acid, water and acetonitrile and analysed for oxathiapiprolin and the IN-E8S72, IN-QPS10, IN-RAB06 and IN-RDT31 degradates by reverse phase UPLC separation coupled to tandem mass spectrometry (LC-MS/MS), with LOQs of 0.01 mg/kg for each analyte.

Degradation half-lives of oxathiapiprolin in the different soils ranged from 4–205 days (geometric mean of 26 days), with no apparent pH dependency (following FOCUS 2006, 2011) Guidelines. Oxathiapiprolin and its metabolites were generally confined to the upper soil segment (0–15 cm) with the highest concentration found in the 0–5 cm segment. Degradates of oxathiapiprolin were slightly more mobile through the soil depths but were rarely found below 15 cm depth in any significant amounts and were not found below 50 cm.

Table 23 Soil half-lives of oxathiapiprolin in field studies in Europe and North America [Ref: DP-31761]

Study location	%Sand:Silt:Clay	OM(%)	pH	Soil Texture	DT_{50} (days)	Model	Study
France, Lucenay	51:30:19	2.1	6.9	Loam	5.5	DFOP	DP-27214
Germany, Lentzke	77:18:5	1.1	6	Loamy sand	23.9	DFOP	DP-26404
Spain, Sevilla	80:9:11	1.4	8.1	Sandy loam	8.1	DFOP	DP-29820
UK, Cambridgeshire	46:24:30	18.2	7.4	Sandy clay loam	101	DFOP	DP-29819
USA, California	50:40:10	0.93	8.5	Loam	30	FOMC	DP-29823
USA, New York	33:50:17	3.3	5.1	Loam	3.9	DFOP	DP-29813
USA, Texas	50:20:30	0.77	8.3	Sandy clay loam	9.8	FOMC	DP-29818
USA, Florida	95:5:0	1.2	6.7	Sand	34.6	FOMC	DP-29817
Canada, Manitoba	46:39:15	7.4	7.9	Loam	205.3	SFO	DP-29814
Canada, BC	65:30:5	3.8	6.5	Sandy loam	169.6	SFO	DP-29816

Table 24 Soil half-lives of oxathiapiprolin degradates in field studies in Europe and North America [Ref: DP-31716]

Study location	IN-RDT31	IN-RAB06	IN-E8S72	IN-QPS10	Study
France, Lucenay	84.9	46.3	150.9	10003	DP-27214
Germany, Lentzke	135	187.9	140.5		DP-26404
Spain, Sevilla	120	50.4	69.1		DP-29820
UK, Cambridgeshire	190	61.9	137.9	241.5	DP-29819
USA, California	17.9	245.5		34.7	DP-29823
USA, New York	112	242.7	74	2000	DP-29813
USA, Texas	64.5	315.2			DP-29818
USA, Florida	50.9	48.1		201.2	DP-29817
Canada, Manitoba	44.5	142.5	88	17.9	DP-29814
Canada, BC	101.9	104.7			DP-29816

In summary, oxathiapiprolin is slightly soluble in water, stable to hydrolysis and is slightly mobile and moderately persistent in soil, with field half-lives of 5–205 days (mean DT_{50} 59 days). Major metabolites in soils are IN-RAB06 (max 13% AR, low-high persistence), IN-RDT31 (max 9.4% AR, moderate-very high persistence), IN-QPS10 (max 8.7% AR, low-high persistence) and IN-E8S72 (max 6.7% AR, high-very high persistence).

Residues of oxathiapiprolin, IN-RDT31, IN-QPS10 and IN-RAB06 generally remain within the top 15 cm with IN-E8S72 being more mobile. Photolysis does not appear to contribute significantly to the degradation of oxathiapiprolin under field conditions.

Rotational crop metabolism

The Meeting received information on the metabolism of oxathiapiprolin in wheat, lettuce and turnip grown as confined rotational crops and in a range of representative field crops grown in oxathiapiprolin-treated soil.

Confined rotational crop studies

Two confined rotational crop studies using wheat, lettuce and turnip were conducted with oxathiapiprolin labelled in the pyrazole-, thiazole- or the isoxazoline- rings at a rate equivalent to 0.21 kg ai/ha (Chapleo *et al*., 2013 [Ref: DP-28381]) or in the pyrazole- or the isoxazoline- rings at a rate equivalent to 0.6 kg ai/ha (Hobbs, 2013 [Ref: DP-31739]).

In these studies, the radiolabel was applied to bare sandy loam soil plots at rates equivalent to 0.21 kg ai/ha or 0.6 kg ai/ha. In the latter study, primary crops (courgettes, lettuce and potatoes) were sown on the day of application and sampled at intervals up to harvest. In both studies, the rotational crops were planted 30, 120 and 365 days after treatment and grown to maturity.

Crops were sampled, pulverised, and total radioactive residues (TRR) determined by combustion analysis or, following extraction, by summing radioactivity in the extracts and unextracted residues. Soil cores (to 30 cm) from the 0.21 kg ai/ha plots were also taken for analysis. Samples were stored for up to 53 days before extraction with acetonitrile and then with acetonitrile:water (3:1). Additional extraction methods were used for some samples, including water (ambient), acetone (*ca.* 40°C, ultrasonication), amylase (*ca.* 50 °C, 2 × 72 h), amyloglucosidase plus cellulase (*ca.* 50 °C, 2 × 48 h), 0.1 N sodium hydroxide (*ca.* 60 °C, 2 × 6 h), and 1.0 N hydrochloric acid (*ca.* 60 °C, 2 × 6 h).

Study 1 (0.21 kg ai/ha) [Ref: DP-28381]

Soil samples (30 cm cores) from the 120DAT [pyrazole^{14}C]-oxathiapiprolin and [thiazole^{14}C]-oxathiapiprolin plots were taken at wheat grain maturity (246 days after treatment) and analysed for TRR and oxathiapiprolin metabolites. Concentrations in the [pyrazole^{14}C]-oxathiapiprolin treated soil used to grow wheat, lettuce, and turnip were 0.252, 0.309, and 0.215 mg eq/kg, respectively.

Corresponding values for [thiazole^{14}C]-oxathiapiprolin treated soil were 0.527, 0.075 (believed to be an outlier), and 0.346 mg eq/kg, respectively.

Most of the soil radioactivity (72-78% TRR) was able to be extracted in acetonitrile:water (9:1). The predominant residue was oxathiapiprolin (8.8-20% TRR) with the main degradates being IN-Q7H09 (10–15% TRR) and an unidentified metabolite (12–14% TRR). Other identified components found at lower levels (less than 8% TRR) were IN-E8S72, IN-WR791, IN-RAB06, IN-RDG40, IN-KJ552 and IN-Q9L80.

Table 25 Radioactive residues in soil 246 days after a bare soil treatment of 0.21 kg ai/ha [^{14}C]-oxathiapiprolin (sampled after the 120 DAT wheat crop mature harvest).

	[Pyrazole^{14}C]-label treated soil		[Thiazole^{14}C]-label treated soil	
	% TRR	mg eq/kg	% TRR	mg eq/kg
Lettuce plots – TRR extracted	72.5	0.224	72.6	0.054
Turnip plots – TRR extracted	76.3	0.164	77.8	0.269
Wheat plots – TRR extracted	75.9	0.191	78.2	0.412
Identified/characterised				
Oxathiapiprolin	20.3	0.051	8.8	0.046
IN-E8S72	0.8	0.002	ND	ND
IN-WR791	1.2	0.003	ND	ND
IN-RAB06	3.9	0.010	2.5	0.013
IN-RDG40	4.3	0.011	3.2	0.017
IN-KJ552	4.7	0.012	ND	ND
IN-Q7H09	14.7	0.037	10.0	0.053
IN-Q9L80	ND	ND	7.5	0.039
ca. 56 min	13.8	0.035	11.8	0.062
Individual unidentified	≤2.0	≤0.005	≤3.7	≤0.020
Total unidentified	9.3	0.022	28.7	0.152
Total extractable	72.9	0.184	72.7	0.383
Unextracted	24.1	0.061	21.8	0.115

In food items (lettuce, turnip tubers, and wheat grain) from soil treated with either [isoxazoline^{14}C]-oxathiapiprolin or [thiazole^{14}C]-oxathiapiprolin, TRRs ranged from 0.003 to 0.013 mg eq/kg (in wheat grain) and in animal feed items, the TRRs ranged from 0.005 mg eq/kg to 0.06 mg eq/kg (in wheat straw).

Residues were higher in the samples from the soils treated with [pyrazole^{14}C]-oxathiapiprolin, up to 0.76 mg eq/kg in wheat straw, 0.26–0.3 mg eq/kg in wheat grain, forage and hay, 0.09–0.12 mg eq/kg in turnip tops, 0.028 mg eq/kg in lettuce, and 0.023 mg eq/kg turnip roots. The higher residues in the samples grown in the [pyrazole^{14}C] treated soil could reflect a higher root uptake of polar metabolites derived from the pyrazole moiety.

Table 26 Total radioactive residues (mg eq/kg) in rotational crops grown in soil treated with [^{14}C]-oxathiapiprolin at a rate equivalent to 0.21 kg ai/ha.

RAC	[Pyrazole^{14}C]-label (mg eq/kg)			[Thiazole^{14}C]-label (mg eq/kg)			[Isoxazoline^{14}C]-label (mg eq/kg)		
	30 DAT	120 DAT	365 DAT	30 DAT	120 DAT	365 DAT	30 DAT	120 DAT	365 DAT
Wheat grain	0.258	0.097	< 0.007	0.007	0.003	< 0.008	0.012	0.013	< 0.006
Immature lettuce	0.028	0.028	< 0.01	0.004	< 0.004	< 0.006	< 0.006	< 0.009	< 0.005
Mature lettuce	0.013	0.022	0.006	0.002	< 0.004	< 0.008	< 0.008	< 0.008	< 0.005
Turnip tubers	0.014	0.023	0.008	0.006	0.004	0.01	0.008	< 0.007	< 0.004
Wheat forage	0.269	0.172	0.022	0.013	0.01	< 0.009	0.007	< 0.01	< 0.006
Wheat hay	0.298	0.172	0.081	0.018	0.012	0.006	0.009	0.007	< 0.008
Wheat straw	0.76	0.59	0.166	0.055	0.055	0.008	0.024	0.04	0.002
Turnip foliage (immature)	0.093	0.084	0.014	0.005	< 0.005	< 0.009	< 0.008	< 0.008	< 0.008
Turnip foliage (maturity)	0.122	0.174	0.016	0.007	< 0.006	< 0.01	< 0.010	< 0.009	< 0.006

The majority of the TRR was solvent extractable (≥ 69% TRR) and in samples containing more than 0.01 mg eq/kg total radioactivity, residues were identified using HPLC retention times and confirmed by LC-MS where possible.

Oxathiapiprolin residues were not detected (< 0.001 mg/kg) in lettuce and turnip tops, were below 0.01 mg/kg and less than 10% TRR in the other rotational crops except turnip roots (120 day sowing) where levels were up to 15% TRR (0.003 mg/kg).

At the 30 and 120 days sowings, IN-WR791 residues above 0.01 mg eq/kg were measured in wheat grain (22–38% TRR, 0.021-0.098 mg eq/kg), in wheat forage (42% TRR, 0.11 mg eq/kg), in turnip tops (19–52% TRR, 0.03–0.05 mg eq/kg) and in wheat hay and straw (2.6–19% TRR, 0.02–0.06 mg eq/kg).

IN-E8S72 contributed 15–20% TRR (0.02–0.04 mg eq/kg) in wheat grain and 11–76% TRR (0.006-0.022 mg eq/kg) in lettuce from these sowings. In the other crops, residues of IN-E8S72 above 10% TRR and more than 0.01 mg eq/kg were found in wheat forage (8.8–12% TRR, 0.02–0.024 mg eq/kg), wheat hay and straw (13–17%TRR, 0.03–0.1 mg eq/kg) and turnip tops (20–73% TRR, 0.02–0.08 mg eq/kg).

IN-SXS67, IN-RZB20, and IN-RZB21/IN-RZD74 (unresolved) were detected at 4–12% TRR (0.004–0.022 mg eq/kg) in wheat grain and 3-5% TRR (0.001 mg eq/kg) in mature lettuce. In the 30 day sowings, IN-SXS67 made up 18–20% TRR (0.05–0.15 mg eq/kg) in wheat forage, hay and straw, increasing to 48–59% TRR (0.1–0.28 mg eq/kg) in the 120 day sowings. In wheat hay and straw, IN-RZB20 was found at 21–27% TRR (0.06–0.2 mg eq/kg) in the 30 day sowings and were 43–55% TRR (0.01–0.03 mg eq/kg) in the 365 day wheat sowings (forage and hay).

Table 27 Characterisation and identification of extracted radioactive residues in rotational crops planted after soil application of 0.21 kg ai/ha [pyrazole^{14}C]-oxathiapiprolin.

Matrix	PBI	Hvst DAT	TRR mg eq/kg [extracted]	Residue – mg eq/kg (%TRR)						
				Parent [a]	IN-E8S72	IN-WR791	IN-SXS67	IN-RZB20	IN-RZB21 IN-RZD74	Not identified [b]
Wheat grain	30	146	0.258 [0.238] [c]	0.005 (1.9%)	0.039 (15%)	0.098 (38%)	0.01 (4.2%)	0.022 (8.6%)	0.012 (4.5%)	0.028 (11%)
	120	246	0.098 [0.075] [c]	< 0.001 (0.3%)	0.019 (20%)	0.021 (22%)	0.011 (12%)	0.005 (4.9%)	0.004 (4.1%)	0.006 (8%)
Lettuce (immature) [d]	30	78	0.028 [0.026]	ND	0.006 (21%)	0.009 (31%)	ND	ND	ND	0.007 (27%)
	120	187	0.032 [0.029]	ND	0.022 (76%)	0.002 (7.5%)	ND	ND	ND	0.005 (18%)
Lettuce (mature)	30	92	0.013 [0.007]	ND	0.001 (11%)	0.002 (12%)	ND	ND	ND	0.001 (32%)
	120	200	0.022 [0.02]	0.002 (5.8%)	0.013 (48%)	0.001 (5.4%)	0.001 (4.7%)	0.001 (2.9%)	0.001 (3%)	0.001 (19%)
Turnip roots	30	110	0.014 [0.012]	ND	0.003 (19%)	0.007 (9.7%)				< 0.001 (14%)
	120	218	0.023 [0.017]	0.003 (15%)	0.004 (18%)	0.002 (9.7%)				0.002 (12%)
Wheat forage	30	70	0.269 [0.259]	0.002 (0.7%)	0.024 (8.8%)	0.114 (42%)	0.049 (18%)	0.028 (10%)	0.015 (5.6%)	0.033 (12%)
	120	168	0.172 [0.165]	ND	0.02 (12%)	0.006 (3.2%)	0.101 (59%)	0.013 (7.5%)	0.011 (6.6%)	0.007 (4.6%)
	365	407	0.022 [0.02]	ND	0.001 (6.5%)	ND	0.002 (7.3%)	0.012 (55%)	ND	0.002 (12%)
Wheat hay	30	85	0.298 [0.281]	0.007 (2.5%)	0.047 (16%)	0.056 (19%)	0.053 (18%)	0.064 (21%)	0.03 (10%)	0.043 (14%)
	120	200	0.172 [0.161]	ND	0.029 (17%)	0.008 (4.9%)	0.096 (56%)	0.015 (8.6%)	0.013 (7.6%)	0.018 (9.1%)
	365	448	0.081 [0.068]	ND	0.004 (5%)	ND	0.015 (18%)	0.034 (43%)	0.007 (8.9%)	0.008 (12%)

Matrix	PBI	Hvst DAT	TRR mg eq/kg [extracted]	Residue – mg eq/kg (%TRR)						
				Parent [a]	IN-E8S72	IN-WR791	IN-SXS67	IN-RZB20	IN-RZB21 IN-RZD74	Not identified [b]
Wheat straw [e]	30	146	0.758 [0.747][(c)]	0.005 (0.7%)	0.1 (13%)	0.044 (5.7%)	0.15 (20%)	0.204 (27%)	0.071 (9.4%)	0.088 (12%)
	120	246	0.59 [0.578][(c)]	ND	0.08 (14%)	0.016 (2.6%)	0.284 (48%)	0.075 (13%)	0.042 (7.1%)	0.033 (5.4%)
	365	469	0.166 [0.162][(c)]	ND	0.011 (7%)	0.003 (1.9%)	0.095 (57%)	0.013 (7.8%)	0.014 (8.3%)	0.026 (16%)
Turnip tops (immature)	30	91	0.093 [0.09]	ND	0.019 (21%)	0.048 (52%)	0.003 (3.3%)	0.011 (12%)	0.003 (3.5%)	0.006 (9.1%)
	120	195	0.09 [0.082]	ND	0.061 (73%)	0.027 (32%)	ND	0.008 (10%)	0.002 (2.9%)	0.006 (6.3%)
	365	441	0.014 [0.013]	ND	0.003 (19%)	0.004 (26%)	ND	0.001 (10%)	0.001 (10%)	0.001 (16%)
Turnip tops (mature)	30	92	0.12 [0.119]	ND	0.025 (20%)	0.055 (45%)	0.012 (9.9%)	0.013 (10%)	0.006 (4.8%)	0.008 (7.5%)
	120	200	0.174 [0.166]	ND	0.083 (48%)	0.032 (19%)	0.01 (6%)	0.011 (6.2%)	0.008 (4.6%)	0.001 (0.3%)
	365	441	0.016 [0.015]	ND	0.002 (12%)	0.007 (46%)	ND	0.002 (11%)	0.005 (32%)	0.001 (5.4%)

[a] The HPLC system used did not separate oxathiapiprolin from some non-polar reference standards

[b] Total unidentified metabolites

[c] Characterised/uncharacterised fractions following solvent, enzyme, base and acid extractions

[d] Metabolite IN-Q7D41 also reported in the 30 DAT immature lettuce at 6% TRR – 0.002 mg eq/kg

[e] Metabolites also reported in the 30 DAT wheat straw were IN-RZD74 (0.036 mg eq/kg – 4.8% TRR), IN-RZB21 (0.013 mg eq/kg – 1.7% TRR) and IN-KJ552 (0.011 mg eq/kg – 1.4% TRR).

Study 2 (0.6 kg ai/ha) [Ref: DP-31739]

In food items (lettuce, turnip tubers, and wheat grain) from soil treated with the equivalent of 0.6 kg ai/ha [pyrazole^{14}C]-oxathiapiprolin, radioactive residues were up to 0.19 mg eq/kg in wheat grain, up to 0.04 mg eq/kg in lettuces and up to 0.02 mg eq/kg in turnip tubers. Lower levels (up to 0.017 mg eq/kg – wheat grain) were present in the commodities from the plots treated with 0.6 kg ai/ha [isoxazoline^{14}C]-oxathiapiprolin.

In animal feed commodities, TRRs in the wheat commodities taken from the pyrazole-label plots were up to 0.23 mg eq/kg (forage), up to 0.26 mg eq/kg (hay) and 0.7 mg eq/kg in straw and up to 0.11 mg eq/kg in turnip tops. The maximum TRR levels for these commodities taken from the isoxazoline-label plots were lower, 0.09 mg eq/kg in straw, less than 0.02 mg eq/kg in forage and hay and 0.01 mg eq/kg in turnip tops.

Table 28 Total radioactive residues (mg eq/kg) in rotational crops grown in soil treated with [^{14}C]-oxathiapiprolin at a rate equivalent to 0.6 kg ai/ha.

RAC	[Pyrazole14C]-label (mg eq/kg)			[Isoxazoline14C]-label (mg eq/kg)		
	30 DAT	120 DAT	365 DAT	30 DAT	120 DAT	365 DAT
Wheat grain	0.135	0.191	0.117	0.016	0.017	0.008
Immature lettuce	0.025	0.036	0.036	0.002	< 0.001	< 0.004
Mature lettuce	0.02	0.031	0.024	0.006	< 0.002	< 0.003
Turnip tubers	0.02	0.011	0.016	0.016	0.006	0.004
Wheat forage	0.066	0.168	0.234	0.017	0.017	0.008
Wheat hay	0.263	0.142	0.226	0.018	0.01	0.011
Wheat straw	0.697	0.668	0.477	0.091	0.094	0.057
Turnip foliage (immature)	0.107	0.024	0.044	0.01	< 0.004	0.009
Turnip foliage (maturity)	0.086	0.031	0.043	0.011	< 0.007	< 0.004

The majority of the TRR was solvent extractable (≥ 68% TRR) and in samples containing more than 0.01 mg eq/kg total radioactivity, residues were identified using HPLC retention times and confirmed by LC-MS where possible.

Oxathiapiprolin residues were not detected (< 0.001 mg/kg) in all rotational crops except wheat forage and turnip tops, where trace levels (up to 0.005 mg/kg) were measured.

Metabolites present at more than 0.01 mg eq/kg and above 10% TRR were IN-SXS67, IN-RZB20, IN-RZB21/IN-RZD74, IN-WR791 and IN-E8S72.

IN-WR791 residues were present at up to 37% TRR in wheat grain (0.03–0.05 mg eq/kg), up to 31% TRR in wheat forage (0.01–0.07 mg eq/kg) and up to 0.02 mg eq/kg in turnip tops (41% TRR in immature tops and 29% TRR in mature tops).

IN-E8S72 residues were present at levels up to 14% TRR (0.02 mg eq/kg) in wheat grain, up to 35% TRR (0.01 mg eq/kg) in immature lettuce, up to 31% TRR (0.07 mg eq/kg) in wheat forage and up to 35% TRR (0.04 mg eq/kg) and up to 42% TRR (0.02 mg eq/kg in immature and mature turnip tops respectively.

IN-SXS67 residues were up to 37% TRR – 0.06 mg eq/kg in wheat forage, up to 35% TRR – 0.09 mg eq/kg in wheat hay, and up to 39% TRR (0.27 mg eq/kg) in wheat straw.

IN-RZB20 residues were present at up to 13% TRR – 0.015 mg eq/kg in wheat grain, up to 19% TRR – 0.05 mg eq/kg in wheat hay, up to 26% TRR – 0.12 mg eq/kg in wheat straw and up to 18% TRR (0.016 mg eq/kg) in turnip tops.

Unresolved IN-RZB21/IN-RZD74 made up 12–16% TRR in wheat forage and fodders and in turnip tops, up to 0.1 mg eq/kg in wheat straw and up to 0.035 mg eq/kg in wheat hay, up to 0.02 mg eq/kg in wheat forage and up to 0.013 mg eq/kg in turnip tops.

Table 29 Characterisation and identification of extracted radioactive residues in rotational crops planted after soil application of 0.6 kg ai/ha [pyrazole^{14}C]-oxathiapiprolin

Matrix	PBI	Hvst DAT	TRR mg eq/kg [extracted]	Residue – mg/kg (TRR)						
				parent	IN-E8S72	IN-WR791	IN-SXS67	IN-RZB20	IN-RZB21 IN-RZD74	IN-KJ552
Wheat grain	30	241	0.135 [0.094]	ND	0.019 14%	0.03 23%	0.013 9.9%	0.014 10%	0.011 8.6%	0.004 2.9%
	120	282	0.191 [0.178]	ND	0.014 7.4%	0.047 25%	0.015 7.9%	0.017 9.2%	0.008 4.5%	0.003 1.7%
	365	464	0.117 [0.079]	ND	0.01 8.2%	0.043 37%	0.011 9.6%	0.015 13%	ND	ND
Lettuce (immature)	30	72	0.025 [0.018]	ND	0.005 20%	0.007 27%	0.001 2.7%	0.003 14%	0.001 4.8%	0.001 2.2%
	120	184	0.036 [0.028]	ND	0.009 25%	0.007 20%	0.002 5%	ND	0.005 14%	0.002 4.9%
	365	421	0.036 [0.031]	ND	0.012 35%	0.008 24%	0.001 4%	0.001 3.2%	0.005 14%	0.001 2.5%
Lettuce (mature)	30	84	0.02 [0.018]	ND	0.004 22%	0.007 34%	ND	0.001 5%	0.004 21%	0.001 3.5%
	120	212	0.031 [0.022]	ND	0.007 21%	0.006 20%	0.001 4.7%	0.001 3.2%	0.005 15%	0.001 4.1%

Matrix	PBI	Hvst DAT	TRR mg eq/kg [extracted]	Residue – mg/kg (TRR)						
				parent	IN-E8S72	IN-WR791	IN-SXS67	IN-RZB20	IN-RZB21 IN-RZD74	IN-KJ552
	365	435	0.024 [0.023]	ND	0.008 33%	0.007 27%	0.001 5.2%	0.001 4.7%	0.004 18%	ND
Turnip roots	30	142	0.02 [0.012]	ND	0.002 9%	0.001 4.3%	0.001 5.6%	0.001 4%	0.003 14%	0.002 9.3%
	120	232	0.011 [0.008]	NA	NA	NA	NA	NA	NA	NA
	365	435	0.016 [0.01]	ND	0.009 14%	0.011 17%	0.004 5.9%	ND	ND	0.003 5%
Wheat forage	30	65	0.066 [0.06]	0.004 5.4%	0.003 4.5%	0.009 14%	0.016 24%	0.007 11%	0.008 12%	0.004 6.2%
	120	157	0.168 [0.158]	ND	0.013 (7.6%)	0.022 (13%)	0.062 (37%)	0.014 (8.5%)	0.023 (14%)	0.013 (7.9%)
	365	401	0.234 [0.207]	0.002 0.9%	0.028 12%	0.073 31%	0.05 21%	0.012 5.2%	0.025 10%	0.016 7%
Wheat hay	30	170	0.263 [0.212]	ND	0.013 5.1%	0.011 4%	0.092 35%	0.049 19%	0.035 13%	0.005 1.8%
	120	197	0.142 [0.12]	ND	0.008 5.4%	0.01 7.1%	0.043 30%	0.018 13%	0.02 14%	0.007 4.6%
	365	421	0.226 [0.189]	ND	0.018 8.2%	0.023 10%	0.058 26%	0.039 17%	0.027 12%	0.007 3.2%
Wheat straw[(d)]	30	240	0.697 [0.641]	ND	0.031 4.5%	0.022 3.1%	0.272 39%	0.116 17%	0.081 12%	0.014 2.1%
	120	282	0.668 [0.654]	ND	0.058 8.7%	0.055 8.3%	0.17 26%	0.145 22%	0.1 15%	0.028 4.2%
	365	464	0.477 [0.364]	ND	0.029 6%	0.025 5.3%	0.126 26%	0.122 26%	0.057 12%	ND
Turnip tops (immature)	30	100	0.107 [0.008]	0.002 1.7%	0.037 35%	0.024 23%	ND	0.024 23%	0.001 1.3%	0.002 1.5%
	120	212	0.024 [0.018]	ND	0.002 8.8%	0.003 14%	0.002 9.9%	0.006 24%	0.003 9.6%	ND
	365	428	0.044 [0.043]	ND	0.007 15%	0.018 41%	0.003 6.1%	0.007 17%	0.005 11%	ND
Turnip tops (mature)	30	142	0.086 [0.084	ND	0.016 19%	0.023 27%	0.007 7.6%	0.016 18%	0.013 16%	0.001 1.5%
	120	232	0.031 [0.027	ND	0.004 12%	0.006 21%	0.003 9.2%	0.008 26%	0.003 11%	ND
	365	435	0.043 [0.043	0.001 1.2%	0.018 42%	0.013 29%	0.002 3.8%	0.002 4.3%	0.007 17%	0.001 2.5%

In rotational crops, the metabolites are predominantly those containing the pyrazole moiety (possibly reflecting preferential root uptake of the more polar pyrazole cleavage products). Cleavage of the parent compound in soil (and to a much lesser extent in rotational crop) results in the formation of IN-RZB20, IN-WR791, IN-RZD74, IN-RZB21, IN-KJ552 and IN-E8S72 (which undergoes *N*-glucose conjugation to form IN-SXS67. Hydroxylation of oxathiapiprolin in the isoxazole ring followed by dehydration may lead to the formation of IN-Q7D41, a minor metabolite.

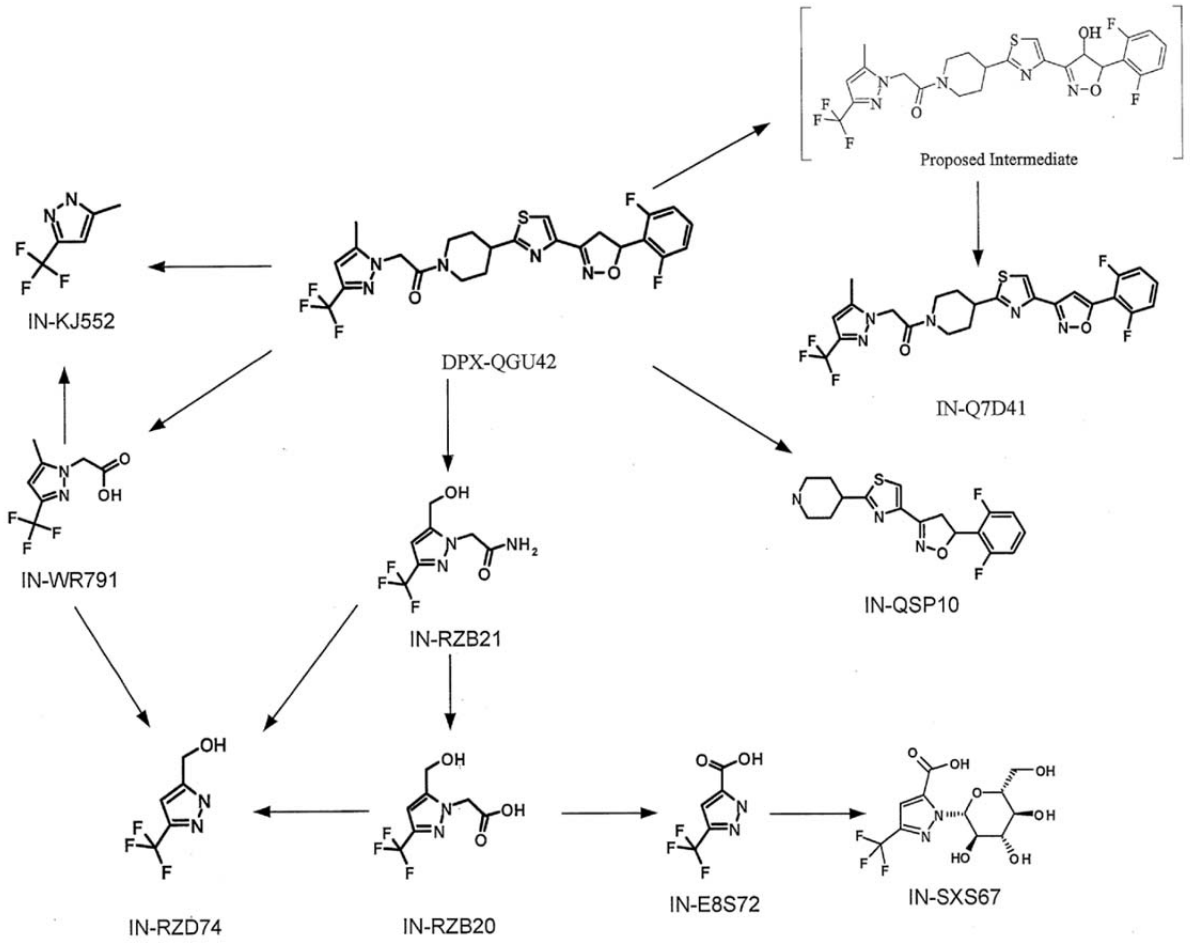


Figure 12 Proposed metabolic pathways for oxathiapiprolin (DPX-QGU42) in confined rotational crops

Field rotational crop studies

In a study reported by Cairns and Hammond, 2012 [Ref: DP-30174], cropped plots (rye and wheat) at two sites in Europe (Germany and Spain) were treated with 3 applications of 0.07 kg ai/ha oxathiapiprolin (OD formulation), 10 days apart (total rate of 0.21 kg ai/ha) and after the primary crops had been harvested for forage and tilled, barley/wheat, spinach/lettuce, carrots/radish and peas/beans were planted 30, 120 and 270–365 days after the last application.

Samples were stored frozen for up to 32 weeks before analysis using method DP-30422 (LC-MS/MS) to measure residues of oxathiapiprolin and metabolites IN-WR791, IN-RDG40, IN-E8S72, IN-Q7H09, IN-SXS67, IN-RZB20 and IN-RZD74. Mean recovery values for all analyte/matrix combinations ranged from 70–107% in samples spiked with 0.01–0.5 mg/kg and the LOQ for each analyte was 0.01 mg/kg.

Oxathiapiprolin residues were not detected in any rotational crop except in one 120 day PBI sample of peas (with pod), where residues of < 0.01 mg/kg were reported. Residues of metabolites IN-

RDG40, IN-Q7H09, IN-RZB20 and IN-RZD74 were also not detected in any rotational crop and IN-WR791 residues were < 0.01 mg/kg.

IN-E8S72 was the predominant residue in leafy vegetables (mean residues up to 0.13 mg/kg), carrot/radish tops (mean residues up to 0.02 mg/kg) and legume pods (mean residues up to 0.02 mg/kg).

IN-SXS67, the glucose conjugate of IN-E8S72 was found in legume vines (mean residues 0.03–0.05 mg/kg) and cereal forage, hay and straw (mean residues up to 0.34 mg/kg) with levels declining at the longer plant-back intervals. It was also the only residue detected in cereal grains (< 0.01 mg/kg).

Table 30 Residues in rotational crops following 3 applications of 0.07 kg ai/ha oxathiapiprolin (10 days apart) to barley or wheat crops in Europe (Total 0.21 kg ai/ha).

Crop Commodity	Harvest (Days after Planting)	DAT	PBI (days)	Average Residues (mg/kg)[a][b]			
				Oxathiapiprolin	IN-SXS67	IN-E8S72	IN-WR791
Root and Tuber Vegetables (carrot, radish)							
Tops	54–85	83–115	30	ND	ND	0.013	ND
	54–85	174–205	120	ND	ND	0.021	ND
	54–85	324–402	270–365	ND	ND	ND	ND
Roots	54–85	83–115	30	ND	ND	ND	ND
	54–85	174–205	120	ND	ND	ND	ND
	54–85	324–402	270–365	ND	ND	ND	ND
Leafy Vegetables (spinach, lettuce)							
Immature leaves	48–54	78–83	30	ND	ND	0.13	< 0.01
	48–54	168–174	120	ND	< 0.01	0.11	< 0.01
	48–54	324–365	270–365	ND	ND	0.012	ND
Mature leaves	56–70	86–99	30	ND	< 0.01	0.086	ND
	56–70	176–190	120	ND	< 0.01	0.071	< 0.01
	56–70	340–373	270–365	ND	ND	0.015	ND
Legume Vegetables (peas, beans)							
Vines	57–70	87–99	30	ND	0.047	< 0.01	ND
	57–70	177–190	120	ND	0.032	0.013	ND
	57–70	340–374	270–365	ND	0.026	< 0.01	ND
Pods with seeds (immature)	57–70	87–99	30	ND	< 0.01	0.018	ND
	57–70	177–190	120	< 0.01	< 0.01	0.014	ND
	57–70	340–374	270–365	ND	< 0.01	ND	ND
Mature peas without pods	85–103	115–132	30	ND	< 0.01	< 0.01	ND
	85–103	205–223	120	ND	< 0.01	< 0.01	0.004
	85–103	373–402	270–365	ND	ND	ND	ND
Small Grain Cereals (barley, wheat)							
Forage	35–54	65–83	30	ND	0.26	0.031	< 0.01
	35–54	155–174	120	ND	0.096	0.012	< 0.01
	35–54	352–324	270–365	ND	0.037	< 0.01	ND
Hay	58–74	87–103	30	ND	0.34	0.015	ND
	58–74	177–194	120	ND	0.21	0.018	ND
	58–74	344–374	270–365	ND	0.094	< 0.01	ND
Grain	91–96	120–126	30	ND	< 0.01	ND	ND
	91–96	211–216	120	ND	< 0.01	ND	ND
	91–96	361–413	270–365	ND	ND	ND	ND
Straw	91–96	120–126	30	ND	0.099	0.019	ND
	91–96	211–216	120	ND	0.095	0.021	ND
	91–96	361–413	270–365	ND	0.02	ND	ND

[a] Residues of metabolites IN-Q7H09, IN-RDG40, IN-RZB20 and IN-RZD74 were not detected in any commodity.

(b) Mean residues from three replicate samples

In a second European study, reported by Spence and Brown, 2012 [[Ref: DP-34687], separate bare soil plots at eight sites in UK and France were treated with oxathiapiprolin (SE formulations), either 4 applications of 0.05 kg ai/ha , 6–10 days apart (total rate of 0.2 kg ai/ha) or with 6

applications of 0.02 kg ai/ha (total 0.12 kg ai/ha) at 3–5-day intervals. After the plots were tilled, peas, sugar beet, cabbage, wheat, green onions, and maize were planted 14–35 days after the last application.

Samples were stored frozen for up to 32 weeks before analysis using method DP-30422 (LC-MS/MS) to measure residues of oxathiapiprolin and metabolites IN-WR791, IN-RDG40, IN-E8S72, IN-Q7H09, IN-SXS67, IN-RZB20 and IN-RZD74. Mean recovery values per analyte/matrix combination ranged from 85–104% in samples spiked with 0.01–0.5 mg/kg and the LOQ for each analyte was 0.01 mg/kg.

Oxathiapiprolin residues were not detected in any rotational crop. Residues of metabolites IN-WR791, IN-RDG40, IN-Q7H09, IN-RZB20, and IN-RZD74 were also not detected in any rotational crop.

Residues of IN-E8S72 and its glucose conjugate IN-SXS67 were either not detected or < 0.01mg/kg in all rotational crops except in wheat and maize forage, where levels of up to 0.02 mg/kg were reported.

Table 31 Residues in rotational crops following bare soil applications of oxathiapiprolin (SE formulation) in Europe.

Crop Commodity	Harvest (Days after planting)	DAT	PBI (days)	Average Residues (mg/kg)[a]		
				Oxathiapiprolin	IN-E8S72	IN-SXS67
0.2 kg ai/ha (4× 0.05 kg ai/ha, 6-10 day RTI)						
Pea vines	96	129	33	ND	0.008	0.009
Pea seeds with pod	96	129	33	ND	ND	ND
Sugar beet leaves	90-143	122-177	32-34	ND	ND	0.003
Cabbage heads	115	143	28	ND	ND	ND
Green onions	98	130	32	ND	ND	ND
Wheat forage	63	77	14	ND	ND	0.017
Wheat straw	162	176	14	ND	ND	ND
Maize forage	101-130	117-154	16-24	ND	ND	0.012
Corn cobs (immature)	101-130	117-154	16-24	ND	ND	ND
Maize grain	116-185	132-209	16-24	ND	ND	ND
0.12 kg ai/ha (6× 0.02 kg ai/ha, 3-5 day RTI)						
Pea vines	96	129	33	ND	0.005	0.006
Pea seeds with pod	96	129	33	ND	ND	ND
Sugar beet leaves	90-143	122-182	32-39	ND	ND	0.003
Cabbage heads	115	143	28	ND	ND	ND
Green onions	98	130	32	ND	0.005	ND
Wheat forage	63	80	17	ND	ND	0.007
Wheat straw	162	179	17	ND	ND	ND
Maize forage	101-130	120-157	19-27	ND	ND	0.012
Corn cobs (immature)	101-130	120-157	19-27	ND	ND	ND
Maize grain	116-185	135-212	19-27	ND	ND	ND

[a] Residues of metabolites IN-WR791, IN-RDG40, IN-Q7H09, IN-RZB20, and IN-RZD74 were not detected in any commodity.

In a study conducted in North America and reported by Shephard, 2013 [Ref: DP-31740], bare soil plots at three sites were treated with oxathiapiprolin (OD formulations) at either 0.28 kg

ai/ha (2× 0.14 kg ai/ha, 7 days apart) or 0.56 kg ai/ha ((2× 0.28 kg ai/ha, 7 days apart). After the plots were tilled, radish, oats, wheat, soya bean, peas, sugar beet, cabbage, wheat, green onions, and maize were planted 8–30 days, 60–120 days and 300–365 days after the last application.

Duplicate samples were stored frozen for up to 47 weeks before analysis using method DP-30422 (LC-MS/MS) to measure residues of oxathiapiprolin and metabolites IN-WR791, IN-RDG40, IN-E8S72, IN-Q7H09, IN-SXS67, IN-RZB20 and IN-RZD74. Mean recovery values per analyte/matrix combination ranged from 79–111% in samples spiked with 0.01–1.0 mg/kg and the LOQ for each analyte was 0.01 mg/kg.

Oxathiapiprolin residues were not detected in any rotational crop except soya bean forage, where residues in hay and straw were either not detected or < 0.01 mg/kg and were up to 0.03 mg/kg in forage (0.56 kg ai/ha plot). Residues of metabolites IN-RDG40, IN-Q7H09, IN-RZB20, IN-RZD74 and IN-WR791 (except soya bean seed) were also not detected in any rotational crop.

Residues of IN-E8S72 and its glucose conjugate IN-SXS67 were the predominant residues in most commodities, generally found at less than 0.02 mg/kg, with higher levels in soya beans, cereal forage and fodders. Highest residues of IN-E8S72 were in soya bean hay (up to 0.16 mg/kg) and cereal hay (up to 0.09 mg/kg) and IN-SXS67 residues were up to 0.09 mg/kg in cereal hay and up to 0.08 mg/kg in soya bean hay in the 0.56 kg ai/ha plots.

Table 32 Residues in rotational crops following bare soil applications of oxathiapiprolin (OD formulations) in North America.

Crop Commodity	Harvest (Days after planting)	DAT	PBI (days)	Average Residues (mg/kg)[a]			
				Oxathiapiprolin	IN-E8S72	IN-SXS67	IN-WR791
0.28 kg ai/ha (2× 0.14 kg ai/ha, 7 day RTI)							
Radish root	27–33	41–44	8–17	ND	ND	0.04	ND
	28–45	103–148	75–103	Samples were not analysed			
	31–40	376–390	336–359	ND	ND	ND	ND
Radish tops	27–33	41–44	8–17	ND	0.006	ND	ND
	28–45	103–148	75–103	Samples were not analysed			
	31–40	376–390	336–359	ND	ND	ND	ND
Cereal forage	33–37	41–52	8–15	ND	ND	0.022	ND
	188–239	328–359	120–140	ND	0.004	0.007	ND
	42–51	376–395	334–344	ND	ND	0.004	ND
Soya bean forage	48–55	57–76	9–21	0.004	0.022	0.007	ND
	27–54	104–117	63–77	0.019	0.01	0.005	ND
	57–62	376–421	319–359	ND	ND	ND	ND
Cereal hay	46–50	54–65	8–15	ND	0.008	0.041	ND
	217–259	357–379	120–140	ND	0.004	0.017	ND
	63–66	397–410	334–344	ND	ND	0.009	ND
Soya bean hay	54–83	63–104	9–21	0.007	0.066	0.02	ND
	54–60	123–131	63–77	ND	0.022	0.009	ND
	70–120	389–461	319–359	ND	ND	ND	ND
Cereal grain	73–82	88–90	8–15	Samples were not analysed			
	264–296	404–416	120–140	ND	ND	ND	ND
	104–112	438–456	334–344	ND	ND	ND	ND
Cereal straw	73–82	88–90	8–15	ND	0.005	0.004	ND
	264–296	404–416	120–140	ND	0.006	0.009	ND

Crop Commodity	Harvest (Days after planting)	DAT	PBI (days)	Average Residues (mg/kg)[a]			
				Oxathiapiprolin	IN-E8S72	IN-SXS67	IN-WR791
	104–112	438–456	334–344	ND	ND	ND	ND
Soya bean with pod (immature)	108–132	117–153	9–21	ND	0.008	0.022	ND
	81–82	144–159	63–77	ND	0.007	0.007	ND
	108–123	427–482	319–359	ND	0.003	ND	ND
Soya bean (seed)	143–158	152–179	9–21	ND	0.016	0.034	ND
	112	175–189	63–77	ND	0.012	0.008	ND
	151–157	470–516	319–359	ND	0.006	0.006	ND
0.56 kg ai/ha (2× 0.28 kg ai/ha, 7 day RTI)							
Radish root	27–33	41–44	8–17	ND	ND	0.003	ND
	28–45	103–148	75–103	0.004	ND	ND	ND
	31–40	376–390	336–359	ND	ND	ND	ND
Radish tops	27–33	41–44	8–17	ND	0.011	ND	ND
	28–45	103–148	75–103	ND	0.003	ND	ND
	31–40	376–390	336–359	ND	0.012	ND	ND
Cereal forage	33–37	41–52	8–15	ND	0.004	0.039	ND
	188–239	328–359	120–140	ND	0.013	0.025	ND
	42–51	376–395	334–344	ND	ND	0.026	ND
Soya bean forage	48–55	57–76	9–21	0.006	0.062	0.015	ND
	27–54	104–117	63–77	0.033	0.026	0.009	ND
	57–62	376–421	319–359	ND	0.009	ND	ND
Cereal hay	46–50	54–65	8–15	ND	0.008	0.09	ND
	217–259	357–379	120–140	ND	0.011	0.047	ND
	63–66	397–410	334–344	ND	ND	0.055	ND
Soya bean hay	54–83	63–104	9–21	0.009	0.16	0.078	ND
	54–60	123–131	63–77	ND	0.046	0.039	ND
	70–120	389–461	319–359	ND	0.009	0.015	ND
Cereal grain	73–82	88–90	8–15	ND	ND	0.006	ND
	264–296	404–416	120–140	ND	0.004	ND	ND
	104–112	438–456	334–344	ND	ND	ND	ND
Cereal straw	73–82	88–90	8–15	ND	0.013	0.018	ND
	264–296	404–416	120–140	ND	0.014	0.022	ND
	104–112	438–456	334–344	ND	0.006	0.008	ND
Soya bean with pod (immature)	108–132	117–153	9–21	ND	0.024	0.037	ND
	81–82	144–159	63–77	ND	0.01	0.009	ND
	108–123	427–482	319–359	ND	0.014	ND	ND
Soya bean (seed)	143–158	152–179	9–21	ND	0.039	0.059	0.005
	112	175–189	63–77	ND	0.01	0.008	ND
	151–157	470–516	319–359	ND	0.015	0.017	ND

[a] Residues of metabolites IN-RDG40, IN-Q7H09, IN-RZB20, and IN-RZD74 were not detected in any commodity.

In a further study involving 32 trial sites in North America and reported by Shephard, 2013 [Ref: DP-33929], bare soil plots were treated with oxathiapiprolin (OD or SE formulations) with added adjuvant at 0.28 kg ai/ha (2× 0.14 kg ai/ha, 6–9 days apart) and after the plots were tilled, sugar beet, lettuce, celery, mustard greens, soya bean, sweet corn, maize, sorghum, wheat, oil seed rape and strawberry were planted 5–10 days after the last application.

Duplicate samples were stored frozen for up to 48 weeks before analysis using method DP-30422 (LC-MS/MS) to measure residues of oxathiapiprolin and metabolites IN-WR791, IN-RDG40, IN-E8S72, IN-Q7H09, IN-SXS67, IN-RZB20 and IN-RZD74. Mean recovery values per analyte/matrix combination ranged from 83–115% in samples spiked with 0.01–2.0 mg/kg and the LOQ for each analyte was 0.01 mg/kg.

Oxathiapiprolin residues were not detected in any rotational crop except soya bean hay and wheat straw, where residues were < 0.01 mg/kg. Residues of metabolites IN-RDG40, IN-Q7H09, IN-RZB20, and IN-RZD74 were also not detected in any rotational crop and residues of IN-WR791 were also either not detected or < 0.01 mg/kg in all commodities except wheat hay (0.012 mg/kg).

Residues of IN-E8S72 and its glucose conjugate IN-SXS67 were the predominant residues in most commodities, but generally below 0.02 mg/kg. Highest IN-SXS67 residues were found in wheat hay (0.3 mg/kg), wheat forage and straw (0.1 mg/kg), and mature soya bean seeds (0.03 mg/kg). Residues of IN-E8S72 were generally lower, 0.02–0.08 mg/kg in wheat forage, hay and straw and were 0.07 mg/kg in soya bean hay.

Table 33 Residues in rotational crops following bare soil applications of 0.28 kg ai/ha oxathiapiprolin in North America (2× 0.14 kg ai/ha, 7 day RTI).

Crop Commodity	Harvest (Days after planting)	DAT	PBI (days)	Average Residues (mg/kg)[a]			
				Oxathiapiprolin	IN-E8S72	IN-SXS67	IN-WR791
Strawberry fruit	83–117	91–122	5-8	ND	0.009	ND	ND
Sugar beet tops	108–146	115–153	5-7	ND	ND	0.016	ND
Lettuce (immature leaves)	35–63	41–73	6-10	ND	0.004	ND	ND
Lettuce (mature leaves)	48–78	54–88	6-10	ND	0.004	ND	ND
Celery (stalk)	76–97	81–103	5-6	ND	ND	ND	0.005
Mustard greens (leaves)	30–43	37–51	7-8	ND	0.02	ND	ND
Soya bean forage	41–55	48–61	6-7	ND	0.011	0.012	ND
Soya bean hay	63–69	70–75	6-7	0.004	0.068	0.042	0.004
Soya bean seeds (immature)	108–112	115–119	6-7	ND	0.017	0.015	ND
Soya bean pods (immature)	108–112	115–119	6-7	ND	0.006	0.014	ND
Soya bean seed (mature)	146–163	153–170	6-7	ND	0.023	0.029	ND
Maize, sorghum forage	68–97	75–113	5-7	ND	ND	0.005	ND
Corn (cobs without husks)	68–89	74–95	5-7	ND	ND	ND	ND
Maize, sorghum stover	108–146	107–157	5-7	ND	ND	ND	ND
Wheat forage	27–50	32–55	5-9	ND	0.022	0.098	0.008
Wheat hay	48–66	60–75	5-9	ND	0.075	0.3	0.012
Wheat straw	54–114	61–123	5-9	0.003	0.045	0.098	ND
Rape seed	88–114	93–123	5-9	ND	0.006	ND	ND

[a] Residues of metabolites IN-RDG40, IN-Q7H09, IN-RZB20, and IN-RZD74 were all not detected in any sample.

In summary, a series of rotational crop field trials were conducted at a range of application rates to anticipate the maximum application rates expected in Europe and North America. Residues of

oxathiapiprolin and metabolites IN-WR791, IN-RDG40, IN-E8S72, IN-Q7H09, IN-SXS67, IN-RZB20 and IN-RZD74 were measured in a number of crops to representing root and tuber vegetables, leafy vegetables, stem vegetables, brassica vegetables, bulb vegetables, legumes/pulses, small-grain cereals, large-grain cereals, oilseeds, and small berries.

Residue results for oxathiapiprolin and three metabolites (IN-WR791, IN-E8S72, and IN-SXS67) in rotational crop samples, scaled to reflect a seasonal application rate of 0.56 kg ai/ha are summarized below.

Residues of oxathiapiprolin in rotational crops at all plant-back intervals were < 0.01 mg/kg in all commodities except legume feed commodities (up to 0.04 mg/kg in soya bean forage and up to 0.015 mg/kg in soya bean hay).

Residues of IN-WR791 were 0.01 mg/kg or less in all rotational crops except leafy vegetables (up to 0.016 mg/kg) and cereal forage and hays (less than 0.03 mg/kg).

Metabolites IN-E8S72 and IN-SXS67 were the predominant residues in most rotational crops. In food commodities, maximum levels of IN-E8S72 were up to 0.02 mg/kg in strawberries, green onions and oilseeds, up to about 0.05 mg/kg in legume vegetables and pulses and up to 0.35 mg/kg in leafy vegetables. In feed commodities, maximum IN-E8S72 residues were 0.05–0.1 mg/kg in legume and cereal forages, root vegetable tops and cereal straws, with higher levels of up to 0.15 mg/kg in legume and cereal hays.

Maximum residues of IN-SXS67 were up to 0.02 mg/kg in cereal grains and leafy vegetables, 0.04–0.08 mg/kg in legume vegetables, pulses and root crops. In animal feed commodities, maximum IN-SXS67 residues above 0.1 mg/kg were present in legume forage (up to 0.13 mg/kg) and in cereal forage (max 0.69 mg/kg), hays (max 0.91 mg/kg) and straws (max 0.26 mg/kg).

Table 34 Summary table: Maximum and mean residues from rotational crop field trials with oxathiapiprolin scaled to a seasonal application rate of 0.56 kg ai/ha

Crop group	Crop commodity	Residues scaled to 560 g ai/ha (mg/kg)							
		Oxathiapiprolin		IN-WR791		IN-E8S72		IN-SXS67	
		max	mean	max	mean	max	mean	max	mean
Food commodities									
Berries	strawberry	< 0.01	< 0.01	< 0.01	< 0.01	**0.018**	0.018	< 0.01	< 0.01
Brassica vegetables	cabbage head	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
Stem vegetables	celery stalk	< 0.01	< 0.01	**0.01**	0.01	< 0.01	< 0.01	< 0.01	< 0.01
Bulb vegetables	green onion tops	< 0.01	< 0.01	< 0.01	< 0.01	**0.023**	0.01	< 0.01	< 0.01
Root vegetables	carrot, radish, sugar beet roots	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	**0.08**	0.013
Legume vegetables	bean, pea, soya bean seeds+pods	< 0.01	< 0.01	< 0.01	< 0.01	**0.048**	0.016	**0.044**	0.016
Leafy vegetables	lettuce, spinach, mustard greens -immature leaves	< 0.01	< 0.01	**0.013**	< 0.01	**0.35**	0.17	< 0.01	< 0.01
	lettuce, spinach, mustard greens -mature leaves	< 0.01	< 0.01	0.016	< 0.01	**0.23**	0.1	**0.016**	< 0.01
Pulses	bean, pea, soya bean seeds	< 0.01	< 0.01	0.011	< 0.01	**0.046**	0.022	**0.068**	0.027
Cereals	barley, wheat, maize grain	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	**0.021**	< 0.01
Oilseeds	rape seed	< 0.01	< 0.01	< 0.01	< 0.01	**0.012**	0.012	< 0.01	< 0.01
Feed commodities									
Root vegetables	carrot, radish, sugar beet tops	< 0.01	< 0.01	< 0.01	< 0.01	**0.056**	0.013	**0.032**	< 0.01
Legumes	beans, peas, soya bean forage	**0.038**	< 0.01	< 0.01	< 0.01	**0.062**	0.024	**0.125**	0.034
	beans, peas, soya bean hay	**0.014**	< 0.01	< 0.01	< 0.01	**0.14**	0.076	**0.084**	0.04
Cereals	barley, wheat, oat, maize, sorghum forage	< 0.01	< 0.01	**0.016**	< 0.01	**0.083**	0.015	**0.69**	0.1
	barley, wheat, oat hay	< 0.01	< 0.01	**0.024**	< 0.01	**0.15**	0.03	**0.91**	0.26
	barley, wheat, oats, maize, sorghum straw	< 0.01	< 0.01	< 0.01	< 0.01	**0.09**	0.021	**0.26**	0.065

E8SEQ = combined IN-E8S72 + IN-SXS67 residues, expressed as IN-E8S72 equivalent residues, LOQ 0.01 mg/kg.

METHODS OF RESIDUE ANALYSIS

Analytical methods

The meeting received analytical method descriptions and validation data for oxathiapiprolin and metabolites IN-SXS67, IN-RZB20, IN-RZD74, IN-E8S72, IN-WR791, IN-RDG40, and IN-Q7H09 in plant and animal matrices and these are summarized below.

Table 35 Summary of analytical methods for oxathiapiprolin and its metabolites, developed for plant and animal matrices

Matrix	Analyte	Method	Principle	LOQ (mg/kg)	Reference
Plant matrices -Low water content -High water content -High oil content -High acid content	Oxathiapiprolin IN-E8S72 IN-WR791 IN-RDG40 IN-Q7H09	DuPont-30422	Acetonitrile/formic acid/water extraction acetonitrile/water dilution reverse-phase LC-MS/MS	0.01 0.01 0.01 0.01 0.01	DP-30422-1
Plant matrices -Low water content -High water content -High oil content -High acid content	Oxathiapiprolin IN-E8S72 IN-WR791 IN-RDG40 IN-Q7H09 IN-SXS67 IN-RZB20 IN-RZD74	DuPont-30422 Supplement 1	Acetonitrile/formic acid/water extraction aqueous formic acid/methanol dilution SPE clean-up (some matrices) reverse-phase LC-MS/MS	0.01 0.01 0.01 0.01 0.01 0.01 0.01 0.01	DP-30422-2
Plant matrices -High water content -High acid content	Oxathiapiprolin	QuEChERS	Acetonitrile/water extraction SPE or freezing out clean-up Acidification/dilution (formic acid/water) LC/MS/MS analysis	0.01	DP-28696
Animal tissues Milk Eggs	Oxathiapiprolin IN-Q7H09 IN-RDG40 IN-RLB67 IN-RAB06	DuPont-31138	Acetone/water/hexane extraction aqueous formic acid/methanol dilution reverse-phase LC-MS/MS	0.01 0.01 0.01 0.01 0.01	DP-31138

Data collection methods

Method DuPont-30422 (Oxathiapiprolin and four metabolites - fresh and processed plant matrices)

This LC-MS/MS method for measuring residues of oxathiapiprolin and its IN-E8S72, IN-WR791, IN-RDG40, IN-Q7H09 metabolites in crops was reported by Henze & Stry, 2011 [Ref: DP-30422-1].

In this method, residues were extracted from crop samples using a solution of formic acid, water, and acetonitrile. The extracts were diluted to 50 mL and an aliquot removed and diluted to 10-mL with acetonitrile and water. An aliquot of the diluted extract was transferred to an auto-sampler vial for analysis. IN-E8S72, IN-WR791, IN-RDG40, IN-Q7H09, and oxathiapiprolin were separated from co-extracts by reversed phase liquid chromatography and detected by electrospray mass spectrometry/mass spectrometry. Quantitation was performed using the mass transition 207→143 for IN-WR791, 179→65 for IN-E8S72, 556→174 for IN-RDG40, 556→155 for IN-Q7H09, 540→500 for oxathiapiprolin. The reported LOQ for each analyte is 0.01 mg/kg.

Good linearity was observed in the range of 0.050 to 5.0 ng/mL for oxathiapiprolin and metabolites. Analysis of control samples resulted in no apparent residues of oxathiapiprolin and metabolites (the response in the area of the oxathiapiprolin and metabolites peak always corresponded to less than 20% of the limit of determination). It can therefore be concluded that few, if any, apparent residues or false positive values would arise.

Method validation recovery data were obtained by the same analyst over the course of two days per matrix. Relative standard deviations of less than 20% were obtained for fortifications made

at the quantification limit for each matrix, as well as at higher levels. Therefore, the repeatability of this method is adequate for the purposes of residue data collection and enforcement of MRLs.

Residue field trial concurrent recovery rates where this method was used are summarized in the discussions on the individual crop field trials. These concurrent recoveries produced similar results to the fortification data obtained during method validation. The average recoveries ranged from 70–120%, with a standard deviation of ≤ 20%. Therefore, the recovery of this method is adequate for the purposes of residue data collection.

Table 36 DuPont-30422 analytical method validation recovery rates in plant matrices.

Matrix	Fortification level (mg/kg)	Number of tests	Average % recovery	Standard deviation	% RSD	Reference
Oxathiapiprolin						
Wheat Grain	0.01, 0.1	5, 5	97, 89	5.9, 1.9	6.1, 2.2	DP-30422-1
Soya beans	0.01, 0.1	5, 5	118, 113	9.4, 6.9	8.0, 6.2	
Wheat Forage	0.01, 0.1	5, 5	97, 94,	3.7, 1.9	3.8, 2.1	
Wheat Straw	0.01, 0.1	5, 5	95, 94	14, 5.9	14, 6.3	
Canola Seed	0.01, 0.1	5, 5	92, 92	8.3, 4.4	8.9, 4.8	
Grapes	0.01, 0.1	5, 5	96, 100	7.0, 6.8	7.3, 6.8	
Tomatoes	0.01, 0.1	5, 5	92, 94	7.2, 2.3	7.8, 2.5	
Potatoes	0.01, 0.1	5, 5	103, 100	2.4, 1.9	2.3, 1.9	
IN-E8S72						
Wheat Grain	0.01, 0.1	5, 5	89, 83	3.6, 3.3	4.1, 3.9	DP-30422-1
Soya beans	0.01, 0.1	5, 5	88, 88	2.7, 5.3	3.1, 6.1	
Wheat Forage	0.01, 0.1	5, 5	102, 95	3.1, 1.9	3.1, 2.1	
Wheat Straw	0.01, 0.1	5, 5	97, 97	4.7, 3.0	4.8, 3.2	
Canola Seed	0.01, 0.1	5, 5	90, 89	5.3, 2.2	5.9, 2.5	
Grapes	0.01, 0.1	5, 5	104, 104	3.7, 2.7	3.6, 2.6	
Tomatoes	0.01, 0.1	5, 5	99, 103	2.2, 2.1	2.3, 2.0	
Potatoes	0.01, 0.1	5, 5	100, 104	3.8, 2.3	3.8, 2.3	
IN-WR791						
Wheat Grain	0.01, 0.1	5, 5	88, 91	2.1, 1.1	2.4, 1.2	DP-30422-1
Soya beans	0.01, 0.1	5, 5	93, 91	8.1, 6.3	8.7, 6.9	
Wheat Forage	0.01, 0.1	5, 5	89, 90	3.0, 0.8	3.4, 0.9	
Wheat Straw	0.01, 0.1	5, 5	90, 90	3.7, 2.4	4.1, 2.7	
Canola Seed	0.01, 0.1	5, 5	88, 88	6.7, 2.5	7.6, 2.8	
Grapes	0.01, 0.1	5, 5	98, 99	7.5, 1.3	7.7, 1.3	
Tomatoes	0.01, 0.1	5, 5	96, 96	5.4, 3.2	5.6, 3.4	
Potatoes	0.01, 0.1	5, 5	102, 97	5.3, 2.8	5.2, 2.9	
IN-RDG40						
Wheat Grain	0.01, 0.1	5, 5	101, 93	11, 4.6	11, 4.9	DP-30422-1
Soya beans	0.01, 0.1	5, 5	111, 94	9.9, 5.7	8.9, 6.1	
Wheat Forage	0.01, 0.1	5, 5	97, 97	5.9, 7.2	6.2, 7.4	
Wheat Straw	0.01, 0.1	5, 5	106, 98	10, 4.4	9.4, 4.5	
Canola Seed	0.01, 0.1	5, 5	103, 101	9.7, 4.1	9.4, 4.1	
Grapes	0.01, 0.1	5, 5	93, 103	12, 6.2	13, 6.0	
Tomatoes	0.01, 0.1	5, 5	97, 94	12, 3.0	12, 3.2	
Potatoes	0.01, 0.1	5, 5	106, 97	6.4, 6.7	6.1, 6.9	
IN-Q7H09						
Wheat Grain	0.01, 0.1	5, 5	90, 92	7.8, 3.4	8.6, 3.6	DP-30422-1
Soya beans	0.01, 0.1	5, 5	98, 99	4.4, 4.5	4.5, 4.6	
Wheat Forage	0.01, 0.1	5, 5	95, 97	2.6, 2.3	2.8, 2.4	
Wheat Straw	0.01, 0.1	5, 5	94, 99	3.8, 5.4	4.1, 5.4	
Canola Seed	0.01, 0.1	5, 5	93, 98	3.2, 3.2	3.4, 3.3	
Grapes	0.01, 0.1	5, 5	94, 99	6.8, 9.8	7.2, 9.9	
Tomatoes	0.01, 0.1	5, 5	93, 97	7.6, 4.6	8.2, 4.8	
Potatoes	0.01, 0.1	5, 5	105, 103	8.4, 2.8	8.0, 2.7	

The DuPont-30422 method was validated by Brown & Woodmansey, 2012, [Ref: DP-31091] for grapes, grape pomace, wheat forage and potato tubers (from 0.01–0.1 mg/kg) using the Charles

River Method 1846a and a slightly modified method (Method 1846b) was validated for watery matrices; grape juice and wine.

These methods involved extraction with water, acetonitrile and formic acid with a 1.0 mL aliquot of the extract being diluted to 10 mL using water, acetonitrile and spiked with 0.01 mL of formic acid before transferring an aliquot of the final extract to an autosampler vial for analysis. Analysis was by liquid chromatography with tandem mass spectrometry employing turbo-ion spray ionization. The compounds IN-E8S72 and IN-WR791 were analysed in the negative ion mode and oxathiapiprolin, IN-Q7H09 and IN-RDG40 were analysed in the positive ion mode. Method validation recovery data from this study are summarized below.

Table 37 DuPont-30422 analytical method – Validation recovery rates in plant matrices.

Matrix	Fortification level (mg/kg)	Number of tests	Average % recovery	Standard deviation	% RSD	Reference
Oxathiapiprolin						
Grapes	0.01, 0.1	5, 5	100, 98	2, 4	2, 4	DP-31091
Grapes Pomace	0.01, 0.1	5, 5	98, 112	15, 6	15, 6	
Wheat Forage	0.01, 0.1	5, 5	99, 109	5, 2	5, 2	
Potatoes Tubers	0.01, 0.1	5, 5	112, 110	7, 5	6, 5	
Grape Juice	0.01, 0.1	5, 5	85, 83	4, 4	4, 5	
Wine	0.01, 0.1	5, 5	93, 85	6, 2	6, 3	
IN-E8S72						
Grapes	0.01, 0.1	5, 5	86, 90	4, 5	5, 5	DP-31091
Grapes Pomace	0.01, 0.1	5, 5	90, 100	4, 3	5, 3	
Wheat Forage	0.01, 0.1	5, 5	90, 93	2, 1	2, 2	
Potatoes Tubers	0.01, 0.1	5, 5	106, 92	5, 3	5, 3	
Grape Juice	0.01, 0.1	5, 5	88, 83	3, 3	3, 3	
Wine	0.01, 0.1	5, 5	94, 85	6, 1	6, 2	
IN-WR791						
Grapes	0.01, 0.1	5, 5	84, 93	3, 4	4, 4	DP-31091
Grapes Pomace	0.01, 0.1	5, 5	90, 100	7, 4	8, 4	
Wheat Forage	0.01, 0.1	5, 5	87, 90	2, 1	2, 1	
Potatoes Tubers	0.01, 0.1	5, 5	109, 99	2, 2	2, 2	
Grape Juice	0.01, 0.1	5, 5	75, 80	2, 4	3, 5	
Wine	0.01, 0.1	5, 5	83, 82	4, 1	5, 2	
IN-RDG40						
Grapes	0.01, 0.1	5, 5	99, 95	3, 5	3, 5	DP-31091
Grapes Pomace	0.01, 0.1	5, 5	105, 107	12, 3	11, 3	
Wheat Forage	0.01, 0.1	5, 5	95, 101	4, 3	4, 3	
Potatoes Tubers	0.01, 0.1	5, 5	106, 93	9, 4	8, 4	
Grape Juice	0.01, 0.1	5, 5	105, 101	3, 4	2, 4	
Wine	0.01, 0.1	5, 5	108, 100	8, 4	7, 4	
IN-Q7H09						
Grapes	0.01, 0.1	5, 5	91, 95	2, 4	2, 4	DP-31091
Grapes Pomace	0.01, 0.1	5, 5	92, 100	7, 3	8, 3	
Wheat Forage	0.01, 0.1	5, 5	94, 100	3, 3	3, 3	
Potatoes Tubers	0.01, 0.1	5, 5	104, 91	6, 3	6, 3	
Grape Juice	0.01, 0.1	5, 5	85, 81	7, 4	8, 5	
Wine	0.01, 0.1	5, 5	95, 85	5, 4	5, 5	

Method DuPont-30422 Supplement No. 1 (Oxathiapiprolin and seven metabolites - fresh and processed plant matrices)

A modification of Method DuPont-30422 (in order to analyse for additional analytes – IN-SXS67, IN-RZB20 and IN-RZD74 in crop matrices) was reported by Henze & Stry, 2013 [Ref: DP-30422-2].

Residues were extracted from samples using a solution of formic acid, water and acetonitrile, diluted with aqueous formic acid and methanol and analysed by reverse-phase LC-MS/MS. Quantitation and confirmation was performed using the following mass transitions:

Analyte	Quantitation	Confirmation
Oxathiapiprolin	540→163	540→500
IN-E8S72	179→65	179→40
IN-WR791	207→143	207→163
IN-RDG40	556→174	556→163
IN-Q7H09	556→155	556→538
IN-SXS67	341→135	341→297
IN-RZB20	222→192	222→42
IN-RZD74	165→65	165→135

A solid phase extraction phase (SPE) clean up step using a graphetized carbon cartridge was used for some matrices in order to negate any possible interference and the polar (IN-SXS67, IN-RZB20, IN-RZD74, IN-WR791, and IN-E8S72) and non-polar (IN-RDG40, IN-Q7H09, and oxathiapiprolin) compounds were eluted separately. As a result, for these matrices the analysis was divided into two injections, polar compounds and non-polar compounds. For the analysis of oranges, grapes, wheat straw, and canola the SPE clean-up was used for the polar compounds. For the analysis of ginseng the SPE clean-up was used for the analysis of the polar and non-polar compounds.

Good linearity was observed (6 point calibrations) in the range of 0.050 to 5.0 ng/mL for oxathiapiprolin and metabolites.

Extraction efficiency was investigated by Chapleo & Inns, 2013 [Ref: DP-33818] by extracting incurred residue samples of lettuce, grape berries, and wheat grain. The residue profiles obtained were compared with those obtained for the same samples in the plant metabolism study. Comparable levels of radioactive residues were liberated using both methods for all 3 commodities. Radioactive residues extracted using the crop residue method and the metabolism method are summarized below:

Table 38 Comparison of residue extraction efficiencies

Commodity	% Extracted		Residue method as % of metabolism method
	Residue method	Metabolism method	
Lettuce Foliage	87.7	89.2	98
Grape Berries	94.8	91.7	103
Wheat Grain	80.3	71.3	113

Radioactive components extracted from the samples using each method were compared by HPLC. The profiles showed similar distribution and levels of metabolites, regardless of which extraction technique was used. All metabolites of interest were identified in at least one of the sample matrices. No difference between the methods exceeded 7.9% TRR (0.016 mg/kg).

Residue field trial concurrent recovery rates where this method was used are summarized in the discussions on the individual crop field trials. These concurrent recoveries produced similar results to the fortification data obtained during method validation. The average recoveries ranged from 70–120%, with a standard deviation of ≤ 20%. Therefore, the recovery of this method is adequate for the purposes of residue data collection.

Table 39 DuPont-30422 Supplement No. 1 analytical method validation recovery rates in plant matrices.

Matrix	Fortification level (mg/kg)	Number of tests	Average % recovery	Standard deviation	% RSD	Reference
Oxathiapiprolin						
Wheat Grain	0.01, 0.1	5, 5	88, 94	3.0, 1.5	3.5, 1.6	DP-30422-2

Matrix	Fortification level (mg/kg)	Number of tests	Average % recovery	Standard deviation	% RSD	Reference
Ginseng (with SPE)	0.01, 0.1	5, 5	97, 99	15, 5.9	16, 6.0	
Canola	0.01, 0.1	5, 5	115, 106	4.3, 0.8	3.7, 0.8	
Wheat Straw	0.01, 0.1	5, 5	89, 96	5.9, 2.0	6.6, 2.1	
Oranges	0.01, 0.1	5, 5	94, 86	4.9, 3.1	5.2, 3.6	
Grapes	0.01, 0.1	5, 5	93, 99	4.3, 1.3	4.7, 1.3	
Tomatoes	0.01, 0.1	5, 5	92, 95	11, 6.5	12, 6.9	
Potatoes	0.01, 0.1	5, 5	106, 105	6.4, 3.9	6.0, 3.7	
IN-SXS67						
Wheat Grain	0.01, 0.1	5, 5	86, 90	7.9, 6.0	9.2, 6.7	DP-30422-2
Ginseng (with SPE)	0.01, 0.1	5, 5	83, 83	9.3, 2.2	11, 2.6	
Canola (with SPE)	0.01, 0.1	5, 5	93, 92	6.5, 6.1	7.0, 6.6	
Wheat Straw (with SPE)	0.01, 0.1	5, 5	104, 98	13, 6.8	13, 7.0	
Oranges (with SPE)	0.01, 0.1	5, 5	90, 88	12, 13	13, 15	
Grapes (with SPE)	0.01, 0.1	5, 5	105, 103	8.8, 5.6	8.4, 5.4	
Tomatoes	0.01, 0.1	5, 5	100, 83	6.3, 5.2	6.3, 6.3	
Potatoes	0.01, 0.1	5, 5	95, 85	5.9, 2.3	6.2, 2.7	
IN-RZB20						
Wheat Grain	0.01, 0.1	5, 5	106, 109	6.2, 9.1	5.8, 9.4	DP-30422-2
Ginseng (with SPE)	0.01, 0.1	5, 5	94, 105	4.0, 6.8	4.2, 6.5	
Canola (with SPE)	0.01, 0.1	5, 5	94, 91	8.9, 3.6	9.5, 4.0	
Wheat Straw (with SPE)	0.01, 0.1	5, 5	104, 89	9.8, 11	9.5, 12	
Oranges (with SPE)	0.01, 0.1	5, 5	83, 76	6.1, 4.1	7.4, 5.4	
Grapes (with SPE)	0.01, 0.1	5, 5	100, 104	10, 4.3	10, 4.2	
Tomatoes	0.01, 0.1	5, 5	91, 90	5.6, 3.6	6.1, 4.1	
Potatoes	0.01, 0.1	5, 5	95, 92	7.0, 6.9	7.4, 7.5	
IN-RZD74						
Wheat Grain	0.01, 0.1	5, 5	97, 99	6.9, 3.0	7.1, 3.1	DP-30422-2
Ginseng (with SPE)	0.01, 0.1	5, 5	82, 91	4.4, 2.3	5.4, 2.5	
Canola, (SPE Used)	0.01, 0.1	5, 5	94, 90	5.4, 0.5	5.7, 0.6	
Wheat Straw (with SPE)	0.01, 0.1	5, 5	91, 89	2.8, 1.6	3.0, 1.8	
Oranges (with SPE)	0.01, 0.1	5, 5	93, 86	3.3, 1.8	3.6, 2.1	
Grapes (with SPE)	0.01, 0.1	5, 5	99, 99	4.2, 2.2	4.2, 2.2	
Tomatoes	0.01, 0.1	5, 5	97, 94	6.9, 3.7	7.1, 3.9	
Potatoes	0.01, 0.1	5, 5	96, 95	5.5, 3.7	5.8, 3.9	
IN-WR791						
Wheat Grain	0.01, 0.1	5, 5	96, 101	4.1, 4.6	4.3, 4.6	DP-30422-2
Ginseng (with SPE)	0.01, 0.1	5, 5	91, 95	3.7, 2.6	4.1, 2.7	
Canola (with SPE)	0.01, 0.1	5, 5	108, 101	5.7, 2.9	5.2, 2.8	
Wheat Straw (with SPE)	0.01, 0.1	5, 5	94, 90	7.9, 6.3	8.4, 7.0	
Oranges (with SPE)	0.01, 0.1	5, 5	101, 96	3.6, 1.9	3.6, 2.0	
Grapes (with SPE)	0.01, 0.1	5, 5	96, 96	4.6, 4.1	4.8, 4.3	
Tomatoes	0.01, 0.1	5, 5	92, 89	5.2, 2.9	5.6, 3.2	
Potatoes	0.01, 0.1	5, 5	94, 90	3.3, 3.1	3.5, 3.5	
IN-E8S72						
Wheat Grain	0.01, 0.1	5, 5	93, 94	8.5, 3.7	9.2, 4.0	DP-30422-2
Ginseng (with SPE)	0.01, 0.1	5, 5	93, 93,	18, 1.6	20, 1.8	
Canola (with SPE)	0.01, 0.1	5, 5	104, 95	4.2, 3.3	4.1, 3.4	
Wheat Straw (with SPE)	0.01, 0.1	5, 5	91, 86	17, 8.3	19, 9.7	
Oranges (with SPE)	0.01, 0.1	5, 5	105, 96	2.7, 3.2	2.6, 3.3	
Grapes (with SPE)	0.01, 0.1	5, 5	98, 97	6.7, 3.8	6.8, 4.0	
Tomatoes	0.01, 0.1	5, 5	99, 100	6.6, 1.9	6.7, 1.9	
Potatoes	0.01, 0.1	5, 5	111, 106	6.3, 3.4	5.7, 3.2	
IN-RDG40						
Wheat Grain	0.01, 0.1	5, 5	101, 101	6.1, 3.2	6.1, 3.2	DP-30422-2
Ginseng (with SPE)	0.01, 0.1	5, 5	100, 98	19, 5.0	19, 5.1	
Canola	0.01, 0.1	5, 5	107, 107	2.4, 3.4	2.2, 3.2	
Wheat Straw	0.01, 0.1	5, 5	99, 95	7.0, 5.5	7.1, 5.8	
Oranges	0.01, 0.1	5, 5	113, 108	7.0, 4.3	6.2, 4.0	
Grapes	0.01, 0.1	5, 5	97, 101	8.5, 3.9	8.8, 3.9	
Tomatoes	0.01, 0.1	5, 5	95, 96	3.4, 4.2	3.6, 4.4	

Matrix	Fortification level (mg/kg)	Number of tests	Average % recovery	Standard deviation	% RSD	Reference
Potatoes	0.01, 0.1	5, 5	106, 104	6.4, 3.3	6.0, 3.2	
IN-Q7H09						
Wheat Grain	0.01, 0.1	5, 5	99, 103	7.1, 3.7	7.2, 3.6	DP-30422-2
Ginseng (with SPE)	0.01, 0.1	5, 5	105, 110	8.9, 13	8.5, 12	
Canola	0.01, 0.1	5, 5	107, 108	6.1, 8.2	5.7, 7.5	
Wheat Straw	0.01, 0.1	5, 5	110, 96	5.6, 5.2	5.1, 5.4	
Oranges	0.01, 0.1	5, 5	87, 82	1.4, 3.0	1.6, 3.7	
Grapes	0.01, 0.1	5, 5	99, 99	3.9, 1.3	4.0, 13	
Tomatoes	0.01, 0.1	5, 5	96, 98	5.0, 4.0	5.2, 4.1	
Potatoes	0.01, 0.1	5, 5	111, 111	6.1, 5.0	5.4, 4.5	

The DuPont-30422 Supplement No. 1 method was validated by Brown & Woodmansey, 2012, [Ref: DP-31091] for carrot roots, spinach leaves, dry beans, wheat grain and wheat straw using the Charles River Method 1846c (using UPLC chromatography rather than HPLC); by Vincent, 2013 [Ref: DP-31545] for a range of fresh and processed plant commodities (see the validation table below) with the SPE clean-up step being included for all samples. An independent laboratory validation study was also conducted by Lissemore & Li, 2013 [Ref: DP-37739] for oxathiapiprolin, IN-SXS67, IN-WR791 and IN-E8S72 in wheat grain, grapes and tomato.

Table 40 DuPont-30422 Supplement No. 1 analytical method – Recovery rates in plant matrices from Validation [DP-31091] and Independent validation [DP-37739] studies.

Matrix	Fortification level, (mg/kg)	Number of tests	Average % recovery	Standard deviation	% RSD	Reference
Oxathiapiprolin						
Carrot Roots (with SPE)	0.01, 0.1	5, 5	79, 91	7, 2	9, 3	DP-31091
Spinach Leaves (with SPE)	0.01, 0.1	5, 5	87, 93	6, 2	7, 2	
Dry Beans (with SPE)	0.01, 0.1	5, 5	98, 100	9, 2	9, 2	
Wheat Grain (with SPE)	0.01, 0.1	5, 5	94, 89	3, 4	4, 5	
Wheat Straw (with SPE)	0.01, 0.1	5, 5	71, 74	3, 2	5, 2	
Wheat Grain	0.01, 0.1	5, 5	78, 84	0.66, 0.85	0.84, 1.0	DP-37739
Grapes	0.01, 0.1	5, 5	95, 100	1.4, 0.91	1.5, 0.91	
Tomatoes	0.01, 0.1	5, 5	106, 101	1.8, 2.2	1.7, 2.2	
Wheat Forage	0.01, 0.1	5, 5	104, 104	4, 2	4, 2	DP-31545
Wheat Grain	0.01, 0.1	5, 5	102, 98	4, 7	4, 7	
Wheat Straw	0.01, 0.1	5, 5	102, 104	4, 7	4, 6	
Potato	0.01, 0.1	5, 5	97, 97	17, 4	17, 4	
Potato Chips	0.01, 0.1	5, 5	96, 96	1, 2	1, 2	
Grapes	0.01, 0.1	5, 5	98, 107	5, 9	5, 8	
Dry Grape Pomace (with SPE)	0.01, 0.1	5, 5	97, 96	6, 7	7, 7	
Tomato	0.01, 0.1	5, 5	108, 102	2, 4	2, 4	
Tomato Juice	0.01, 0.1	5, 5	107, 105	3, 1	3, 1	
Spinach	0.01, 0.1	5, 5	93, 111	8, 4	8, 3	
Broccoli	0.01, 0.1	5, 5	106, 98	4, 9	4, 9	
Dry Bulb Onion	0.01, 0.1	5, 5	95, 104	6, 3	6, 3	DP-31545
Whole Pepper	0.01, 0.1	5, 5	109, 110	2, 1	2, 1	
Dried Soya bean Seed	0.01, 0.1	5, 5	93, 97	10, 8	11, 8	
Dried Beans	0.01, 0.1	5, 5	102, 103	6, 7	6, 7	
Dried Ginseng Root (with SPE)	0.01, 0.1	5, 5	111, 100	12, 14	11, 14	
Dried Tobacco Leaves	0.01, 0.1	5, 5	106, 107	9, 2	8, 2	
IN-SXS67						
Carrot Roots (with SPE)	0.01, 0.1	5, 5	83, 93	3, 4	3, 4	DP-31091
Spinach Leaves (with SPE)	0.01, 0.1	5, 5	101, 95	4, 4	4, 4	
Dry Beans (with SPE)	0.01, 0.1	5, 5	84, 86	5, 2	6, 3	
Wheat Grain (with SPE)	0.01, 0.1	5, 5	88, 85	4, 3	5, 3	
Wheat Straw (with SPE)	0.01, 0.1	5, 5	80, 85	4, 2	6, 3	
Wheat Forage	0.01, 0.1	5, 5	90, 90	7, 3	8, 3	DP-31545
Wheat Grain	0.01, 0.1	5, 5	91, 93	13, 2	14, 2	
Wheat Straw	0.01, 0.1	5, 5	94, 88	10, 2	11, 2	

Matrix	Fortification level, (mg/kg)	Number of tests	Average % recovery	Standard deviation	% RSD	Reference
Potato	0.01, 0.1	5, 5	99, 100	4, 2	4, 2	
Potato Chips	0.01, 0.1	5, 5	111, 105	4, 5	3, 5	DP-31545
Grapes	0.01, 0.1	5, 5	94, 95	6, 2	7, 3	
Dry Grape Pomace (with SPE)	0.01, 0.1	5, 5	88, 104	9, 3	10, 3	
Tomato	0.01, 0.1	5, 5	114, 98	8, 2	7, 2	
Tomato Juice	0.01, 0.1	5, 5	101, 99	9, 3	9, 3	
Spinach	0.01, 0.1	5, 5	92, 94	4, 3	5, 4	
Broccoli	0.01, 0.1	5, 5	93, 91	3, 1	4, 1	
Dry Bulb Onion	0.01, 0.1	5, 5	104, 105	3, 2	3, 2	
Whole Pepper	0.01, 0.1	5, 5	99, 99	3, 1	3, 2	
Dried Soya bean Seed	0.01, 0.1	5, 5	81, 87	7, 4	8, 4	
Dried Beans	0.01, 0.1	5, 5	91, 96	2, 4	2, 4	
Dried Ginseng Root (with SPE)	0.01, 0.1	5, 5	106, 106	5, 8	5, 8	
Dried Tobacco Leaves	0.01, 0.1	5, 5	111, 116	12, 3	10, 3	
Wheat Grain (with SPE)	0.01, 0.1	5, 5	96, 102	0.36, 0.84	0.38, 0.82	DP-37739
Grapes (with SPE)	0.01, 0.1	5, 5	96, 96	1.0, 0.99	1.0, 0.99	
Tomatoes (with SPE)	0.01, 0.1	5, 5	96, 102	0.45, 1.7	0.47, 1.7	
IN-RZB20						
Carrot Roots (with SPE)	0.01, 0.1	5, 5	83, 92	4, 3	4, 4	DP-31091
Spinach Leaves (with SPE)	0.01, 0.1	5, 5	80, 89	4, 6	5, 7	
Dry Beans (with SPE)	0.01, 0.1	5, 5	92, 90	4, 4	4, 4	
Wheat Grain (with SPE)	0.01, 0.1	5, 5	71, 86	4, 2	6, 3	
Wheat Straw (with SPE)	0.01, 0.1	5, 5	82, 84	10, 2	12, 3	
Wheat Forage	0.01, 0.1	5, 5	88, 93	7, 3	7, 3	DP-31545
Wheat Grain	0.01, 0.1	5, 5	86, 91	7, 3	8, 3	
Wheat Straw	0.01, 0.1	5, 5	92, 90	9, 4	10, 4	
Potato	0.01, 0.1	5, 5	104, 103	8, 4	8, 4	
Potato Chips	0.01, 0.1	5, 5	91, 96	4, 4	5, 5	
Grapes	0.01, 0.1	5, 5	93, 93	7, 3	7, 3	
Dry Grape Pomace (with SPE))	0.01, 0.1	5, 5	100, 95	7, 2	7, 2	
Tomato	0.01, 0.1	5, 5	101, 94	6, 3	6, 4	
Tomato Juice	0.01, 0.1	5, 5	102, 104	9, 2	9, 1	
Spinach	0.01, 0.1	5, 5	95, 97	8, 2	8, 2,	
Broccoli	0.01, 0.1	5, 5	96, 96	5, 2	6, 2	
Dry Bulb Onion	0.01, 0.1	5, 5	104, 105	11, 2	10, 2	DP-31545
Whole Pepper	0.01, 0.1	5, 5	96, 102	7, 4	7, 4	
Dried Soya bean Seed	0.01, 0.1	5, 5	83, 85	6, 3	8, 3	
Dried Beans	0.01, 0.1	5, 5	90, 91	10, 3	11, 3	
Dried Ginseng Root (with SPE)	0.01, 0.1	5, 5	96, 105	7, 7	7, 7	
Dried Tobacco Leaves	0.01, 0.1	5, 5	84, 86	5, 3	6, 4	
IN-RZD74						
Carrot Roots (with SPE)	0.01, 0.1	5, 5	87, 101	9, 2	11, 2	DP-31091
Spinach Leaves (with SPE)	0.01, 0.1	5, 5	93, 91	4, 4	4, 4	
Dry Beans (with SPE)	0.01, 0.1	5, 5	95, 98	3, 2	3, 2	
Wheat Grain (with SPE)	0.01, 0.1	5, 5	91, 94	5, 3	6, 3	
Wheat Straw (with SPE)	0.01, 0.1	5, 5	85, 88	5, 1	6, 2	
Wheat Forage	0.01, 0.1	5, 5	93, 92	6, 5	6, 6	DP-31545
Wheat Grain	0.01, 0.1	5, 5	91, 94	8, 4	9, 4	
Wheat Straw	0.01, 0.1	5, 5	105, 80	4, 2	4, 2	
Potato	0.01, 0.1	5, 5	98, 104	7, 6	7, 5	
Potato Chips	0.01, 0.1	5, 5	98, 97	2, 5	2, 5	DP-31545
Grapes	0.01, 0.1	5, 5	86, 81	8, 7	9, 9	
Dry Grape Pomace (with SPE)	0.01, 0.1	5, 5	104, 100	5, 2	5, 2	
Tomato	0.01, 0.1	5, 5	97, 95	5, 3	5, 3	
Tomato Juice	0.01, 0.1	5, 5	103, 103	2, 2	2, 2	
Spinach	0.01, 0.1	5, 5	94, 94	2, 2	2, 2	
Broccoli	0.01, 0.1	5, 5	74, 71	1, 2	1, 2	
Dry Bulb Onion	0.01, 0.1	5, 5	102, 106	2, 3	2, 2	
Whole Pepper	0.01, 0.1	5, 5	101, 100	2, 3	2, 3	
Dried Soya bean Seed	0.01, 0.1	5, 5	94, 80	7, 4	7, 5	

Matrix	Fortification level, (mg/kg)	Number of tests	Average % recovery	Standard deviation	% RSD	Reference
Dried Beans	0.01, 0.1	5, 5	91, 92	4, 3	4, 4	
Dried Ginseng Root (with SPE)	0.01, 0.1	5, 5	98, 102	6, 11	6, 11	
Dried Tobacco Leaves	0.01, 0.1	5, 5	104, 101	8, 2	7, 2	
IN-WR791						
Carrot Roots (with SPE)	0.01, 0.1	5, 5	71, 88	5, 3	7, 3	DP-31091
Spinach Leaves (with SPE)	0.01, 0.1	5, 5	88, 82	6, 6	7, 8	
Dry Beans (with SPE)	0.01, 0.1	5, 5	83, 95	5, 3	6, 4	
Wheat Grain (with SPE)	0.01, 0.1	5, 5	80, 85	11, 4	13, 5	
Wheat Straw (with SPE)	0.01, 0.1	5, 5	86, 80	13, 6	16, 7	
Wheat Forage	0.01, 0.1	5, 5	94, 89	5, 2	6, 2	DP-31545
Wheat Grain	0.01, 0.1	5, 5	87, 90	6, 7	7, 8	
Wheat Straw	0.01, 0.1	5, 5	92, 94	5, 2	5, 2	
Potato	0.01, 0.1	5, 5	101, 100	4, 3	4, 3	
Potato Chips	0.01, 0.1	5, 5	100, 101	4, 5	4, 5	
Grapes	0.01, 0.1	5, 5	98, 97	1, 2	1, 2	
Dry Grape Pomace (with SPE)	0.01, 0.1	5, 5	100, 98	6, 4	6, 4	
Tomato	0.01, 0.1	5, 5	97, 97	6, 2	6, 2	
Tomato Juice	0.01, 0.1	5, 5	106, 103	3, 2	3, 2	
Spinach	0.01, 0.1	5, 5	93, 91	7, 2	7, 2	
Broccoli	0.01, 0.1	5, 5	96, 98	4, 1	4, 1	
Wheat Grain	0.01, 0.1	5, 5	97, 97	0.92, 1.2	0.95, 1.2	DP-37739
Grapes ,	0.01, 0.1	5, 5	92, 100	0.7, 1.3	0.8, 1.3	
Tomatoes	0.01, 0.1	5, 5	100, 102	1.5, 1.7	1.5, 1.7	
Dry Bulb Onion	0.01, 0.1	5, 5	102, 107	6, 3	6, 3	DP-31545
Whole Pepper	0.01, 0.1	5, 5	101, 103	6, 2	6, 2	
Dried Soya bean Seed	0.01, 0.1	5, 5	77, 82	6, 4	8, 5	
Dried Beans	0.01, 0.1	5, 5	97, 96	5, 4	5, 4	
Dried Ginseng Root (with SPE)	0.01, 0.1	5, 5	102, 109	7, 4	6, 4	
Dried Tobacco Leaves	0.01, 0.1	5, 5	116, 107	3, 2	3, 2	
IN-E8S72						
Carrot Roots (with SPE)	0.01, 0.1	5, 5	73, 91	4, 4	5, 4	DP-31091
Spinach Leaves (with SPE)	0.01, 0.1	5, 5	88, 82	3, 5	3, 6	
Dry Beans (with SPE)	0.01, 0.1	5, 5	89, 93	4, 2	5, 3	
Wheat Grain (with SPE)	0.01, 0.1	5, 5	78, 84	5, 4	7, 5	
Wheat Straw (with SPE)	0.01, 0.1	5, 5	97, 86	5, 3	5, 4	
Wheat Forage	0.01, 0.1	5, 5	89, 88	6, 3	6, 3	DP-31545
Wheat Grain	0.01, 0.1	5, 5	88, 91	9, 7	10, 7	
Wheat Straw	0.01, 0.1	5, 5	102, 98	5, 2	5, 2	
Potato	0.01, 0.1	5, 5	105, 104	2, 3	2, 3	
Potato Chips	0.01, 0.1	5, 5	102, 99	3, 6	3, 6	DP-31545
Grapes	0.01, 0.1	5, 5	106, 105	5, 2	5, 2	
Dry Grape Pomace (with SPE)	0.01, 0.1	5, 5	114, 113	3, 5	2, 4	
Tomato	0.01, 0.1	5, 5	98, 97	2, 2	2, 2	
Tomato Juice	0.01, 0.1	5, 5	103, 105	2, 2	2, 2	
Spinach	0.01, 0.1	5, 5	98, 99	2, 2	2, 2	
Broccoli	0.01, 0.1	5, 5	100, 99	5, 2	5, 2	
Dry Bulb Onion	0.01, 0.1	5, 5	101, 86	5, 3	5, 3	
Whole Pepper	0.01, 0.1	5, 5	109, 107	3, 4	3, 4	
Dried Soya bean Seed	0.01, 0.1	5, 5	84, 91	8, 4	10, 4	
Dried Beans	0.01, 0.1	5, 5	97, 100	3, 5	3, 5	
Dried Ginseng Root (with SPE)	0.01, 0.1	5, 5	114, 120	3, 6	3, 5	
Dried Tobacco Leaves	0.01, 0.1	5, 5	104, 112	7, 3	7, 2	
Wheat Grain	0.01, 0.1	5, 5	75, 84	1.4, 1.1	1.9, 1.3	DP-37739
Grapes ,	0.01, 0.1	5, 5	93, 100	0.16, 0.88	0.17, 0.88	
Tomatoes	0.01, 0.1	5, 5	98, 103	1.3, 2.6	1.3, 2.5	
IN-RDG40						
Carrot Roots (with SPE)	0.01, 0.1	5, 5	76, 73	6, 3	8, 3	DP-31091
Spinach Leaves (with SPE)	0.01, 0.1	5, 5	102, 97	8, 7	8, 8	
Dry Beans (with SPE)	0.01, 0.1	5, 5	90, 93	3, 2	4, 2	
Wheat Grain (with SPE)	0.01, 0.1	5, 5	91, 93	2, 14	2, 15	

Matrix	Fortification level, (mg/kg)	Number of tests	Average % recovery	Standard deviation	% RSD	Reference
Wheat Straw (with SPE)	0.01, 0.1	5, 5	75, 75	4, 1	5, 1	
Wheat Forage	0.01, 0.1	5, 5	105, 99	4, 2	4, 2	DP-31545
Wheat Grain	0.01, 0.1	5, 5	113, 113	2, 3	2, 2	
Wheat Straw	0.01, 0.1	5, 5	86, 85	2, 3	2, 3	
Potato	0.01, 0.1	5, 5	95, 101	14, 2	15, 2	
Potato Chips	0.01, 0.1	5, 5	109, 108	3, 2	3, 2	
Grapes	0.01, 0.1	5, 5	115, 116	4, 10	3, 9	
Dry Grape Pomace (with SPE)	0.01, 0.1	5, 5	111, 108	4, 2	3, 2	
Tomato	0.01, 0.1	5, 5	108, 109	2, 2	2, 2	
Tomato Juice	0.01, 0.1	5, 5	119, 117	5, 0	4, 0	
Spinach	0.01, 0.1	5, 5	108, 100	5, 1	4, 1	
Broccoli	0.01, 0.1	5, 5	103, 105	4, 2	4, 2	
Dry Bulb Onion	0.01, 0.1	5, 5	116, 116	4, 1	3, 1	DP-31545
Whole Pepper	0.01, 0.1	5, 5	106, 105	3, 0	3, 0	
Dried Soya bean Seed	0.01, 0.1	5, 5	92, 94	12, 3	13, 3	
Dried Beans	0.01, 0.1	5, 5	115, 114	5, 2	4, 1	
Dried Ginseng Root (with SPE)	0.01, 0.1	5, 5	106, 105	13, 10	12, 10	
Dried Tobacco Leaves	0.01, 0.1	5, 5	84, 93	4, 2	5, 2	
IN-Q7H09						
Carrot Roots (with SPE)	0.01, 0.1	5, 5	85, 93	12, 2	14, 2	DP-31091
Spinach Leaves (with SPE)	0.01, 0.1	5, 5	94, 96	2, 3	2, 3	
Dry Beans (with SPE)	0.01, 0.1	5, 5	92, 91	3, 3	4, 3	
Wheat Grain (with SPE)	0.01, 0.1	5, 5	106, 91	5, 6	5, 6	
Wheat Straw (with SPE)	0.01, 0.1	5, 5	79, 75	5, 2	6, 3	
Wheat Forage	0.01, 0.1	5, 5	88, 92	3, 2	3, 2	DP-31545
Wheat Grain	0.01, 0.1	5, 5	99, 100	4, 2	4, 2	
Wheat Straw	0.01, 0.1	5, 5	88, 87	5, 2	6, 3	
Potato	0.01, 0.1	5, 5	97, 104	11, 3	12, 3	
Potato Chips	0.01, 0.1	5, 5	93, 93	7, 1	7, 2	DP-31545
Grapes	0.01, 0.1	5, 5	105, 107	3, 9	3, 8	
Dry Grape Pomace (with SPE)	0.01, 0.1	5, 5	99, 101	11, 2	12, 2	
Tomato	0.01, 0.1	5, 5	95, 103	7, 0	8, 0	
Tomato Juice	0.01, 0.1	5, 5	111, 108	3, 1	3, 1	
Spinach	0.01, 0.1	5, 5	79, 80	3, 7	3, 8	
Broccoli	0.01, 0.1	5, 5	95, 97	8, 1	8, 1	
Dry Bulb Onion	0.01, 0.1	5, 5	100, 102	6, 1	6, 1	
Whole Pepper	0.01, 0.1	5, 5	103, 103	6, 1	6, 1	
Dried Soya bean Seed	0.01, 0.1	5, 5	76, 79	6, 2	8, 2	
Dried Beans	0.01, 0.1	5, 5	106, 100	4, 2	4, 2	
Dried Ginseng Root (with SPE)	0.01, 0.1	5, 5	94, 98	3, 10	3, 10	
Dried Tobacco Leaves	0.01, 0.1	5, 5	86, 94	5, 4	6, 4	

Analytical (concurrent) recoveries in supervised crop trials

Analytical recovery rates were measured in all the supervised crop field trials, with control samples being fortified with oxathiapiprolin and metabolites IN-E8S72, IN-WR791, IN-RDG40, IN-Q7H09, IN-SXS67, IN-RZB20, IN-RZD74 at 0.01 mg/kg and at higher levels (up to 25 mg/kg) that generally reflected the range of expected residues in the commodities tested. For each study, average recoveries per fortification level generally fell within the 70–120% range, with a relative standard deviation of 20% or less. Mean recovery rates for each analyte are summarized in the individual crop residue supervised trials evaluations.

Method DuPont-31138 (Oxathiapiprolin and four metabolites – animal tissues, milk and eggs)

This LC-MS/MS method for measuring residues of oxathiapiprolin and its IN-RAB06, IN-RLB67, IN-RDG40 and IN-Q7H09 metabolites in animal tissues, milk and eggs was reported by Henze and Stry, 2012 [Ref: DP-31138].

In this method, Oxathiapiprolin and metabolites were obtained from animal tissue, milk and egg samples by extraction with acetonitrile:hexane and acetonitrile:water. The extracts were diluted to 50 mL and an aliquot was removed and diluted to 7 mL using an aqueous formic acid solution and methanol. An aliquot of the diluted extract was transferred to an auto-sampler vial for analysis.

Oxathiapiprolin, IN-RAB06, IN-RLB67, IN-RDG40 and IN-Q7H09 were separated from co-extracts by reversed phase liquid chromatography and detected by electrospray mass spectrometry/mass spectrometry (MS/MS). Quantitation and confirmation was performed using the following mass transitions:

Analyte	Quantitation	Confirmation
Oxathiapiprolin	540→163	540→500
IN-RAB06	568→524	568→135
IN-RLB67	572→554	572→163
IN-RDG40	556→174	556→163
IN-Q7H09	556→155	556→538

Good linearity was observed in the range of 0.05 ng/mL to 5.0 ng/mL for oxathiapiprolin and no residues were detected in control samples; the response in the areas of the oxathiapiprolin and metabolites peaks always corresponded to less than 20% of the limit of determination. This method is capable of determining oxathiapiprolin and metabolite residues in milk, eggs, and meat tissues with a limit of quantification of 0.01 mg/kg for milk, eggs, and meat tissues. Recovery rates were within guideline requirements (70–120%, RSD ≤ 20%).

Table 41 DuPont-31138 analytical method validation recovery rates in animal matrices.

Matrix	Number of tests	Fortification level (mg/kg)	Average % recovery	Standard deviation	% RSD	Reference
Oxathiapiprolin						
Whole Eggs	5, 5	0.01, 0.1	97, 100	5.0, 6.2	5.1, 6.2	DP-31138
Whole Milk	5, 5	0.01, 0.1	110, 104	11, 13	10, 13	
Heavy Cream	5, 5	0.01, 0.1	104, 100	6.5, 4.4	6.2, 4.4	
Beef Fat	5, 5	0.01, 0.1	101, 105	11, 4.9	11, 4.7	
Beef Muscle	5, 5	0.01, 0.1	115, 103	9.1, 10	7.9, 9.9	
Beef Kidney	5, 5	0.01, 0.1	95, 95	4.2, 3.6	4.4, 3.8	
Beef Liver	5, 5	0.01, 0.1	100, 107	6.7, 2.6	6.7, 2.4	
IN-RAB06						
Whole Eggs	5, 5	0.01, 0.1	95, 96	8.0, 8.0	8.4, 8.4	DP-31138
Whole Milk	5, 5	0.01, 0.1	101, 99	5.2, 1.6	5.1, 1.6	
Heavy Cream	5, 5	0.01, 0.1	100, 99	4.6, 4.6	4.6, 4.6	
Beef Fat	5, 5	0.01, 0.1	101, 102	5.9, 1.1	5.9, 1.1	
Beef Muscle	5, 5	0.01, 0.1	99, 82	13, 4.1	13, 5.0	
Beef Kidney	5, 5	0.01, 0.1	98, 102	4.7, 2.6	4.8, 2.5	
Beef Liver	5, 5	0.01, 0.1	102, 104	19, 4.9	18, 4.7	
IN-RLB67						
Whole Eggs	5, 5	0.01, 0.1	108, 100	8.1, 2.0	7.5, 2.0	DP-31138
Whole Milk	5, 5	0.01, 0.1	98, 98	4.5, 3.8	4.6, 3.9	
Heavy Cream	5, 5	0.01, 0.1	100, 101	1.8, 5.2	1.8, 5.1	
Beef Fat	5, 5	0.01, 0.1	102, 107	9.8, 5.8	9.6, 5.5	
Beef Muscle	5, 5	0.01, 0.1	119, 99	18, 2.6	15, 2.6	
Beef Kidney	5, 5	0.01, 0.1	99, 101	5.5, 4.9	5.6, 4.9	
Beef Liver	5, 5	0.01, 0.1	103, 110	11, 5.4	10, 4.9	
IN-RDG40						
Whole Eggs	5, 5	0.01, 0.1	88, 95	11, 4.0	13, 4.1	DP-31138
Whole Milk	5, 5	0.01, 0.1	106, 99	13, 4.2	12, 4.3	

Matrix	Number of tests	Fortification level (mg/kg)	Average % recovery	Standard deviation	% RSD	Reference
Heavy Cream	5, 5	0.01, 0.1	106, 100	14, 3.7	13, 3.7	
Beef Fat	5, 5	0.01, 0.1	107, 109	5.6, 2.7	5.3, 2.5	
Beef Muscle	5, 5	0.01, 0.1	109, 98	4.9, 5.6	4.5, 5.7	
Beef Kidney	5, 5	0.01, 0.1	92, 89	4.1, 7.6	4.5, 8.6	
Beef Liver	5, 5	0.01, 0.1	117, 118	6.8, 6.1	5.8, 5.2	
IN-Q7H09						
Whole Eggs	5, 5	0.01, 0.1	106, 100	7.8, 1.7	7.3, 1.7	DP-31138
Whole Milk	5, 5	0.01, 0.1	109, 100	8.0, 2.3	7.3, 2.3	
Heavy Cream	5, 5	0.01, 0.1	100, 103	8.5, 4.4	8.5, 4.3	
Beef Fat	5, 5	0.01, 0.1	103, 109	4.6, 2.4	4.5, 2.2	
Beef Muscle	5, 5	0.01, 0.1	108, 105	5.8, 3.9	5.4, 3.7	
Beef Kidney	5, 5	0.01, 0.1	96, 101	7.9, 7.1	8.2, 7.1	
Beef Liver	5, 5	0.01, 0.1	113, 116	3.3, 3.6	2.9, 3.1	

The DuPont-31138 method was independently validated by Harris, 2012, [Ref: DP-32355] for muscle, liver, milk and eggs but with modified HPLC conditions to measure IN-RDG40 residues in liver and eggs and a change to the secondary MS/MS transition for determining residues of IN-RLB67 and IN-RDG40 in liver.

Table 42 DuPont-31138 analytical method – Independent validation recovery rates in animal matrices.

Matrix	Number of tests	Fortification level (mg/kg)	Average % recovery	Standard deviation	% RD	Reference
Oxathiapiprolin						
Beef Muscle	5, 5	0.01, 0.1	109, 111	11, 9.4	11, 8.5	DP-32355
Beef Liver	5, 5	0.01, 0.1	97, 107	5.6, 1.8	5.8, 1.7	
Whole Milk	5, 5	0.01, 0.1	97, 91	7.0, 5.1	7.2, 5.6	
Whole Eggs	5, 5	0.01, 0.1	104, 103	3.6, 7.3	3.5, 7.1	
IN-RAB06						
Beef Muscle	5, 5	0.01, 0.1	112, 115	3.0, 2.2	2.7, 1.9	DP-32355
Beef Liver,	5, 5	0.01, 0.1	113, 111	2.6, 1.9	2.3, 1.7	
Whole Milk	5, 5	0.01, 0.1	108, 108	4.2, 1.1	3.9, 1.0	
Whole Eggs	5, 5	0.01, 0.1	114, 119	6.5, 2.4	5.7, 2.0	
IN-RLB67						
Beef Muscle	5, 5	0.01, 0.1	105, 103	8.0, 3.8	7.6, 3.7	DP-32355
Beef Liver	5, 5	0.01, 0.1	97, 100	16, 4.6	17, 4.6	
Whole Milk	5, 5	0.01, 0.1	107, 101	9.1, 1.6	8.5, 1.6	
Whole Eggs	5, 5	0.01, 0.1	102, 104	5.2, 1.5	5.1, 1.4	
IN-RDG40						
Beef Muscle	5, 5	0.01, 0.1	101, 106	4.6, 6.8	4.6, 6.4	DP-32355
Beef Liver	5, 5	0.01, 0.1	124, 113	8.6, 3.6	6.9, 3.2	
Whole Milk	5, 5	0.01, 0.1	102, 104	3.1, 1.6	3.0, 1.6	
Whole Eggs	5, 5	0.01, 0.1	116, 112	5.9, 4.3	5.1, 3.8	
IN-Q7H09						
Beef Muscle	5, 5	0.01, 0.1	105, 103	4.7, 3.5	4.5, 3.4	DP-32355
Beef Liver	5, 5	0.01, 0.1	94, 104	4.2, 3.5	4.5, 3.4	
Whole Milk	5, 5	0.01, 0.1	98, 97	4.0, 1.5	4.1, 1.5	
Whole Eggs	5, 5	0.01, 0.1	108, 109	4.9, 6.3	4.5, 5.8	

Enforcement methods

Multi-residue method DFG S 19 (plant matrices)

The multi-residue method DFG S 19 (LC-MS/MS module) was investigated and validated for the determination of oxathiapiprolin and metabolites IN-SXS67, IN-RZB20, IN-RZD74, IN-E8S72, IN-WR791, IN-RDG40, and IN-Q7H09 in crop matrices by Weber, 2012 [Ref: DP-31140].

Representative high water/low fat content samples (apple and citrus fruit) were extracted using module E 1, low water/low fat content samples (barley grain) were extracted using module E2 and low water/low fat content sample rape seed) were extracted using module E 5. Ethyl acetate/cyclohexane and sodium chloride liquid-liquid partition was used for all samples except rape seed where residues were partitioned with dichloromethane. Samples were cleaned-up using gel permeation chromatography (module GPC), re-suspended in methanol/formic acid and analysed by LC-MS/MS. Quantitation and confirmation was performed using the following mass transitions:

Analyte	Quantitation	Confirmation
Oxathiapiprolin	540→163	540→500
IN-E8S72	179→65	179→40
IN-WR791	207→143	207→163
IN-RDG40	556→174	556→163
IN-Q7H09	556→155	556→538 (556→516 for orange)
IN-SXS67	341→135	341→297
IN-RZB20	222→192	222→42
IN-RZD74	165→65	165→135

The method showed good linearity in the range of 0.25–100 ng/mL for all analytes (correlation coefficients >0.994 and no significant interferences were noted at the retention time corresponding to all analytes in any control samples (the response in the area of the oxathiapiprolin or metabolites peak always corresponded to less than 20% of the limit of determination). The mean recoveries for all matrices tested and at all fortification levels ranged from 70 and 110%, within the acceptable range, with relative standard deviations of < 20% except for rape seed (high oil content), where average recoveries were less than 70%. The limits of quantitation (LOQs) were 0.01 mg/kg for all matrices tested.

Table 43 Multi-residue method DFG S 19 analytical recovery rates for oxathiapiprolin and metabolites in crop matrices

Matrix	Fortification level (mg/kg)	Number of tests	Average % recovery	Standard deviation	% RSD	Reference
Oxathiapiprolin						
Apples	0.01, 0.1	5, 5	80, 77	11, 4.6	13, 6.0	DP-31140
Citrus	0.01, 0.1	5, 5	86, 85	7.6, 9.4	8.8, 11	
Barley Grain	0.01, 0.1	5, 5	77, 74	5.8, 5.7	7.6, 7.7	
Oil Seed Rape	0.01, 0.1	5, 5	51, 43	11, 2.4	22, 5.6	
IN-Q7H09						
Apples	0.01, 0.1	5, 5	77, 76	9.9, 3.3	13, 4.3	DP-31140
Citrus	0.01, 0.1	5, 5	94, 93	5.9, 8.2	6.2, 8.8	
Barley Grain	0.01, 0.1	5, 5	74, 80	4.1, 5.6	5.6, 7.1	
Oil Seed Rape	0.01, 0.1	5, 5	56, 47	12.1, 5.5	22, 12	
IN-RDG40						
Apples	0.01, 0.1	5, 5	85, 85	14, 3.7	16, 4.4	DP-31140
Citrus	0.01, 0.1	5, 5	101, 98	5.3, 8.6	5.2, 8.7	
Barley Grain	0.01, 0.1	5, 5	75, 76	7.1, 3.3	9.5, 4.3	
Oil Seed Rape	0.01, 0.1	5, 5	52, 52	3.8, 7.1	7.3, 14	
IN-WR791						
Apples	0.01, 0.1	5, 5	88, 84	4.5, 4.6	5.2, 5.5	DP-31140
Citrus	0.01, 0.1	5, 5	105, 108	4.8, 5.0	4.5, 4.6	
Barley Grain	0.01, 0.1	5, 5	92, 88	6.3, 2.5	3.8, 2.9	
Oil Seed Rape	0.01, 0.1	5, 5	89, 94	13, 4.7	15, 4.9	
IN-RZD74						
Apples	0.01, 0.1	5, 5	91, 88	2.2, 3.5	2.4, 4.0	DP-31140
Citrus	0.01, 0.1	5, 5	104, 102	3.0, 3.6	2.9, 3.5	
Barley Grain	0.01, 0.1	5, 5	96, 89	5.6, 1.7	5.9, 1.9	

Matrix	Fortification level (mg/kg)	Number of tests	Average % recovery	Standard deviation	% RSD	Reference
Oil Seed Rape	0.01, 0.1	5, 5	83, 78	9.9, 3.4	12, 4.4	
IN-RZB20						
Apples	0.01, 0.1	5, 5	92, 91	6.1, 2.3	6.6, 2.6	DP-31140
Citrus	0.01, 0.1	5, 5	82, 79	2.5, 5.7	3.0, 7.2	
Barley Grain	0.01, 0.1	5, 5	85, 81	4.8, 2.5	5.7, 3.1	
Oil Seed Rape	0.01, 0.1	5, 5	70, 73	13, 7.4	18, 10	
IN-E8S72						
Apples	0.01, 0.1	5, 5	90, 91	4.6, 3.3	5.1, 3.6	DP-31140
Citrus	0.01, 0.1	5, 5	104, 106	1.8, 2.6	1.8, 2.4	
Barley Grain	0.01, 0.1	5, 5	102, 100	5.2, 1.1	5.1, 1.1	
Oil Seed Rape	0.01, 0.1	5, 5	96, 101	7.2, 1.9	7.5, 1.9	
IN-SXS67						
Apples	0.01, 0.1	5, 5	75, 62	29, 12	38, 19	DP-31140
Citrus	0.01, 0.1	5, 5	22, 24	1.3, 4.1	6.0, 17	
Barley Grain	0.01, 0.1	5, 5	76, 40	13, 5.1	18, 13	
Oil Seed Rape	0.01, 0.1	5, 5	566, 41	229, 3.3	41, 8.2	

Inns, 2013 [Ref: DP-36106] investigated the extraction efficiency of this method by extracting samples of lettuce (high water content), grape berries (high acid content) and wheat straw (low water content) containing incurred residues of radiolabeled oxathiapiprolin from the metabolism studies. The residue profiles obtained were compared with those obtained for the same samples in the plant metabolism study. The levels of the parent and metabolites obtained using the residue method were within 80 to 120% of those found in the metabolism study for watery and acidic crops but did not adequately extract incurred oxathiapiprolin residues from dry crops. Extraction efficiency in a representative high oil content matrix was not investigated because of the lack of incurred residue samples.

Table 44 Analytical method DFG S 19 - extraction efficiency

Commodity	% Total radioactivity extracted		
	DFG S 19 method	Metabolism method	DFG S 19 method as % of metabolism method
Lettuce Foliage	91.3	88.4	103.3
Grape Berries	91.1	93.0	98.0
Wheat Straw	58.8	72.3	81.3

An independent laboratory validation of Method DFG S 19 for measuring residues of oxathiapiprolin and metabolites tomato, barley grain and rape seed was conducted by Richter, 2013 [Ref: DP-37477] and reported good linearity in the range of 0.25–25 ng/mL for all analytes (correlation coefficients > 0.99) and no significant interferences at the relevent retention times. The mean recoveries for all matrices tested and at all fortification levels ranged from 70 and 110% (RSDs < 20%) except for IN-SXS67 and for oxathiapiprolin, IN-RDG40, IN-Q7H09 in rape seed (high oil content), where average recoveries were less than 70%. The limits of quantitation (LOQs) were 0.01 mg/kg for all matrices tested.

Table 45 Analytical method DFG S 19 – Independent validation recovery rates in crop matrices.

Matrix	Fortification level (mg/kg)	Number of tests	Average % recovery	Standard deviation	% RSD	Reference
Oxathiapiprolin						
Tomatoes	0.01, 0.1	5, 5	115, 118	2, 2	2, 2	DP-37477
Barley Grain	0.01, 0.1	5, 5	70, 80	4, 6	5, 7	
Oil Seed Rape	0.01, 0.1	5, 5	21, 22	1, 1	6, 3	
IN-Q7H09						
Tomatoes	0.01, 0.1	5, 5	110, 110	2, 1	2, 1	DP-37477
Barley Grain	0.01, 0.1	5, 5	72, 76	2, 2	3, 2	
Oil Seed Rape	0.01, 0.1	5, 5	31, 36	5, 2	16, 5	

Matrix	Fortification level (mg/kg)	Number of tests	Average % recovery	Standard deviation	% RSD	Reference
IN-RDG40						
Tomatoes	0.01, 0.1	5, 5	110, 111	1, 2	1, 2	DP-37477
Barley Grain	0.01, 0.1	5, 5	76, 82	2, 2	2, 2	
Oil Seed Rape	0.01, 0.1	5, 5	40, 35	2, 2	4, 5	
IN-WR791						
Tomatoes	0.01, 0.1	5, 5	110, 108	3, 5	3, 5	DP-37477
Barley Grain	0.01, 0.1	5, 5	109, 109	2, 4	2, 4	
Oil Seed Rape	0.01, 0.1	5, 5	75, 80	2, 9	3, 11	
IN-RZD74						
Tomatoes	0.01, 0.1	5, 5	106, 94	5, 3	5, 3	DP-37477
Barley Grain	0.01, 0.1	5, 5	99, 90	4, 9	4, 10	
Oil Seed Rape	0.01, 0.1	5, 5	78, 73	4, 6	5, 8	
IN-RZB20						
Tomatoes	0.01, 0.1	5, 5	91, 88	9, 4	10, 5	DP-37477
Barley Grain	0.01, 0.1	5, 5	84, 83	5, 4	6, 5	
Oil Seed Rape	0.01, 0.1	5, 5	76, 76	2, 10	2, 13	
IN-E8S72						
Tomatoes	0.01, 0.1	5, 5	119, 110	6, 6	5, 5	DP-37477
Barley Grain	0.01, 0.1	5, 5	114, 109	3, 10	3, 9	
Oil Seed Rape	0.01, 0.1	5, 5	98, 93	3, 9	3, 10	
IN-SXS67						
Tomatoes	0.01, 0.1	5, 5	69, 53	5, 6	7, 11	DP-37477
Barley Grain	0.01, 0.1	5, 5	28, 40	4, 2	16, 5	
Oil Seed Rape	0.01, 0.1	5, 5	50, 43	10, 8	19, 19	

QuEChERS multiresidue method

The use of the QuEChERS multiresidue method was evaluated for measuring residues of oxathiapiprolin in commodities of plant origin by Schwarz, 2009 [Ref: DP-28696].

Residues of oxathiapiprolin were extracted with acetonitrile/ water. After the addition of $MgSO_4$, NaCl and buffering citrate salts (pH = 5–5.5), the mixture was shaken intensively and centrifuged for phase separation. An aliquot of the organic phase was cleaned-up by freezing out (for fat and wax containing material) and dispersive SPE with PSA, $MgSO_4$, and additionally with C_{18}-material for whole oranges. The final extracts were acidified with formic acid and diluted with water for LC/MS/MS analysis, with two ion transitions monitored for analyte quantitation and confirmation.

Inns, 2013 [Ref: DP-36106] investigated the extraction efficiency of this method by extracting samples of lettuce (high water content), grape berries (high acid content) and wheat grain (low water content) containing incurred residues of radiolabelled oxathiapiprolin from the metabolism studies. The residue profiles obtained were compared with those obtained for the same samples in the plant metabolism study. The levels of the parent and metabolites obtained using the residue method were within 80 to 120% of those found in the metabolism study for watery and acidic crops but did not adequately extract incurred oxathiapiprolin residues from dry crops. Extraction efficiency in a representative high oil content matrix was not investigated.

Table 46 QuEChERS analytical method extraction efficiency

Commodity	% Total radioactivity extracted		
	QuEChERS method	Metabolism method	QuEChERS method as % of metabolism method
Lettuce Foliage	71.8	88.4	81.2
Grape Berries	72.1	93.0	77.5
Wheat Straw	12.2	72.3	16.9

Good linearity was observed in the range of 0.5 to 100 ng/mL for oxathiapiprolin and no residues were detected in control samples; the response in the area of the oxathiapiprolin peak always

corresponded to less than 20% of the limit of determination. The limit of quantification of the method is 0.01 mg/kg for oxathiapiprolin. Recovery rates achieved by the same analyst over the course of two days per matrix meet the recognised criteria of 70 to 120%, with a standard deviation of ≤ 20%.

Table 47 QuEChERS analytical method validation recovery rates for oxathiapiprolin in plant matrices

Matrix	Fortification level (mg/kg)	Number of tests	Average % recovery	Standard deviation	% RSD	Reference
Oxathiapiprolin (540→163 amu – Quantitation)						
Lettuce	0.01, 0.1	5, 5	103, 103	4, 4	4, 4	DP-28696
Wheat Grain	0.01, 0.1	5, 5	90, 88	3, 3	3, 3	
Whole Orange	0.01, 0.1	5, 5	105, 104	4, 2	4, 2	
Maize Grain	0.01, 0.1	5, 5	108, 108	2, 2	2, 2	
Oxathiapiprolin (540→500 amu – Confirmation)						
Lettuce	0.01, 0.1	5, 5	106, 102	2, 4	2, 4	DP-28696
Wheat Grain	0.01, 0.1	5, 5	88, 88	4, 2	4, 2	
Whole Orange	0.01, 0.1	5, 5	107, 104	3, 3	3, 3	
Maize Grain	0.01, 0.1	5, 5	107, 107	3, 2	3, 2	

Multi-residue method DFG S 19 (animal matrices)

The multi-residue method DFG S 19 (L 00.00-34) was investigated and validated for the determination of oxathiapiprolin and IN-Q7H09, IN-RDG40, IN-RLB67, and IN-RAB06 in muscle, fat, liver, milk and eggs by Weber, 2012 [Ref: DP-31951].

Samples of muscle (bovine), liver, milk, and eggs were extracted with acetone (module E1). Sufficient water was added beforehand to maintain a constant 2:1 ration of acetone:water during the extraction procedure. For liquid/liquid partition, ethyl acetate/cyclohexane and sodium chloride were added. Fat samples were extracted with ethyl acetate/cyclohexane (module E6). Samples were cleaned-up by gel permeation chromatography using ethyl acetate/cyclohexane (1/1, v/v) as eluant and analysed by LC-MS/MS with quantitation and confirmation using the following mass transitions:

Analyte	Quantitation	Confirmation
Oxathiapiprolin	540→163	540→500
IN-RAB06	570→350	570→167
IN-RLB67	572→554	572→396
IN-RDG40	556→174	556→163
IN-Q7H09	556→155	556→538

The method showed good linearity in the range of 0.25–300 ng/mL for all analytes (correlation coefficients > 0.999 and no significant interferences were observed at the relevent retention times. The mean recoveries for all matrices tested and at all fortification levels ranged from 70 and 110% (RSD < 20%) except for IN-Q7H09 in fat, where the average recovery was 67% in the 0.01 mg/kg spiked samples. The limits of quantitation (LOQs) were 0.01 mg/kg for all matrices tested.

Table 48 Multi-residue method DFG S 19 analytical recovery rates for oxathiapiprolin and metabolites in animal matrices

Matrix	Number of tests	Fortification level (mg/kg)	Average % recovery	Standard deviation	% RSD	Reference
Oxathiapiprolin						
Meat (Muscle)	5, 5	0.01, 0.1	83, 75	3.5, 3.4	4.3, 4.5	DP-31951
Fat	5, 5	0.01, 0.1	73, 75	7.2, 4.0	9.9, 5.4	
Liver	5, 5	0.01, 0.1	91, 93	7.3, 2.1	8.1, 2.2	
Milk	5, 5	0.01, 0.1	90, 80	10, 5.0	12, 6.3	
Eggs	5, 5	0.01, 0.1	94, 82	5.5, 5.4	5.8, 6.7	
IN-Q7H09						
Meat (Muscle)	5, 5	0.01, 0.1	77, 73	4.0, 4.0	5.1, 5.6	DP-31951
Fat	5, 5	0.01, 0.1	67, 75	7.3, 5.5	11, 7.4	

Matrix	Number of tests	Fortification level (mg/kg)	Average % recovery	Standard deviation	% RSD	Reference
Liver	5, 5	0.01, 0.1	90, 84	9.8, 1.1	11, 1.3	
Milk	5, 5	0.01, 0.1	92, 86	4.6, 3.0	5.0, 3.5	
Eggs	5, 5	0.01, 0.1	97, 80	6.0, 6.9	6.2, 8.6	
IN-RDG40						
Meat (Muscle)	5, 5	0.01, 0.1	73, 77	12, 8.8	16, 12	DP-31951
Fat	5, 5	0.01, 0.1	78, 75	11, 3.8	15, 5.1	
Liver	5, 5	0.01, 0.1	97, 92	11, 4.9	11, 5.3	
Milk	5, 5	0.01, 0.1	92, 80	5.9, 3.9	6.4, 4.9	
Eggs	5, 5	0.01, 0.1	98, 78	7.9, 6.4	8.1, 8.3	
IN-RLB67						
Meat (Muscle)	5, 5	0.01, 0.1	79, 75	4.5, 3.4	5.8, 4.5	DP-31951
Fat	5, 5	0.01, 0.1	74, 78	10, 5.4	14, 7.0	
Liver	5, 5	0.01, 0.1	94, 86	8.3, 1.8	8.8, 1.2	
Milk	5, 5	0.01, 0.1	100, 88	6.3, 4.2	6.3, 4.7	
Eggs	5, 5	0.01, 0.1	96, 80	7.1, 5.5	7.4, 6.8	
IN-RAB06						
Meat (Muscle)	5, 5	0.01, 0.1	78, 75	4.5, 4.4	5.8, 5.9	DP-31951
Fat	5, 5	0.01, 0.1	67, 72	8.3, 3.9	12, 5.4	
Liver	5, 5	0.01, 0.1	78, 74	6.5, 0.4	8.2, 0.6	
Milk	5, 5	0.01, 0.1	83, 80	8.8, 4.8	11, 6.0	
Eggs	5, 5	0.01, 0.1	92, 78	5.4, 4.6	5.8, 5.9	

Inns, 2013 [Ref: DP-36106] investigated the extraction efficiency of this method by extracting samples of However the method did not were compared with those obtained for the same samples in the goat metabolism study. The levels of the parent and metabolites obtained using the residue method were within 95 to 135% of those found in the metabolism study, indicating that the DFG S 19 residue method provides acceptable extraction efficiency.

Commodity	% Total Radioactivity Extracted		DFG S19 Method as % of Metabolism Method
	DFG S19 Method	Metabolism Method	
Muscle	100.0	74.1	135.0
Kidney	85.0	89.4	95.1
Fat	100.0	98.2	101.8
Milk	100.0	94.7	105.6

An independent laboratory validation of Method DFG S 19 for measuring residues of oxathiapiprolin and metabolites IN-Q7H09, IN-RDG40, IN-RLB67, and IN-RAB06 in muscle, fat, milk and eggs was conducted by Asekunowo & Bacher, 2013 [Ref: DP-37476] and reported good linearity in the range of 0.2–20 ng/mL for all analytes (correlation coefficients > 0.99) and no significant interferences at the relevent retention times. The mean recoveries for all matrices tested and at all fortification levels ranged from 79 and 109% (RSDs < 18%) The limits of quantitation (LOQs) were 0.01 mg/kg for all matrices tested.

Table 49 Analytical method DFG S 19 – Independent validation recovery rates in muscle, fat, milk and eggs

Matrix	Number of tests	Fortification level (mg/kg)	Average % recovery	Standard deviation	% RSD	Reference
Oxathiapiprolin						
Meat (Muscle)	5, 5	0.01, 0.1	91, 91	2, 2	2, 2	DP-37476
Fat	5, 5	0.01, 0.1	100, 89	5, 3	5, 3	
Milk	5, 5	0.01, 0.1	86, 109	6, 9	7, 8	
Eggs	5, 5	0.01, 0.1	98, 99	16, 7	17, 7	
IN-Q7H09						
Meat (Muscle)	5, 5	0.01, 0.1	94, 91	6, 3	6, 3	DP-37476
Fat	5, 5	0.01, 0.1	100, 87	5, 3	5, 4	
Milk	5, 5	0.01, 0.1	89, 108	4, 9	5, 8	
Eggs	5, 5	0.01, 0.1	101, 100	16, 5	16, 5	

Matrix	Number of tests	Fortification level (mg/kg)	Average % recovery	Standard deviation	% RSD	Reference
IN-RDG40						
Meat (Muscle)	5, 5	0.01, 0.1	83, 98	9, 17	11, 17	DP-37476
Fat	5, 5	0.01, 0.1	100, 90	6, 3	6, 3	
Milk	5, 5	0.01, 0.1	93, 106	7, 8	7, 8	
Eggs	5, 5	0.01, 0.1	100, 104	17, 5	17, 5	
IN-RLB67						
Meat (Muscle)	5, 5	0.01, 0.1	91, 92	2, 3	2, 3	DP-37476
Fat	5, 5	0.01, 0.1	101, 85	6, 3	6, 3	
Milk	5, 5	0.01, 0.1	89, 104	4, 7	5, 7	
Eggs	5, 5	0.01, 0.1	83, 86	14, 2	17, 2	
IN-RAB06						
Meat (Muscle)	5, 5	0.01, 0.1	110, 95	7, 14	6, 15	DP-37476
Fat	5, 5	0.01, 0.1	82, 82	9, 7	11, 9	
Milk	5, 5	0.01, 0.1	74, 84	5, 8	7, 9	
Eggs	5, 5	0.01, 0.1	102, 117	12, 16	12, 14	

Stability of residues in stored analytical samples

The Meeting received information on the stability of residues of oxathiapiprolin, IN-Q7H09, IN-RDG40, IN-E8S72, IN-RZB20, IN-RZD74, IN-SXS67, and IN-WR791 in representative commodities with high water (wheat forage, tomato), high starch (potato, wheat grain), high protein (dry bean seed), high oil (soya bean seed) , high acid (grape) content and low moisture content (wheat straw, dry grape pomace), stored at freezer temperatures of - 20 °C (or below) for up to 18 months.

In a study reported by Vincent, 2013 [Ref: DP-30046], separate representative crop sample replicates were fortified with 0.1 mg/kg oxathiapiprolin, IN-Q7H09, IN-RDG40, IN-E8S72, IN-RZB20, IN-RZD74, IN-SXS67, and IN-WR791 and stored at -20 ± 10 °C. At intervals during the storage period, 2 stored fortified and 3 control samples were removed from storage for analysis. Two of the control samples were freshly fortified with oxathiapiprolin, IN-E8S72, IN-Q7H09, IN-RDG40, IN-RZB20, IN-RZD74, IN-SXS67, and IN-WR791 at 0.1 mg/kg and the five samples were analysed using the same analytical procedures as those employed for crop residue studies (methods DuPont-30422 or DuPont-30422, Supplement No. 1).

All except ten percent of concurrent recoveries fell within the range of acceptability specified by the protocol (70–110%) and the stored recoveries for oxathiapiprolin and metabolites (IN-Q7H09, IN-RDG40, IN-E8S72, IN-RZB20, IN-RZD74, IN-SXS67, and IN-WR791) in representative crop samples stored frozen for up to 18 months were greater than 70% except in the wheat straw samples stored for 12 and 18 months (62–65% IN-RZD74 being recovered) and in the soya bean seed sample stored for 6 months (65% IN-RDG40 being recovered). Overall normalised recoveries in representative crop samples stored frozen for up to 18 months ranged from 73 to 120%.

Table 50 Residue stability of oxathiapiprolin and metabolites in a range of fresh and processed plant matrices spiked at 0.1 mg/kg and stored at -20 °C for up to 18 months

Matrix	Day 0	3 month storage		6 month storage		12 month storage		18 month storage	
	%Residues remaining	%Residues remaining	Procedural %Recovery	%Residues remaining	Procedural %Recovery	%Residues remaining	Procedural %Recovery	%Residues remaining	Procedural %Recovery
Wheat forage									
Oxathiapiprolin	100.75	91.5	91.5	104.5	110	97	107.5	99.5	103
IN-Q7H09	106.25	97.5	94.5	95.5	102	94	102	84.5	92.5
IN-RDG40	102.75	97	90.5	97	96	101.5	95.5	81	80.5
IN-E8S72	100.5	106	101	98.5	101	107	106.5	115.5	113
IN-RZB20	103.5	99.5	100.5	110	112	104.5	113	118	118
IN-RZD74	91	84.5	93	96.5	105	90	96.5	99	103.5

Matrix	Day 0	3 month storage		6 month storage		12 month storage		18 month storage	
	%Residues remaining	%Residues remaining	Procedural %Recovery	%Residues remaining	Procedural %Recovery	%Residues remaining	Procedural %Recovery	%Residues remaining	Procedural %Recovery
IN-SXS67	113	99	109	99.5	106	91.5	92.5	119	123
IN-WR791	99.75	102.5	100.5	101.5	104	99	103	110	104.5
Tomatoes									
Oxathiapiprolin	116	104	108.5	109.5	111	105	102.5	103.5	101.5
IN-Q7H09	108.5	114	116.5	102	104.5	103	100.5	102	96
IN-RDG40	110	107.5	106	93	97	95	93.5	97.5	96
IN-E8S72	106.5	107	112	103	103.5	103.5	100.5	109	108
IN-RZB20	110	100.5	105	116	112.5	109.5	102.5	112.5	108
IN-RZD74	106	91.5	106	96.5	105.5	87.5	100	91	99.5
IN-SXS67	103.5	93	98.5	102.5	108.5	90.5	98.5	111	107
IN-WR791	105.5	102	103	96	96.5	105	103	101.5	102
Soya bean seed									
Oxathiapiprolin	91	97	91.5	76.5	78	110	99	94	89
IN-Q7H09	84	89.5	78	75	76	98.5	87.5	94	85
IN-RDG40	89	77.5	79.5	65	75.5	92.5	91.5	82.5	82
IN-E8S72	88	88	85.5	77	81.5	90.5	97	115.5	110
IN-RZB20	87.5	100.5	95	93	94.5	83.5	87.5	106	105.5
IN-RZD74	76	90.5	96.5	77	92.5	71	84	81	88.5
IN-SXS67	89.5	74.5	79	74.5	76.5	78.5	84	97.5	99
IN-WR791	79.5	87	82.5	72.5	79	89	91	104	112
Dried bean seed									
Oxathiapiprolin	110.5	96	104	108.5	106.5	101	98	104.5	102.5
IN-Q7H09	115	97.5	100	108	106	90	90	110	101.5
IN-RDG40	116.5	97	100.5	93	95.5	85	90	90.5	93
IN-E8S72	109.5	95	99	105	103	109.5	129	127.5	115.5
IN-RZB20	94	108	116.5	117.5	121	101.5	113.5	104.5	108
IN-RZD74	96	102.5	107.5	104.5	108.5	93.5	99.5	98	102.5
IN-SXS67	105	84	94.5	96	95.5	98.5	109	112.5	120.5
IN-WR791	103.5	95.5	100.5	99.5	97	101	103.5	119	116
Potato tubers									
Oxathiapiprolin	98	92.5	107.5	79.5	98.5	86	90	86.5	90
IN-Q7H09	101.25	95	107	90	100	91	97.5	87	102
IN-RDG40	104.25	111.5	117.5	105	104.5	93	89.5	94.5	90.5
IN-E8S72	106.25	101.5	108	112	110	103	105	100	91
IN-RZB20	112	107.5	110.5	110	106	103	107.5	95.5	105
IN-RZD74	101.5	102	112	99.5	103.5	99	104.5	90	88.5
IN-SXS67	111	108	109	103	107.5	97.5	98	92	100.5
IN-WR791	106	93.5	100.5	107.5	109.5	103.5	107	94.5	93.5
Wheat grain									
Oxathiapiprolin	92	80	85.5	94	105	110.5	107.5	104.5	99
IN-Q7H09	102.5	79	85	97.5	103	109	98	106	98
IN-RDG40	110.5	77.5	84	91.5	99	98.5	101.5	101	97.5
IN-E8S72	87	81	81.5	83.5	82	88	88	96.5	92

Matrix	Day 0	3 month storage		6 month storage		12 month storage		18 month storage	
	%Residues remaining	%Residues remaining	Procedural %Recovery	%Residues remaining	Procedural %Recovery	%Residues remaining	Procedural %Recovery	%Residues remaining	Procedural %Recovery
IN-RZB20	96	90	89	94.5	102	102.5	96.5	93	92
IN-RZD74	83	80.5	89	81.5	98	89	91.5	88.5	87
IN-SXS67	84.5	79.5	79	84	83	105	97.5	101.5	106
IN-WR791	84	82	83	75.5	75.5	86	87	88.5	88
Grapes									
Oxathiapiprolin	104.75	86.5	92	103.5	108.5	96	104.5	100.5	96
IN-Q7H09	107	94.5	94.5	99.5	108.5	98.5	104	101.5	99.5
IN-RDG40	107.5	98	95.5	111.5	104	108	99	108.5	102.5
IN-E8S72	107.25	95	96	110.5	110	107.5	101.5	100	101
IN-RZB20	102.5	99.5	99	108	107.5	114.5	120.5	111	109.5
IN-RZD74	82	76	89.5	98	106	90	101	82.5	85
IN-SXS67	101.5	99.5	105	106	104	112	103	113.5	106
IN-WR791	103.75	96.5	96.5	106	108.5	101.5	96.5	103.5	107.5
Grape dry pomace									
Oxathiapiprolin	99.25	86.5	96	80.5	96	89.5	101.5	101.5	100.5
IN-Q7H09	101.5	99	102.5	89.5	92	91.5	95.5	90.5	97.5
IN-RDG40	105.5	103	102.5	107	99.5	104.5	97	104.5	93.5
IN-E8S72	101	107	108	99.5	103	98.5	98.5	101	107.5
IN-RZB20	105.5	86	92	97	93.5	105	97.5	115.5	111.5
IN-RZD74	95	85	100	83.5	92	89	100.5	87	89.5
IN-SXS67	94	102.5	107.5	114	111.5	108.5	114	110.5	98
IN-WR791	102	106	107.5	99.5	101	86	86	92	97.5
Wheat straw									
Oxathiapiprolin	86	87	88.5	78	85	89	78	77.5	77
IN-Q7H09	75	94	93	85.5	86.5	83.5	79.5	84	82
IN-RDG40	71	90.5	83.5	80.5	85	88.5	80	79	77.5
IN-E8S72	97	88	92.5	98.5	96.5	126.5	107	114	110
IN-RZB20	93.5	105.5	109	99	100.5	92	86	91.5	99
IN-RZD74	71.5	83.5	99.5	69	81	64	79	62.5	84.5
IN-SXS67	92	94.5	99	92.5	97	93.5	88	91.5	92.5
IN-WR791	95	108.5	110	84	84	111	109.5	103	102.5

Results are mean values from two replicate analyses

In summary, residues of oxathiapiprolin and metabolites IN-Q7H09, IN RDG40, IN E8S72, IN-RZB20, IN RZD74, IN SXS67, and IN WR791were stable in analytical samples stored frozen (-18 to -20 °C) for at least the storage intervals used in the supervised residue trials, with residues in the stored samples usually more than 80% of the spiked sample levels. In general, residue stability was shown for at least 18 months in representative commodities with high water content (wheat forage, tomato), high starch content (potato, wheat grain), high protein content (dry bean seed), high oil content (soya bean seed) , high acid content (grape) and low moisture content (wheat straw, dry grape pomace). The exception was for the metabolite IN-RDG40 in soya bean seed (high oil content), where residues were stable for up to 3 months.

Stability of residues in fresh analytical sub-samples

The Meeting received supplementary information on an in-house laboratory study [Unreferenced] investigating the stability of oxathiapiprolin residues in cut samples prior to freezing. In this study, six 5 g portions of homogenized cantaloupe pulp were fortified at 2 mg/kg. Three samples were extracted and analysed immediately without heating or storage and three samples were placed in a water bath for four hours at 30 °C (simulating the possible field air temperatures) before extraction and analysis.

Oxathiapiprolin residues were stable for at least four hours when in 100% contact with the melon pulp maintained at 30 °C.

Table 51 Oxathiapiprolin residue recovery rates in homogenised melon pulp samples stored at 30 °C.

Sample ID	Fresh Fortification (2.0 mg/kg)	4 hours at 30 °C
Sample 1	102	98
Sample 2	98	93
Sample 3	98	95
Average	99	95
STDEV	2.3	2.5
%RSD	2.3	2.6
	Normalized Recovery	96

USE PATTERNS

Information on GAP in Australia, Canada, China and USA was available to the Meeting on the use of oxathiapiprolin (SC or OD formulations). The Meeting also noted that oxathiapiprolin registrations exist in a number of countries in Asia, the Pacific, Central and South America and are pending in Europe and other countries.

The following table summarizes the representative critical GAPs in Australia, Canada, China, new Zealand and USA for crops relevant to the available residue field trials.

Table 52 Representative registered uses of oxathiapiprolin (100 g ai/litre OD and 200 g ai/litre SC formulations)

Crop	Country	Application [a]		Max/crop		PHI (days)	Comments
		g ai/ha	Type	no	g ai/ha		
Grapes	China		foliar	2		14	4-5 g ai/hL (2000-3000 dilution), 10 day RTI
Bulb vegetables	Can	17.5-35	foliar	4	140	0	Max 2 sequential applications. 5-10 day RTI.
	USA	15-35	foliar	4	140	0	
	Aus	35	foliar	3		10	Max 2 sequential applications. 10-14 day RTI. Max 2 sprays/season for onions
Bulb onions	NZ	35	foliar	2		14	10 day RTI
Head and Stem Brassica vegetables	Can	17.5-35	foliar	4	140	0	Max 2 sequential applications. 5-10 day RTI.
	USA	15-35	foliar	4	140	0	
Brassica vegetables	Aus	35	foliar	3		0	Max 2 sequential applications. 7-10 day RTI.
Cucurbit [b] vegetables	Can	8.75-35	foliar	4	140	0	Max 2 sequential applications. 3-14 day RTI. Do not use in conjunction with soil treatments.
	USA	15-35	foliar	4	140	0	
	Can	70-280	soil	4	560	0	In transplant water, band or in-furrow at planting, drip irrigation or directed band application. Max 2 sequential applications. Min 7 day RTI.
	USA	35-280	soil	4	560	0	
Cucurbit vegetables	Aus	35	foliar	3		1	Max 2 sequential applications. 7-10 day RTI.
Cucumber (outdoor)	China	15-30	foliar	2		3	10 day RTI
Cucumber (indoor)	China	15-30	foliar	4		3	2 sprays in spring, 2 sprays in autumn
Fruiting [b] vegetables	Can	8.75-35	foliar	4	140	0	Max 2 sequential applications. 5-14 day RTI. Do not use in conjunction with soil treatments
	USA	15-35	foliar	4	140	0	
	Can	70-280	soil	4	560	0	In transplant water, band or in-furrow at planting,

Crop	Country	Application [a]		Max/crop		PHI (days)	Comments
		g ai/ha	Type	no	g ai/ha		
	USA	35-280	soil	4	560	0	drip irrigation or directed band application. Max 2 sequential applications. Min 7 day RTI.
Chili pepper	China	22-37.5	foliar	3		5	10 day RTI
Tomato	China	15-30	foliar	3		5	
Leafy Greens [b]	Can	17.5-35	foliar	4	140	0	Max 2 sequential applications. 3-14 day RTI. Do not use in conjunction with soil treatments
	USA	15-35	foliar	4	140	0	
	Can	70-280	soil	4	560	0	In transplant water, band or in-furrow at planting, drip irrigation or directed band application. Max 2 sequential applications. Min 7 day RTI.
	USA	35-280	soil	4	560	0	
Leafy vegetables	Aus	35	foliar	3		3	Max 2 sequential applications. 7-10 day RTI.
Peas (Pisum spp) with or without pods	Can	17.5-35	foliar	4	140	0	Max 2 sequential applications. 5-7 day RTI.
	USA	18-35	foliar	4	140	0	
Tuberous and Corm vegetables	USA	12-35	foliar	4	200	5	Max 2 sequential applications. 5-14 day RTI.
		50	foliar	2			Apply at nickel-sized tuber (flowering) and repeat 10-14 days later
Potato	Can	12-35	foliar	4	200	5	Max 2 sequential applications. 7-14 day RTI.
	China	22.5-30	foliar	3		10	10 day RTI
Ginseng	Can	70-280	foliar	4	560	14	7-14 dayRTI
	USA	35-280	foliar	4	560	14	14 day RTI

Head and Stem Brassica vegetables = Broccoli; broccoli, Chinese; brussels sprouts; cabbage; cabbage, Chinese (napa); cabbage, Chinese mustard; cauliflower; cavalo broccolo; kohlrabi.

Bulb vegetables = **Onion bulb subgroup** [daylily, bulb; fritillaria, bulb; garlic, bulb; garlic, great-headed, bulb; garlic, serpent, bulb; lily, bulb; onion, bulb; onion, Chinese, bulb; onion, pearl; onion, potato, bulb; shallot, bulb; cultivars, varieties, and/or hybrids of these] and **Onion green subgroup** [chive, fresh leaves; chive, Chinese, fresh leaves; elegans hosta; fritillaria, leaves; kurrat; lady's leek; leek; leek, wild; Onion, Beltsville bunching; onion, fresh; onion, green; onion, macrostem; onion, tree, tops; onion, Welsh, tops; shallot, fresh leaves; cultivars, varieties, and/or hybrids of these].

Cucurbit vegetables = **Melons subgroup** [citron melon; muskmelons (includes cantaloupe etc.); watermelon] and **Squash/Cucumber subgroup** [chayote (fruit); chinese waxgourd (chinese preserving melon); cucumber; gherkin; gourds, edible (includes hyotan, cucuzza, hechima, chinese okra); Momordica spp. (includes balsam apple, balsam pear, bittermelon, chinese cucumber); pumpkin; squash, summer; squash, winter (includes butternut squash, calabaza, hubbard squash, acorn squash, spaghetti squash].

Fruiting vegetables = **Tomato subgroup** [bush tomato; cocona; currant tomato; garden huckleberry; goji berry; groundcherry; naranjilla; sunberry; tomatillo; tomato; tree tomato; cultivars, varieties, and/or hybrids of these.] and **Pepper/Eggplant subgroup** [African eggplant; bell pepper; eggplant; Martynia; nonbell pepper; okra; pea eggplant; pepino; roselle; scarlet eggplant; cultivars, varieties, and/or hybrids of these].

Leafy Greens = Amaranth; arugula; chervil; chrysanthemum, edible-leaved; chrysanthemum, garland; corn salad; cress, garden; cress, upland; dandelion; dock; endive; lettuce; orach; parsley; purslane, garden; purslane, winter; radicchio (red chicory); spinach; spinach, New Zealand; spinach, vine.

Tuberous and Corm vegetables = Arracacha; arrowroot; artichoke, Chinese; artichoke, Jerusalem; canna, edible; cassava, bitter and sweet; chayote (root); chufa; dasheen; ginger; leren; potato; sweet potato; tanier; turmeric; yam bean; yam, true.

[a] Apply a minimum spray mix of 140 L/ha (ground), 94 L/ha (ground – air assisted), 19L/ha (air)

[b] Use either soil applications or foliar applications but not both. Only OD formulation authorised for indoor use on tomatoes, peppers, cucumbers and summer squash.

RESIDUES RESULTING FROM SUPERVISED TRIALS

The Meeting received information on supervised field trials involving soil or foliar treatments of oxathiapiprolin to the following crops.

Group	Crop	Countries	Table no
Small fruit vine climbing	Grape	China, Europe	53-56

Group	Crop	Countries	Table no
Bulb vegetables	Onion, dry bulb	Nth America	57
	Spring onion	Nth America	58
Brassica vegetables	Broccoli	Nth America	59
	Cabbage	Nth America	60
	Cauliflower	Nth America	61
Fruiting vegetables, Cucurbits	Cucumber	Europe, Nth America	62-65
	Summer squash	Europe, Nth America	66-69
	Melons	Europe, Nth America	70-73
Fruiting vegetables, other than Cucurbits	Peppers	Nth America	74-75
	Tomato	Europe, Nth America	76-79
Leafy vegetables	Lettuce	Europe, Nth America	80-84
	Spinach	Nth America	85-87
Legume vegetables	Peas	Nth America	88-89
Root and tuber vegetables	Potato	Europe, USA	90-93
	Ginseng	Nth America	94-95

The supervised trials were well documented with laboratory and field reports. Laboratory reports included method validation including procedural recoveries with spiking at residue levels similar to those occurring in samples from the supervised trials. Dates of analyses or duration of residue sample storage were also provided. Although trials included control plots, no control data are recorded in the tables unless residues in control samples exceeded the LOQ. In such cases, the residues found are noted as "c=nn mg/kg". Residue data are recorded unadjusted for recovery.

Results from replicated field plots are presented as individual values. Residues and application rates have been reported as provided in the study reports except for finite values below the LOQ, where these have been rportd as < 0.01 mg/kg. The results from trials used for the estimation of maximum residue levels (underlined) have been rounded to two significant digits (or if close to the LOQ, rounded to one significant digit) in the Appraisal. The designation "ND" is used for treated samples for which the residue was <LOD (below the limit of detection); < 0.003 mg/kg. For calculating means of two replicate values, ½ LOD (0.0015 mg/kg) was used for samples with no detectable (ND) residues.

When multiple applications were made to a crop, the application rate, spray concentration and spray volume were not always identical from one application to the next. In most trials, the actual treatment rates were within 10% of the listed 'target' application rates, but if not, the actual treatment rates are listed.

Berries and other small fruits

Grapes

In supervised trials on grapes conducted in China between 2012 and 2015, two or three foliar applications of oxathiapiprolin (OD formulations) were applied at 10-day intervals to vines. Samples of grapes (min 6 bunches or 2 kg) were frozen within 12 hours and berries were analysed for oxathiapiprolin and metabolites IN-WR791 and IN-E8S72, within 18 months of harvest using method DuPont-30422 (LC-MS/MS) and with an LOQ of 0.01 mg/kg. Mean recoveries from control grape samples fortified with oxathiapiprolin or metabolites at levels of 0.01–1.0 mg/kg ranged from 92–107%.

Table 53 Residues in grapes from supervised trials in China involving two or three foliar applications of oxathiapiprolin (OD formulations).

GRAPES Country, year Location (Variety) Trial Ref	Application			DAT	Residues (mg/kg) [(c)]		
	no	g ai/ha	g ai/hL		Oxathiapiprolin	IN-E8S72	IN-WR791
GAP: China	2		4-5	PHI: 14d			
China, 2012 HeNan (Rongming) DP-006-12F282-1315	2	37.5	5	10 14 21	0.37 <u>0.17</u> 0.072	< 0.01 < 0.01 < 0.01	< 0.01 0.012 < 0.01
	3	37.5	5	10 14 21	0.39 0.35 0.27	< 0.01 < 0.01 < 0.01	0.013 0.013 < 0.01
	2	56	7.5	10 14 21	0.65 0.4 0.27	< 0.01 < 0.01 < 0.01	0.012 0.015 < 0.01
	3	56	7.5	10 14 21	0.77 0.45 0.32	< 0.01 < 0.01 < 0.01	0.013 0.018 < 0.01
China, 2013 HeNan (Rongming) DP-006-12F282-1315	2	37.5	5	10 14 21	0.45 <u>0.37</u> 0.31	< 0.01 < 0.01 < 0.01	0.013 0.015 < 0.01
	3	37.5	5	10 14 21	0.84 0.53 0.36	< 0.01 < 0.01 < 0.01	0.011 0.014 0.013
	2	56	7.5	10 14 21	0.81 0.61 0.43	< 0.01 < 0.01 < 0.01	< 0.01 0.011 0.015
	3	56	7.5	10 14 21	1.1 0.75 0.5	< 0.01 < 0.01 < 0.01	0.011 0.017 0.012
Shandong, China, 2012 China, 2012 Shangdong (Red grape) DP-006-12F282-1315	2	37.5	5	10 14 21	0.52 <u>0.38</u> 0.13	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01
	3	37.5	5	10 14 21	0.71 0.5 0.26	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01
	2	56	7.5	10 14 21	0.64 0.6 0.24	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01
	3	56	7.5	10 14 21	0.81 0.7 0.4	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01
China, 2013 Shangdong (Red grape) DP-006-12F282-1315	2	37.5	5	10 14 21	0.53 <u>0.5</u> 0.33	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01
	3	37.5	5	10 14 21	0.96 0.61 0.39	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 0.02
	2	56	7.5	10 14 21	0.98 0.69 0.43	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01
	3	56	7.5	10 14 21	1.1 0.73 0.48	< 0.01 < 0.01 < 0.01	0.011 < 0.01 < 0.01
China, 2015 Xingcheng (Red grape) DP-Grape-CN-2015	2	37.5	5	21	0.053	< 0.01	< 0.01
China, 2015 Jinzhong (Muscat) DP-Grape-CN-2015	2	37.5	5	21	0.038	< 0.01	< 0.01

GRAPES Country, year Location (Variety) Trial Ref	Application			DAT	Residues (mg/kg) [c]		
	no	g ai/ha	g ai/hL		Oxathiapiprolin	IN-E8S72	IN-WR791
China, 2015 Zhenjiang (Kyoho) DP-Grape-CN-2015	2	37.5	5	21	0.24	< 0.01	0.014
China, 2015 Tianjin (Muscat) DP-Grape-CN-2015	2	37.5	5	21	0.13	< 0.01	0.024

[a] Average of triplicate samples.

In supervised trials on grapes conducted in Europe in 2010 and 2011, three foliar applications of oxathiapiprolin (OD formulations) were applied at intervals of 8–13 days to vines using motorised back-pack sprayers to apply 1500–1600 litres of spray mix/ha, with reduced spray volumes of about 400 litres/ha being applied in some trials. A number of these trials were reverse decline studies.

Samples of grapes (minimum of 12 bunches or 2 kg) were frozen within 9 hours and analysed for oxathiapiprolin and metabolites IN-WR791, IN-RDG40, IN-E8S72 and IN-Q7H09 within 14 months of harvest using method DuPont-30422 (LC-MS/MS) and with an LOQ of 0.01 mg/kg. In many of the trials, grapes were also collected from vines about a year after the last application and analysed for oxathiapiprolin and a wider range of metabolites (IN-WR791, IN-RDG40, IN-E8S72, IN-RZB20, IN-RZD74, IN-SXS67 and IN-Q7H09) using method DuPont-30422 Supplement 1.

Mean recoveries from control grape samples fortified with oxathiapiprolin at levels of 0.01–1.0 mg/kg ranged from 99–105% and the mean recovery rates for the metabolites ranged from 89–112% in samples spiked with 0.01–0.5 mg/kg.

Table 54 Residues in grapes from supervised trials in Europe involving three foliar applications of oxathiapiprolin (OD formulations).

GRAPES Country, year Location (Variety) Trial Ref	Application			DAT	Residues (mg/kg)				
	g ai/ha	water (L/ha)	g ai/hL		Oxathia-piprolin	IN-RDG40	IN-Q7H09	IN-E8S72	IN-WR791
Germany, 2010 Saxony-Anhalt, (White Wine Grapes/ Müller-Thurgau DP-30043-Test-01a	32	1640-1680	1.9	-0 0	0.27 0.25	ND ND	ND ND	ND ND	< 0.01 ND
	30-33	1550-1680	1.9	14	0.17	ND	ND	ND	ND
	30-31	1530-1620	1.9	21	0.16	ND	ND	ND	< 0.01
	30	1525-1570	1.9	28	0.091	ND	ND	ND	ND
	30	1525	1.9	59	0.028	ND	ND	ND	< 0.01
	30-31	1525-1590	1.9	91	ND	ND	ND	ND	ND
Germany, 2010 Saxony-Anhalt, (White Wine Grapes/ Müller-Thurgau) DP-30043-Test-01b	51-52	1620-1645	3.2	-0 0	0.18 0.32	ND ND	ND ND	ND ND	< 0.01 ND
	52	1630-1645	3.2	14	0.7	ND	ND	ND	ND
	51-53	1600-1680	3.2	21	0.25	ND	ND	ND	< 0.01
	49-52	1540-1645	3.2	28	0.31	ND	ND	ND	< 0.01
	48-50	1525-1580	3.2	59	0.1	ND	ND	ND	< 0.01
	48-50	1525-1590	3.2	91	< 0.01	ND	ND	ND	ND
Hungary, 2010 Györ-Moson-Sopron (White Wine Grapes/ Grüner Veltliner) DP-30043-Test-02a	31-32	1605-1635	2	-0 0	0.039 0.065	ND ND	ND ND	ND ND	ND ND
	31-32	1620-1650	2	13	0.038	ND	ND	ND	ND
	30-31	1560-1620	1.9	21	0.037	ND	ND	ND	ND
	31-32	1600-1640	2	30	0.015	ND	ND	ND	< 0.01
	31-32	1615-1640	2	57	0.013	ND	ND	ND	< 0.01
	32	1630-1650	2	89	0.013	ND	ND	ND	< 0.01

GRAPES Country, year Location (Variety) Trial Ref	Application			DAT	Residues (mg/kg)				
	g ai/ha	water (L/ha)	g ai/hL		Oxathia-piprolin	IN-RDG40	IN-Q7H09	IN-E8S72	IN-WR791
Hungary, 2010 Györ-Moson-Sopron (White Wine Grapes/ Grüner Veltliner) DP-30043-Test-02b	51-52	1615-1650	3.2-3.3	-0 0	0.06 0.12	ND ND	ND ND	ND ND	ND ND
	49-52	1565-1655	3.1-3.3	13	0.14	ND	ND	ND	< 0.01
	50-52	1590-1660	3.1-3.3	21	0.061	ND	ND	ND	< 0.01
	48-52	1530-1660	3.0-3.3	30	0.093	ND	ND	< 0.01	0.012
	51-52	1625-1650	3.2-3.3	57	0.023	ND	ND	ND	0.011
	51-52	1620-1635	3.2	89	0.024	ND	ND	ND	< 0.01
France, 2010 Bourgogne (Red Wine Grapes/ Gamay) DP-30043-Test-03a	30-31	345-350	8.7	-0 0	0.034 0.066	ND ND	ND ND	ND ND	ND ND
	30-31	350-360	8.7	14	0.022	ND	ND	ND	< 0.01
	30	345-350	8.7	20	0.042	ND	ND	ND	ND
	30-31	345-355	8.7	27	0.012	ND	ND	ND	ND
	30-31	340-355	8.7	60	< 0.01	ND	ND	ND	< 0.01
	31-32	355-365	8.7	90	< 0.01	ND	ND	ND	ND
France, 2010 Bourgogne (Red Wine Grapes/ Gamay) DP-30043-Test-03b	50-52	345-355	15	-0 0	0.11 0.14	ND ND	ND ND	ND ND	ND < 0.01
	52	355-360	15	14	0.049	ND	ND	ND	< 0.01
	51-52	350-355	15	20	0.22	ND	ND	ND	< 0.01
	52-53	355-365	15	27	0.075	ND	ND	ND	< 0.01
	51	350-355	15	60	0.044	ND	ND	ND	< 0.01
	51	350	15	90	0.021	ND	ND	ND	< 0.01
France, 2010 Aquitane (Red wine grapes/ Malbec) DP-30043-Test-04a	31	350-355	8.7	-0 0	0.16 0.26	ND ND	< 0.01 0.012	ND ND	ND ND
	31	350-355	8.7	13[(a)]	0.13	ND	< 0.01	ND	ND
	31	350	8.7	20	0.077	ND	ND	ND	ND
	31	350-355	8.7	28	0.059	ND	ND	ND	ND
	31	355-360	8.7	60	0.013	ND	ND	ND	ND
	31-32	350-360	8.7	89	0.024	ND	ND	ND	ND
France, 2010 Aquitane (Red wine grapes/ Malbec) DP-30043-Test-04b	51-52	350-355	15	-0 0	0.13 0.26	ND ND	< 0.01 < 0.01	ND ND	ND ND
	51-52	350-360	15	13[a]	0.52	ND	0.021	ND	ND
	51-52	350-355	15	20	0.077	ND	ND	ND	< 0.01
	51	345-350	15	28	0.051	ND	0.016	ND	< 0.01
	51-53	350-365	15	60	0.061	ND	ND	ND	< 0.01
	51	345-350	15	89	0.15	ND	ND	ND	0.011
Spain, 2010 Catalunya (Red Wine Grapes/ Cabernet Sauvignon) DP-30043-Test-05a	31	1590-1595	1.9	-0 0	0.095 0.072	ND ND	ND ND	ND ND	ND ND
	31	1600	1.9	14	0.13	ND	ND	ND	ND
	31	1595-1605	1.9	20	0.22	ND	ND	ND	ND
	31	1595-1605	1.9	27	0.068	ND	ND	ND	ND
	31	1590-1605	1.9	58	0.042	0.029	ND	ND	< 0.01
	31	1590	1.9	90	0.044	ND	ND	ND	< 0.01
Spain, 2010 Catalunya (Red Wine Grapes/ Cabernet Sauvignon) DP-30043-Test-05b	50-51	1595-1600	3.2	-0 0	0.049 0.25	ND ND	ND ND	ND ND	ND ND
	51	1600	3.2	14	0.28	ND	ND	ND	< 0.01
	51	1600	3.2	20	0.31	ND	ND	ND	< 0.01
	50-51	1595-1605	3.2	27	0.15	ND	ND	ND	< 0.01
	50	1595-1600	3.2	58	0.13	ND	ND	ND	< 0.01
	50-51	1575-1600	3.2	90	0.089	ND	ND	ND	< 0.01
Greece, 2010 Anchialos,Thessaloniki (White Wine Grapes/ Roditis) DP-30043-Test-06a	31	1595-1605	1.9	-0 0	0.036 0.095	ND ND	ND ND	ND ND	ND ND
	31-32	1600-1650	1.9	14	0.043	ND	ND	ND	ND
	31	1590-1625	1.9	21	0.05	ND	ND	ND	ND
	31-32	1590-1670	1.9	28	0.038	ND	ND	ND	ND

GRAPES Country, year Location (Variety) Trial Ref	Application			DAT	Residues (mg/kg)				
	g ai/ha	water (L/ha)	g ai/hL		Oxathia-piprolin	IN-RDG40	IN-Q7H09	IN-E8S72	IN-WR791
	31	1595-1620	1.9	60	0.044	ND	ND	ND	< 0.01
	30-32	1525-1655	1.9	81	< 0.01	ND	ND	ND	ND
Greece, 2010 Anchialos,Thessaloniki (White Wine Grapes/ Roditis) DP-30043-Test-06b	50-51	1695-1615	3.2	-0 0	0.049 0.058	ND ND	ND ND	ND ND	ND ND
	49-51	1535-1615	3.2	14	0.036	ND	ND	ND	ND
	50-51	1585-1610	3.2	21	0.094	ND	ND	ND	ND
	50-52	1575-1635	3.2	28	0.085	ND	ND	ND	< 0.01
	51	1605-1620	3.2	60	0.064	ND	ND	ND	0.012
	48-52	1530-1655	3.2	81	0.025	ND	ND	ND	< 0.01
Germany, 2011 Wittlich (Red Table Grapes/ Regent) DP-30043-Test-07	59-61	1475-1535	4	-0 0 367[c]	0.37 0.65 ND	ND ND ND	ND ND ND	ND ND ND	< 0.01 0.01 ND
	60-63	1500-1575	4	14	0.34	ND	ND	ND	0.018
	60-61	1495-1535	4	21	0.25	ND	ND	ND	0.014
	60-63	1495-1565	4	29	0.16	ND	ND	ND	0.014
	62-63	1550-1565	4	60	0.21	ND	ND	ND	0.018
	62	1500-1555	4	90	0.19	ND	ND	ND	0.012
France, 2011 Vineuse, Bourgogne (Red Wine Grapes/ Pinot Noir) DP-30043-Test-08	50	350	14	-0 0 379[c]	0.044 0.067 ND	ND ND ND	ND ND ND	ND ND ND	ND ND ND
	49-52	345-360	14	14	0.15	ND	ND	ND	ND
	48-50	340-350	14	21	0.12	ND	ND	ND	ND
	49-51	345-360	14	31	0.16	ND	ND	ND	< 0.01
	50-51	350-360	14	56	0.11	ND	ND	ND	< 0.01
	51	355	14	83	0.043	ND	ND	ND	< 0.01
Germany, 2011 Saxony-Anhalt (White Wine Grapes/ Gutedel) DP-30043-Test-09	59-64	1460-1560	4.1	14 379[c]	0.24 ND	ND ND	ND ND	ND ND	ND ND
Hungary, 2011 Györ-Moson-Sopron (White Wine Grapes/ Sauvignon Blanc) DP-30043-Test-10	60-63	1490-1560	4	14 388[c]	0.35 ND	ND ND	ND ND	ND ND	0.011 ND
France, 2011 Charmes, Bourgogne (White Wine Grapes/ Chardonnay) DP-30043-Test-11	49-50	345-350	14	14 395[c]	0.12 ND	ND ND	ND ND	ND ND	ND ND
Czech Republic, 2011 Lednice (Table Grapes/ Nero) DP-30043-Test-12	59-61	1490-1530	4	14 374[c]	0.51 ND	ND ND	ND ND	ND ND	0.01 ND
France, 2011 Combe de Gaby, Midi-Pyrenees (Red Wine Grapes/ Malbec) DP-30043-Test-13	49-51	345-355	14	-0 0 379[c]	0.41 0.53 ND	ND ND ND	ND ND ND	ND ND ND	ND ND ND
	51	355	14	14	0.63	ND	ND	ND	ND
	50-51	350-360	14	22	0.81	ND	ND	ND	ND
	51	355-360	14	28	1.7	ND	ND	ND	ND
	50-51	350-355	14	61	0.073	ND	ND	ND	ND
	50-51	350-360	14	90[b]	0.057	ND	ND	ND	ND
Italy, 2011 Corropoli, Terramo (Red Wine Grapes/ Montepulciano d'Abruzzo)	59-63	1450-1540	4.0-4.2	-0 0 354[c]	0.31 0.53 ND	ND ND ND	ND ND ND	ND ND ND	ND ND ND

GRAPES Country, year Location (Variety) Trial Ref	Application			DAT	Residues (mg/kg)				
	g ai/ha	water (L/ha)	g ai/hL		Oxathia-piprolin	IN-RDG40	IN-Q7H09	IN-E8S72	IN-WR791
DP-30043-Test-14	59-61	1440-1490	3.9-4.1	14	0.31	ND	ND	ND	< 0.01
	60-63	1475-1545	4.0-4.2	21	0.4	ND	ND	ND	< 0.01
	59-63	1450-1545	4.0-4.2	28	0.41	ND	ND	ND	< 0.01
	58-60	1430-1510	3.9-4.0	60	0.1	ND	ND	ND	0.01
	62	1430-1475	4.1	90	0.11	ND	ND	ND	< 0.01
Spain. 2011 Los Palacios, Andalucía (Red Wine Grapes/ Merlot) DP-30043-Test-15	60	1500-1510	4	14 378[c]	0.41 ND	ND ND	ND ND	ND ND	ND ND
Spain, 2011 Verdu, Catalunya (Red Wine Grapes/ Shyra) DP-30043-Test-16	60	1500	4	14 367[c]	0.21 ND	ND ND	ND ND	ND ND	ND ND
Italy, 2011 Abruzzo, Teramo (Table Grapes/ Italia) DP-30043-Test-17	59-60	1450-1480	3.9-4.0	14 363[c]	0.11 ND	ND ND	ND ND	ND ND	ND ND
Greece, 2011 Anchialos, Thessaloniki (White Wine Grapes/ Roditis) DP-30043-Test-18	60-61	1500-1525	4	14	0.11	ND	ND	ND	ND

[a] Retreatment intervals of 10 days and 5 days

[b] Retreatment intervals of 6 days and 13 days

[c] Berries sampled taken from the next season crop - residues of metabolites IN-RZB20, IN-RZD74 and IN-SXS67 were ND in all samples.

Additional trials on grapes were conducted in Europe in 2013, involving two foliar applications of oxathiapiprolin (OD formulations) applied at intervals of 9–11 days to vines using motorised back-pack sprayers to apply 1500-1600 litres of spray mix/ha, with reduced spray volumes of about 400 litres/ha being applied in some trials.

Samples of grapes (minimum of 12 bunches or 1 kg) were frozen within 6 hours and analysed for oxathiapiprolin and its IN-WR791 and IN-E8S72 metabolites within 8 months of harvest using method Dupont-30422 Supplement 1 (LC-MS/MS) and with an LOQ of 0.01 mg/kg. The mean recovery rates from control grape samples fortified with oxathiapiprolin, IN-WR791 or IN-E8S72 at levels of 0.01 and 2.0 mg/kg were 101%, 78% and 76% respectively.

Table 55 Residues in grapes from supervised trials in Europe involving two foliar applications of oxathiapiprolin (OD formulations)

GRAPES Country, year Location (Variety) Trial Ref	Application			DAT	Residues (mg/kg)		
	g ai/ha	water (L/ha)	g ai/hL		Oxathiapiprolin	IN-E8S72	IN-WR791
GAP: China			4-5	PHI:14d			
Czech Republic, 2013 Bzenec Jihomoravsky kraj (Wine grape/ Riesling) DP-37748-Test-03	61-64	1530-1595	4	14	0.021	ND	ND

GRAPES Country, year Location (Variety) Trial Ref	Application			DAT	Residues (mg/kg)		
	g ai/ha	water (L/ha)	g ai/hL		Oxathiapiprolin	IN-E8S72	IN-WR791
France, 2013 Bourgogne (Wine grapes/ Chardonnay) DP-37748-Test-04	49-48	345-340	14	15	0.037	ND	ND
France, 2013 Champagne-Ardennes (Wine Grape/ Pinot Noir) DP-37748-Test-01	52	360	14	14	0.034	ND	ND
France, 2013 Rhône Alpes (Wine grapes/ Gamay) DP-37748-Test-05	50-51	348	14.28	14	0.029	ND	ND
Germany, 2013 Saxony-Anhalt (Wine Grape/ Pinot Noir) DP-37748-Test-02	60-57	1500-1440	4	14	0.2	ND	ND
Greece, 2013 Central Macedonia (Table grape/ Vitus vinifera) DP-37748-Test-06	60	1490	4	14	0.21	ND	ND
Italy,2013 Lombardia Wine grape/ Verdea) DP-37748-Test-08	59-61	1485-1530	4	14	0.2	ND	< 0.01
Spain, 2013 Andalucía (Table grape/ Red Globe) DP-37748-Test-07	61-60	1515-1485	4	14	0.23	ND	ND

Grape leaves

In the supervised trials on grapes conducted in Europe between 2001 and 2013 involving two or three foliar applications of oxathiapiprolin (OD formulations), samples of grape leaves (min 0.5 kg) were taken from vines one day after the last applications from the reverse decline plots treated 60-90 days before grape harvest.

The samples were frozen within 9 hours and analysed for oxathiapiprolin and metabolites (IN-WR791, IN-RDG40, IN-E8S72 and IN-Q7H09 within 14 months of harvest using method DuPont-30422 Supplement 1 (LC-MS/MS) and with an LOQ of 0.01 mg/kg. In some trials, leaves were also collected from vines about a year after the last application and analysed for oxathiapiprolin and a wider range of metabolites (IN-WR791, IN-RDG40, IN-E8S72, IN-RZB20, IN-RZD74, IN-SXS67 and IN-Q7H09) using method DuPont-30422 Supplement 1.

Mean recovery rates for oxathiapiprolin in spiked grape leaf samples ranged from 95% to 102% and were 86–107% for the metabolites (spike levels of 0.01–25 mg/kg).

Table 56 Residues in grape leaves from supervised trials in Europe involving two or three foliar applications of oxathiapiprolin (OD formulations).

GRAPE LEAVES Country, year Location (Variety) Trial Ref	Application				DAT	Residues (mg/kg)				
	no	g ai/ha	water (L/ha)	g ai/hL		Oxathia-piprolin	IN-RDG40	IN-Q7H09	IN-E8S72	IN-WR791

GRAPE LEAVES Country, year Location (Variety) Trial Ref	Application				DAT	Residues (mg/kg)				
	no	g ai/ha	water (L/ha)	g ai/hL		Oxathia-piprolin	IN-RDG40	IN-Q7H09	IN-E8S72	IN-WR791
France, 2010 Bourgogne (Wine Grapes/ Gamay) DP-30043-Test-03a	3	30-31	340-355	8.7	1	5.4	< 0.01	0.016	< 0.01	0.019
France, 2010 Bourgogne (Wine Grapes/ Gamay) DP-30043-Test-03b	3	51	350-355	15	1	9.5	< 0.01	0.035	< 0.01	0.049
France, 2011 Combe de Gaby, Midi-Pyrenees (Wine Grapes/ Malbec) DP-30043-Test-13	3	50-51	350-355	14	1 379[a]	12 (c=0.011) ND	0.014 ND	ND ND	ND ND	0.024 ND
France, 2011 Vineuse, Bourgogne (Wine Grapes/ Pinot Noir) DP-30043-Test-08	3	50-51	350-360	14	1	14	< 0.01	ND	ND	0.026
France, 2013 Rhône Alpes (Wine grapes/ Gamay) DP-37748-Test-05	2	51-50	355-350	14	-0 0 1 3 7 10	0.73 4.1 5.0 5.6 1.0 1.0			< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 0.019 0.023 0.011 0.014
Germany, 2010 Saxony-Anhalt, (Wine Grapes/ Müller-Thurgau DP-30043-Test-01a	3	30-31	1525-1590	1.9	1	6.0	0.012	0.063	ND	0.015
Germany, 2010 Saxony-Anhalt, (Wine Grapes/ Müller-Thurgau) DP-30043-Test-01b	3	48-50	1525-1590	3.2	1	10	0.015	0.086	ND	0.015
Germany, 2011 Wittlich (Table Grapes/ Regent) DP-30043-Test-07	3	62-63	1550-1565	4	1	9.4	0.01	ND	< 0.01	0.051
Greece, 2010 Anchialos,Thessaloniki (Wine Grapes/ Roditis) DP-30043-Test-06a	3	30-32	1525-1655	1.9	1	8.3	0.017	0.06	ND	0.019
Greece, 2010 Anchialos,Thessaloniki (Wine Grapes/ Roditis) DP-30043-Test-06b	3	48-52	1530-1655	3.2	1	13	0.02	0.097	ND	0.018
Greece, 2013 Central Macedonia (Table grape/ Vitis vinifera) DP-37748-Test-06	2	60	1490	4	-0 0 1 3 7 10	7.1 13 15 15 16 16			< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.013 0.017 0.026 0.029 0.02 0.016
Hungary, 2010 Györ-Moson-Sopron (Wine Grapes/ Grüner Veltliner) DP-30043-Test-02a	3	31-32	1615-1640	2	1	2.6	ND	0.023	< 0.01	0.028
Hungary, 2010 Györ-Moson-Sopron (Wine Grapes/ Grüner Veltliner) DP-30043-Test-02b	3	51-52	1625-1650	3.2-3.3	1	6.9	< 0.01	0.034	0.012	0.07

GRAPE LEAVES Country, year Location (Variety) Trial Ref	Application				DAT	Residues (mg/kg)				
	no	g ai/ha	water (L/ha)	g ai/hL		Oxathia-piprolin	IN-RDG40	IN-Q7H09	IN-E8S72	IN-WR791
Italy, 2011 Abruzzo, Teramo (Table Grapes/ Italia) DP-30043-Test-17	3	59-60	1450-1480	3.9-4.0	363[a]	ND	ND	ND	ND	ND
Italy, 2011 Corropoli, Terramo (Wine Grapes/ Montepulciano d'Abruzzo) DP-30043-Test-14	3	62	1430-1475	4.1	1 354[a]	12 ND	< 0.01 ND	ND ND	< 0.01 ND	0.097 ND
Italy,2013 Lombardia (2013) DP-37748-Test-08	2	59-60	1460-1505	4	-0 0 1 3 7 9	0.22 5.5 3.1 1.5 1.2 0.74			< 0.01 0.019 0.01 < 0.01 0.045 0.037	0.01 0.019 0.019 0.042 0.05 0.035
Spain, 2010 Catalunya (Wine Grapes/ Cabernet Sauvignon) DP-30043-Test-05a	3	31	1590	1.9	1	15	0.015	0.071	ND	0.013
Spain, 2010 Catalunya (Wine Grapes/ Cabernet Sauvignon) DP-30043-Test-05b	3	50-51	1575-1600	3.2	1	24	0.02	0.11	ND	0.016
Spain, 2011 Verdu, Catalunya (Wine Grapes/ Shyra) DP-30043-Test-16	3	60	1500	4	367[a]	ND	ND	ND	ND	ND
Spain, 2013 Andalucía (Table grape/ Red Globe) DP-37748-Test-07	2	60	1500-1495	4	-0 0 1 3 8 10	10 13 11 12 12 9.1			ND ND ND < 0.01 0.01 < 0.01	0.014 0.011 0.012 0.021 0.027 0.022
Spain. 2011 Los Palacios, Andalucía (Wine Grapes/ Merlot) DP-30043-Test-15	3	60	1500-1510	4	378[a]	ND	ND	ND	ND	ND

[a] Leaf samples taken from the next season growth - residues of metabolites IN-RZB20, IN-RZD74 and IN-SXS67 were ND in all samples.

Bulb vegetables

Onion, dry bulb

In supervised trials on bulb onions conducted in North America in 2011 and 2012, four foliar applications of oxathiapiprolin (OD formulations) with added adjuvant were applied at 4–6 day intervals intervals using tractor-mounted or backpack sprayers with 3–7 nozzle flat booms (broadcast treatments) or drop nozzles (directed treatments).

Duplicate samples (minimum of 24 bulbs or 1.8 kg) were field dried for 3–10 days, trimmed to remove tops and roots before freezing and storage for up to 18 weeks before analysis for oxathiapiprolin and its metabolites (IN-WR791, IN-RDG40, IN-E8S72, IN-RZB20, IN-RZD74, IN-SXS67 and IN-Q7H09) using method DuPont-30422 Supplement 1 (LC-MS/MS). Mean recovery rates for each analyte ranged from 88% to 110% in samples spiked with 0.01–2 mg/kg. The LOQs were 0.01 mg/kg for all analytes.

Table 57 Residues in dry onion bulbs from supervised trials in North America involving 4 foliar applications of oxathiapiprolin (OD formulations) with adjuvant, applied at 4–6 day intervals.

ONIONS Country, year Location (Variety) Trial Ref	Application			Matrix	DAT	Residues (mg/kg)[a]			
						Oxathiapiprolin		IN-E8S72	
	g ai/ha	water (L/ha)	season total (g ai/ha)			values	mean	values	mean
GAP: USA	15-35		140		PHI: 0d	Max 4 sprays/season. 5-10day RTI			
Canada, 2011 Delhi, ON (LaSalle) DP IR-4 PR-10617-ON20	35	400-410	141 Broadcast	dry Bulb	0 6 10 14	0.016, 0.012 < 0.01, < 0.01 ND, < 0.01 ND, ND	0.014 < 0.01 < 0.01 ND	ND, ND ND, ND ND, ND ND, ND	ND ND ND ND
Canada, 2011 Harrow, ON (Lasalle) DP IR-4 PR-10617-ON21 [not independent]	33-35	490-510	136 Directed	dry Bulb	0 5	< 0.01, < 0.01 < 0.01, ND	< 0.01 < 0.01	ND, ND ND, ND	ND ND
Canada, 2011 Harrow, ON (Pulsar) DP IR-4 PR-10617-ON22	34	490-500	134 Directed	dry Bulb	0 5	< 0.01, 0.012 < 0.01, < 0.01	0.011 < 0.01	ND, ND ND, ND	ND ND
Canada, 2011 Ste-Clotilde, QC (Hyb. Gunnison) DP IR-4 PR-10617-QC06 [not independent]	35-36	355-360	141 Directed	dry Bulb	0 4	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND
Canada, 2011 Ste-Clotilde, QC (Hyb. Talon) DP IR-4 PR-10617-QC07	35-39	355-390	146 Directed	dry Bulb	0 4	0.011, 0.013 ND, ND	0.012 ND	< 0.01, < 0.01 ND, ND	< 0.01 ND
USA, 2011 Freeville, NY (Vespucci) DP IR-4 PR-10617-NY10	33-35	500-570	137 Directed	dry Bulb	0 5	0.018, 0.023 < 0.01, 0.012	0.02 0.011	ND, ND ND, ND	ND ND
USA, 2011 Las Cruces, NM (Sweetpak) DP IR-4 PR-10617-NM08	34-35	230-280	139 Directed	dry Bulb	0 6	0.021, 0.02 < 0.01, < 0.01	0.02 < 0.01	ND, ND ND, ND	ND ND
USA, 2011 Moxee, WA (Candy F1) DP IR-4 PR-10617-WA*16	34-35	295-355	137 Broadcast	dry Bulb	0 5	ND, < 0.01 ND, < 0.01	ND < 0.01	ND, ND ND, ND	ND ND
USA, 2011 Parlier, CA (Candy) DP IR-4 PR-10617-CA72	35-36	370-385	141 Broadcast	dry Bulb	0 4	0.01, 0.01 < 0.01, < 0.01	0.01 < 0.01	ND, ND ND, ND	ND ND
USA, 2011 Parma, ID (Granero) DP IR-4 PR-10617-ID11	34-36	275-285	139 Broadcast	dry Bulb	0 6	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND

ONIONS Country, year Location (Variety) Trial Ref	Application			Matrix	DAT	Residues (mg/kg)[a]			
						Oxathiapiprolin		IN-E8S72	
	g ai/ha	water (L/ha)	season total (g ai/ha)			values	mean	values	mean
USA, 2012 Holtville, CA (BZ 55) DP IR-4 PR-10617-CA71	34-36	480	139 Directed	dry Bulb	0 4	0.026, 0.026 0.015, 0.016	0.026 0.016	ND, ND ND, ND	ND ND
USA, 2012 Weslaco, TX (Granex Yellow) DP IR-4 PR-10617-TX01	34-36	340-355	140 Directed	dry Bulb	0 5	< 0.01, < 0.01 < 0.01, < 0.01	≤ 0.01 < 0.01	ND, ND ND, ND	ND ND

[a] Residues of metabolites IN-WR791, IN-Q7H09, IN-RDG40, IN-RZB20, IN-RZD74 and IN-SXS67 were ND in all samples

Spring onions

In supervised trials on spring onions conducted in North America in 2011 and 2012, four directed foliar applications of oxathiapiprolin (OD formulations) with added adjuvant were applied at 4–6 day intervals intervals using tractor-mounted or backpack sprayers with 3–7 dropped nozzles (directed treatments). Duplicate green onion samples (min plants or 1.8 kg) were lightly washed and trimmed before freezing (within 2 hours) and storage for up to 17 months before analysis for oxathiapiprolin and its metabolites (IN-WR791, IN-RDG40, IN-E8S72, IN-RZB20, IN-RZD74, IN-SXS67 and IN-Q7H09) using method DuPont-30422 Supplement 1 (LC-MS/MS). Mean recovery rates for each analyte ranged from 88% to 110% in samples spiked with 0.01–2 mg/kg. The LOQs were 0.01 mg/kg for all analytes.

Table 58 Residues in spring onions from supervised trials in North America involving 4 foliar applications of oxathiapiprolin (OD formulations) with adjuvant, applied at 4–6 day intervals.

GREEN ONIONS Country, year Location (Variety) Trial Ref	Application			DAT	Residues (mg/kg)[a]					
					Oxathiapiprolin		IN-E8S72		IN-WR791	
	g ai/ha	water (L/ha)	season total (g ai/ha)		values	mean	values	mean	values	mean
GAP: USA	15-35		140		Max 4 sprays/season. 5-10day RTI					
Canada, 2011 Ste-Clotilde, QC (Green Banner) DP IR-4 PR-10617-QC08	37-38	370-380	150	0 4	0.47, 0.43 0.35, 0.36	0.45 0.36	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND
USA, 2011 Laingsburg, MI (Long white bunching) DP IR-4 PR-10617-MI35	36	275-280	144	0 6	0.85, 0.86 0.37, 0.41	0.85 0.39	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND
USA, 2011 Salinas, CA (Evergreen) DP IR-4 PR-10617-CA*74 [not independent]	34-35	490-655	138	0 4	0.43, 0.38 0.24, 0.21	0.4 0.23	ND, ND ND, ND	ND < 0.01	ND, ND ND, ND	ND ND
USA, 2011 Salinas, CA (Guardsman) DP IR-4 PR-10617-CA*73	35-36	245-390	140	0 6	0.51, 0.64 0.27, 0.34	0.57 0.31	ND, ND < 0.01 (2)	ND < 0.01	ND, ND < 0.01, ND	ND ND
USA, 2011 Weslaco, TX (White Spear) DP IR-4 PR-10617-TX14	35	250	140	0 5 10 14	0.58, 0.68 0.4, 0.49 0.33, 0.39 0.19, 0.17	0.63 0.45 0.36 0.18	ND, ND ND, ND ND, ND ND, ND	ND ND ND ND	ND, ND ND, ND ND, ND ND, ND	ND ND ND ND

[a] Residues of metabolites IN-Q7H09, IN-RDG40, IN-RZB20, IN-RZD74 and IN-SXS67 were ND in all samples

Brassica vegetables

Broccoli

In supervised trials on broccoli conducted in North America in 2011, four foliar applications of oxathiapiprolin (OD formulations) with added adjuvant were applied at 4–6 day intervals intervals using tractor-mounted or backpack sprayers with 3–8 nozzle booms.

Duplicate samples (minimum of 12 flowerheads+stems) frozen within 1 hour and stored for up 20 weeks before analysis for oxathiapiprolin and its metabolites (IN-WR791, IN-RDG40, IN-E8S72, IN-RZB20, IN-RZD74, IN-SXS67 and IN-Q7H09) using method DuPont-30422 Supplement 1 (LC-MS/MS). Mean recovery rates for each analyte ranged from 92% to 110% in samples spiked with 0.01–2 mg/kg. The LOQs were 0.01 mg/kg for all analytes. In two trials, additional samples were taken and washed under running cold water for 15–20 seconds, and drained for 2 minutes before being frozen and stored for subsequent analysis.

Table 59 Residues in broccoli heads from supervised trials in North America involving 4 foliar applications of oxathiapiprolin (OD formulations) with adjuvant, applied at 4–6 day intervals

BROCCOLI Country, year Location (Variety) Trial Ref	Application			Matrix	DAT	Residues (mg/kg) [a]			
						Oxathiapiprolin		IN-WR791	
	g ai/ha	water (L/ha)	season total (g ai/ha)			values	mean	values	mean
GAP: USA	15-35		140		PHI: 0d	Max 4 sprays/season. 5-10day RTI			
Canada, 2011 Lynden, ON (Everest) DP-32067-Trial 04	34-36	200	139	Head	0 5	0.22, 0.21 0.22, 0.21	0.21 <u>0.22</u>	ND, ND ND, ND	ND ND
USA, 2011 Alton, NY (Bay Meadow) DP-32067-Trial 01	35	280	140	Head Washed Head	0 5 0	0.84, 0.78 0.34, 0.43 0.13, 0.095	<u>0.81</u> 0.38 0.11	ND, ND ND, ND < 0.01, < 0.01	ND ND < 0.01
USA, 2011 Kerman, CA (Green Magic) DP-32067-Trial 19	35-36	275-285	141	Head	0 5 10 15 29	0.22, 0.25 0.11, 0.18 0.093, 0.1 0.048, 0.073 0.024, 0.022	<u>0.23</u> 0.14 0.098 0.061 0.023	ND, ND ND, ND < 0.01, < 0.01 < 0.01, < 0.01 < 0.01, 0.011	ND ND < 0.01 < 0.01 0.01
USA, 2011 King City, CA (Patron) DP-32067-Trial 05	35	370	140	Head Washed Head	0 5 0	0.056, 0.076 0.027, 0.02 0.057, 0.092	0.066 0.023 <u>0.075</u>	< 0.01, < 0.01 < 0.01, < 0.01 < 0.01, < 0.01	< 0.01 < 0.01 < 0.01
USA, 2011 Madera, CA (Heritage) DP-32067-Trial 07	35-36	280	142	Head	0 5	0.17, 0.18 0.081, 0.067	<u>0.17</u> 0.074	ND, ND < 0.01, < 0.01	ND < 0.01

[a] Residues of metabolites IN-Q7H09, IN-RDG40, IN-E8S72, IN-RZB20, IN-RZD74 and IN-SXS67 were ND in all samples

Cabbage

In supervised trials on head cabbage conducted in North America in 2011, four foliar applications of oxathiapiprolin (OD formulations) with added adjuvant were applied at 4–6 day intervals intervals using tractor-mounted or backpack sprayers with 3–8 nozzle booms.

Duplicate samples (minimum of 12 heads plus wrapper leaves) frozen within 1 hour and stored for up to 20 weeks before analysis for oxathiapiprolin and its metabolites (IN-WR791, IN-RDG40, IN-E8S72, IN-RZB20, IN-RZD74, IN-SXS67 and IN-Q7H09) using method DuPont-30422 Supplement 1 (LC-MS/MS). Mean recovery rates for each analyte ranged from 98% to 104% in

samples spiked with 0.01–2 mg/kg. The LOQs were 0.01 mg/kg for all analytes. In three trials, additional samples were taken and washed under running cold water for 15–20 seconds, and drained for 2 minutes before being frozen and stored for subsequent analysis.

Table 60 Residues in cabbage heads from supervised trials in North America involving 4 foliar applications of oxathiapiprolin (OD formulations) with adjuvant, applied at 4–6 day intervals

CABBAGE Country, year Location (Variety) Trial Ref	Application			Matrix	DAT	Residues (mg/kg) [a]					
						Oxathiapiprolin		IN-WR791		IN-E8S72	
	g ai/ha	water (L/ha)	season total g ai/ha			values	mean	values	mean	values	mean
GAP: USA	15-35		140		PHI: 0d	Max 4 sprays/season. 5-10day RTI					
Canada, 2011 Branchton, ON (BC-63) DP-32067-Trial 15	34-37	200	142	Head Head	0 4	0.32, 0.32 0.12, 0.17	0.32 0.14	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND
Canada, 2011 New Glasgow, PEI (Lennox) DP-32067-Trial 11	34-38	200	143	Head Head	0 5	0.05, 0.04 0.02, 0.01	0.044 0.017	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND
Canada, 2011 St. Marc-sur- Richelieu, QC (Bourbon) DP-32067-Trial 20	34-36	250	139	Head Head Head Head Head	0 5 10 15 30	0.05, 0.06 0.03, 0.07 0.04, 0.03 0.01, 0.01 ND, < 0.01	0.057 0.049 0.035 0.013 ND	< 0.01 (2) < 0.01 (2) < 0.01 (2) 0.01, < 0.01 < 0.01 (2)	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	ND, ND ND, ND ND, ND ND, ND ND, ND	ND ND ND ND ND
USA, 2011 Chula, GA (Cheers) DP-32067-Trial 12	34-36	220-360	140	Head Head	0 6	0.05, 0.07 0.02, 0.01	0.062 0.014	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND
USA, 2011 Germansville, PA (Blue Lagoon) DP-32067-Trial 10	35-36	215	142	Head Head Washed Head	0 6 0	0.11, 0.14 0.01, 0.12 0.09, 0.11	0.12 0.11 0.1	< 0.01, ND < 0.01 (2) < 0.01, ND	ND < 0.01 ND	ND, ND ND, ND ND, ND	ND ND ND
USA, 2011 Oviedo, FL (Copenhagen) DP-32067-Trial 13	34-35	280	137	Head Head Washed Head	0 5 0	0.46, 0.38 0.2, 0.18 0.29, 0.26	0.42 0.19 0.28	< 0.01 (2) < 0.01 (2) < 0.01 (2)	< 0.01 < 0.01 < 0.01	ND, ND ND, ND ND, ND	ND ND ND
USA, 2011 Porterville, CA (Supreme Vantage) DP-32067-Trial 18	35	305	140	Head Head	0 5	0.12, 0.06 0.12, 0.11	0.09 0.12	ND, ND ND, ND	ND ND	< 0.01 (2) ND, ND	< 0.01 ND
USA, 2011 Raymondville, TX (Cheers) DP-32067-Trial 16	35-36	187	143	Head Head	0 5	0.27, 0.3 0.23, 0.19	0.29 0.21	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND
USA, 2011 Rochelle, IL (Blue Vantage) DP-32067-Trial 14	35-36	290	142	Head Head Washed Head	0 5 0	0.17, 0.16 0.05, 0.05 < 0.01, 0.01	0.16 0.05 < 0.01	ND, ND ND, ND ND, ND	ND ND ND	ND, ND ND, ND ND, ND	ND ND ND
USA, 2011 Uvalde, TX (Blue Vantage) DP-32067-Trial 17	35-36	187	142	Head Head	0 4	0.2, 0.24 0.2, 0.21	0.22 0.2	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND

[a] Residues of metabolites IN-Q7H09, IN-RDG40, IN-RZB20, IN-RZD74 and IN-SXS67 were ND in all samples

Cauliflower

In supervised trials on cauliflowers conducted in North America in 2011, four foliar applications of oxathiapiprolin (OD formulations) with added adjuvant were applied at 4–6 day intervals intervals using tractor-mounted or backpack sprayers with 3–8 nozzle booms.

Duplicate samples (minimum of 12 flowerheads+stems) frozen within 1 hour and stored for up to 20 weeks before analysis for oxathiapiprolin and its metabolites (IN-WR791, IN-RDG40, IN-E8S72, IN-RZB20, IN-RZD74, IN-SXS67 and IN-Q7H09) using method DuPont-30422 Supplement 1 (LC-MS/MS). Mean recovery rates for each analyte ranged from 99% to 109% in samples spiked with 0.01–2 mg/kg. The LOQs were 0.01 mg/kg for all analytes.

Table 61 Residues in cauliflower heads from supervised trials in North America involving 4 foliar applications of oxathiapiprolin (OD formulations) with adjuvant, applied at 4–6 day intervals

CAULIFLOWER Country, year Location (Variety) Trial Ref	Application			Matrix	DAT	Residues (mg/kg) [a]			
						Oxathiapiprolin		IN-WR791	
	g ai/ha	water (L/ha)	season total (ai/ha)			values	mean	values	mean
GAP: USA	15-35		140		PHI: 0d	Max 4 sprays/season. 5-10day RTI			
Canada, 2011 St. Marc-sur-Richelieu, QC (Freemont) DP-32067-Trial 03	35-36	250	143	Head Washed Head	0 5 0	0.076, 0.084 < 0.01, 0.016 0.073, 0.086	<u>0.08</u> 0.012 0.079	< 0.01, ND < 0.01, ND ND, < 0.01	ND ND ND
USA, 2011 Corning, CA (Chieftain) DP-32067-Trial 06	35	230	139	Head	0 5	0.11, 0.17 0.07, 0.11	<u>0.14</u> 0.09	< 0.01, ND < 0.01, < 0.01	ND < 0.01
USA, 2011 Hillsboro, OR (Concert) DP-32067-Trial 09	35-36	230	142	Head	0 5	0.078, 0.086 0.025, 0.021	<u>0.082</u> 0.023	ND, ND ND, ND	ND ND
USA, 2011 Marysville, OH (Snow Crown) DP-32067-Trial 02	35	190-200	140	Head	0 5	0.096, 0.087 0.052, 0.038	<u>0.091</u> 0.045	< 0.01, < 0.01 < 0.01, < 0.01	< 0.01 < 0.01
USA, 2011 Porterville, CA (Pacencia) DP-32067-Trial 08	35	300	141	Head	0 5	0.073, 0.082 0.064, 0.055	<u>0.077</u> 0.059	ND, ND < 0.01, < 0.01	ND < 0.01

[a] Residues of metabolites IN-Q7H09, IN-RDG40, IN-E8S72, IN-RZB20, IN-RZD74 and IN-SXS67 were ND in all samples

Fruiting vegetables, Cucurbits

Cucumber

In supervised trials on indoor (protected) cucumbers conducted in Europe in 2011 and 2012, three foliar applications of oxathiapiprolin (OD formulations) without added adjuvant were applied at 6–8 day intervals using motorised or pressurised backpack sprayers with single-nozzle hand lances.

Samples (minimum of 12 fruit or 2 kg) were frozen within 7 hours and stored for up to 44 weeks before analysis for oxathiapiprolin and its metabolites using method DuPont-30422 (LC-MS/MS). In the 2011 trials, metabolites IN-WR791, IN-RDG40, IN-E8S72 and IN-Q7H09 were measured, while in the 2012 trials, metabolites IN-Q7H09 and IN-RDG40 were not investigated. Mean recovery rates for each analyte ranged from 86% to 100% in samples spiked with 0.01–0.5 mg/kg. The LOQs were 0.01 mg/kg for all analytes.

Table 4. Residues in protected cucumbers from supervised trials in Europe involving 3 foliar applications of oxathiapiprolin (OD formulations), applied at 6–8 day intervals.

CUCUMBERS Country, year Location (Variety) Trial Ref	Application				DAT	Residues (mg/kg)[a]
	g ai/ha	water (L/ha)	g ai/hL	season total (g ai/ha)		Oxathiapiprolin
Belgium, 2011 Antwerpen (Proloog) DP-31733-Test 01	29-31	975-1040	3	90.37	-0 0 1 3 7 10	ND 0.022 0.015 0.013 < 0.01 < 0.01
Italy, 2012 Sicily (Solverde) DP-31733-Test 08	30-31	1000	3	90.81	3	0.02
South France, 2011 Rhône-Alpes (Lustic) DP-31733-Test 04	29-31	965-1030	3	89.97	-0 0 1 3 7 10	0.015 0.028 0.016 0.011 < 0.01 < 0.01
South Spain, 2012 Andalucia (Poseidon) DP-31733-Test 10	30	995-1005	3	90.07	3	0.041

[a] Residues of metabolites IN-Q7H09, IN-RDG40, IN-E8S72 and IN-WR791 were ND in all samples

In supervised trials on field and protected cucumbers conducted in USA in 2011, four foliar applications of oxathiapiprolin (OD formulations) with added adjuvant were applied at 2–4 day intervals intervals using motorised or pressurised backpack sprayers with 1–4 nozzle hand lances. In the field trials, pressurised or motorised tractor-mounted 6-nozzle boom sprayers were also used.

Duplicate samples (minimum of 12 units or 2 kg) were frozen within 3 hours (after quartering of larger fruit in the field in some trials), stored for up to 38 weeks before analysis for oxathiapiprolin and its metabolites (IN-WR791, IN-RDG40, IN-E8S72, IN-RZB20, IN-RZD74, IN-SXS67 and IN-Q7H09) using method DuPont-30422 Supplement 1 (LC-MS/MS). Mean recovery rates for each analyte ranged from 98% to 105% in samples spiked with 0.01–0.1 mg/kg. The LOQs were 0.01 mg/kg for all analytes.

Table 63 Residues in protected cucumbers (whole fruit) from supervised trials in North America, involving four foliar applications of oxathiapiprolin (OD formulations) with added adjuvant, applied at 2–4 day intervals

CUCUMBER Country, year Location (Variety) Trial Ref	Application			DAT	Residues (mg/kg) [a]	
	g ai/ha	water (L/ha)	season total (g ai/ha)		Oxathiapiprolin	
					values	mean
GAP: USA	15-35		140	PHI: 0d	Max 4 sprays/season. 3-14 day RTI	
Canada, 2011 Harrow, ON (Camaro large English) DP IR-4 PR-10618-ON31 (August applications)	35	2000	140	0 3 7 15 29	0.045, 0.034 0.028, 0.038 < 0.01, ND ND, ND ND, ND	<u>0.039</u> 0.033 ND ND ND
Canada, 2011 Harrow, ON Camaro large English) DP IR-4 PR-10618-ON32 (June applications)	35	1990-2015	140	0 3	0.023, 0.021 0.018, 0.024	<u>0.022</u> 0.021

CUCUMBER Country, year Location (Variety) Trial Ref	Application			DAT	Residues (mg/kg) [a]	
	g ai/ha	water (L/ha)	season total (g ai/ha)		Oxathiapiprolin	
					values	mean
USA, 2011 Parlier, CA (Manar F1 small English) DP IR-4 PR-10618-CA84	34-38	470-520	142	0 3	0.039, 0.043 0.021, 0.018	0.041 0.019
USA, 2011 Salisbury, MD (Jawell small English) DP IR-4 PR-10618-MD20	35	540	140	0 3	0.044, 0.044 0.016, 0.02	0.044 0.018

Information provided to show residues of parent stable in quartered fruit for the intervals between sampling and freezing

[a] Residues of metabolites IN-Q7H09, IN-RDG40, IN-RZB20, IN-RZD74, IN-WR791, IN-E8S72 and IN-SXS67 were ND in all samples

Table 64 Residues in cucumbers (whole fruit) from supervised field trials in North America, involving four foliar applications of oxathiapiprolin (OD formulations) with added adjuvant, applied at 2-4 day intervals

CUCUMBER Country, year Location (Variety) Trial Ref	Application			DAT	Residues (mg/kg) [a]	
	g ai/ha	water (L/ha)	season total (g ai/ha)		Oxathiapiprolin	
					values	mean
GAP: USA	15-35		140	PHI: 0d	Max 4 sprays/season. 3-14 day RTI	
Canada, 2011 Agassiz, BC (Sassy pickling) DP IR-4 PR-10618-BC12-F	33-36	340-360	139	0 3	0.047, 0.087 0.062, 0.059	0.067 0.06
Canada, 2011 Delhi, ON (Marketmore 76 slicing) DP IR-4 PR-10618-ON29-F [not independent]	35-36	400-405	141	0 3	< 0.01, < 0.01 < 0.01, < 0.01	< 0.01 < 0.01
Canada, 2011 Delhi, ON (Speedway slicing) DP IR-4 PR-10618-ON30-F	35-36	400-405	141	0 3 7 14 29	0.011, 0.013 < 0.01, < 0.01 ND, ND ND, ND ND, ND	0.012 < 0.01 ND ND ND
USA, 2011 Charleston, SC (Poinsett 76) DP IR-4 PR-10618-SC*06-F	36	350-355	144	0 3	0.012, 0.014 < 0.01, < 0.01	0.013 < 0.01
USA, 2011 Citra, FL (Dasher II slicing) DP IR-4 PR-10618-FL22-F	36	375-380	144	0 3	0.027, 0.019 < 0.01, 0.013	0.023 0.011
USA, 2011 Holt, MI (Expedition pickling) DP IR-4 PR-10618-MI36-F	35-36	280-290	141	0 3	0.083, 0.096 0.067, 0.064	0.09 0.066

CUCUMBER Country, year Location (Variety) Trial Ref	Application			DAT	Residues (mg/kg) [a]	
	g ai/ha	water (L/ha)	season total (g ai/ha)		Oxathiapiprolin	
					values	mean
USA, 2011 Raleigh, NC (Calypso pickling) DP IR-4 PR-10618-NC19-F	34-36	245-255	140	0 4	0.032, 0.029 0.011, < 0.01	0.03 0.01
USA, 2011 Salisbury, MD (Stonewall slicing) DP IR-4 PR-10618-MD19-F	34	390-400	136	0 3	0.032, 0.028 0.01, 0.011	0.03 0.011
USA, 2011 Tifton, GA (Straight Eight slicing) DP IR-4 PR-10618-GA*04-F	35-36	300-305	141	0 3	< 0.01, < 0.01 ND, < 0.01	< 0.01 ND
USA, 2011 Weslaco, TX (Genuine slicing) DP IR-4 PR-10618-TX17-F	35	250-255	140	0 3	0.028, 0.029 0.019, 0.017	0.029 0.018
USA, 2011 Weslaco, TX (Wealthy pickling) DP IR-4 PR-10618-TX*18-F	35-36	355-365	143	0 3	0.038, 0.044 0.013, 0.014	0.041 0.014
USA, 2011 Wooster, OH (Dasher II slicing) DP IR-4 PR-10618-OH*05-F	35-37	360-380	142	0 3	< 0.01, < 0.01 ND, ND	< 0.01 ND

Information provided to show residues of parent stable in quartered fruit for the intervals between sampling and freezing

[a] Residues of metabolites IN-Q7H09, IN-RDG40, IN-RZB20, IN-RZD74, IN-WR791, IN-E8S72 and IN-SXS67 were ND in all samples

In the supervised trials on field cucumbers conducted in USA in 2011, separate plots were treated with oxathiapiprolin (SC or OD formulations) as soil drenches or through drip irrigation systems, with two applications being made 6–8 days apart. Dilute concentrations of oxathiapiprolin were either poured around the base of the plants or injected into the drip irrigations systems and followed by irrigation (0.25–0.5 acre-inch) with water.

Samples (minimum of 12 units or 2 kg) were frozen (after quartering of larger fruit in the field in some trials), within 3 hours and stored for up to 38 weeks before analysis for oxathiapiprolin and its metabolites (IN-WR791, IN-RDG40, IN-E8S72, IN-RZB20, IN-RZD74, IN-SXS67 and IN-Q7H09) using method DuPont-30422 Supplement 1 (LC-MS/MS). Mean recovery rates for each analyte ranged from 98% to 105% in samples spiked with 0.01–0.1 mg/kg. The LOQs were 0.01 mg/kg for all analytes.

Table 65 Residues in cucumbers (whole fruit) from supervised trials in North America, involving two soil drench/irrigation treatments oxathiapiprolin, applied 6–8 days apart.

CUCUMBER Country, year Location (Variety) Trial Ref	Application				DAT	Residues (mg/kg) [a]			
	form	method	g ai/ha	season total (g ai/ha)		Oxathiapiprolin		IN-E8S72	
						values	mean	values	mean
GAP: USA			70-280	560	PHI: 0d	Max 4 applications/season. Min 7d RTI			

CUCUMBER Country, year Location (Variety) Trial Ref	Application				DAT	Residues (mg/kg) [a]			
	form	method	g ai/ha	season total (g ai/ha)		Oxathiapiprolin		IN-E8S72	
						values	mean	values	mean
Canada, 2011 Agassiz, BC (Sassy pickling) DP IR-4 PR-10618-BC12-F	SC	drench	280-284	564	0 3	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND
Canada, 2011 Delhi, ON (Marketmore 76) DP IR-4 PR-10618-ON29-F	SC	drip	229-289	518	0 3	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND
Canada,2011 Delhi, ON Speedway slicing DP IR-4 PR-10618-ON30-F [not independent]	SC	drip	289	578	0 3 7 14 29	ND, ND ND, ND ND, ND ND, ND ND, ND	ND ND ND ND ND	ND, ND ND, ND ND, ND ND, ND ND, ND	ND ND ND ND ND
USA, 2011 Charleston, SC (Poinsett 76) DP IR-4 PR-10618-SC*06-F	SC	drench	281	562	0 3	< 0.01, < 0.01 < 0.01, < 0.01	< 0.01 < 0.01	ND, ND < 0.01, < 0.01	ND < 0.01
USA, 2011 Citra, FL (Dasher II slicing) DP IR-4 PR-10618-FL22-F	OD	drench	281	562	0 3	< 0.01, < 0.01 < 0.01, 0.021	< 0.01 0.012	ND, ND ND, ND	ND ND
USA, 2011 Holt, MI (Expedition pickling) DP IR-4 PR-10618-MI36-F	SC	drench	284	568	0 3	ND, ND < 0.01, ND	ND < 0.01	ND, ND ND, ND	ND ND
USA, 2011 Salisbury, MD (Stonewall slicing) DP IR-4 PR-10618-MD19-F	SC	drip	281	562	0 3	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND
USA, 2011 Tifton, GA (Straight Eight) DP IR-4 PR-10618-GA*04-F	SC	drench	284	568	0 3	< 0.01, < 0.01 ND, < 0.01	< 0.01 ND	ND, ND ND, ND	ND ND
USA, 2011 Weslaco, TX (Genuine slicing) DP IR-4 PR-10618-TX17-F	OD	drip	281	562	0 3	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND
USA, 2011 Weslaco, TX (Wealthy pickling) DP IR-4 PR-10618-TX*18-F	OD	drip	281	562	0 3	< 0.01, ND ND, ND	< 0.01 ND	ND, ND ND, ND	ND ND
USA, 2011 Wooster, OH (Dasher II slicing) DP IR-4 PR-10618-OH*05-F	SC	drench	283	566	0 3	< 0.01, < 0.01 ND, ND	< 0.01 ND	ND, ND ND, ND	ND ND

Information provided to show residues of parent stable in quartered fruit for the intervals between sampling and freezing

[a] Residues of metabolites IN-Q7H09, IN-RDG40, IN-RZB20, IN-RZD74, IN-WR791 and IN-SXS67 were ND in all samples

Summer squash

In supervised trials on indoor (protected) summer squash (courgettes) conducted in Europe in 2011 and 2012, three foliar applications of oxathiapiprolin (OD formulations) without added adjuvant were applied at 6–8 day intervals using motorised or pressurised backpack sprayers with single-nozzle hand lances or a six-nozzle mini-boom.

Samples (minimum of 12 fruit or 2 kg) were frozen within 7 hours and stored for up to 44 weeks before analysis for oxathiapiprolin and its metabolites using method DuPont-30422 (LC-

MS/MS). In the 2011 trials, metabolites IN-WR791, IN-RDG40, IN-E8S72, and IN-Q7H09 were measured, while in the 2012 trials, the metabolites IN-Q7H09 and IN-RDG40 were not investigated. Mean recovery rates for each analyte ranged from 86% to 100% in samples spiked with 0.01–0.5 mg/kg. The LOQs were 0.01 mg/kg for all analytes.

Table 66 Residues in protected summer squash (courgettes) from supervised trials in Europe involving 3 foliar applications of oxathiapiprolin (OD formulations), applied at 6–8 day intervals.

SUMMER SQUASH Country, year Location (Variety) Trial Ref	Application				DAT	Residues (mg/kg) [a]
	g ai/ha	water (L/ha)	g ai/hL	season total (g ai/ha)		Oxathiapiprolin
Greece, 2011 Thessaloniki (Jedida F1) DP-31733-Test 02	30	1000	3	90.03	-0 0 1 3 7 10	< 0.01 0.015 < 0.01 0.011 ND ND
Italy, 2011 Sicily (Richgreen) DP-31733-Test 05	30	990-1000	3	89.19	3	0.024
Italy, 2012 Sicily (President) DP-31733-Test 07	30	985-1000	3	89.5	-0 0 1 3 7 10	0.024 0.078 0.059 0.062 0.016 < 0.01
Spain, 2011 Andalucia (Sinatra) DP-31733-Test 03	30-31	100-1020	3	90.68	3	0.01

[a] Residues of metabolites IN-RDG40, IN-WR791, IN-E8S72 and IN-Q7H09 were ND in all samples

In supervised trials on summer squash (courgettes) in the field, conducted in Europe in 2011 and 2012, three foliar applications of oxathiapiprolin (OD formulations) without added adjuvant were applied at 6–8 day intervals intervals using motorised or pressurised backpack sprayers with single-nozzle hand lances or six-nozzle mini-booms.

Samples (minimum of 12 fruit or 2 kg) were frozen within 7 hours and stored for up to 36 weeks before analysis for oxathiapiprolin and its metabolites using method DuPont-30422 (LC-MS/MS). In the 2011 trials, metabolites IN-WR791, IN-RDG40, IN-E8S72 and IN-Q7H09 were measured, while in the 2012 trials, the metabolites IN-Q7H09 and IN-RDG40 were not investigated. Mean recovery rates for each analyte ranged from 95% to 103% in samples spiked with 0.01–0.5 mg/kg. The LOQs were 0.01 mg/kg for all analytes.

Table 67 Residues in field summer squash (courgettes) from supervised trials in Europe involving 3 foliar applications of oxathiapiprolin (OD formulations), applied at 6–8 day intervals

SUMMER SQUASH Country, year Location (Variety) Trial Ref	Application				DAT	Residues (mg/kg) [a]	
	g ai/ha	water (L/ha)	g ai/hL	season total (g ai/ha)		Oxathiapiprolin	IN-WR791
Italy, 2011 Lombardia (President) DP-31732-Test 01	20	975-1020	2	59.87	-0 0	ND 0.016	< 0.01 ND
	20-21	975-1040	2	60.69	1	0.014	ND
	20-21	975-1030	2	60.38	3	< 0.01	ND
	20	1000-1020	2	60.48	8	ND	ND
	20	985-1025	2	60.08	10	ND	< 0.01

SUMMER SQUASH Country, year Location (Variety) Trial Ref	Application				DAT	Residues (mg/kg) [a]	
	g ai/ha	water (L/ha)	g ai/hL	season total (g ai/ha)		Oxathiapiprolin	IN-WR791
France, 2011 Nord-Pas de Calais (Supremo) DP-31732-Test 02	19-20	965-1020	2	59.57	-0 0	< 0.01 0.024	ND ND
	20	985=1010	2	59.68	1	0.013	ND
	19-20	970-985	2	58.75	3	< 0.01	ND
	20-21	985-1030	2	59.67	7	ND	ND
	19	965-970	2	58.04	9	ND	ND
France, 2011 Rhône-Alpes (Mirza) DP-31732-Test 03	19-20	970-1010	2	59.47	3	< 0.01	ND
South Spain, 2011 Andalucia (Nieves) DP-31732-Test 04	20	990-1010	2	59.98	-0 0	ND 0.011	ND ND
	20	990-1010	2	59.97	1	< 0.01	ND
	20	995-1000	2	59.77	3	< 0.01	ND
	20	1000-1015	2	60.38	7	ND	ND
	20	990-1000	2	56.67	10	ND	ND
Italy, 2011 Sicily (Comune) DP-31732-Test 05	20	995-1010	2	60.17	3	< 0.01	ND
Greece, 2011 Central Macedonia (Ezra F1) DP-31732-Test 06	19-20	975-1010	2	59.55	3	0.016	ND
France, 2012 Picardie (Supremo) DP-31732-Test 07	19-21	950-1030	2	59.69	-0 0	< 0.01 0.018	ND ND
	20	985-1030	2	60.32	1	0.025	ND
	19-21	955-1030	2	59.27	3	< 0.01	ND
	19-20	950-1030	2	59.07	7	0.02	ND
	20-21	995-1045	2	60.64	10	< 0.01	ND
Spain, 2012 Andalucia (Amalia) DP-31732-Test 09	20	995-1000	2	59.8	3	< 0.01	ND
Italy, 2012 Sicily (Diamant) DP-31732-Test 10	20	985-995	2	59.23	3	0.013	ND
Italy, 2012 Lombardia (President) DP-31732-Test 11	20	980-995	2	59.07	3	< 0.01	ND

[a] Residues of metabolites IN-RDG40, IN-E8S72 and IN-Q7H09 were ND in all samples

In supervised trials on field summer squash conducted in USA in 2011, four foliar applications of oxathiapiprolin (OD formulations) with added adjuvant were applied at 2–4 day intervals intervals using tractor-mounted, motorised or pressurised backpack sprayers with 3–6 nozzle booms.

Duplicate samples (minimum of 12 units or 2 kg) were frozen (after quartering of larger fruit in the field in some trials) within 5 hours and stored for up to 36 weeks before analysis for oxathiapiprolin and its metabolites (IN-WR791, IN-RDG40, IN-E8S72, IN-RZB20, IN-RZD74, IN-SXS67 and IN-Q7H09) using method DP30422 Supplement 1 (LC-MS/MS). Mean recovery rates for each analyte ranged from 99% to 109% in samples spiked with 0.01–0.2 mg/kg. The LOQs were 0.01 mg/kg for all analytes.

Table 5. Residues in summer squash from supervised field trials in North America, involving four foliar applications of oxathiapiprolin (OD formulations) with added adjuvant, applied at 2–4 day intervals.

SUMMER SQUASH Country, year Location (Variety) Trial Ref	Application			DAT	Residues (mg/kg) [a]					
					Oxathiapiprolin		IN-E8S72		IN-WR791	
	g ai/ha	water (L/ha)	season total (g ai/ha)		values	mean	values	mean	values	mean
GAP: USA	15-35		140	PHI: 0d	Max 4 sprays/season. 3-14 day RTI					
Canada, 2011 Agassiz, BC (Golden Dawn III) DP IR-4 PR-10619-11-BC13	36-39	295-335	149	0 3 6 13 28	0.04, 0.04 0.02, 0.01 < 0.01 (2) < 0.01, ND ND, ND	0.039 0.018 < 0.01 ND ND	ND, ND ND, ND ND, ND ND, ND ND, ND	ND ND ND ND ND	ND, ND ND, ND ND, ND ND, ND ND, ND	ND ND ND ND ND
Canada, 2011 Delhi, ON (Payroll Hybrid) DP IR-4 PR-10619-11-ON33	35-36	400-410	143	0 3	0.03, 0.04 0.01, 0.01	0.033 0.011	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND
Canada, 2011 Harrow, ON (Select) DP IR-4 PR-10619-11-ON34	35-37	300-320	144	0 3	0.03, 0.03 < 0.01, < 0.01	0.031 < 0.01	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND
USA, 2011 Charleston, SC (Superpik F1) DP IR-4 PR-10619-11-SC*07	36	350-355	144	0 3	0.08, 0.08 0.04, 0.04	0.083 0.039	ND, ND ND, ND	ND ND	ND, ND < 0.01 (2)	ND < 0.01
USA, 2011 Citra, FL (Gold Star) DP IR-4 PR-10619-11-FL23	35-36	370-375	142	0 3	< 0.01 (2) < 0.01 (2)	0.01 < 0.01	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND
USA, 2011 Davis, CA (Black Beauty) DP IR-4 PR-10619-11-CA85	34-37	295-315	142	0 3	0.02, 0.05 0.02, 0.01	0.039 0.015	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND
USA, 2011 Freeville, NY (Multipik) DP IR-4 PR-10619-11-NY12	35	335-360	140	0 3	0.02, 0.02 < 0.01 (2)	0.02 < 0.01	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND
USA, 2011 Madison, WI (Golbar Hybrid) DP IR-4 PR-10619-11-WI13	35	250-270	140	0 3	0.03, 0.02 0.01, 0.02	0.023 0.013	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND
USA, 2011 Salisbury, MD (Conquerer III) DP IR-4 PR-10619-11-MD21	35	230-235	140	0 3	0.1, 0.13 0.02, 0.02	0.12 0.02	ND, ND < 0.01 (2)	ND < 0.01	< 0.01 (2) < 0.01 (2)	< 0.01 < 0.01
USA, 2011 Wooster, OH (Envy) DP IR-4 PR-10619-11-OH*06	34-35	355-385	138	0 3	0.03, 0.04 ND, ND	0.03 ND	ND, ND ND, ND	ND ND	< 0.01 (2) < 0.01 (2)	< 0.01 < 0.01

Information provided to show residues of parent stable in quartered fruit for the intervals between sampling and freezing

[a] Residues of metabolites IN-Q7H09, IN-RDG40, IN-RZB20, IN-RZD74 and IN-SXS67 were ND in all samples

In the supervised trials on field summer squash conducted in USA in 2011, separate plots were treated with oxathiapiprolin (SC formulations - OD formulation at one site) as soil drenches or through drip irrigation systems, with two pre-harvest applications being made 6–8 days apart. In three additional trials,soil drench or drip irrigation treatments were made at different growth stages, ranging from planting through to pre-harvest. Dilute concentrations of oxathiapiprolin were either poured around the base of the plants or injected into the drip irrigations systems and followed by irrigation (0.25–0.5 acre-inch) with water.

Samples (minimum of 12 units or 2 kg) were frozen (after quartering of larger fruit in the field in some trials) within 5 hours and stored for up to 36 weeks before analysis for oxathiapiprolin and its metabolites (IN-WR791, IN-RDG40, IN-E8S72, IN-RZB20, IN-RZD74, IN-SXS67 and IN-Q7H09) using method DP30422 Supplement 1 (LC-MS/MS). Mean recovery rates for each analyte ranged from 98% to 105% in samples spiked with 0.01–0.1 mg/kg. The LOQs were 0.01 mg/kg for all analytes.

Table 69 Residues in summer squash (whole fruit) from supervised trials in North America, involving two soil drench/irrigation treatments oxathiapiprolin, applied 6–8 days apart.

SUMMER SQUASH Country, year Location (Variety) Trial Ref	Application			DAT	Residues (mg/kg) [a]					
	method	g ai/ha	season total (g ai/ha)		Oxathiapiprolin		IN-E8S72		IN-WR791	
					values	mean	values	mean	values	mean
GAP: USA	soil	70-280	560	PHI: 0d	Max 4 applications/season. Min 7d RTI					
Canada, 2011 Agassiz, BC (Golden Dawn III) DP IR-4 PR-10619-11-BC13	drench	283-289	571	0 3 8 15 30	ND, < 0.01 ND, ND ND, ND ND, ND ND, ND	ND ND ND ND ND	ND, ND ND, ND ND, ND ND, ND ND, ND	ND ND ND ND ND	ND, ND ND, ND ND, ND ND, ND ND, ND	ND ND ND ND ND
Canada, 2011 Delhi, ON (Payroll Hybrid) DP IR-4 PR-10619-11-ON33	drip	289	578	0 3	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND
Canada, 2011 Harrow, ON (Select) DP IR-4 PR-10619-11-ON34	drench	280	560	0	< 0.01, ND ND, ND	< 0.01 ND	ND, ND ND, ND	ND ND	ND, < 0.01 ND, ND	< 0.01 ND
USA, 2011 Charleston, SC (Superpik F1) DP IR-4 PR-10619-11-SC*07	drench	281	562	0 3	0.01, 0.01 0.02, 0.01	0.01 0.017	ND, ND < 0.01,ND	ND ND	ND, ND 0.02, 0.01	ND 0.014
USA, 2011 Citra, FL (Gold Star) OD Formulation DP IR-4 PR-10619-11-FL23	drench	280	560	0 3	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND
USA, 2011 Davis, CA (Black Beauty) DP IR-4 PR-10619-11-CA85	drip	282	564	0 3	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND
USA, 2011 Freeville, NY (Multipik) DP IR-4 PR-10619-11-NY12	drench	280	560	0 3	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND

SUMMER SQUASH Country, year Location (Variety) Trial Ref	Application			DAT	Residues (mg/kg) [a]					
	method	g ai/ha	season total (g ai/ha)		Oxathiapiprolin		IN-E8S72		IN-WR791	
					values	mean	values	mean	values	mean
USA, 2011 Madison, WI (Golbar Hybrid) DP IR-4 PR-10619-11-WI13	drench	278-279	557	0 3	0.04, 0.01 < 0.01 (2)	0.026 < 0.01	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND
USA, 2011 Salisbury, MD (Conquerer III) DP IR-4 PR-10619-11-MD21	drench	284	568	0 3	ND, 0.01 ND, ND	< 0.01 ND	ND, ND ND, ND	ND ND	< 0.01, ND < 0.01 (2)	ND < 0.01
USA, 2011 Wooster, OH (Envy) DP IR-4 PR-10619-11-OH*06	drench	283	566	0 3	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND	ND, ND < 0.01, ND	ND ND
USA, 2011 Germansville, PA (Super Pik) DP-33132-Trial 01	drench	280	560	0 7 14	ND, ND ND, ND ND, ND	ND ND ND	ND, ND ND, ND ND, ND	ND ND ND	ND, ND ND, ND ND, ND	ND ND ND
	drench-T3	280	560	49	ND, ND	ND	ND, ND	ND	ND, ND	ND
	drench-T4	280	560	28 35 42	ND, ND ND, ND ND, ND	ND ND ND	< 0.01, ND ND, ND ND, ND	ND ND ND	< 0.01 (2) ND, ND ND, ND	< 0.01 ND ND
	drench-T5	280	560	6 13 20	ND, ND ND, ND ND, ND	ND ND ND	ND, ND ND, ND ND, ND	ND ND ND	ND, ND ND, ND ND, ND	ND ND ND
Canada, 2011 Branchton, ON (Senater Zucchini) DP-33132-Trial 02	drip	283	566	0 7 14	ND, ND ND, ND ND, ND	ND ND ND	ND, ND ND, ND ND, ND	ND ND ND	ND, ND ND, ND ND, ND	ND ND ND
	drip-T3	283	566	27	ND, ND	ND	< 0.01 (2)	< 0.01	< 0.01, ND	ND
	drip-T4	283	566	6 13 20	ND, ND ND, ND ND, ND	ND ND ND	ND, ND ND, ND ND, ND	ND ND ND	ND, ND ND, ND ND, < 0.01	ND ND ND
USA, 2011 Porterville, CA (Black Beauty) DP-33132-Trial 03 One application	drench	280	280	0 7 14	ND, < 0.01 0.06, 0.1 0.1, 0.04	ND 0.079 0.071	ND, ND ND, ND ND, ND	ND ND ND	ND, ND ND, ND ND, ND	ND ND ND
	drench-T2	280	280	70	ND, ND	ND	< 0.01, ND	ND	ND, ND	ND
	drench-T4	280	280	47 54 61	ND, ND ND, ND ND, ND	ND ND ND	< 0.01 (2) < 0.01 (2) < 0.01 (2)	< 0.01 < 0.01 < 0.01	ND, ND ND, ND ND, ND	ND ND ND
	drench-T5	280	280	27 34 41	ND, ND < 0.01 (2) ND, 0.04	ND < 0.01 0.019	< 0.01 (2) < 0.01 (2) < 0.01 (2)	< 0.01 < 0.01 < 0.01	ND, ND ND, ND ND, ND	ND ND ND
	drench T6	280	280	7 14 21	< 0.01, ND ND, < 0.01 0.01, 0.03	< 0.01 ND 0.022	ND, ND ND, ND < 0.01, ND	ND ND ND	ND, ND ND, ND ND, ND	ND ND ND
USA, 2012 Porterville, CA (Black Beauty) DP-33132-Trial 03	drench	280	560	0 7	0.01, 0.02 ND, ND	0.013 ND	0.02, < 0.01 0.01, 0.01	0.013 0.011	0.01, < 0.01 0.01, 0.01	0.01 0.013
	drench-T3	280	560	84	0.04, 0.08	0.061	ND, ND	ND	ND, ND	ND
	drench-T4	280	560	63 70	ND, ND ND, ND	ND ND	0.01, 0.01 0.03, 0.02	0.013 0.023	ND, ND < 0.01 (2)	ND < 0.01
	drench-T5	280	560	42 49	ND, ND ND, ND	ND ND	0.01, 0.02 0.01, 0.02	0.015 0.017	ND, ND ND, ND	ND ND
	drench-T6	280	560	21 28	< 0.01, 0.01 ND, ND	0.01 ND	< 0.01, 0.02 0.02, 0.01	0.012 0.016	ND, ND ND, ND	ND ND

Information provided to show residues of parent stable in quartered fruit for the intervals between sampling and freezing

[a] Residues of metabolites IN-Q7H09, IN-RDG40, IN-RZB20, IN-RZD74 and IN-SXS67 were ND in all samples

T3 First applications at planting

T4 First application 21 days after planting

T5 First application 42 days after planting

T6 First application 63 days after planting

T7 First application 84 days after planting

Melons

In supervised trials on protected melons, conducted in Europe in 2011 and 2012, three foliar applications of oxathiapiprolin (OD formulations) without added adjuvant were applied at 6–8 day intervals using motorised or pressurised backpack sprayers with single-nozzle hand lances or 6-nozzle mini-booms.

Samples (minimum of 12 fruit or 2 kg) were separated into pulp and peel (with the seeds discarded), sub-sampled by slicing or quartering, frozen within 9 hours and stored for up to 40 weeks before analysis for oxathiapiprolin and its metabolites using method DP30422 (LC-MS/MS). In the 2011 trials, metabolites IN-WR791, IN-RDG40, IN-E8S72 and IN-Q7H09 were measured, while in the 2012 trials, the metabolites IN-Q7H09 and IN-RDG40 were not investigated. Mean recovery rates for each analyte ranged from 88% to 102% in samples spiked with 0.01–0.5 mg/kg. The LOQs were 0.01 mg/kg for all analytes.

Table 70 Residues in protected melons from supervised trials in Europe involving 3 foliar applications of oxathiapiprolin (OD formulations), applied at 6–8 day intervals

MELONS Country, year Location (Variety) Trial Ref	Application				DAT	Oxathiapiprolin residues (mg/kg) [a]		
	g ai/ha	water (L/ha)	g ai/hL	season total (g ai/ha)		Peel	Pulp	Whole fruit
France, 2011 Midi-Pyrénées (Alonso) DP-31746-Test 01	29.5-30	980-995	3	89.26	-0 0 1 3 7 10	0.02 0.039 0.023 0.029 0.024 0.017	ND ND ND ND ND ND	0.011 0.02 0.011 0.013 0.01 < 0.01
France, 2012 Provence Alpes Cote d'Azure (Maltese) DP-31746-Test 09	31	995-1010	3.1	93.06	3	0.026	ND	0.014
Greece, 2011 Thessaloniki (Lavigal) DP-31746-Test 05	29.5-30	990-1010	3	89.93	3	< 0.01	ND	< 0.01
Greece, 2012 Thessaloniki (Lavigal) DP-31746-Test 07	29.5-30	980-1000	3	89.23	-0 0 1 3 7 9	0.061 0.12 0.14 0.11 0.13 0.1	ND < 0.01 < 0.01 ND ND < 0.01	0.039 0.069 0.089 0.072 0.085 0.068
Italy, 2011 Sicily (Cabrero) DP-31746-Test 03	30	995-1000	3	89.76	-0 0 1 3 7 10	0.016 0.013 0.031 0.025 0.036 0.04	ND ND ND ND ND ND	0.01 < 0.01 0.018 0.015 0.02 0.021

MELONS Country, year Location (Variety) Trial Ref	Application				DAT	Oxathiapiprolin residues (mg/kg) [a]		
	g ai/ha	water (L/ha)	g ai/hL	season total (g ai/ha)		Peel	Pulp	Whole fruit
Italy, 2012 Sicily (Polis) DP-31746-Test 08	30	990-1000	3	90.03	3	0.036	ND	0.022
Spain, 2011 Adra (Valverde) DP-31746-Test 02	30	995-1005	3	90.07	-0 0 1 3 7 10	0.052 0.02 0.016 < 0.01 < 0.01 < 0.01	ND ND ND ND ND ND	0.021 < 0.01 < 0.01 < 0.01 ND < 0.01
Spain, 2011 La Mojonera (Homer) DP-31746-Test 04	30	1000	3	89.87	3	0.01	ND	< 0.01

Information provided to show residues of parent stable in quartered fruit for the intervals between sampling and freezing

[a] Residues of metabolites IN-Q7H09, IN-RDG40, IN-E8S72 and IN-WR791 were ND in all samples

In supervised field trials on melons, conducted in Europe in 2011 and 2012, three foliar applications of oxathiapiprolin (OD formulations) without added adjuvant were applied at 6–8 day intervals intervals using motorised or pressurised backpack sprayers with single-nozzle hand lances or 6–8 nozzle booms.

Samples (minimum of 12 fruit or 2 kg) were separated into pulp and peel (with the seeds discarded), sub-sampled by slicing or quartering, frozen within 8 hours and stored for up to 28 weeks before analysis for oxathiapiprolin and its metabolites using method DP30422 (LC-MS/MS). In the 2011 trials, metabolites IN-WR791, IN-RDG40, IN-E8S72 and IN-Q7H09 were measured, while in the 2012 trials, the metabolites IN-Q7H09 and IN-RDG40 were not investigated. Mean recovery rates for each analyte ranged from 91% to 106% in samples spiked with 0.01–0.5 mg/kg. The LOQs were 0.01 mg/kg for all analytes.

Table 71 Residues in field melons from supervised trials in Europe involving 3 foliar applications of oxathiapiprolin (OD formulations), applied at 6-8 day intervals

MELONS Country, year Location (Variety) Trial Ref	Application				DAT	Oxathiapiprolin residues (mg/kg) [a]		
	g ai/ha	water (L/ha)	g ai/hL	season total (g ai/ha)		Peel	Pulp	Whole fruit
France, 2011 Maine et Loire (Hugo) DP-31745-Test 01	20	890-900	2	59.95	-0 0 1 3 7 10	< 0.01 0.038 0.032 0.03 0.022 0.015	ND ND ND ND ND ND	< 0.01 0.014 0.013 0.012 < 0.01 < 0.01
France, 2012 Maine et Loire (Gandalf) DP-31745-Test 06	19-20.5	940-1065	2	60.54	-0 0 1 3 7 10	0.012 0.027 0.034 0.025 0.021 0.017	ND ND ND ND ND ND	< 0.01 0.014 0.018 0.013 0.012 < 0.01
Greece, 2011 Thessaloniki (Lavigal) DP-31745-Test 03	20	995-1000	2	59.79	-0 0 1 3 8 10	0.031 0.031 0.01 0.012 0.019 < 0.01	ND ND ND ND ND ND	0.017 0.018 < 0.01 < 0.01 0.011 < 0.01

MELONS Country, year Location (Variety) Trial Ref	Application				DAT	Oxathiapiprolin residues (mg/kg) [a]		
	g ai/ha	water (L/ha)	g ai/hL	season total (g ai/ha)		Peel	Pulp	Whole fruit
Greece, 2012 Thessaloniki (Lavigal) DP-31745-Test 08	20	1000	2	59.86	3	< 0.01	ND	< 0.01
Italy, 2011 Sicily (Gialetto) DP-31745-Test 05	20	1000-1005	2	60.07	3	0.1	ND	0.059
Italy, 2012 Sicily (Helios) DP-31745-Test 09	20	1010-1015	2	60.49	3	< 0.01	ND	< 0.01
Spain, 2011 Aguadulce (Ricura) DP-31745-Test 02	20	995-1000	2	59.97	-0 0 1 3 7 9	0.011 0.011 < 0.01 < 0.01 < 0.01 0.011	ND ND ND ND ND ND	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01
Spain, 2011 Andalucia (Sancho) DP-31745-Test 04	20	995-1005	2	59.87	3	0.014	ND	< 0.01
Spain, 2012 Andalucia (Sancho) DP-31745-Test 10	20	995-1000	1.99	59.7	3	< 0.01	ND	< 0.01

Information provided to show residues of parent stable in quartered fruit for the intervals between sampling and freezing

[a] Residues of metabolites IN-Q7H09, IN-RDG40, IN-E8S72 and IN-WR791 were ND in all samples

In supervised trials on field melons conducted in North America in 2011, four foliar applications of oxathiapiprolin (OD formulations) with added adjuvant were applied at 2–4 day intervals intervals using tractor-mounted, motorised or pressurised backpack sprayers with 2–6 nozzle booms.

Duplicate samples (minimum of 12 whole fruit or 2 kg) were separated into pulp and peel (with the seeds discarded), sub-sampled by slicing or quartering, frozen within 3.5 hours and stored for up to 73 weeks (18 weeks for the pulp samples) before analysis for oxathiapiprolin and its metabolites (IN-WR791, IN-RDG40, IN-E8S72, IN-RZB20, IN-RZD74, IN-SXS67 and IN-Q7H09) using method DP30422 Supplement 1 (LC-MS/MS). Mean recovery rates for each analyte ranged from 100% to 107% in samples spiked with 0.01–0.4 mg/kg. The LOQs were 0.01 mg/kg for all analytes.

Table 72 Residues in field melons from supervised field trials in North America, involving four foliar applications of oxathiapiprolin (OD formulations) with added adjuvant, applied at 2–4 day intervals.

MELONS Country, year Location (Variety) Trial Ref	Application			Matrix	DAT	Residues (mg/kg) [a]			
	g ai/ha	water (L/ha)	season total (g ai/ha)			Oxathiapiprolin		IN-WR791	
						values	mean	values	mean
GAP: USA	15-35		140		PHI: 0d	Max 4 sprays/season. 3-14 day RTI			
Canada, 2011 Delhi, ON (Sugar Cube) Trial 10620.11-ON01	34-36	405-410	140	Whole Fruit	0 3	0.045, 0.03 0.04, 0.044	0.038 <u>0.042</u>	ND, < 0.01 < 0.01, ND	ND ND

MELONS Country, year Location (Variety) Trial Ref	Application			Matrix	DAT	Residues (mg/kg) [a]			
	g ai/ha	water (L/ha)	season total (g ai/ha)			Oxathiapiprolin		IN-WR791	
						values	mean	values	mean
Canada, 2011 St-Jean-sur-Richelieu, QC (Hyb.Athena) Trial 10620.11-QC15	34-36	345-360	140	Whole Fruit	0 3	0.081, 0.081 0.08, 0.089	0.081 <u>0.085</u>	ND, ND ND, ND	ND ND
USA, 2011 Clinton, NC (Athena) Trial 10620.11-NC20	35-36	220	142	Whole Fruit Pulp	0 3 0	0.057, 0.048 0.041, 0.033 ND, ND	<u>0.052</u> 0.037 ND	ND, < 0.01 ND, ND ND, ND	ND ND ND
USA, 2011 Davis, CA (Durango) Trial 10620.11-CA88	34-35	290-300	139	Whole Fruit	0 3	0.04, 0.032 0.03, 0.039	<u>0.036</u> 0.034	ND, ND ND, ND	ND ND
USA, 2011 Fremont, OH (Odyssey) Trial 10620.11-OH*07	35-38	370-415	146	Whole Fruit	0 3	0.014, 0.014 < 0.01, 0.01	<u>0.014</u> < 0.01	< 0.01, < 0.01 < 0.01, < 0.01	< 0.01 < 0.01
USA, 2011 Holt, MI (Sugar Cube) Trial 10620.11-MI37	34-36	280-285	140	Whole Fruit	0 3	0.062, 0.056 0.055, 0.055	<u>0.059</u> 0.055	ND, ND ND, ND	ND ND
USA, 2011 Holtville, CA (Gold Express) Trial 10620.11-CA86	34-36	480-500	141	Whole Fruit Pulp	0 3 0	0.13, 0.1 0.073, 0.087 < 0.01, < 0.01	<u>0.12</u> 0.08 < 0.01	ND, ND ND, ND ND, ND	ND ND ND
USA, 2011 Holtville, CA (Navigator) Trial 10620.11-CA87 [not independent]	33-36	185-215	138	Whole Fruit	0 3	0.074, 0.13 0.065, 0.12	0.1 0.091	ND, ND ND, ND	ND ND
USA, 2011 Salisbury, MD (Athena) Trial 10620.11-MD22	34-35	230-235	138	Whole Fruit Pulp	0 3 0	0.042, 0.024 0.017, 0.028 ND, ND	<u>0.033</u> 0.022 ND	ND, ND ND, ND ND, ND	ND ND ND
USA, 2011 Tifton, GA (Green flesh honeydew) Trial 10620.11-GA*05	35-36	300-305	141	Whole Fruit	0 3	0.031, 0.031 0.032, 0.035	0.031 <u>0.033</u>	ND, ND ND, ND	ND ND
USA, 2011 Weslaco, TX (Primo) Trial 10620.11-TX*20	35	323	140	Whole Fruit	0 3 7 14 28	0.074, 0.063 0.011, 0.01 < 0.01, < 0.01 0.023, 0.022 < 0.01	<u>0.068</u> 0.011 < 0.01 0.022 < 0.01	ND, ND ND, ND ND, ND ND, ND ND	ND ND ND ND ND
USA, 2011 Weslaco, TX (Sarah's choice) Trial 10620.11-TX19	35-36	255-26	142	Whole Fruit	0 3	0.012, 0.017 < 0.01, < 0.01	<u>0.015</u> < 0.01	ND, ND ND, ND	ND ND

Information provided to show residues of parent stable in quartered fruit for the intervals between sampling and freezing

[a] Residues of metabolites Q7H09, RDG40, E8S72, RZB20, RZD74 and SXS67 were ND in all samples

In the supervised trials on field melons (cantaloupes) conducted in USA in 2011, separate plots were treated with oxathiapiprolin (SC or OD formulations) as soil drenches or through drip irrigation systems, with two applications being made 6–8 days apart. Dilute concentrations of oxathiapiprolin were either poured around the base of the plants or injected into the drip irrigations systems and followed by irrigation (0.25–0.5 acre-inch) with water.

Duplicate samples (minimum of 12 fruit or 2 kg) were sliced longitudinally or quartered, with the sub-sampled specimens being frozen within 3.5 hours and stored for up to 73 weeks before

analysis for oxathiapiprolin and its metabolites (IN-WR791, IN-RDG40, IN-E8S72, IN-RZB20, IN-RZD74, IN-SXS67 and INQ7H09) using method DP30422 Supplement 1 (LC-MS/MS). Mean recovery rates for each analyte ranged from 100% to 107% in samples spiked with 0.01–0.4 mg/kg. The LOQs were 0.01 mg/kg for all analytes.

Table 73 Residues in melons from supervised trials in North America, involving two soil drench/irrigation treatments oxathiapiprolin, applied 6-8 days apart.

MELONS Country, year Location (Variety) Trial Ref	Application				Matrix	DAT	Residues (mg/kg) [a]	
	form	type	g ai/ha	season total (g ai/ha)			Oxathiapiprolin	
							values	mean
GAP: USA	Max 4/season @		35-280	560		PHI: 0d	Min 7 day RTI	
Canada, 2011 Delhi, ON (Sugar Cube) Trial 10620.11-ON01	SC	drip	289	578	Whole Fruit	0 3 14	ND, ND 0.011, ND ND, ND	ND < 0.01 ND
Canada, 2011 St-Jean-sur-Richelieu, QC (Hyb.Athena) Trial 10620.11-QC15	SC	drip	279	558	Whole Fruit	0 3	ND, ND ND, ND	ND ND
USA, 2011 Clinton, NC (Athena) Trial 10620.11-NC20	SC	drench	285-286	571	Whole Fruit	0 3	ND, ND ND, ND	ND ND
USA, 2011 Davis, CA (Durango) Trial 10620.11-CA88	SC	drip	281	562	Whole Fruit	0 3 26	ND, ND ND, ND ND, ND	ND ND ND
USA, 2011 Fremont, OH (Odyssey) Trial 10620.11-OH*07	SC	drench	282-283	565	Whole Fruit	0 3	0.015, 0.034 0.032, 0.036	0.024 0.034
USA, 2011 Holt, MI (Sugar Cube) Trial 10620.11-MI37	SC	drench	283-284	567	Whole Fruit	0 3	0.021, 0.011 0.032, < 0.01	0.016 0.019
USA, 2011 Holtville, CA (Gold Express) Trial 10620.11-CA86 [different site/spray dates]	OD	drip	257-285	542	Whole Fruit	0 3	ND, < 0.01 ND, < 0.01	< 0.01 ND
USA, 2011 Holtville, CA (Navigator) Trial 10620.11-CA87	OD	drip	280	560	Whole Fruit	0 3	ND, ND ND, 0.028	ND 0.015
USA, 2011 Salisbury, MD (Athena) Trial 10620.11-MD22	SC	drip	281	562	Whole Fruit	0 3 11	ND, ND ND, ND ND, ND	ND ND ND
USA, 2011 Tifton, GA (Green flesh honey dew) Trial 10620.11-GA*05	SC	drench	281	562	Whole Fruit	0 3 10	ND, ND ND, ND ND, ND	ND ND ND
USA, 2011 Weslaco, TX (Primo) Trial 10620.11-TX*20	SC	drip	281	562	Whole Fruit	0 3 7 14 27	ND, < 0.01 0.025, 0.01 ND, ND ND, ND ND	< 0.01 0.017 ND ND ND
USA, 2011 Weslaco, TX (Sarah's choice) Trial 10620.11-TX19	SC	drip	282-283	565	Whole Fruit	0 3	ND, ND ND, ND	ND ND

Information provided to show residues of parent stable in quartered fruit for the intervals between sampling and freezing

[a] Residues of metabolites IN-WR791, Q7H09, RDG40, E8S72, RZB20, RZD74 and SXS67 were <LOD in all samples

Fruiting vegetables, other than Cucurbits

Peppers

In supervised trials on field and greenhouse peppers conducted in North America in 2011, four foliar applications of oxathiapiprolin (OD formulations) with added adjuvant were applied at 2–4 day intervals intervals using tractor-mounted, motorised or pressurised backpack sprayers with 3–8 nozzle booms.

Samples (minimum of 12 whole fruit or 2 kg) were frozen within 3 hours and stored for up to 31 weeks before analysis for oxathiapiprolin and its metabolites (IN-WR791, IN-RDG40, IN-E8S72, IN-RZB20, IN-RZD74, IN-SXS67 and IN-Q7H09) using method DP30422 Supplement 1 (LC-MS/MS). Mean recovery rates for each analyte ranged from 90% to 108% in samples spiked with 0.01–0.2 mg/kg. The LOQs were 0.01 mg/kg for all analytes.

Table 74 Residues in peppers (bell and non-bell) from supervised field and greenhouse trials in North America, involving four foliar applications of oxathiapiprolin (OD formulations) with added adjuvant, applied at 2–4 day intervals

PEPPERS Country, year Location (Variety) Trial Ref	Application			DAT	Residues (mg/kg)[a]					
					Oxathiapiprolin		IN-E8S72		IN-WR791	
	g ai/ha	Water (L/ha)	season total (g ai/ha)		values	mean	values	mean	values	mean
GAP: USA	15-35		140	PHI: 0d	Max 4 sprays/season. 3-14 day RTI					
Canada, 2011 Delhi, ON (Non-Bell/ El Jefe) DP IR-4 PR-10621.11-ON24	36	405-410	140	0 5	0.047, 0.063 0.047, 0.042	0.055 0.045	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND
Canada, 2011 Harrow, ON (Bell/ Revolution) DP IR-4 PR-10621.11-ON23	35-36	290-310	142	0 5 10 15 37	0.025, 0.015 < 0.01, 0.011 < 0.01, < 0.01 < 0.01,< 0.01 ND, ND	0.02 0.01 < 0.01 < 0.01 ND	ND, ND ND, ND ND, ND ND, ND ND, ND	ND ND ND ND ND	ND, ND ND, ND < 0.01 (2) < 0.01 (2) < 0.01 (2)	ND ND < 0.01 < 0.01 < 0.01
Canada, 2011 L'Acadie, QC (Bell/ Red Knight) DP IR-4 PR-10621.11-QC09	35-37	445-475	143	0 6	0.1, 0.13 0.059, 0.076	0.12 0.068	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND
Canada, 2011 L'Acadie, QC (Non-Bell/ Hyb. Carmen) DP IR-4 PR-10621.11-QC10	48 35-37	460 470-450	156	0 5	0.13, 0.12 0.075, 0.1	0.12 0.088	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND
USA, 2011 Charleston, SC (Non-Bell/ Sweet banana) DP IR-4 PR-10621.11-SC*04	35-36	345-350	143	0 4 11 15 30	0.077, 0.04 0.022, 0.031 0.014, 0.023 0.011, 0.013 < 0.01, < 0.01	0.059 0.027 0.019 0.012 < 0.01	ND, ND ND, ND ND, ND < 0.01, ND < 0.01 (2)	ND ND ND ND < 0.01	ND, ND ND, < 0.01 < 0.01 (2) < 0.01 (2) < 0.01 (2)	ND ND < 0.01 < 0.01 < 0.01
USA, 2011 Citra, FL (Bell/ Aristotle) DP IR-4 PR-10621.11-FL18	36	425	144	0 5	0.036, 0.032 0.033, 0.025	0.034 0.029	ND, ND ND, ND	ND ND	ND, ND < 0.01 (2)	ND < 0.01
USA, 2011 Citra, FL (Non-Bell/ Compadre) DP IR-4 PR-10621.11-FL19	34-35	235	143	0 5	0.019, 0.037 0.033, 0.026	0.028 0.029	ND, ND ND, ND	ND ND	ND, ND < 0.01 (2)	ND < 0.01
USA, 2011 Clinton, GA (Bell/ Intruder) DP IR-4 PR-10621.11-NC17	36	265	144	0 4	0.013, 0.019 < 0.01, 0.013	0.016 0.011	ND, ND ND, ND	ND ND	< 0.01 (2) < 0.01 (2)	< 0.01 < 0.01
USA, 2011 Freemont, OH (Bell/ Tomcat) DP IR-4 PR-10621.11-OH*04	36-37	360-425	147	0 5	0.017, 0.015 < 0.01, < 0.01	0.016 < 0.01	ND, ND ND, ND	ND ND	ND, ND < 0.01 (2)	ND < 0.01

PEPPERS Country, year Location (Variety) Trial Ref	Application			DAT	Residues (mg/kg)[a]					
					Oxathiapiprolin		IN-E8S72		IN-WR791	
	g ai/ha	Water (L/ha)	season total (g ai/ha)		values	mean	values	mean	values	mean
USA, 2011 Holtville, CA (Bell/ Encore) DP IR-4 PR-10621.11-CA77	35-36	215-225	142	0 5	0.032, 0.026 0.045, 0.043	0.029 0.044	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND
USA, 2011 Irvine, CA (Bell/ Carlwander) Trial 10621.11-CA75	35-36	325-335	141	0 5	0.035, 0.02 0.02, 0.021	0.027 0.02	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND
USA, 2011 Las Cruces, NM (Non-Bell/ Big Jim) DP IR-4 PR-10621.11-NM09	2×35 1×50 1×36	365-395 410 410	156	0 5	0.092, 0.077 0.072, 0.055	0.084 0.063	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND
USA, 2011 Las Cruces, NM (Non-Bell/ Joe E. Parker) DP IR-4 PR-10621.11-NM10 [not independent]	36-37	210-265	146	0 5	0.028, 0.031 0.026, 0.027	0.029 0.027	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND
USA, 2011 Madison, WI (Bell/ Tomcat) DP IR-4 PR-10621.11-WI12	35	260-265	140	0 5	0.034, 0.04 0.013, 0.018	0.037 0.015	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND
USA, 2011 Tifton, GA (Bell/ Tomcat) DP IR-4 PR-10621.11-GA*03	35-36	360-365	141	0 4	0.056, 0.044 0.021, 0.017	0.05 0.019	ND, ND ND, ND	ND ND	< 0.01 (2) 0.012, 0.01	< 0.01 0.011
USA, 2011 Weslaco, TX (Bell/ Snapper F1) DP IR-4 PR-10621.11-TX16	35-36	265	141	0 5	0.043, 0.053 0.038, 0.036	0.048 0.037	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND
Greenhouse										
USA, 2011 Parlier, CA (Bell/ Derby) DP IR-4 PR-10621.11-CA76	35-36	380-390	142	0 4	0.14, 0.1 0.064, 0.051	0.12 0.057	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND
USA, 2011 Salisbury, MD (Bell/ Alliance) DP IR-4 PR-10621.11-MD17	34-36	420-450	138	0 4	0.027, 0.027 0.07, 0.052	0.027 0.061	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND

[a] Residues of metabolites IN-Q7H09, IN-RDG40, IN-RZB20, IN-RZD74 and IN-SXS67 were ND in all samples

In the supervised trials on field peppers conducted in North America in 2011, separate plots were treated with oxathiapiprolin (SC or OD formulations) as soil drenches or through drip irrigation systems, with two pre-harvest applications being made 6–8 days apart. Dilute concentrations of oxathiapiprolin were either poured around the base of the plants or injected into the drip irrigations systems and followed by irrigation (0.25–0.5 acre-inch) with water.

Samples (minimum of 12 whole fruit or 2 kg) were frozen within 3 hours and stored for up to 31 weeks before analysis for oxathiapiprolin and its metabolites (IN-WR791, IN-RDG40, IN-E8S72, IN-RZB20, IN-RZD74, IN-SXS67 and IN-Q7H09) using method DP30422 Supplement 1 (LC-MS/MS). Mean recovery rates for each analyte ranged from 90% to 108% in samples spiked with 0.01–0.2 mg/kg. The LOQs were 0.01 mg/kg for all analytes.

Table 75 Residues in field peppers (bell and non-bell) from supervised trials in North America, involving two pre-harvest soil drench/irrigation treatments oxathiapiprolin, applied 6–8 days apart

PEPPERS Country, year Location (Variety) Trial Ref	Application				DAT	Residues (mg/kg) [a]					
						Oxathiapiprolin		IN-E8S72		IN-WR791	
	Form	Method	g ai/ha	season total (g ai/ha)		values	mean	values	mean	values	mean
GAP: USA			35-280	560	PHI: 0d	Max 4 applications per season. Min 7 day RTI					
Canada, 2011 Delhi, ON (Non-Bell/ El Jefe) DP IR-4 PR-10621.11-ON24	SC	drip	284	568	0 5	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND
Canada, 2011 Harrow, ON (Bell/ Revolution) DP IR-4 PR-10621.11-ON23	OD	drench	385-280	665	0 5 10 15 27	ND, ND ND, ND ND, ND ND, < 0.01 ND, ND	ND ND ND < 0.01 ND	ND, ND ND, ND ND, ND ND, ND ND, ND	ND ND ND ND ND	ND, ND ND, ND ND, ND < 0.01, < 0.01 0.01, < 0.01	ND ND ND < 0.01 0.01
Canada, 2011 L'Acadie, QC (Bell/ Red Knight) DP IR-4 PR-10621.11-QC09	SC	drip	279	558	0 6	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND
Canada, 2011 L'Acadie, QC (Non-Bell/ Carmen) DP IR-4 PR-10621.11-QC10	SC	drip	279	558	0 5	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND
USA, 2011 Charleston, SC (Non-Bell/ Sw banana) DP IR-4 PR-10621.11-SC*04	SC	drench	273	546	0 4 9 13 28	ND, ND ND, ND ND, ND ND, ND ND, ND	ND ND ND ND ND	ND, ND < 0.01 (2) < 0.01 (2) < 0.01 (2) < 0.01 (2)	ND < 0.01 < 0.01 < 0.01 0.01	< 0.01, 0.01 < 0.01, 0.012 0.013, 0.011 0.016, 0.019 0.022,0.026	< 0.01 0.01 0.012 0.017 0.024
USA, 2011 Citra, FL (Bell/ Aristotle) DP IR-4 PR-10621.11-FL18	OD	drench	287	574	0 5	0.015, 0.019 < 0.01, < 0.01	0.017 < 0.01	ND, ND ND, ND	ND ND	ND, ND < 0.01, < 0.01	ND < 0.01
USA, 2011 Citra, FL (Non-Bell/ Compadre) DP IR-4 PR-10621.11-FL19	OD	drench	282	563	0 5	ND, < 0.01 ND, ND	ND ND	ND, ND ND, ND	ND ND	ND, ND 0.015, 0.017	ND 0.016
USA, 2011 Freemont, OH (Bell/ Tomcat) DP IR-4 PR-10621.11-OH*04	OD	drench	282	564	0 5	ND, < 0.01 ND, ND	< 0.01 ND	ND, ND ND, ND	ND ND	ND, ND ND, < 0.01	ND ND
USA, 2011 Holtville, CA (Bell/ Encore) DP IR-4 PR-10621.11-CA77	OD	drip	280	560	0 5	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND
USA, 2011 Las Cruces, NM (Non-Bell/ Big Jim) DP IR-4 PR-10621.11-NM09	OD	drip	280	560	0 6	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND
USA, 2011 Las Cruces, NM (Non-Bell/ Joe Parker) DP IR-4 PR-10621.11-NM10 [not independent]	OD	drip	280	560	0 5	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND
USA, 2011 Madison, WI (Bell/ Tomcat) DP IR-4 PR-10621.11-WI12	SC	drench	280	560	0 5	< 0.01, ND ND, < 0.01	ND < 0.01	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND

PEPPERS Country, year Location (Variety) Trial Ref	Application				DAT	Residues (mg/kg) [a]					
						Oxathiapiprolin		IN-E8S72		IN-WR791	
	Form	Method	g ai/ha	season total (g ai/ha)		values	mean	values	mean	values	mean
USA, 2011 Raleigh, GA (Bell/ Intruder) DP IR-4 PR-10621.11-NC17	SC	drench	283	566	0 4	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND	< 0.01, < 0.01 0.013, 0.013	< 0.01 0.013
USA, 2011 Riverside, CA (Bell/ Carlwander) DP IR-4 PR-10621.11-CA75	SC	drip	280	560	0 5	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND
USA, 2011 Tifton, GA (Bell/ Tomcat) DP IR-4 PR-10621.11-GA*03	SC	drench	280	560	0 4	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND	ND, ND < 0.01, < 0.01	ND < 0.01
USA, 2011 Weslaco, TX (Bell/ Snapper F1) DP IR-4 PR-10621.11-TX16	SC	drip	281	562	0 5	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND

[a] Residues of metabolites IN-Q7H09, IN-RDG40, IN-RZB20, IN-RZD74 and IN-SXS67 were ND in all samples

In supervised trials on protected tomatoes, conducted in Europe in 2011 and 2012, three foliar applications of oxathiapiprolin (OD formulations) without added adjuvant were applied at 6–8 day intervals intervals using motorised or pressurised backpack sprayers with single-nozzle hand lances.

Samples (minimum of 2 kg) were frozen within 9 hours and stored for up to 28 weeks before analysis for oxathiapiprolin and its metabolites using method DuPont-30422 (LC-MS/MS). In the 2011 trials, metabolites IN-WR791, IN-RDG40, IN-E8S72 and IN-Q7H09 were measured, while in the 2012 trials, the metabolites IN-Q7H09 and IN-RDG40 were not investigated. Mean recovery rates for each analyte ranged from 96% to 101% in samples spiked with 0.01–1.0 mg/kg. The LOQs were 0.01 mg/kg for all analytes.

Table 76 Residues in protected tomatoes from supervised trials in Europe involving 3 foliar applications of oxathiapiprolin (OD formulations), applied at 6–8 day intervals.

TOMATOES Country, year Location (Variety) Trial Ref	Application				DAT	Residues (mg/kg)
	g ai/ha	water (L/ha)	g ai/hL	season total (g ai/ha)		Oxathiapiprolin
Belgium, 2011 Antwerpen (Adniro) DP-31737-Test 01	30-31	1015-1030	3	91.7	-0 0 1 3 7 10	0.023 0.016 0.019 0.019 < 0.01 0.013
Belgium, 2012 Antwerpen (SG 309522) Cherry tomato DP-31737-Test 08	30-31	1000-1025	3	91.47	3	0.081
France, 2011 Provence-Alpes-Cote d'Azur (Idoia) Cherry tomato DP-31737-Test 04	30	980-1010	3	89.15	3	0.083

TOMATOES Country, year Location (Variety) Trial Ref	Application				DAT	Residues (mg/kg)
	g ai/ha	water (L/ha)	g ai/hL	season total (g ai/ha)		Oxathiapiprolin
France, 2012 Rhône-Alpes (Felicia) DP-31737-Test 07	30-31	1000-1015	3	90.85	-0 0 1 3 7 10	0.032 0.047 0.07 0.045 0.048 0.047
Greece, 2011 Thessaloniki (Primadona) DP-31737-Test 05	30	1000	3	89.97	3	0.017
Greece, 2012 Thessaly (Belladonna) DP-31737-Test 09	30-31	1000-1030	3	90.63	3	0.01
Italy, 2011 Sicily (Genio) Cherry tomato DP-31737-Test 03	29-30	980-1000	3	88.84	-0 0 1 3 7 10	0.032 0.066 0.047 0.062 0.073 0.041
Spain, 2011 Andalucia (Betina) Cherry tomato DP-31737-Test 02	30	990-1000	3	90.06	-0 0 1 3 7 10	0.041 0.1 0.055 0.064 0.056 0.031

[a] Residues of metabolites IN-Q7H09, IN-RDG40, IN-E8S72 and IN-WR791 were ND in all samples

In supervised field trials on tomatoes, conducted in Europe in 2011 and 2012, three foliar applications of oxathiapiprolin (OD formulations) without added adjuvant were applied at 6–9 day intervals intervals using motorised or pressurised backpack sprayers with single-nozzle hand lances.

Samples (minimum of 2 kg) were frozen within 5 hours and stored for up to 32 weeks before analysis for oxathiapiprolin and its metabolites using either method DuPont-30422 or DuPont-30422 Supplement 1 (LC-MS/MS). In the 2011 trials, metabolites IN-WR791, IN-RDG40, IN-E8S72 and IN-Q7H09 were measured, while in the 2012 trials, the metabolites IN-Q7H09 and IN-RDG40 were not investigated. Mean recovery rates for each analyte ranged from 71% to 112% in samples spiked with 0.01–1.0 mg/kg. The LOQs were 0.01 mg/kg for all analytes.

Table 77 Residues in field tomatoes from supervised trials in Europe involving 3 foliar applications of oxathiapiprolin (OD formulations), applied at 6–8 day intervals.

TOMATOES Country, year Location (Variety) Trial Ref	Application				DAT	Residues (mg/kg) [a]
	g ai/ha	water (L/ha)	g ai/hL	season total (g ai/ha)		Oxathiapiprolin
France, 2011 Rhone-Alpes (Perfect peel) DP-31736-Test 1	29-31	970-1020	3	90.06	-0 0	< 0.01 0.018
	30-31	100-1025	3	91.08	1	0.025
	30-31	1010-1025	3	91.49	3	0.024
	30	995-1010	3	90.68	7	< 0.01
	30	990-1010	3	90.37	10	< 0.01
Spain, 2011 Andalucía (Boca)	30	1000	3	90.57	-0 0	< 0.01 0.032
	30	1000	3	90.47	1	0.017

TOMATOES Country, year Location (Variety) Trial Ref	Application				DAT	Residues (mg/kg) [a]
	g ai/ha	water (L/ha)	g ai/hL	season total (g ai/ha)		Oxathiapiprolin
DP-31736-Test 2	30	1000-1010	3	90.67	3	0.012
	30	1000	3	90.47	7	0.016
	30	1000-1010	3	90.78	10	0.012
Greece, 2011 Central Macedonia (Clodin) DP-31736-Test 3	30	1000-1020	3	90.6	-0 0	< 0.01 < 0.01
	30	1000	3	90.4	1	< 0.01
	30	1000	3	90.56	3	0.017
	30	1000-1010	3	90.42	8	0.021
	30	1000-1010	3	90.35	11	0.013
Spain, 2011 Andalucía (Albatros) DP-31736-Test 4	30	1010	3	90.79	3	0.02
Italy, 2011 Catania (Missouri) DP-31736-Test 5	30	995-1000	3	89.57	3	0.036
Italy, 2013 Palagonia, Sicily (Missouri) DP-36222-Test 2	30	995-1000	3	90.17	-0 0	0.031 0.056
	30	990-1010	3	89.95	1	0.079
	30	1000-1010	3	90.39	3	0.068
	30	985-1010	3	89.73	7	0.064
	30	995-1000	3	89.45	11	0.06
Spain, 2013 Andalucia (Bocca) DP-36222-Test 3	30	995-1000	3	90.04	3	< 0.01
Greece, 2013 Central Macedonia (Unspecified) DP-36222-Test 4	30	1000	3	89.96	3	0.015

[a] Residues of metabolites IN-Q7H09, IN-RDG40, IN-E8S72 and IN-WR791 were ND in all samples

In supervised trials on field and greenhouse tomatoes conducted in North America in 2011, four foliar applications of oxathiapiprolin (OD formulations) with added adjuvant were applied at 4–6 day intervals intervals using tractor-mounted or pressurised backpack sprayers with 3–8 nozzle booms.

Duplicate samples (minimum of 2 kg) were frozen within 4 hours and stored for up to 36 weeks before analysis for oxathiapiprolin and its metabolites (IN-WR791, IN-RDG40, IN-E8S72, IN-RZB20, IN-RZD74, IN-SXS67 and IN-Q7H09) using method DP30422 Supplement 1 (LC-MS/MS). Mean recovery rates for each analyte ranged from 101% to 113% in samples spiked with 0.01–2.4 mg/kg. The LOQs were 0.01 mg/kg for all analytes.

Table 78 Residues in tomatoes from supervised field and greenhouse trials in North America, involving four foliar applications of oxathiapiprolin (OD formulations) with added adjuvant, applied at 2–4 day intervals

TOMATO Country, year Location (Variety) Trial Ref	Application				DAT	Residues (mg/kg) [a]			
						Oxathiapiprolin		IN-WR791	
	g ai/ha	water (L/ha)	g ai/hL	season total (g ai/ha)		values	mean	values	mean
GAP: USA	15-35			140	PHI: 0d	Max 4 sprays/season. 5-14 day RTI			

TOMATO Country, year Location (Variety) Trial Ref	Application				DAT	Residues (mg/kg) [a]			
						Oxathiapiprolin		IN-WR791	
	g ai/ha	water (L/ha)	g ai/hL	season total (g ai/ha)		values	mean	values	mean
Canada, 2011 Branchton, ON (Biltmore) (standard) DP-32064-Trial 09	35-36	250	14-14.5	141	0 5	0.034, 0.03 0.017, 0.015	0.032 0.016	ND, < 0.01 < 0.01, < 0.01	< 0.01 < 0.01
Canada, 2011 Branchton, ON (Sweet Million) (small) DP-32064-Trial 08	32-38	250	13-15	144	0 5	0.09, 0.066 0.078, 0.067	0.078 0.073	ND, ND ND, ND	ND ND
Canada, 2011 Burford, ON (Biltmore) (standard) DP-32064-Trial 07	34-37	250	13.5-14.5	140	0 5	0.049, 0.046 0.044, 0.036	0.048 0.04	ND, ND ND, ND	ND ND
Canada, 2011 Burford, ON (Sweet Million) (small) DP-32064-Trial 10	35-36	250	14-14.5	143	0 5	0.14, 0.14 0.15, 0.14	0.14 0.14	ND, ND ND, ND	ND ND
Canada, 2011 Portage la Prairie, MB (Early Girl) (standard) DP-32064-Trial 11	34-36	250	13.5-14	139	0 5	ND, ND < 0.01, < 0.01	ND < 0.01	ND, ND ND, ND	ND ND
Canada, 2011 St. Marc-sur-Richelieu, QC (Florida 47) (standard) DP-32064-Trial 05	35-36	250	14-14.5	143	0 5	0.025, 0.022 0.018, 0.021	0.023 0.02	ND, ND ND, ND	ND ND
Canada, 2011 St. Marc-sur-Richelieu, QC (Primo Red) (standard) DP-32064-Trial 06	35-36	250	14-14.5	141	0 5	0.028, 0.035 0.025, 0.029	0.032 0.027	ND, ND ND, ND	ND ND
USA, 2011 Alton, NY (Polbig F1) (standard) DP-32064-Trial 01	35-36	280-285	12.5	141	0 5	0.026, 0.022 < 0.01, 0.012	0.024 0.011	ND, ND < 0.01, ND	ND ND
USA, 2011 Athens, GA (Roma) (small) DP-32064-Trial 02	35-36	225-350	10-16	140	0 5	0.018, 0.025 0.023, 0.017	0.022 0.02	ND, ND ND, ND	ND ND
USA, 2011 Chico, CA (Sun 3633 Roma) (small) DP-32064-Trial 18	35-36	187	19	142	0 5	0.27, 0.35 0.19, 0.14	0.31 0.16 [b]	ND, ND ND, ND	ND ND
USA, 2011 Kerman, CA (Early Girl) (standard) DP-32064-Trial 24	35	280	12.6	140	0 5 10 15 30	0.06, 0.09 0.058, 0.056 0.035, 0.05 0.035, 0.036 0.031, 0.033	0.075 0.057 0.043 0.036 0.032	ND, ND ND, ND ND, ND ND, ND ND, ND	ND ND ND ND ND

TOMATO Country, year Location (Variety) Trial Ref	Application				DAT	Residues (mg/kg) [a]			
						Oxathiapiprolin		IN-WR791	
	g ai/ha	water (L/ha)	g ai/hL	season total (g ai/ha)		values	mean	values	mean
USA, 2011 King City, CA (AB2) (standard) DP-32064-Trial 14	35	280-300	11.5-12	139	0 5	0.045, 0.039 0.03, 0.041	<u>0.042</u> 0.036	ND, ND ND, ND	ND ND
USA, 2011 Live Oak, FL (Florida 47) (standard) DP-32064-Trial 04	35	400-415	8.5	139	0 5	0.039, 0.029 0.03, 0.025	<u>0.034</u> 0.027	< 0.01, < 0.01 < 0.01, < 0.01	< 0.01 < 0.01
USA, 2011 Madera, CA (Quality 27) (small) DP-32064-Trial 13	35	280-285	12.3	139	0 5	0.029, 0.034 0.029, 0.031	<u>0.032</u> 0.03	ND, ND ND, ND	ND ND
USA, 2011 Marysville, OH (Husky Cherry Red) (small, cherry) DP-32064-Trial 23	35	200	17.5	140	0 5 10 15 30	0.044, 0.05 0.037, 0.035 0.022, 0.021 0.016, 0.019 < 0.01, < 0.01	<u>0.047</u> 0.036 0.022 0.018 < 0.01	ND, ND ND, ND ND, ND ND, ND ND, ND	ND ND ND ND ND
USA, 2011 Oviedo, FL (Better Boy) (standard) DP-32064-Trial 03	34	280	10-12	136	0 5	0.033, 0.045 0.019, 0.015	<u>0.039</u> 0.017	ND, ND < 0.01, ND	ND ND
USA, 2011 Paso Robles, CA (Galilea Roma) (small) DP-32064-Trial 17	34-35	374	9.2-9.3	138	0 5	0.034, 0.036 0.031, 0.025	<u>0.035</u> 0.028	ND, ND ND, ND	ND ND
USA, 2011 Porterville, CA (Roma) (small) DP-32064-Trial 16	35	260-310	11.5-13.5	141	0 5	0.1, 0.1 0.089, 0.059	<u>0.1</u> 0.074	ND, ND ND, ND	ND ND
USA, 2011 Visalia, CA (Cherry) (small) DP-32064-Trial 15	35-36	265-320	11-13.5	141	0 5	0.13, 0.088 0.15, 0.089	0.11 <u>0.12</u>	ND, ND ND, ND	ND ND
Greenhouse									
USA, 2011 Detroit Lakes, MN (Dasher) (small, grape) DP-32064-Trial 22-G	36-37	470	7.7-7.9	147	0 5 10 15 30	0.033, 0.037 0.054, 0.073 0.081, 0.077 0.037, 0.053 0.025, 0.02	0.035 0.063 <u>0.079</u> 0.045 0.023	ND, ND ND, ND ND, ND ND, ND ND, ND	ND ND ND ND ND
USA, 2011 Detroit Lakes, MN (Geronimo) (large) DP-32064-Trial 21- G	34-36	470	7.4-7.8	143	0 5	< 0.01, ND 0.011, < 0.01	ND <u>< 0.01</u>	ND, ND ND, ND	ND ND
USA, 2011 North Rose, NY (Early Girl) (large) DP-32064-Trial 19-G	35	450	7.8	140	0 5	< 0.01, 0.01 < 0.01, 0.013	< 0.01 <u>< 0.01</u>	ND, ND ND, ND	ND ND

TOMATO Country, year Location (Variety) Trial Ref	Application				DAT	Residues (mg/kg) [a]			
						Oxathiapiprolin		IN-WR791	
	g ai/ha	water (L/ha)	g ai/hL	season total (g ai/ha)		values	mean	values	mean
USA, 2011 North Rose, NY (Supersweet 100VF) (small) DP-32064-Trial 20- G	35	450	7.8	140	0 5	0.021, 0.016 0.031, 0.031	0.019 0.031	ND, ND ND, ND	ND ND

[a] Residues of metabolites IN-E8S72, IN-Q7H09, IN-RDG40, IN-RZB20, IN-RZD74 and IN-SXS67 were ND in all samples

[b] Reported residues in Trial 18 significantly higher than other trial results but no identified reason for this inconsistency

In the supervised trials on field tomatoes conducted in North America in 2011, separate plots were treated with oxathiapiprolin (SC or OD formulations) as soil drenches or through drip irrigation systems, with two pre-harvest or near-planting applications being made 6–8 days apart. In three additional trials,soil drench or drip irrigation treatments were made at different growth stages, ranging from planting through to pre-harvest. Dilute concentrations of oxathiapiprolin were either applied around the base of the plants or injected into the drip irrigations systems and in most trials, followed by irrigation (0.25–0.5 acre-inch) with water.

Samples (minimum of 2 kg) were frozen within 4 hours and stored for up to 36 weeks before analysis for oxathiapiprolin and its metabolites (IN-WR791, IN-RDG40, IN-E8S72, IN-RZB20, IN-RZD74, IN-SXS67 and IN-Q7H09) using method DP30422 Supplement 1 (LC-MS/MS). Mean recovery rates for each analyte ranged from 101% to 113% in samples spiked with 0.01–2.5 mg/kg. The LOQs were 0.01 mg/kg for all analytes.

Table 79 Residues in field tomatoes from supervised trials in North America, involving two soil drench/irrigation treatments oxathiapiprolin (SC formulations), applied 6–8 days apart

TOMATO Country, year Location (Variety) Trial Ref	Application			DAT	Residues (mg/kg) [a]							
					Oxathiapiprolin		IN-E8S72		IN-SXS67		IN-WR791	
	method	g ai/ha	season total g ai/ha		values	mean	values	mean	values	mean	values	mean
GAP: USA		35-280	560	PHI: 0d	Max 4 applications per season. Min 7 day RTI							
USA, 2011 Alton, NY (Polbig F1) (standard) DP-32064-T-01	drench-T2	280	560	0 5	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND
	drench-T3	280	560	97	ND, ND	ND	< 0.01, ND	ND	ND, ND	ND	ND, ND	ND
USA, 2011 Athens, GA (Roma) (small) DP-32064-T 02	drench-T2	276-278	554	0 5	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND	ND, ND < 0.01 (2)	ND < 0.01
USA, 2011 Oviedo, FL (Better Boy) (standard) DP-32064-T-03	drench-T2	283	566	0 5	ND, ND < 0.01 (2)	ND < 0.01	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND	ND, ND < 0.01 (2)	ND < 0.01
USA, 2011 Live Oak, FL (Florida 47) (standard) DP-32064-T 04	drench-T2	281	561	0 5	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND
Canada, 2011 St. Marc-sur-Richelieu, QC (Florida 47) (standard) DP-32064-T-05	drench-T2	280	560	0 5	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND
	drench-T3	280	560	86	ND, ND	ND	ND, ND	ND	ND, ND	ND	< 0.01, ND	ND

TOMATO Country, year Location (Variety) Trial Ref	Application			DAT	Residues (mg/kg)[a]							
					Oxathiapiprolin		IN-E8S72		IN-SXS67		IN-WR791	
	method	g ai/ha	season total g ai/ha		values	mean	values	mean	values	mean	values	mean
Canada, 2011 St. Marc-sur-Richelieu, QC (Primo Red) (standard) DP-32064-T-06	drench-T2	280	560	0 5	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND
Canada, 2011 Burford, ON (Biltmore) (standard) DP-32064-T-07	drip-T2	280	560	0 5	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND
Canada, 2011 Branchton, ON (Sweet Million) (small) DP-32064-T-08	drip-T2	283	566	0 5	ND, < 0.01 ND, ND	< 0.01 ND	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND
Canada, 2011 Branchton, ON (Biltmore) (standard) DP-32064-T-09	drip-T2	283	566	0 5	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND
Canada, 2011 Burford, ON (Sweet Million) (small) DP-32064-T-10	drip-T2	280	560	0 5	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND
Canada, 2011 Portage la Prairie, MB (Early Girl) (standard) DP-32064-T-11	drench-T2	280	560	0 5	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND
USA, 2011 Madera, CA (Quality 27) (small) DP-32064-T-13	drip-T2	280	560	0 5	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND
	drip-T3	280	560	85	ND, ND	ND	< 0.01, 0.012	0.011	< 0.01 (2)	< 0.01	0.015, 0.018	0.016
USA, 2011 King City, CA (AB2) (standard) DP-32064-T-14	drench-T2	280	560	0 5	0.014, < 0.01 < 0.01 (2)	< 0.01 < 0.01	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND
USA, 2011 Visalia, CA (Cherry) (small) DP-32064-T-15	drip-T2	280	560	0 5	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND
USA, 2011 Porterville, CA (Roma) (small) DP-32064-T-16	drip-T2	280	560	0 5	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND
	drip-T3	280	560	114	ND, ND	ND	ND, ND	ND	ND, ND	ND	ND, ND	ND
USA, 2011 Paso Robles, CA (Galilea Roma) (small) DP-32064-T-17	drip-T2	280	560	0 5	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND
USA, 2011 Chico, CA (Sun 3633 Roma) (small) DP-32064-T-18	drench-T2	280	560	0 5	0.44, 0.04 0.19, 0.14	0.24 0.16[b]	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND
	drench-T3	280	560	108	0.14, 0.032	0.086	ND, ND	ND	ND, ND	ND	ND, ND	ND

TOMATO Country, year Location (Variety) Trial Ref	Application			DAT	Residues (mg/kg)[a]							
					Oxathiapiprolin		IN-E8S72		IN-SXS67		IN-WR791	
	method	g ai/ha	season total g ai/ha		values	mean	values	mean	values	mean	values	mean
USA, 2011 Marysville, OH (Husky Cherry Red) (small, cherry) DP-32064-T-23	drench-T2	280	560	0 5 10 15 30	0.02, 0.037 0.011, 0.019 0.011,< 0.01 < 0.01, ND < 0.01, ND	0.028 0.015 < 0.01 < 0.01 ND	ND, ND ND, ND ND, ND ND, ND ND, ND	ND ND ND ND ND	ND, ND ND, ND ND, ND ND, ND ND, ND	ND ND ND ND ND	ND, ND ND, ND ND, ND ND, ND ND, ND	ND ND ND ND ND
USA, 2011 Kerman, CA (Early Girl) (standard) DP-32064-T-24	drench-T2	305	610	0 5 10 15 30	ND, ND ND, ND ND, ND ND, ND ND, ND	ND ND ND ND ND	ND, ND ND, ND ND, ND ND, ND ND, ND	ND ND ND ND ND	ND, ND ND, ND ND, ND ND, ND ND, ND	ND ND ND ND ND	ND, ND ND, ND ND, ND ND, ND ND, ND	ND ND ND ND ND
USA, 2011 Germansville, PA (Roma) DP-33132-T-01	drench-T2	280	560	0 7 13	ND, ND ND, ND ND, ND	ND ND ND	ND, ND ND, ND ND, ND	ND ND ND	ND, ND ND, ND ND, ND	ND ND ND	ND, ND ND, ND ND, ND	ND ND ND
	drench-T3	280	560	77	ND, ND	ND	< 0.01 (2)	< 0.01	ND, ND	ND	0.011, 0.01	0.01
	drench-T4	280	560	56 63 69	ND, ND ND, ND ND, ND	ND ND ND	ND, < 0.01 < 0.01 (2) < 0.01 (2)	ND < 0.01 < 0.01	ND, ND ND, ND ND, ND	ND ND ND	< 0.01,0.015 < 0.01, 0.01 < 0.01 (2)	0.012 < 0.01 < 0.01
	drench-T5	280	560	34 41 47	ND, ND ND, ND ND, ND	ND ND ND	ND, ND ND, ND ND, ND	ND ND ND	ND, ND ND, ND ND, ND	ND ND ND	ND, ND ND, ND ND, ND	ND ND ND
	drench-T6	280	560	14 21 27	ND, ND ND, ND ND, ND	ND ND ND	ND, ND ND, ND ND, ND	ND ND ND	ND, ND ND, ND ND, ND	ND ND ND	ND, ND ND, ND ND, ND	ND ND ND
Canada, 2011 Branchton, ON (HP30) DP-33132-T-02	drip-T2	283	566	0 7 14	ND, ND ND, ND ND, ND	ND ND ND	ND, ND ND, ND ND, ND	ND ND ND	ND, ND ND, ND ND, ND	ND ND ND	ND, ND ND, ND ND, ND	ND ND ND
	drip-T3	283	566	83	ND, ND	ND	ND, ND	ND	ND, ND	ND	< 0.01, ND	ND
	drip-T4	283	566	62 69 76	ND, ND ND, ND ND, ND	ND ND ND	ND, ND ND, ND ND, ND	ND ND ND	ND, ND ND, ND ND, ND	ND ND ND	ND, ND ND, ND ND, ND	ND ND ND
	drip-T5	283	566	41 48 55	ND, ND ND, ND ND, ND	ND ND ND	ND, ND ND, ND ND, ND	ND ND ND	ND, ND ND, ND ND, ND	ND ND ND	ND, ND ND, ND ND, ND	ND ND ND
	drip-T6	283	566	20 27 34	ND, ND ND, ND < 0.01, ND	ND ND ND	ND, ND ND, ND ND, ND	ND ND ND	ND, ND ND, ND ND, ND	ND ND ND	ND, ND ND, ND ND, ND	ND ND ND
USA, 2011 Porterville, CA (Roma) DP-33132-T-03	drench-T2	560	560	0 7 14	0.038, 0.07 0.046, 0.04 0.018, 0.074	0.054 0.043 0.046	< 0.01,0.012 < 0.01, ND < 0.01 (2)	< 0.01 ND < 0.01	ND, ND ND, ND ND, ND	ND ND ND	ND, ND ND, ND ND, ND	ND ND ND
	drench-T7	280	560	35 42 49	0.033, 0.016 0.041, 0.023 0.042, 0.028	0.025 0.032 0.035	ND, ND ND, < 0.01 ND, ND	ND ND ND	ND, ND ND, ND ND, ND	ND ND ND	< 0.01 (2) < 0.01 (2) < 0.01 (2)	< 0.01 < 0.01 < 0.01
USA, 2011 Porterville, CA (Roma) DP-33132-T-03 1 application	drench-T3	280	280	112	ND, ND	ND	ND, < 0.01	ND	ND, ND	ND	ND, ND	ND
	drench-T4	280	280	89 96 103	ND, ND ND, ND ND, ND	ND ND ND	ND, ND < 0.01, ND < 0.01, ND	ND ND ND	ND, ND ND, ND ND, ND	ND ND ND	ND, ND ND, ND ND, ND	ND ND ND
	drench-T5	280	280	69 76 83	ND, ND ND, ND ND, ND	ND ND ND	ND, ND ND, ND ND, ND	ND ND ND	ND, ND ND, ND ND, ND	ND ND ND	ND, ND ND, ND ND, ND	ND ND ND
	drench-T6	280	280	49 56 63	< 0.01, 0.027 ND, < 0.01 ND, ND	0.018 < 0.01 ND	ND, ND ND, ND ND, ND	ND ND ND	ND, ND ND, ND ND, ND	ND ND ND	ND, ND < 0.01, ND ND, ND	ND ND ND
USA, 2012 Porterville, CA (Roma) DP-33132-T-03	drench-T3	280	560	84	0.035, 0.044	0.04	ND, ND	ND	< 0.01 (2)	< 0.01	ND, ND	ND
	drench-T4	280	560	63 70	ND, ND ND, ND	ND ND	0.031, 0.028 0.025, 0.026	0.03 0.026	0.012, 0.013 0.021, 0.021	0.013 0.021	0.013, 0.014 0.012, 0.01	0.014 0.011
	drench-T5	280	560	42 49	< 0.01, ND ND, ND	< 0.01 ND	0.022, 0.025 0.026, 0.011	0.023 0.018	0.013, 0.012 0.017, 0.011	0.012 0.014	< 0.01 (2) < 0.01 (2)	< 0.01 < 0.01
	drench-T6	280	560	21 28	0.021, 0.027 0.02, ND	0.024 0.011	0.01, 0.01 < 0.01 (2)	0.01 < 0.01	< 0.01 (2) 0.012, < 0.01	< 0.01 < 0.01	< 0.01 (2) < 0.01, ND	< 0.01 < 0.01

[a] Residues of metabolites IN-RDG40, IN-Q7H09, IN-RZB20, and IN-RZD74 were <LOD in any sample

[b] Reported residues in Trial 18 significantly higher than other trial results. No identified reason for this inconsistency

T2 Last application at harvest

T3 First applications at planting

T4 First application 21 days after planting

T5 First application 42 days after planting
T6 First application 63 days after planting
T7 First application 84 days after planting

Leafy vegetables

Lettuce

In supervised field trials on leaf (open head) lettuce, conducted in Europe in 2011 and 2012, two foliar applications of oxathiapiprolin (OD or SE formulations) without added adjuvant were applied at 6–8 day intervals intervals using motorised or pressurised backpack sprayers with 3–10 nozzle booms.

Samples (minimum of 2 kg of leaves from at least 12 plants) were were frozen within 7 hours and stored for up to 40 weeks before analysis for oxathiapiprolin and its metabolites IN-WR791, IN-RDG40, IN-E8S72 and IN-Q7H09 using method DP30422 (LC-MS/MS). In the 2012 trials, residues of metabolites IN-RDG40 and and IN-Q7H09 were not measured. Mean recovery rates for each analyte ranged from 91% to 102% in samples spiked with 0.01–5.0 mg/kg. The LOQs were 0.01 mg/kg for all analytes.

Table 80 Residues in field leaf lettuce from supervised trials in Europe involving 2 foliar applications of oxathiapiprolin, applied at 6–8 day intervals.

LETTUCE Country, year Location (Variety) Trial Ref	Application					DAT	Residues (mg/kg) [a]		
	form	g ai/ha	water (L/ha)	g ai/hL	season total (g ai/ha)		Oxathiapiprolin	IN-RDG40	IN-E8S72
France, 2011 Le Maisnil (open-leaf/ Quelio) DP-31734 Test 01a	SE	14.23 15.3	948 1018	1.5 1.5	29.53	-0 0	0.051 0.41	ND ND	ND ND
	SE	14.66 14.98	982 998	1.49 1.5	29.64	1	0.24	ND	ND
	SE	15.62 14.77	1045 985	1.49 1.5	30.39	3	0.31	ND	ND
	SE	15.19 15.3	1017 1023	1.49 1.5	30.49	8	0.11	ND	ND
	SE	15.19 14.87	1014 990	1.5 1.5	30.06	10	0.13	ND	ND
France, 2011 Le Maisnil (open-leaf/ Quelio) DP-31734 Test 01b	SE	23.33 23.54	968 977	2.41 2.41	46.87	-0 0	0.11 0.81	ND ND	ND ND
	SE	24.29 24.5	1009 1018	2.41 2.41	48.79	1	0.57	ND	ND
	SE	23.11 24.18	962 1006	2.4 2.4	47.29	3	0.51	ND	ND
	SE	22.9 24.18	952 1003	2.41 2.41	47.08	8	0.29	ND	ND
	SE	24.61 24.29	1021 1010	2.41 2.4	48.9	10	0.18	ND	ND
France, 2011 Pys (open-leaf/ Maruschka[b]) DP-31734 Test 02a	SE	14.34 14.66	952 976	1.51 1.5	29	7	0.048	ND	ND
France, 2011 Pys (open-leaf/ Maruschka) DP-31734 Test 02b	SE	22.79 24.4	948 1014	2.4 2.41	47.19	7	0.069	ND	ND
France, 2011 Pys (open-leaf/ Maruschka) DP-31734 Test 02c	OD	15.1 15.3	1000 1014	1.51 1.51	30.4	7	0.063	ND	ND
Spain 2011 Benavent de Segria	OD	15.2 15.1	1009 1003	1.51 1.51	30.3	-0 0	0.01 0.29	ND ND	ND ND

LETTUCE Country, year Location (Variety) Trial Ref	Application					DAT	Residues (mg/kg) [a]		
	form	g ai/ha	water (L/ha)	g ai/hL	season total (g ai/ha)		Oxathiapiprolin	IN-RDG40	IN-E8S72
(open-leaf/ Fulla de Roure) DP-31734 Test 03a	OD	15.4 14.48	1024 965	1.5 1.5	29.88	1	0.24	ND	ND
	OD	14.99 15.2	997 1008	1.5 1.51	30.19	3	0.13	ND	ND
	OD	15.2 15.2	1009 1012	1.51 1.5	30.4	8	0.015	ND	ND
	OD	14.59 15.2	967 1008	1.51 1.51	29.79	10	< 0.01	ND	ND
Spain 2011 Benavent de Segria (open-leaf/ Fulla de Roure) DP-31734 Test 03b	OD	24.28 24.07	1013 1003	2.4 2.4	48.35	-0 0	0.017 0.37	ND ND	ND ND
	OD	24.48 24.07	1020 1006	2.4 2.39	48.55	1	0.36	ND	ND
	OD	23.77 24.28	991 1011	2.4 2.4	48.05	3	0.28	ND	ND
	OD	24.28 24.17	1013 1007	2.4 2.4	48.45	8	0.044	ND	ND
	OD	23.46 23.77	979 991	2.4 2.4	47.23	10	0.022	ND	ND
Spain, 2011 Zaidin (open-leaf/ Arenas) DP-31734 Test 04a	SE	14.77 14.98	989 1000	1.49 1.5	29.75	7	< 0.01	ND	ND
Spain, 2011 Zaidin (open-leaf/ Arenas) DP-31734 Test 04b	OD	15.4 15.1	1026 1002	1.5 1.51	30.5	7	0.011	ND	ND
Spain, 2011 Zaidin (open-leaf/ Arenas) DP-31734 Test 04c	OD	24.79 23.77	1032 990	2.4 2.4	48.56	7	0.024	ND	ND
Italy, 2011 Mediglia (open-leaf/ Lollo) DP-31734 Test 05a	OD	15.1 14.99	1004 997	1.5 1.5	30.09	-0 0	0.055 0.25	ND ND	ND ND
	OD	15.3 15.4	1019 1021	1.5 1.51	30.7	1	0.21	ND	ND
	OD	15.3 14.48	1014 963	1.51 1.5	29.78	3	0.17	ND	ND
	OD	15.3 15.1	1017 1002	1.5 1.51	30.4	8	0.05	ND	ND
	OD	14.79 15.4	981 1023	1.51 1.51	30.19	10	0.063	ND	ND
Italy, 2011 Mediglia (open-leaf/ Lollo) DP-31734 Test 05b	OD	23.56 24.17	983 1008	2.4 2.4	47.73	-0 0	0.063 0.47	ND ND	ND ND
	OD	24.38 25.09	1015 1046	2.4 2.4	49.47	1	0.31	ND	ND
	OD	23.66 23.15	989 967	2.39 2.39	46.81	3	0.22	ND	ND
	OD	24.07 23.66	1005 989	2.4 2.39	47.73	8	0.14	ND	ND
	OD	24.17 23.46	1010 980	2.39 2.39	47.63	10	0.096	ND	ND
Germany, 2011 Motterwitz (open-leaf/ Lollo Bionda) DP-31734 Test 06a	SE	16.39 15.87	1020 988	1.61 1.61	32.26	-0 0	0.033 0.85	ND ND	ND ND
	SE	16.51 16.77	1028 1044	1.61 1.61	33.28	1	0.59	ND	ND
	SE	15.61 15.61	972 972	1.61 1.61	31.22	3	0.52	ND	ND
	SE	15.74 16.77	980 1044	1.61 1.61	32.51	7	0.044	ND	ND

LETTUCE Country, year Location (Variety) Trial Ref	Application					DAT	Residues (mg/kg) [a]		
	form	g ai/ha	water (L/ha)	g ai/hL	season total (g ai/ha)		Oxathiapiprolin	IN-RDG40	IN-E8S72
	SE	16.77 15.36	1044 956	1.61 1.61	32.13	10	0.07	ND	ND
Germany, 2011 Motterwitz (open-leaf/ Lollo Bionda) DP-31734 Test 06b	SE	26.6 24.55	1036 956	2.57 2.57	51.15	-0 0	0.088 1.4	ND ND	ND ND
	SE	26.81 26.81	1044 1044	2.57 2.57	53.62	1	1.1	< 0.01	ND
	SE	26.6 24.76	1036 964	2.57 2.57	51.36	3	0.91	< 0.01	ND
	SE	25.78 26.81	1004 1044	2.57 2.57	52.59	7	0.092	ND	ND
	SE	26.19 26.91	1020 1048	2.57 2.57	53.1	10	0.21	ND	ND
Greece, 2011 Chalkidona (Field lettuce/ Simpson) DP-31734 Test 07a	OD	15.02 15.06	998 1001	1.5 1.5	30.08	-0 0	0.018 0.59	ND ND	ND ND
	OD	15.07 15.02	1001 999	1.5 1.5	30.09	1	0.31	ND	ND
	OD	15.29 15.07	1016 1001	1.5 1.5	30.36	3	0.32	ND	ND
	OD	15.02 15.08	999 1002	1.5 1.5	30.1	7	0.062	ND	ND
	OD	15 14.95	997 993	1.5 1.5	29.95	10	0.016	ND	ND
Greece, 2011 Chalkidona (open-leaf/ Simpson) DP-31734 Test 07b	OD	24.08 23.95	1005 999	2.4 2.4	48.03	-0 0	0.1 0.85	ND ND	ND ND
	OD	24.02 23.21	1002 997	2.4 2.4	47.23	1	0.49	ND	ND
	OD	24.27 23.92	1013 998	2.4 2.4	48.19	3	0.43	ND	ND
	OD	23.99 23.92	1001 998	2.4 2.4	47.91	7	0.085	ND	ND
	OD	23.99 24.18	1001 1009	2.4 2.4	48.17	10	0.025	ND	< 0.01
Germany, 2011 Straelen (open-leaf/ Eichblatt Rot Prunai) DP-31734 Test 08a	SE	15.08 15.28	1007 1020	1.5 1.5	30.36	-0 0	0.019 0.25	ND ND	ND ND
	SE	14.98 15.18	1000 1013	1.5 1.5	30.16	1	0.12	ND	ND
	SE	14.68 14.98	980 1000	1.5 1.5	29.66	3	0.061	ND	ND
	SE	14.88 15.18	993 1013	1.5 1.5	30.06	8	0.043	ND	ND
	SE	14.88 15.08	993 1007	1.5 1.5	29.96	10	0.027	ND	ND
Germany, 2011 Straelen (open-leaf/ Eichblatt Rot Prunai) DP-31734 Test 08b	SE	24.29 24.29	1013 1013	2.4 2.4	48.58	-0 0	0.048 0.41	ND ND	ND ND
	SE	24.45 24.45	1020 1020	2.4 2.4	48.9	1	0.27	ND	ND
	SE	24.29 24.29	1013 1013	2.4 2.4	48.58	3	0.23	ND	ND
	SE	23.97 24.13	1000 1007	2.4 2.4	48.1	8	0.13	ND	ND
	SE	23.97 24.45	1000 1020	2.4 2.4	48.42	10	0.11	ND	ND
Germany, 2011 Wachtendonk (Field lettuce/ Kitare) DP-31734 Test 09a	SE	15.18 15.28	1013 1020	1.5 1.5	30.46	7	0.022	ND	ND

LETTUCE Country, year Location (Variety) Trial Ref	Application					DAT	Residues (mg/kg) [a]		
	form	g ai/ha	water (L/ha)	g ai/hL	season total (g ai/ha)		Oxathiapiprolin	IN-RDG40	IN-E8S72
Germany, 2011 Wachtendonk (open-leaf/ Kitare) DP-31734 Test 09b	SE	24.48 24.16	1020 1007	2.4 2.4	48.64	7	0.04	ND	ND
Germany, 2011 Wachtendonk (open-leaf/ Kitare) DP-31734 Test 09c	OD	15.09 14.89	1007 933	1.5 1.5	29.98	7	0.015	ND	ND
Italy, 2011 Roncoferraro (open-leaf/ Ballerina) DP-31734 Test 10a	SE	14.66 15.73	978 1048	1.5 1.5	30.39	7	0.027	ND	ND
Italy, 2011 Roncoferraro (open-leaf/ Ballerina) DP-31734 Test 10b	OD	15.1 15.2	1004 1009	1.5 1.51	30.3	7	0.021	ND	ND
Italy, 2011 Roncoferraro (open-leaf/ Ballerina) DP-31734 Test 10c	OD	24.17 23.26	1008 971	2.4 2.4	47.43	7	0.072	ND	ND
Greece, 2012 Chalkidona (open-leaf/ 210) DP-31734 Test 11a	OD	14.35 15.62	955 1040	1.5 1.5	29.97	7	0.03		ND
Greece, 2012 Chalkidona (open-leaf/ 210) DP-31734 Test 11b	SE	15.3 14.87	1017 994	1.5 1.5	30.17	7	0.19		ND
France, 2012 Lucenay (open-leaf/ Feuille de Chene) DP-31734 Test 13	OD	15.53 15.63	1006 1010	1.54 1.55	31.16	-0 0	0.17 0.42		ND ND
	OD	15.63 15.42	1011 997	1.55 1.55	31.05	1	0.3		ND
	OD	15.0 15.32	972 989	1.54 1.55	30.32	3	0.33		ND
	OD	15.42 15.74	998 1015	1.55 1.55	31.16	7	0.18		ND
	OD	15.74 15.42	1015 995	1.55 1.55	31.16	10	0.12		ND
Spain, 2012 Vilanova de Segria (open-leaf/ Madrigon) DP-31734 Test 14	OD	15.11 15.32	977 991	1.55 1.55	30.43	7	0.014		ND
UK, 2012 Hollesley (Field lettuce/ Madrigon) DP-31734 Test 16	SE	15.05 15.15	1001 1009	1.5 1.5	30.2	-0 0	0.046 0.3		ND ND
	SE	15.24 15.34	1010 1019	1.51 1.51	30.58	1	0.26		ND
	SE	15.05 14.57	1000 965	1.51 1.51	29.62	3	0.082		ND
	SE	15.44 15.15	1028 1005	1.5 1.51	30.59	7	0.15		ND
	SE	16.02 15.54	1063 1031	1.51 1.51	31.56	10	0.12		ND
Germany, 2012 Straelen (Field lettuce/ Lollo Rosso) DP-31734 Test 17	SE	15.18 15.38	1013 1027	1.5 1.5	30.56	7	0.035		ND

[a] Residues of metabolites IN-WR791 and IN-Q7H09 were ND in all samples

[b] The variety reported in Test 02 also described as Scarole (open-leaf endive)

In supervised trials on field lettuce conducted in North America in 2011, four foliar applications of oxathiapiprolin (OD formulations) with added adjuvant were applied at 2–5 day intervals intervals using tractor-mounted, motorised or pressurised backpack sprayers with 2–8 nozzle booms.

Duplicate samples (minimum of 12 heads or plants) were trimmed to remove the roots and the heads or tops were halved or quartered, with sub-sampled specimens being frozen within 5 hours and stored for up to 21 weeks (27 weeks for leaf lettuce) before analysis for oxathiapiprolin and its metabolites (IN-WR791, IN-RDG40, IN-E8S72, IN-RZB20, IN-RZD74, IN-SXS67 and IN-Q7H09) using method DP30422 Supplement 1 (LC-MS/MS). Mean recovery rates for each analyte ranged from 91% to 110% in samples spiked with 0.01–0.2 mg/kg. The LOQs were 0.01 mg/kg for all analytes.

Table 81 Residues in field head lettuce from supervised trials in North America, involving four foliar applications of oxathiapiprolin (OD formulations), applied at 2–5 day intervals

HEAD LETTUCE Country, year Location (Variety) Trial Ref	Application			Matrix	DAT	Residues (mg/kg) [a]			
	g ai/ha	water (L/ha)	season total g ai/ha			Oxathiapiprolin		IN-WR791	
						values	mean	values	mean
GAP: USA	15-35		140		PHI: 0d	Max 4 sprays/season. 3-14 day RTI			
Canada, 2011 Agassiz, BC (Odyssey) PR-10653-BC11	35-37	450-460	144	head	0 3	1.3, 1.5 1.0, 0.96	1.4 0.98	ND, ND ND, ND	ND ND
Canada, 2011 Harrow, ON (Odyssey) PR-10653-ON28	35-36	405-415	143	head washed	0 3 0	0.49, 0.52 0.2, 0.18 0.13, 0.13	0.5 0.19 0.13	ND, ND ND, ND ND, ND	ND ND ND
Canada, 2011) Harrow, ON (Caliente) PR-10653-ON27 [not independent]	35-36	400-410	141	head washed	0 3 0	0.49, 0.27 0.18, 0.16 0.16, 0.12	0.38 0.17 0.14	ND, ND ND, ND ND, ND	ND ND ND
USA, 2011 Citra, FL (Unspecified) PR-10653-FL21	36	280-285	144	head washed, chopped	0 2 0	0.85, 0.56 0.13, 0.095 0.15, 0.42	0.7 0.11 0.28	ND, ND ND, ND ND, ND	ND ND ND
USA, 2011 Freeville, NY (Head Lettuce, Fall Green) PR-10653-NY11	35	345-390	140	head	0 2	0.53, 0.62 0.47, 0.62	0.57 0.54	ND, ND ND, ND	ND ND
USA, 2011 Holt, CA (Jupiter) PR-10653-CA80	35-37	220-230	142	head	0 3	0.37, 0.22 0.26, 0.21	0.3 0.24	ND, ND ND, ND	ND ND
USA, 2011 Las Cruces, NM (Salinas) PR-10653-NM12	34-36	220-280	141	head washed	0 2 0	0.2, 0.25 0.057, 0.13 < 0.01, < 0.01	0.23 0.096 < 0.01	ND, ND ND, ND ND, ND	ND ND ND
USA, 2011 Riverside, CA (Fall green) PR-10653-CA78	35-36	280-290	144	head	0 4	0.61, 1.0 0.69, 0.65	0.82 0.67	ND, ND ND, ND	ND ND
USA, 2011 Salinas, CA (corona) PR-10653-CA*83	35-36	445-470	141	head	0 3 6 15 27	0.88, 0.78 0.55, 0.46 0.4, 0.15 0.31, 0.16 0.15, 0.12	0.83 0.5 0.28 0.24 0.14	ND, ND ND, ND ND, ND ND, ND ND, ND	ND ND ND ND ND

HEAD LETTUCE Country, year Location (Variety) Trial Ref	Application			Matrix	DAT	Residues (mg/kg) [a]			
	g ai/ha	water (L/ha)	season total g ai/ha			Oxathiapiprolin		IN-WR791	
						values	mean	values	mean
USA, 2011) Raleigh, NC (Raider) PR-10653-NC18	35-36	305-315	143	head	0 3	0.25, 0.3 0.012, 0.017	0.28 0.014	ND, ND ND, ND	ND ND
USA, 2011) Salisbury, MD (Black seeded Simpson) PR-10653-MD18	35-36	390	141	head	0 3	1.1, 1.5 0.31, 0.31	1.3 0.31	< 0.01, < 0.01 ND, < 0.01	< 0.01 < 0.01

Information provided to show residues of parent stable in quartered fruit for the intervals between sampling and freezing

[a] Residues of metabolites IN-RDG40, IN-Q7H09, IN-SXS67, IN-RZB20, IN-E8S72 and IN-RZD74 were not detected in any sample

Table 82 Residues in field leaf (open head) lettuce from supervised trials in North America, involving four foliar applications of oxathiapiprolin (OD formulations), applied at 2–5 day intervals

LEAF LETTUCE Country, year Location (Variety) Trial Ref	Application			Matrix	DAT	Residues (mg/kg) [a]			
	g ai/ha	water (L/ha)	season total (g ai/ha)			Oxathiapiprolin		IN-WR791	
							Mean		Mean
GAP: USA	15-35		140		PHI: 0d	Max 4 sprays/season. 3-14 day RTI			
Canada, 2011 Agassiz, BC (Two Star) PR-10653-BC10	36-37	450-475	145	leaves	0 3	1.1, 1.2 0.82, 0.76	1.2 0.79	ND, ND ND, ND	ND ND
Canada, 2011 Harrow, ON (Lasting Green) PR-10653-ON25	35	400	140	leaves	0 3	1.9, 1.9 1.0, 1.4	1.9 1.2	ND, ND ND, ND	ND ND
Canada, 2011 Jordan Station, ON (Green Towers) PR-10653-ON35	36-38	455-470	147	leaves	0 3	0.41, 0.67 0.2, 0.2	0.54 0.2	ND, ND ND, ND	ND ND
Canada, 2011 Ste. Clotilde, QC (Sunbelt) PR-10653-QC13	35-37	350-370	144	leaves	0 3 7 14 23	0.65, 0.97 0.52, 0.55 0.2, 0.12 0.028, 0.081 < 0.01, < 0.01	0.81 0.53 0.16 0.055 < 0.01	ND, ND ND, ND ND, ND ND, ND ND, ND	ND ND ND ND ND
Canada, 2011) Ste. Clotilde, QC (Green Tower) PR-10653-QC14 not independent]	36-38	365-375	149	leaves	0 2	0.52, 0.53 0.53, 0.58	0.53 0.55	ND, ND ND, ND	ND ND
USA, 2011 Citra, FL Two star) PR-10653-FL20	35-36	660-675	141	leaves washed, chopped	0 4 0	1.9, 2.1 1.6, 2.0 2.0, 1.8	2.0 1.8 1.9	ND, ND ND, ND ND, ND	ND ND ND
USA, 2011 Holt, CA (Bergam's) PR-10653-CA79	35-37	280-300	143	leaves	0 3	0.68, 0.69 0.75, 0.84	0.69 0.8	ND, ND ND, ND	ND ND
USA, 2011 Parlier, CA (Red Sails) PR-10653-CA81	35-36	380-395	142	leaves	0 3	3.1, 2.9 2.1, 1.8	3.0 2.0	ND, ND ND, ND	ND ND

LEAF LETTUCE Country, year Location (Variety) Trial Ref	Application			Matrix	DAT	Residues (mg/kg) [a]			
	g ai/ha	water (L/ha)	season total (g ai/ha)			Oxathiapiprolin		IN-WR791	
							Mean		Mean
USA, 2011 Riverside, CA (Winter Density) PR-10653-CA82	35-36	400-405	143	leaves	0 3	1.9, 2.0 1.4, 1.5	1.9 1.4	ND, ND ND, ND	ND ND
USA, 2011) Charleston, SC (New Red Fire) PR-10653-SC*04	35-37	350-360	146	leaves	0 4	1.7, 1.9 0.56, 0.77	1.8 0.66	ND, ND ND, < 0.01	ND < 0.01
USA, 2011) Las Cruces, NM (Oakleaf) PR-10653-NM11	35-37	375-425	142	leaves washed	0 2 0	1.9, 1.8 1.5, 1.3 0.59, 0.84	1.9 1.4 0.72	ND, ND ND, ND ND, ND	ND ND ND

Information provided to show residues of parent stable in quartered fruit for the intervals between sampling and freezing

[a] Residues of metabolites IN-RDG40, IN-Q7H09, IN-SXS67, IN-RZB20, IN-E8S72 and IN-RZD74 were <LOD in any sample

In the supervised trials on lettuce conducted in USA in 2011, separate plots were treated with oxathiapiprolin (SC or OD formulations) as pre-harvest soil drenches or through drip irrigation systems, with two applications being made 6–8 days apart. In three additional trials, soil drench or drip irrigation treatments were made at different growth stages, ranging from planting through to pre-harvest. Dilute concentrations of oxathiapiprolin were either poured around the base of the plants or injected into the drip irrigations systems and followed by irrigation (0.25–0.5 acre-inch) with water.

Duplicate samples (minimum of 12 heads or plants) were trimmed to remove the roots and the heads or tops were halved or quartered, with sub-sampled specimens being frozen within within 5 hours and stored for up to 21 weeks (27 weeks for leaf lettuce) before analysis for oxathiapiprolin and its metabolites (IN-WR791, IN-RDG40, IN-E8S72, IN-RZB20, IN-RZD74, IN-SXS67 and IN-Q7H09) using method DP30422 Supplement 1 (LC-MS/MS). Mean recovery rates for each analyte ranged from 91% to 110% in samples spiked with 0.01–0.2 mg/kg. The LOQs were 0.01 mg/kg for all analytes.

Table 83 Residues in field head lettuce (with wrapper leaves) from supervised trials in North America, involving two pre-harvest soil drench/irrigation treatments oxathiapiprolin, applied 6–8 days apart

HEAD LETTUCE Country, year Location (Variety) Trial Ref	Application			DAT	Residues (mg/kg) [a]	
	form (method)	g ai/ha	season total (g ai/ha)		Oxathiapiprolin	
					values	mean
GAP: USA	Max 4 applications @	35-280	560	PHI: 0d	Min 7 day RTI	
Canada, 2011 Agassiz, BC (Odyssey) PR-10653-BC11	SC (drench)	289	578	0 3	0.47, 0.39 0.2, 0.16	0.43 0.18
Canada, 2011 Harrow, ON (Odyssey) PR-10653-ON28	SC (drench)	280	560	0 3	< 0.01, ND ND, ND	< 0.01 ND
Canada, 2011) Harrow, ON (Caliente) PR-10653-ON27 [not independent]	SC (drench)	280	560	0 3	ND, ND ND, ND	ND ND

HEAD LETTUCE Country, year Location (Variety) Trial Ref	Application			DAT	Residues (mg/kg) [a]	
	form (method)	g ai/ha	season total (g ai/ha)		Oxathiapiprolin	
					values	mean
USA, 2011 Citra, FL (Head Lettuce/) PR-10653-FL21	SC (drench)	281	562	0 2	ND, ND ND, ND	ND ND
USA, 2011 Freeville, NY (Head Lettuce, Fall Green) PR-10653-NY11	SC (drench)	283	566	0 2	< 0.01, < 0.01 < 0.01, ND	< 0.01 < 0.01
USA, 2011 Holt, CA (Jupiter) PR-10653-CA80	SC (drip)	281	562	0 3	ND, ND ND, ND	ND ND
USA, 2011 Las Cruces, NM (Salinas) PR-10653-NM12	OD (drip)	281	562	0 2	ND, ND ND, ND	ND ND
USA, 2011 Riverside, CA (Fall green) PR-10653-CA78	SC (drip)	283	566	0 4	ND, ND ND, ND	ND ND
USA, 2011 Salinas, CA (corona) PR-10653-CA*83	SC (drench)	284	567	0 3 6 15 27	0.47, 0.27 0.26, 0.33 0.32, 0.31 0.098, 0.086 0.037, 0.027	0.37 0.29 0.31 0.092 0.032
USA, 2011) Raleigh, NC (Raider) PR-10653-NC18	SC (drench)	284	568	0 3	ND, ND ND, ND	ND ND
USA, 2011) Salisbury, MD (Black seeded Simpson) PR-10653-MD18	SC (drench)	273	546	0 3	< 0.01, < 0.01 ND, ND	< 0.01 ND

Information provided to show residues of parent stable in quartered fruit for the intervals between sampling and freezing

[a] Residues of metabolites IN-RDG40, IN-WR791, IN-Q7H09, IN-SXS67, IN-RZB20, IN-E8S72 and IN-RZD74 were <LOD in any sample

Table 84 Residues in field leaf (open head) lettuce from supervised trials in North America, involving two pre-harvest soil drench/irrigation treatments oxathiapiprolin, applied 6–8 days apart

LETTUCE Country, year Location (Variety) Trial Ref	Application			DAT	Residues (mg/kg) [a]			
	form (method)	g ai/ha	season total (g ai/a)		Oxathiapiprolin		IN-E8S72	
					values	mean	values	mean
GAP: USA		35-280	560	PHI: 0d	Max 4 applications/season. Min 7 day RTI			
Canada, 2011 Agassiz, BC (Two Star) PR-10653-BC10	SC (drench)	289	578	0 3	0.35, 0.38 0.19, 0.26	0.37 0.23	ND, ND ND, ND	ND ND
USA, 2011 Holt, CA (Bergam's) PR-10653-CA79	SC (drip)	283	565	0 3	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND

LETTUCE Country, year Location (Variety) Trial Ref	Application			DAT	Residues (mg/kg) [a]			
	form (method)	g ai/ha	season total (g ai/a)		Oxathiapiprolin		IN-E8S72	
					values	mean	values	mean
USA, 2011 Parlier, CA (Red Sails) PR-10653-CA81	OD (drip)	282	564	0 3	0.092, 0.054 0.037, 0.033	0.073 0.035	ND, ND ND, ND	ND ND
USA, 2011 Riverside, CA (Winter Density) PR-10653-CA82	SC (drip)	282	564	0 3	< 0.01, < 0.01 < 0.01, ND	< 0.01 ND	ND, ND ND, ND	ND ND
USA, 2011 Citra, FL (Two star) PR-10653-FL20	SC (drench)	283	565	0 3	0.038, 0.14 0.05, 0.023	0.091 0.037	ND, ND ND, ND	ND ND
USA, 2011) Las Cruces, NM (Oakleaf) PR-10653-NM11	OD (drip)	280	560	0 2	< 0.01, ND ND, ND	< 0.01 ND	ND, ND ND, ND	ND ND
Canada, 2011) Harrow, ON (Lasting Green) PR-10653-ON25	SC (drench)	280	560	0 3	0.017, 0.012 ND, < 0.01	0.014 ND	ND, ND ND, ND	ND ND
Canada, 2011 Jordan Station, ON (Green Towers) PR-10653-ON35	SC (drench)	280	559	0 3	0.01, < 0.01 < 0.01, ND	< 0.01 ND	ND, ND ND, ND	ND ND
Canada, 2011 Ste. Clotilde, QC (Sunbelt) PR-10653-QC13	SC (drip)	280	560	0 3 7 14 23	ND, ND ND, ND ND, ND ND, ND ND, ND	ND ND ND ND ND	ND, ND ND, ND ND, ND ND, ND ND, ND	ND ND ND ND ND
Canada, 2011) Ste. Clotilde, QC (Green Tower) PR-10653-QC14 [See QC13]	SC (drip)	280	560	0 2	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND
USA, 2011) Charleston, SC (New Red Fire) PR-10653-SC*04	OD (drench)	273	546	0 3	< 0.01, 0.028 ND, < 0.01	0.016 ND	ND, ND ND, < 0.01	ND < 0.01
USA, 2011 Germansville, PA (Red Sails) DP-33132-Trial 01	SC (drip)	280	560	0 6 14	ND, ND ND, ND ND, ND	ND ND ND	ND, ND ND, ND ND, ND	ND ND ND
	SC (drench) T3	280	560	49	ND, ND	ND	< 0.01, < 0.01	< 0.01
	SC (drip) T4	280	560	28 34 42	ND, ND ND, ND < 0.01, < 0.01	ND ND < 0.01	< 0.01, < 0.01 < 0.01, < 0.01 < 0.01, < 0.01	< 0.01 < 0.01 < 0.01
	SC (drip) T5	280	560	6 12 20	< 0.01, ND ND, ND ND, ND	< 0.01 ND ND	ND, ND ND, ND ND, ND	ND ND ND
Canada, 2011 Branchton, ON (Butterhead) DP-33132-Trial 02	SC (drip)	278	556	0 7 14	< 0.01, < 0.01 0.02, 0.031 0.011, 0.011	< 0.01 0.025 0.011	ND, ND ND, ND ND, ND	ND ND ND
	SC (drip) T3	278	556	47	0.014, 0.015	0.015	0.018, 0.02	0.019
	SC (drip) T4	278	556	27 34 41	0.062, 0.053 0.081, 0.13 0.056, 0.068	0.058 0.1 0.062	0.013, < 0.01 0.01, 0.011 < 0.01, < 0.01	0.011 0.01 < 0.01
USA, 2011 Porterville, CA (Red Sails) DP-33132-Trial 03 [b]	SC (drench)	280	560	0 7 14	3.7, 2.0 3.5, 3.1 5.0, 2.7	2.9 3.3 3.8	ND, ND ND, ND ND, ND	ND ND ND
	SC (drench) T3	280	560	70	0.025, 0.038	0.032	0.09, 0.1	0.097

LETTUCE Country, year Location (Variety) Trial Ref	Application			DAT	Residues (mg/kg) [a]			
	form (method)	g ai/ha	season total (g ai/a)		Oxathiapiprolin		IN-E8S72	
					values	mean	values	mean
	SC (drench) T4	280	560	49	0.049, 0.085	0.067	0.023, 0.034	0.028
				56	0.035, 0.1	0.069	0.026, 0.034	0.03
				63	0.15, 0.064	0.11	0.035, 0.023	0.029
	SC (drench) T5	280	560	28	0.67, 0.87	0.774	ND, < 0.01	< 0.01
				35	0.39, 0.2	0.3	< 0.01, < 0.01	< 0.01
				42	0.27, 0.1	0.18	< 0.01, < 0.01	< 0.01

[a] Residues of metabolites IN-RDG40, IN-WR791, IN-Q7H09, IN-SXS67, IN-RZB20, and IN-RZD74 were <LOD in any sample

[b] Reported residues in Trial 03 significantly higher than other trial results. No identified reason for this inconsistency but contamination during soil application suspected. Trial results have been excluded.

T3 First applications at planting

T4 First application 21 days after planting

T5 First application 42 days after planting

Spinach

In supervised trials on field spinach conducted in North America in 2011, four foliar applications of oxathiapiprolin (OD formulations) with added adjuvant were applied at 2–4 day intervals intervals using tractor-mounted 8-nozzle booms or pressurised backpack or mounted sprayers with 3–6 nozzle booms.

Duplicate samples (minimum of 1 kg from at least 12 plants) were frozen within within 3.5 hours and stored for up to 25 weeks before analysis for oxathiapiprolin and its metabolites (IN-WR791, IN-RDG40, IN-E8S72, IN-RZB20, IN-RZD74, IN-SXS67 and IN-Q7H09) using method DP30422 Supplement 1 (LC-MS/MS). Mean recovery rates for each analyte ranged from 91% to 112% in samples spiked with 0.01–20 mg/kg. The LOQs were 0.01 mg/kg for all analytes.

Table 85 Residues in field spinach from supervised trials in North America, involving four foliar applications of oxathiapiprolin (OD formulations) with adjuvant, applied at 2-4 day intervals

SPINACH Country, year Location (Variety) Trial Ref	Application			Matrix	DAT	Residues (mg/kg) [a]					
	g ai/ha	water (L/ha)	season total (g ai/ha)			Oxathiapiprolin		IN-RDG40		IN-WR791	
						values	mean	values	mean	values	mean
GAP: USA	15-35		140		PHI: 0d	Max 4 sprays/season. 3-14 day RTI					
Canada, 2011 Abbotsford, BC (Unipack 12) DP-31730-Trial 09	34-35	195-200	136.4	leaves	0 3	6.6, 6.5 3.8, 4.0	6.5 3.9	< 0.01, < 0.01 < 0.01, ND	< 0.01 < 0.01	ND, ND ND, ND	ND ND
Canada, 2011 Puslinch, ON (Greyhound) DP-31730-Trial 04	36	200	144.7	leaves washed	0 4 0	1.4, 1.3 0.84, 0.94 1.0, 1.0	1.4 0.89 1.0	ND, ND ND, ND ND, ND	ND ND ND	ND, ND ND, ND ND, ND	ND ND ND
Canada, 2011 St. Marc-sur-Richelieu, QC (Crocodile) DP-31730-Trial 03	34-36	250	140.8	leaves	0 3	1.7, 1.5 0.94, 1.1	1.6 1.0	ND, ND ND, ND	ND ND	ND, ND < 0.01, < 0.01	ND < 0.01
USA, 2011 Chula, GA (Vancouver) DP-31730-Trial 02	35-36	225-360	141.1	leaves washed	0 3 0	2.1, 2.2 1.8, 1.6 1.5, 1.5	2.2 1.8 1.5	ND, ND ND, ND ND, ND	ND ND ND	ND, ND ND, ND ND, ND	ND ND ND

SPINACH Country, year Location (Variety) Trial Ref	Application			Matrix	DAT	Residues (mg/kg)[a]					
	g ai/ha	water (L/ha)	season total (g ai/ha)			Oxathiapiprolin		IN-RDG40		IN-WR791	
						values	mean	values	mean	values	mean
USA, 2011 Frenchtown, NJ (Tyee) DP-31730-Trial 01	36-37	281	144.8	leaves	0 3	2.9, 3.6 1.6, 1.7	3.2 1.6	ND, ND ND, ND	ND ND	< 0.01, < 0.01 < 0.01, < 0.01	< 0.01 < 0.01
USA, 2011 Jerome, ID (Unipack-151) DP-31730-Trial 07	35-36	205-210	140.9	leaves	0 3	3.3, 3.6 2.8, 3.4	3.5 3.1	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND
USA, 2011 Kerman, CA (Shasta Hybrid) DP-31730-Trial 08	35-36	280-285	141.9	leaves	0 3	5.7, 7.0 3.7, 6.9	6.4 5.3	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND
USA, 2011 Porterville, CA (Shasta) DP-31730-Trial 10	34-36	310-320	140.3	leaves	0 3 7 14 30	6.0, 5.4 3.1, 2.7 2.2, 2.2 1.4, 1.8 0.88, 0.72	5.7 2.9 2.2 1.6 0.8	ND, ND ND, ND ND, ND ND, ND ND, ND	ND ND ND ND ND	ND, ND < 0.01, < 0.01 < 0.01, < 0.01 < 0.01, < 0.01 < 0.01, < 0.01	ND < 0.01 < 0.01 < 0.01 < 0.01
USA, 2011 Raymondville, TX (Melody) DP-31730-Trial 06	36	187	143.4	leaves	0 3	3.8, 4.2 2.9, 2.5	4.0 2.7	ND, ND < 0.01, < 0.01	ND < 0.01	< 0.01, < 0.01 < 0.01, < 0.01	< 0.01 < 0.01
USA, 2011 Uvalde, TX (DMC6607) DP-31730-Trial 05	34-36	187	140	leaves washed	0 3 0	2.6, 2.1 1.2, 1.6 2.1, 2.7	2.3 1.4 2.4	ND, ND ND, ND ND, ND	ND ND ND	< 0.01, < 0.01 < 0.01, < 0.01 < 0.01, ND	< 0.01 < 0.01 ND

[a] Residues of metabolites IN-Q7H09, IN-E8S72, IN-SXS67, IN-RZB20, and IN-RZD74 were <LOD in any sample

Washed: samples held under cold running tap water for 15-20 seconds and drained for at least 2 minutes

In the supervised trials on spinach conducted in USA in 2011, separate plots were treated with oxathiapiprolin (SC formulations) as soil applications 6–8 days before maturity and at maturity. Dilute concentrations of oxathiapiprolin were either band sprayed, shank-injected around the base of the plants or added to the drip irrigations systems and followed by irrigation (0.25–0.5 acre-inch).

Duplicate samples (minimum of 1 kg from at least 12 plants) were frozen within within 3.5 hours and stored for up to 25 weeks before analysis for oxathiapiprolin and its metabolites (IN-WR791, IN-RDG40, IN-E8S72, IN-RZB20, IN-RZD74, IN-SXS67 and IN-Q7H09) using method DP30422 Supplement 1 (LC-MS/MS). Mean recovery rates for each analyte ranged from 91% to 112% in samples spiked with 0.01–20 mg/kg. The LOQs were 0.01 mg/kg for all analytes.

Table 86 Residues in field spinach leaves from supervised trials in North America, involving two close-to-harvest soil applications of oxathiapiprolin (SC formulations) applied 6–8 days apart

SPINACH Country, year Location (Variety) Trial Ref	Application				DAT	Residues (mg/kg)[a]				
	method	g ai/ha	water (L/ha)	season total (g ai/ha)		Oxathiapiprolin		IN-WR791		
						values	mean	mean	values	mean
GAP: USA		35-280		560	PHI: 0d	Max 4 applications/season. Min 7 day RTI				
Canada, 2011 Abbotsford, BC (Unipack 12) DP-31730-Trial 09	drip	280	NA	560.4	0 3	< 0.01, ND ND, < 0.01	< 0.01 ND	ND ND	ND, ND ND, ND	ND ND

SPINACH Country, year Location (Variety) Trial Ref	Application				DAT	Residues (mg/kg) [a]				
	method	g ai/ha	water (L/ha)	season total (g ai/ha)		Oxathiapiprolin		IN-WR791		
						values	mean	mean	values	mean
Canada, 2011 Puslinch, ON (Greyhound) DP-31730-Trial 04	drip	278	9000	556	0 4	0.085, 0.11 0.12, 0.1	0.095 0.11	ND ND	ND, ND ND, ND	ND ND
Canada, 2011 St. Marc-sur-Richelieu, QC (Crocodile) DP-31730-Trial 03	band	285	3350-3390	569.2	0 3	1.7, 1.8 2.1, 1.8	1.8 2.0	ND ND	ND, ND < 0.01, < 0.01	ND < 0.01
USA, 2011 Chula, GA (Vancouver) DP-31730-Trial 02	directed	275	175-180	549.4	0 3	2.1, 2.0 2.4, 1.9	2.1 2.2	ND ND	ND, ND < 0.01, ND	ND ND
USA, 2011 Frenchtown, NJ (Tyee) DP-31730-Trial 01	band	278	514	556.8	0 3	1.7, 1.4 0.82, 1.0	1.6 0.91	ND ND	< 0.01, < 0.01 < 0.01, < 0.01	< 0.01 < 0.01
USA, 2011 Jerome, ID (Unipack-151) DP-31730-Trial 07	band	288	2935-2945	575.4	0 3	1.4, 2.0 1.7, 2.0	1.7 1.8	ND ND	ND, ND ND, ND	ND ND
USA, 2011 Kerman, CA (Shasta Hybrid) DP-31730-Trial 08	shank soil inject	280	374	559.3	0 3	< 0.01, < 0.01 0.013, 0.012	< 0.01 0.013	ND ND	ND, ND ND, ND	ND ND
USA, 2011 Porterville, CA (Shasta) DP-31730-Trial 10	drip	280	NA	560.4	0 3 7 14 30	ND, ND < 0.01, < 0.01 < 0.01, < 0.01 ND, 0.01 < 0.01, < 0.01	ND < 0.01 < 0.01 < 0.01 < 0.01	ND ND ND ND ND	ND, ND ND, ND ND, ND ND, ND ND, ND	ND ND ND ND ND
USA, 2011 Raymondville, TX (Melody) DP-31730-Trial 06	shank soil inject	285	421	569.6	0 3	ND, ND < 0.01, ND	ND < 0.01	ND ND	ND, ND ND, ND	ND ND
USA, 2011 Uvalde, TX (DMC6607) DP-31730-Trial 05	shank soil inject	280-285	185-315	563.6	0 3	0.16, 0.079 0.11, 0.073	0.12 0.092	ND ND	ND, ND ND, ND	ND ND

[a] Residues of metabolites IN-Q7H09, IN-RDG40, IN-E8S72, IN-SXS67, IN-RZB20, and IN-RZD74 were <LOD in any sample

In three of the spinach trials conducted in USA in 2011, separate plots were also treated with oxathiapiprolin (SC formulations) as directed band sprays or shank-injections to bare soil at planting and 6–8 days later.

Duplicate samples (minimum of 1 kg from at least 12 plants) were frozen within within 3.5 hours and stored for up to 25 weeks before analysis for oxathiapiprolin and its metabolites (IN-WR791, IN-RDG40, IN-E8S72, IN-RZB20, IN-RZD74, IN-SXS67 and IN-Q7H09) using method DP30422 Supplement 1 (LC-MS/MS). Mean recovery rates for each analyte ranged from 91% to 112% in samples spiked with 0.01–20 mg/kg. The LOQs were 0.01 mg/kg for all analytes.

Table 87 Residues in field spinach from supervised trials in North America, involving two soil drench/irrigation treatments oxathiapiprolin, applied at planting and 6–8 days apart

SPINACH Country, year Location (Variety) Trial Ref	Application				DAT	Residues (mg/kg) [a]			
	method	g ai/ha	water (L/ha)	season total {g ai/ha)		Oxathiapiprolin		IN-E8S72	
							mean		mean
GAP: USA		35-280		560	PHI: 0d	Max 4 applications/season. Min 7 day RTI			
Canada, 2011 St. Marc-sur-Richelieu, QC (Crocodile) DP-31730-Trial 03	Band	286	3320-3390	567.5	30	0.036, 0.033	0.034	< 0.01, < 0.01	< 0.01
USA, 2011 Frenchtown, NJ (Tyee) DP-31730-Trial 01	Band	288	421	574.8	34	0.015, < 0.01	0.011	0.027, 0.025	0.026
USA, 2011 Kerman, CA (Shasta Hybrid) DP-31730-Trial 08	Shank soil inject	282	375	564.4	62	ND, ND	ND	ND, ND	ND

[a] Residues of metabolites IN-Q7H09, IN-RDG40, IN-WR791, IN-SXS67, IN-RZB20, and IN-RZD74 were <LOD in any sample

Legume vegetables

Peas

In supervised trials on garden peas conducted in North America in 2011, four foliar applications of oxathiapiprolin (OD formulations) with added adjuvant were applied at 2–4 day intervals intervals using pressurised backpack or mounted sprayers with 3–6 nozzle booms.

Duplicate samples (minimum of 1 kg) were frozen within within 3.5 hours and stored for up to 25 weeks before analysis for oxathiapiprolin and its metabolites (IN-WR791, IN-RDG40, IN-E8S72, IN-RZB20, IN-RZD74, IN-SXS67 and IN-Q7H09) using method DuPont-30422 Supplement 1 (LC-MS/MS). Mean recovery rates for each analyte ranged from 88% to 116% in samples spiked with 0.01 to 0.1 mg/kg (peas) or 1.0 mg/kg (peas with pod). The LOQs were 0.01 mg/kg for all analytes.

Table 88 Residues in peas (with pod) from supervised trials in North America involving 4 foliar applications of oxathiapiprolin (OD formulations) with adjuvant, applied at 4–6 day intervals

PEAS Country, year Location (Variety) Trial Ref	Application			DAT	Residues (mg/kg) [a]			
	g ai/ha	water (L/ha)	season total (g ai/ha)		oxathiapiprolin		IN-WR791	
					values	mean	values	mean
GAP: USA	15-35		140	PHI: 0d	Max 4 sprays/season. 7-10 day RTI			
Canada 2011 Bouctouche, NB (Oregon Sugar II) DP-AAFC11-013R-248	35-36	200-205	142	0	0.21, 0.2	0.2	< 0.01, < 0.01	< 0.01
Canada 2011 Harrow, ON (Sugar Sprint) DP-AAFC11-013R-250	37-39	260-275	153	0	0.32, 0.27	0.3	0.012, 0.013	0.012

PEAS Country, year Location (Variety) Trial Ref	Application			DAT	Residues (mg/kg) [a]			
	g ai/ha	water (L/ha)	season total (g ai/ha)		oxathiapiprolin		IN-WR791	
					values	mean	values	mean
Canada 2011 Sainte-Clotilde, QC (Snow Green) DP-AAFC11-013R-253	36-38	260-270	140	0 3 6 9 14	0.27, 0.32 0.14, 0.22 0.06, 0.06 0.026, 0.043 ND, ND	0.3 0.18 0.06 0.035 ND	ND, ND ND, ND ND, ND ND, ND ND, ND	ND ND ND ND ND
Canada 2011 Sainte-Clotilde, QC (Spring) DP-AAFC11-013R-254 [not independent]	36-38	255-270	147	0	0.26, 0.27	0.26	ND, ND	ND
USA, 2011 Wapato, WA (Oregon Giant) DP-AAFC11-013R-335	33-38	210-245	143	0	0.55, 0.55	0.55	ND, ND	ND
USA, 2011 Charleston, SC (Sugar Sprint) DP-AAFC11-013R-337	36	245-255	144	0	0.27, 0.29	0.28	ND, ND	ND

[a] Residues of metabolites IN-Q7H09, IN-RDG40, IN-SXS67, IN-RZB20, IN-E8S72 and IN-RZD74 were not detected in any sample

Table 89 Residues in peas (without pod) from supervised trials in North America involving 4 foliar applications of oxathiapiprolin (OD formulations) with adjuvant, applied at 4–6 day intervals

PEAS Country, year Location (Variety) Trial Ref	Application			DAT	Residues (mg/kg)[a]			
	g ai/ha	water (L/ha)	season total (g ai/ha)		oxathiapiprolin		IN-WR791	
					values	mean	values	mean
GAP: USA	15-35		140	PHI: 0d	Max 4 sprays/season. 7-10 day RTI			
Canada 2011 Agassiz,BC Canada, 2011 (Progress # 9) DP-AAFC11-013R-255	36-39	275-295	149	0	< 0.01, < 0.01	< 0.01	ND, ND	ND
Canada 2011 Kentville, NS (Legacy) DP-AAFC11-013R-249	35-36	250-255	141	0	< 0.01, 0.01	< 0.01	< 0.01, < 0.01	< 0.01
Canada 2011 Sainte-Clotilde, QC (Premium) DP-AAFC11-013R-251 [not independent]	37-41	260-270	153	0	0.011, < 0.01	0.01	< 0.01, < 0.01	< 0.01
Canada 2011 Sainte-Clotilde, QC (Strike) DP-AAFC11-013R-252	34-41	245-290	150	0	0.013, 0.012	0.012	< 0.01, < 0.01	< 0.01
USA, 2011 Tifton, GA (Pinkeye Purple Hull) DP-AAFC11-013R-336	35	280-285	140	0	0.022, 0.029	0.026	ND, ND	ND
USA, 2011 Wapato, WA (Progress # 9) DP-AAFC11-013R-334	32-38	160-170	140	0	0.026, 0.023	0.025	ND, ND	ND

[a] Residues of metabolites IN-Q7H09, IN-RDG40, IN-SXS67, IN-RZB20, IN-E8S72 and IN-RZD74 were <LOD in any sample

Root and tuber vegetables

Potato

In supervised field trials on potatoes, conducted in Europe in 2010 and 2011, four foliar applications of oxathiapiprolin (OD or SE formulations) without added adjuvant were applied at 8–12 day intervals intervals using tractor-mounted or pressurised backpack sprayers with 6–10 nozzle booms.

Samples (minimum of 2 kg) were frozen within 9 hours and stored for up to 32 weeks before analysis for oxathiapiprolin and its metabolites using method DuPont-30422 (LC-MS/MS) to measure residues of oxathiapiprolin, IN-WR791, IN-RDG40, IN-E8S72 and IN-Q7H09. Mean recovery rates for each analyte ranged from 79% to 119% in samples spiked with 0.01–0.1 mg/kg. The LOQs were 0.01 mg/kg for all analytes.

Table 90 Residues in potatoes from supervised trials in Europe involving 3 foliar applications of oxathiapiprolin (OD formulations), applied at 8–12 day intervals.

POTATOES Country, year Location (Variety) Trial Ref	Application					Matrix	DAT	Residues (mg/kg)[a]	
	form	g ai/ha	water (L/ha)	g ai/hL	season total (g ai/ha)			Oxathiapiprolin	IN-WR791
UK, 2010 Borders (Saxon) DP-30044-Test 01	SE	32-36	280-315	11	134.64	Tubers	-0 0 1 3	ND ND ND ND	ND ND ND ND
France, 2010 Nord-Pas de Calais (Bintje) DP-30044-Test 02	SE	33	285-290	11	130.97	Tubers	-0 0 1 3	ND ND ND ND	ND ND ND ND
Germany, 2010 Saxony (Alegria) DP-30044-Test 03	SE	34-35	305-315	11	137.3	Tubers	-0 0 1 3	ND ND ND ND	ND ND ND ND
France, 2010 Rhône-Alpes (Mona Lisa) DP-30044-Test 04	SE	33	285-295	11	131.83	Tubers	-0 0 1 3	ND ND ND ND	ND ND ND ND
Italy, 2010 Lombardia (Desy) DP-30044-Test 05	SE	34-36	300-315	11	139.09	Tubers	-0 0 1 3	ND ND ND ND	ND ND ND ND
Spain, 2010 Catalunya (Frisia) DP-30044-Test 06	SE	34-35	295-305	11	136.97	Tubers	-0 0 1 3	ND ND ND ND	ND ND ND ND
UK, 2011 East Lothian (Cultra) DP-30044-Test 07	SE	48-50	285-300	17	197.31	Tubers	-0 0 1 3 7 13 21	ND ND ND ND ND ND ND	ND ND ND ND ND ND ND
France, 2011 Nord-Pas de Calais (Victoria) DP-30044-Test 08	SE	48-51	285-310	17	200.95	Tubers	-0 0 1 3 7 14 21	ND ND ND ND ND ND ND	ND ND ND ND ND ND ND

POTATOES Country, year Location (Variety) Trial Ref	Application					Matrix	DAT	Residues (mg/kg) [a]	
	form	g ai/ha	water (L/ha)	g ai/hL	season total (g ai/ha)			Oxathiapiprolin	IN-WR791
Germany, 2011 Saxony (Marabel) DP-30044-Test 09	SE	50-54	290-310	17	207.7	Tubers	-0 0 1 3 7 14 21	ND ND ND ND ND ND ND	ND ND ND ND ND ND ND
Greece, 2011 Pella (Spunta) DP-30044-Test 10	SE	50-51	300	17	201.53	Tubers	-0 0 1 3 7 14 21	ND ND ND ND ND ND ND	ND ND ND ND ND ND ND
Greece, 2011 Imathia (Spunta) DP-30044-Test 11	SE	50-51	300-305	17	202.59	Tubers	1	ND	ND
	OD	50-51	300	17	202.02		1	ND	ND
Netherlands, 2011 Gennep (Agria) DP-30044-Test 12	SE	50-51	290-310	17	200.85	Tubers	1	ND	ND
	OD	50-51	300-305	17	202.34		1	ND	ND
Italy, 2011 Piemonte (Primura) DP-30044-Test 13	SE	49-52	295-315	17	203.73	Tubers	-0 0 1 3 6 14 21	ND ND ND ND ND ND ND	ND ND ND ND ND ND ND
Spain, 2011 Andalucia (Spunta) DP-30044-Test 14	SE	50-52	300-310	17	203.94	Tubers	-0 0 1 3 7 14 19	ND ND ND ND ND ND ND	ND ND ND ND ND ND ND
Spain, 2011 Aragon (Kenebec) DP-30044-Test 15	SE	49-52	295-310	17	202.44	Tubers	1	ND	< 0.01
	OD	49-51	295-305	17	202.06		1	ND	ND
Poland, 2011 Wielkopolska (Pasja) DP-30044-Test 16	SE	49-52	295-315	17	203.9	Tubers	1	ND	ND
	OD	48-51	285-305	17	195.4		1	ND	ND
France, 2011 Rhône-Alpes (Safrane) DP-30044-Test 17	SE	50-51	300-310	17	202.67	Tubers	1	ND	ND
	OD	49-50	290-300	17	197.57		1	ND	ND
Hungary, 2011 Györ-Moson-Sopron (Agria) DP-30044-Test 18	SE	50-52	300-310	17	201.8	Tubers	1	ND	< 0.01
	OD	49-52	295-315	17	200.7		1	ND	ND

[a] Residues of metabolites IN-Q7H09, IN-RDG40 and IN-E8S72 were <LOD in any sample

In supervised trials on potatoes conducted in North America in 2012, four foliar applications of oxathiapiprolin (OD formulations) with added adjuvant were applied either at 4–6 day intervals at BBCH 13–15, at 5–6 day intervals over flowering or close-to-harvest using pressurised backpack or mounted sprayers with 4–8 nozzle booms.

Duplicate samples (minimum of 1 kg from 12 plants) were frozen within within 2 hours and stored for up to 29 weeks before analysis for oxathiapiprolin and its IN-WR791 and IN-E8S72 metabolites using method DuPont-30422 Supplement 1 (LC-MS/MS). Mean recovery rates for each analyte ranged from 103% to 110% in samples spiked with 0.01 to 0.2 mg/kg. The LOQs were 0.01 mg/kg for all analytes.

Table 91 Residues in potato tubers from supervised trials in North America involving 4 early season (BBCH 13–15) foliar applications of oxathiapiprolin (OD formulations) with adjuvant, applied at 4–6 day intervals

POTATOES Country, year Location (Variety) Trial Ref	Application			DAT	Residues (mg/kg)					
	g ai/ha	water (L/ha)	season total (g ai/ha)		Oxathiapiprolin		IN-E8S72		IN-WR791	
					values	mean	values	mean	values	mean
GAP: USA	50		200	Apply twice over flowering, 10-14 days apart						
Canada, 2012 Fort Saskatchewan, AB (Russet Nocota) DP-31762-Trial 21	49-52	310	202	63	ND, ND	ND	ND, ND	ND	ND, ND	ND
USA, 2012 Alton, NY (Superior) DP-31762-Trial 03	51	280	204	63	ND, ND	ND	ND, ND	ND	ND, ND	ND
USA, 2012 Ephrata, WA (Russet Burbank) DP-31762-Trial 14	49-52	375	202	77	ND, ND	ND	< 0.01, < 0.01	< 0.01	< 0.01, < 0.01	< 0.01
USA, 2012 Payette, ID (Russet Norkotah) DP-31762-Trial 16	49-50	325-330	199	61	ND, ND	ND	ND, ND	ND	ND, ND	ND
USA, 2012 Verona, WI (Superior) DP-31762-Trial 10	49-51	330-365	198	68	ND, ND	ND	ND, ND	ND	ND, ND	ND

Table 92 Residues in potato tubers from supervised trials in North America involving 4 mid-season (over flowering) foliar applications of oxathiapiprolin (OD formulations) with adjuvant, applied at 4–6 day intervals

POTATOES Country, year Location (Variety) Trial Ref	Application			DAT	Residues (mg/kg)					
	g ai/ha	water (L/ha)	season total (g ai/ha)		Oxathiapiprolin		IN-E8S72		IN-WR791	
					values	mean	values	mean	values	mean
GAP: USA	50		200	Apply twice over flowering, 10-14 days apart						
Canada, 2012 Fort Saskatchewan, AB (Russet Nocota) DP-31762-Trial 21	50-51	310	201	36	ND, < 0.01	ND	ND, ND	ND	ND, ND	ND
USA, 2012 Alton, NY (Superior) DP-31762-Trial 03	51	280	203	48	ND, ND	ND	ND, ND	ND	ND, < 0.01	ND
USA, 2012 Ephrata, WA (Russet Burbank) DP-31762-Trial 14	50-52	375	204	39	ND, ND	ND	ND, ND	ND	< 0.01, < 0.01	< 0.01
USA, 2012 Payette, ID (Russet Norkotah) DP-31762-Trial 16	50-51	325-330	202	34	ND, ND	ND	ND, ND	ND	ND, ND	ND

POTATOES Country, year Location (Variety) Trial Ref	Application			DAT	Residues (mg/kg)					
	g ai/ha	water (L/ha)	season total (g ai/ha)		Oxathiapiprolin		IN-E8S72		IN-WR791	
					values	mean	values	mean	values	mean
USA, 2012 Verona, WI (Superior) DP-31762-Trial 10	50	350-390	200	52	ND, ND	ND	ND, ND	ND	ND, ND	ND

Table 93 Residues in potato tubers from supervised trials in North America involving 4 late-season foliar applications of oxathiapiprolin (OD formulations) with adjuvant, applied at 4–6 day intervals

POTATOES Country, year Location (Variety) Trial Ref	Application			DAT	Residues (mg/kg)					
	g ai/ha	water (L/ha)	season total (g ai/ha)		Oxathiapiprolin		IN-E8S72		IN-WR791	
					values	mean	values	mean	values	mean
GAP: USA	12-35		200	PHI: 5d	Max 4 sprays/season. 5-14 day RTI					
Canada, 2012 Abbotsford, BC (Russet Burbank) DP-31762-Trial 20	49-51	400-410	201	4	ND, ND	ND	ND, ND	ND	ND, ND	ND
Canada, 2012 Branchton, ON (Norland) DP-31762-Trial 07	50-53	300	207	5	ND, ND	ND	ND, ND	ND	ND, ND	ND
Canada, 2012 Centreville, NS (FL1867) DP-31762-Trial 01	50-51	470	203	5	ND, ND	ND	ND, ND	ND	< 0.01, < 0.01	< 0.01
Canada, 2012 Fort Saskatchewan, AB (Russet Nocota) DP-31762-Trial 21	50-51	310	203	5	< 0.01, < 0.01	< 0.01	ND, ND	ND	ND, ND	ND
Canada, 2012 Fort Saskatchewan, AB (Russet Nocota) DP-31762-Trial 22 [not independent]	50-52	310	203	5	< 0.01, < 0.01	< 0.01	ND, ND	ND	ND, ND	ND
Canada, 2012 New Glasgow, PE (Norland) DP-31762-Trial 02	48-57	335-400	206	5	ND, ND	ND	ND, ND	ND	ND, ND	ND
Canada, 2012 Portage la Prairie, MB (Russet Ranger) DP-31762-Trial 08	49-50	295-300	199	5	ND, ND	ND	ND, ND	ND	ND, ND	ND
Canada, 2012 St. Marc-sur-Richelieu, QC (Chiefton) DP-31762-Trial 06	52	300	208	5	ND, ND	ND	ND, ND	ND	ND, ND	ND
USA, 2012 Acequia, ID (Russet Burbank) DP-31762-Trial 18	48-49	340-410	196	5	ND, ND	ND	ND, ND	ND	ND, ND	ND
USA, 2012 Alton, NY (Superior) DP-31762-Trial 03	50-51	280	202	5	ND, ND	ND	ND, ND	ND	ND, ND	ND

POTATOES Country, year Location (Variety) Trial Ref	Application			DAT	Residues (mg/kg)					
	g ai/ha	water (L/ha)	season total (g ai/ha)		Oxathiapiprolin		IN-E8S72		IN-WR791	
					values	mean	values	mean	values	mean
USA, 2012 Chula, GA (Red Pontiac) DP-31762-Trial 04	35-36	330-355	142	5	ND, ND	ND	ND, ND	ND	ND, ND	ND
USA, 2012 Ephrata, WA (Russet Burbank) DP-31762-Trial 14 [not independent]	50-51	375	203	5	ND, ND	ND	ND, ND	ND	ND, ND	ND
USA, 2012 Ephrata, WA (Russet Norkotah) DP-31762-Trial 13	50	375	200	5	ND, ND	ND	ND, ND	ND	ND, ND	ND
USA, 2012 Geneva, MN (Cascade) DP-31762-Trial 09	50	280-285	201	-0 0 3 5 9 14	ND, ND ND, ND ND, ND ND, ND ND, ND ND, ND	ND ND ND ND ND ND	ND, ND ND, ND ND, ND ND, ND ND, ND ND, ND	ND ND ND ND ND ND	ND, ND ND, ND ND, ND ND, ND ND, ND ND, ND	ND ND ND ND ND ND
USA, 2012 Hobe Sound, FL (Snowden) DP-31762-Trial 05	35	445-460	141	5	ND, ND	ND	ND, ND	ND	ND, ND	ND
USA, 2012 Jerome, ID (Ranger Russet) DP-31762-Trial 11	50	290-300	200	5	ND, ND	ND	ND, ND	ND	ND, ND	ND
USA, 2012 Jerome, ID (Red Pontiac) DP-31762-Trial 15	49-51	300-310	200	5	ND, ND	ND	ND, ND	ND	ND, ND	ND
USA, 2012 Minidoka, ID (Russet Norkotah TX296) DP-31762-Trial 19	49-52	340-415	201	5	ND, ND	ND	ND, ND	ND	ND, ND	ND
USA, 2012 Payette, ID (Dark Red Norland) DP-31762-Trial 17 [not independent]	50-51	330	202	-0 0 3 5 10 14	ND, ND ND, ND ND, ND ND, ND ND, ND ND, ND	ND ND ND ND ND ND	ND, ND ND, ND ND, ND ND, ND ND, ND ND, ND	ND ND ND ND ND ND	ND, ND ND, ND ND, ND ND, ND ND, ND ND, ND	ND ND ND ND ND ND
USA, 2012 Payette, ID (Russet Norkotah) DP-31762-Trial 16	49-51	320-330	199	5	ND, ND	ND	ND, ND	ND	ND, ND	ND
USA, 2012 Porterville, CA (Russet Norkotah 296) DP-31762-Trial 12	35	360-365	140	5	ND, ND	ND	ND, ND	ND	ND, ND	ND
USA, 2012 Verona, WI (Superior) DP-31762-Trial 10	67 49-54	543 385-395	219	5	ND, ND	ND	ND, ND	ND	ND, ND	ND

Ginseng

In supervised trials on ginseng conducted in North America in 2011, two foliar applications of oxathiapiprolin (OD formulations) with added adjuvant were applied 14–15 days apart in late summer

(close-to-harvest) using pressurised backpack sprayers with 4-nozzle booms. In two of these trials, additional applications (2) were made in spring/early summer, 56 and 85 days before the close-to-harvest treatments.

Duplicate samples (minimum of 1 kg from at least 12 plants) were washed and commercially dried at about 35 °C for 3 days (to achieve a moisture content of 10–30%) before being frozen and stored for up to 16 weeks before analysis for oxathiapiprolin and its DPX-QGU42, INQ7H09, IN-RDG40, IN-E8S72, IN-RZB20, IN-RZD74, IN-SXS67, and IN-WR791 metabolites using method DuPont-30422 Supplement 1 (LC-MS/MS). Mean recovery rates for each analyte ranged from 80% to 111% in samples spiked with 0.01 to 0.5 mg/kg. The LOQs were 0.01 mg/kg for all analytes.

Table 94 Residues in dried ginseng roots from supervised trials in North America involving two spring/summer foliar applications of oxathiapiprolin (OD formulations) with adjuvant, applied at 14–15 day intervals.

GINSENG Country, year Location (Variety) Trial Ref	Application			Matrix	DAT	Residues (mg/kg) [a]			
	g ai/ha	water (L/ha)	season total (g ai/ha)			Oxathiapiprolin		IN-WR791	
						values	mean	values	mean
GAP: USA	35-280		560	PHI: 14d		14 day RTI			
Canada, 2011 Delhi, ON (Land Race) DP IR-4 PR-10616-ON19-02	286 277	1437 1391	563	dried roots	13	0.042, 0.056	0.049	< 0.01, < 0.01	< 0.01
USA, 2011 Eldron, WI (American Ginseng) DP IR-4 PR-10616-MI33	281 289	802 825	571	dried roots	13	0.044, 0.044	0.044	ND, ND	ND
USA, 2011 Mosinee, WI (American Ginseng) DP IR-4 PR-10616-MI34-02	285 286	525 528	571	dried roots	14	0.043, 0.044	0.043	0.01, < 0.01	< 0.01
USA, 2011 Wausau, WI (American Ginseng) DP IR-4 PR-10616-MI32	279 269	1073 1037	548	dried roots	0 6 13 20	0.026, 0.025 0.033, 0.054 0.058, 0.025 0.053, 0.07	0.026 0.044 0.041 0.061	ND, ND ND, ND ND, ND ND, ND	ND ND ND ND

[a] Residues of metabolites IN-RDG40, IN-E8S72, IN-RZB20, IN-RZD74, IN-SXS67 and IN-Q7H09 were <LOD in any sample

Table 95 Residues in dried ginseng roots from supervised trials in North America involving two spring/summer and two late-season foliar applications of oxathiapiprolin (OD formulations)

GINSENG Country, year Location (Variety) Trial Ref	Application			DAT	Residues (mg/kg) [a]					
	g ai/ha	water (L/ha)	season total (g ai/ha)		Oxathiapiprolin		IN-E8S72[b]	IN-RZD74[b]	IN-SXS67[b]	IN-WR791[b]
					values	mean	mean	mean	mean	mean
GAP: USA	35-280		560	PHI: 14d	14 day RTI					
USA, 2011 Mosinee, WI (American Ginseng) DP IR-4 PR-10616-MI34—03 85d between 2nd and 3rd sprays	277 271 + 276 278	512 501 + 510 514	1102	14	0.079, 0.064	0.072	0.02	< 0.01	< 0.01	0.028

GINSENG Country, year Location (Variety) Trial Ref	Application			DAT	Residues (mg/kg) [a]					
	g ai/ha	water (L/ha)	season total (g ai/ha)		Oxathiapiprolin		IN-E8S72 [b]	IN-RZD74 [b]	IN-SXS67 [b]	IN-WR791 [b]
					values	mean	mean	mean	mean	mean
Canada, 2011 Delhi, ON (Land Race) DP IR-4 PR-10616-ON19-03 56d between 2nd and 3rd sprays	280 280 + 281 284	1406 1406 + 1412 1423	1125	13	0.13, 0.15	0.14	< 0.01	ND	ND	0.011

[a] Residues of metabolites IN-Q7H09, IN-RDG40 and IN-E8S72 were <LOD in any sample

[b] Mean values from two duplicate analyses

FATE OF RESIDUES IN STORAGE AND PROCESSING

Nature of the residue during processing

High-temperature hydrolysis

High-temperature hydrolysis of oxathiapiprolin was investigated by Anand, 2010) [Ref: DP-29273]. In the study, solutions of 0.1 mg/L [pyrazole-^{14}C]-oxathiapiprolin and [thiazole-^{14}C]-oxathiapiprolin were prepared prepared in 0.01 M citrate buffer (pH 4, 5, and 6) with 1% co-solvent (DMF) and incubated in the dark, 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.).

Samples were analysed at zero time, after 20 min. (pH 4 and 6), and 1 h (pH 5) by LSC and HPLC. Identification of parent was by co-chromatography with authentic standard. The limit of quantification (LOQ) for both labels was 2% AR.

Recovery of the applied radioactivity (AR) ranged from 94.7 to 97.2% for both radiolabels and no hydrolysis products were detected. Oxathiapiprolin was shown to be stable under conditions simulating pasteurisation, baking, brewing, boiling and sterilisation.

Table 96 Distribution of radioactivity for [^{14}C]-oxathiapiprolin in buffer solutions – high temperature hydrolysis (% radioactivity)

Component	pH 4 (90 °C)		pH 5 (100 °C)		pH 6 (120 °C)	
	Time 0	20 minute	Time 0	60 minutes	Time 0	20 minute
[pyrazole-^{14}C]-oxathiapiprolin						
Oxathiapiprolin	96.0	94.8	95.9	94.7	95.0	93.8
Others [a]	ND[c]	ND	ND	ND	ND	ND
Total % recovery [b]	96.0	94.8	95.9	94.7	95.0	93.8
[thiazole-5-^{14}C]-oxathiapiprolin						
Oxathiapiprolin	97.2	96.2	96.0	95.1	95.3	94.2
Others [a]	ND[c]	ND	ND	ND	ND	ND
Total % recovery [b]	97.2	96.2	96.0	95.1	95.3	94.2

[a] Sum of any minor peaks

[b] Recovery of administered radioactivity as determined by LSC analyses of the dosing solution.

Peel and pulp residue distribution

Melon

In a series of field trials conducted in Europe and USA on melons, residues in whole fruit, pulp and in peel were analysed separately to investigate the distribution of residues of oxathiapiprolin and metabolites. These results are reported in Tables 71–72.

In melons sampled from 0–10 days after the last application, residues of oxathiapiprolin in whole fruit ranged from < 0.01 mg/kg to 0.09 mg/kg and residues in the pulp were either not detectable or < 0.01 mg/kg (LOQ) in all samples. Residues of the metabolites IN-Q7H09, IN-RDG40, IN-E8S72, IN-WR791, IN-RZB20, IN-RZD74, and IN-SXS67 were also not detected (< 0.003 mg/kg) in any melon pulp samples.

Household washing, trimming and cooking

Brassica and leafy vegetables

In a series of field trials conducted in North America, residues in cabbage broccoli and cauliflower heads were measured before and after washing under cold water for 15–20 seconds. Details on these trials are reported below.

Table 97 Residues before and after washing broccoli, cauliflower and cabbage heads and lettuce from supervised trials in North America involving 4 foliar applications of oxathiapiprolin (OD formulations) with adjuvant

Country, year Location (Variety) Trial Ref	Application			Matrix	DAT	Residues (mg/kg) [a]					
						Oxathiapiprolin		IN-WR791		IN-E8S72	
	g ai/ha	water (L/ha)	season total (g ai/ha)			values	mean	values	mean	values	mean
Broccoli											
USA, 2011 Alton, NY Broccoli (Bay Meadow) DP-32067-Trial 01	35	280	140	head	0	0.84, 0.78	0.81	ND, ND	ND	ND, ND	ND
				washed head	0	0.13, 0.095	0.11	< 0.01, < 0.01	< 0.01	ND, ND	ND
USA, 2011 King City, CA Broccoli (Patron) DP-32067-Trial 05	35	370	140	head	0	0.056, 0.076	0.066	< 0.01, < 0.01	< 0.01	ND, ND	ND
				washed head	0	0.057, 0.092	0.075	< 0.01, < 0.01	< 0.01	ND, ND	ND
Cauliflower											
Canada, 2011 St. Marc-sur- Richelieu, QC Cauliflower (Freemont) DP-32067-Trial 03	35-36	250	143	head	0	0.076, 0.084	0.08	< 0.01, ND	ND	ND, ND	ND
				washed head	0	0.073, 0.086	0.079	ND, < 0.01	ND	ND, ND	ND
Cabbage											
USA, 2011 Germansville, PA Cabbage (Blue Lagoon) DP-32067-Trial 10	35-36	215	142	head	0	0.11, 0.14	0.12	< 0.01, ND	ND	ND, ND	ND
				washed head	0	0.093, 0.11	0.1	< 0.01, ND	ND	ND, ND	ND
USA, 2011 Oviedo, FL Cabbage (Copenhagen) DP-32067-Trial 13	34-35	280	137	head	0	0.46, 0.38	0.42	< 0.01, < 0.01	< 0.01	ND, ND	ND
				washed head	0	0.29, 0.26	0.28	< 0.01, < 0.01	< 0.01	ND, ND	ND

Country, year Location (Variety) Trial Ref	Application			Matrix	DAT	Residues (mg/kg) [a]					
						Oxathiapiprolin		IN-WR791		IN-E8S72	
	g ai/ha	water (L/ha)	season total (g ai/ha)			values	mean	values	mean	values	mean
USA, 2011 Rochelle, IL Cabbage (Blue Vantage) DP-32067-Trial 14	35-36	290	142	head washed head	0 0	0.17, 0.16 < 0.01, 0.01	0.16 < 0.01	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND
Head lettuce (with wrapper leaves)											
USA, 2011 Citra, FL (Head Lettuce/) PR-10653-FL21	36	280-285	144	leaves washed, chopped	0 0	0.85, 0.56 0.15, 0.42	0.7 0.28	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND
USA, 2011 Las Cruces, NM (Salinas) PR-10653-NM12	34-36	220-280	141	leaves washed	0 0	0.2, 0.25 < 0.01, < 0.01	0.23 0.006	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND
Canada, 2011) (Harrow, ON (Caliente) PR-10653-ON27	35-36	400-410	141	leaves washed	0 0	0.49, 0.27 0.16, 0.12	0.38 0.14	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND
Canada, 2011 Harrow, ON (Odyssey) PR-10653-ON28	35-36	405-415	143	leaves washed	0 0	0.49, 0.52 0.13, 0.13	0.5 0.13	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND
Leaf lettuce											
USA, 2011 Citra, FL (Two star) PR-10653-FL20	35-36	660-675	141	leaves washed, chopped	0 0	1.9, 2.1 2.0, 1.8	2 1.9	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND
USA, 2011) (Las Cruces, NM (Oakleaf) PR-10653-NM11	35-37	375-425	142	leaves washed	0 0	1.9, 1.8 0.59, 0.84	1.9 0.72	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND
Spinach											
USA, 2011 Chula, GA (Vancouver) DP-31730-Trial 02	35-36	225-360	141.1	leaves washed	0 0	2.1, 2.2 1.5, 1.5	2.2 1.5	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND
Canada, 2011 Puslinch, ON (Greyhound) DP-31730-Trial 04	36	200	144.7	leaves washed	0 0	1.4, 1.3 1.0, 1.0	1.4 1.0	ND, ND ND, ND	ND ND	ND, ND ND, ND	ND ND
USA, 2011 Uvalde, TX (DMC6607) DP-31730-Trial 05	34-36	187	140	leaves washed	0 0	2..6, 2.1 2.1, 2.7	2.3 2.4	< 0.01, < 0.01 < 0.01, ND	0.004 ND	ND, ND ND, ND	ND ND

[a] Residues of metabolites IN-Q7H09, IN-RDG40, IN-RZB20, IN-RZD74 and IN-SXS67 were ND in all samples

The median or average processing factors for oxathiapiprolin in cabbage, cauliflower/broccoli, head lettuce, leaf lettuce, and spinach prepared for consumption range from 0.3 to 1.0. Residues of the metabolites IN-Q7H09, IN-RDG40, IN-E8S72, IN-WR791, IN-RZB20, IN-RZD74, and IN-SXS67 were < 0.01 mg/kg in these washed vegetables.

Residues after processing

The Meeting received processing studies on grapes, potatoes and tomatoes, information on the effects of washing (brassica and leafy vegetables) and on the residue distribution in peel and flesh (melons).

Grape

In supervised trials on grapes conducted in Europe and reported by Spence & Woodmansey, 2012 [Ref: DP-30045], three foliar applications of 50 g ai/ha (or 180 g ai/ha oxathiapiprolin (OD formulations) were applied using single nozzle back-pack sprayers or mist blowers, 9–12 days apart with the last application 19–21 days before harvest.

Grape bunches (min 50 kg) were collected, shipped under ambient conditions and processed into juice, wine and raisin within 24 hours of sampling.

Juice was prepared by manually stemming and crushing the bunches, adding pectolytic enzymes, heating to 50 °C and sterilising for 2 hours (in jars) at 45–60 °C before pressing. The raw juice collected from the press was clarified by heating for 5 minutes at about 85 °C, chilling (to below 10 °C) and the clarified juice decanted, filtered and pasteurised.

Grapes were processed into raisins after stemming by soaking in a potassium bicarbonate solution (50 g/L) for about 1 minute and oven-drying at 60 °C until at least 35% moisture had been removed (30–48 hours).

Red wine was produced by crushing and stemming the grape bunches and adding potassium metabisulphite (0.06 g/L) and dry yeast to initiate fermentation. This was was monitored (with sugar added) until completion (when the wine density had stabilised below 1000) and the must was pressed to obtain the AF (after alcoholic fermentation) wine. Malolactic fermentation (MF) was accelerated by inoculation with lactic bacteria and once completed, 0.1 g/L potassium metabisulphate was added to the wine. After racking for at least 4 days, the wine was seperated from the lees and further clarified by racking and filtration after the addition of dry gelatine (0.1 g/L) and potassium metabisulphate (0.04 g/L).

The same process was used to produce white wine, but with the crushed grapes being first pressed, the must treated with pectolytic enzymes (0.02 g/L) and potassium metabisulphite (0.12 g/L), allowed to settle for 24 hours before decanting and the addition of the yeats.

Samples of grapes, raisins, juice, wine and other processing fractions frozen and stored for up to 13 months before analysis for oxathiapiprolin, IN-RDG40, IN-Q7H09, IN-E8S72 and IN-WR791 using DuPont method 30422. Mean recovery rates for each analyte ranged from 93% to 117% in samples spiked with 0.01–7.0 mg/kg. The LOQs were 0.01 mg/kg for all analytes.

Table 98 Residues in fresh and processed grapes from supervised trials in Europe involving three foliar applications of oxathiapiprolin (OD formulations) and PHIs of 19–21 days.

GRAPES Country, year Location (Variety) Trial Ref	Application		Matrix	Average Residues (mg/kg)					Processing factors
	g ai/ha	water (L/ha)		Oxathia-piprolin	IN-RDG40	IN-Q7H09	IN-E8S72	IN-WR791	Oxathia-piprolin
Germany, 2010 Saxony-Anhalt, (White Wine Grapes/ Müller-Thurgau DP-30045-Test-01	50	1575-1610	Grapes	0.14[(a)]	ND [a]	ND [a]	ND [a]	< 0.01 [a]	
			Stems	0.84	< 0.01	0.022	ND	< 0.01	6.0
			Raw juice	0.036	ND	ND	ND	< 0.01	0.26
			Wet pomace [b]	0.43	ND	< 0.01	ND	< 0.01	3.1
			Grape juice	0.018	ND	ND	ND	< 0.01	0.13
			Raisins	0.57	ND	0.015	ND	0.022	4.1
			Must	0.064	ND	ND	ND	ND	0.46
			Wet pomace [c]	0.28	ND	< 0.01	ND	< 0.01	2.0
			Dry pomace	2.3	ND	0.021	ND	0.024	16
			AF wine	0.032	ND	ND	ND	< 0.01	0.23
			MF wine	0.026	ND	ND	ND	< 0.01	0.19
			White wine	0.024	ND	ND	ND	< 0.01	0.17

GRAPES Country, year Location (Variety) Trial Ref	Application g ai/ha	Application water (L/ha)	Matrix	Average Residues (mg/kg) Oxathia-piprolin	IN-RDG40	IN-Q7H09	IN-E8S72	IN-WR791	Processing factors Oxathia-piprolin
France, 2010	180	350	Grapes	0.39[a]	ND[a]	ND[a]	< 0.01 [a]	0.024[a]	
Rhone Alpes			Stems	0.7	ND	0.014	< 0.01	0.062	1.8
(Red Wine			Raw juice	0.11	ND	ND	ND	0.027	0.28
Grapes/ Gamay)			Wet pomace [b]	0.76	ND	ND	< 0.01	0.036	1.9
			Grape juice	0.071	ND	ND	< 0.01	0.039	0.18
DP-30045-Test-02			Raisins	0.62	ND	ND	0.01	0.13	1.6
			Must	0.24	ND	ND	< 0.01	0.024	0.62
			Wet pomace [c]	2.1	ND	0.011	< 0.01	0.046	5.4
			Dry pomace	6.5	0.011	0.042	0.022	0.19	17
			AF wine	0.072	ND	ND	< 0.01	0.033	0.18
			MF wine	0.04	ND	ND	< 0.01	0.031	0.1
			Red wine	0.038	ND	ND	< 0.01	0.034	0.1
France, 2011	50	300	Grapes	0.085[a]	ND[a]	ND[a]	ND[a]	< 0.01 [a]	
Vineuse,			Stems	0.25	ND	ND	ND	< 0.01	2.9
Bourgogne			Raw juice	0.023	ND	ND	ND	ND	0.27
White Wine			Wet pomace [b]	0.088	ND	ND	ND	< 0.01	1.0
Grapes/			Grape juice	0.012	ND	ND	ND	< 0.01	0.14
Chardonnay)			Raisins	0.11	ND	ND	ND	0.01	1.3
			Must	0.049	ND	ND	ND	< 0.01	0.58
DP-30045-Test-03			Wet pomace [c]	0.13	ND	ND	ND	< 0.01	1.5
			Dry pomace	0.3	ND	ND	ND	0.012	3.5
			AF wine	0.009	ND	ND	ND	< 0.01	0.1
			MF wine	0.008	ND	ND	ND	ND	0.09
			White wine	0.007	ND	ND	ND	ND	0.08
Spain, 2011	50	1590-1630	Grapes	0.14 [a]	ND [a]	ND [a]	ND [a]	< 0.01 [a]	
Verdu,			Stems	0.35	ND	ND	ND	0.018	2.5
Catalunya			Raw juice	0.064	ND	ND	ND	< 0.01	0.46
(Red Wine			Wet pomace [b]	0.19	ND	ND	ND	< 0.01	1.4
Grapes/			Grape juice	0.031	ND	ND	ND	< 0.01	0.22
Tempranillo)			Raisins	0.13	ND	ND	ND	0.015	0.93
			Must	0.27	ND	ND	ND	< 0.01	1.9
DP-30045-Test-04			Wet pomace [c]	0.56	ND	ND	ND	< 0.01	4.0
			Dry pomace	1.7	ND	ND	ND	0.025	12
			AF wine	0.047	ND	ND	ND	< 0.01	0.34
			MF wine	0.026	ND	ND	ND	0.01	0.19
			Red wine	0.025	ND	ND	ND	0.01	0.18

[a] Mean residues from 3 replicate samples

[b] Wet pomace after juice extraction

[c] Wet pomace from wine production

Potato

In a study reported by Shephard, 2012 [Ref: DP-31987], potatoes from three field trials in USA were cooked or processed into flakes, chips and French fries and residues of oxathiapiprolin and its metabolites were measured in these processed commodities and other processing fractions.

In these trials, 1.4–1.6 kg ai/ha oxathiapiprolin were applied as in-furrow treatments at planting (as a seed-piece treatment in one trial) followed by foliar application of 1.4 kg ai/ha when the tubers were half-grown and the start of flowering and four further foliar applications about a further five foliar pre-harvest applications at 5–6 day intervals until 5 days before harvest.

Duplicate samples of 75 kg potatoes were shipped under ambient conditions and cooked or processed into flakes, chips or French fries within 24 hours of sampling.

Potato tubers were cleaned, washed, peeled with an steam/abrasive peeler. The potato peel was collected, pressed hydraulically in a fruit press and blended with trim waste. The peeled potatoes were cut into slabs (1–1.5 cm thick) and spray-washed with cold water to remove free starch. The

slabs were then pre-cooked at 70–77 °C in a steam-jacketed kettle for about 20 minutes. The pre-cooked slabs were cooled to less than 32 °C then steam-cooked at atmospheric pressure at 94–100 °C for 40 minutes. The cooked potato slabs were then mashed, mixed with additives and fed into a drum dryer to produce a thin sheet, which was initially broken into large flakes by hand. The flakes were then fed into a hammermill for uniform milling of the flakes.

Chips were prepared from the peeled potatoes by slicing into 1.6 mm chips using a food cutter, rinsing in warm water to remove free starch, and frying for about 90 seconds at 163–191 °C.

French fries were prepared by pre-cooking peeled potatoes in water at 54 °C for 40 minutes, cutting them into 5 mm strips using a food cutter, spray-washing them to remove free starch, blanching them in 79–85 °C for 5 minutes and dipping them in a solution of sodium pyrophosphate (0.5%) and dextrose (0.5%) for 30 seconds at 71–74 °C. The strips were then air-dried to about 15% moisture content and par-fried in vegetable oil for 45–50 seconds at 188–191 °C. the fried strips were then drained and cooled before being packed and frozen.

Field samples were taken from each trial and cool-stored at or below 10 °C, with samples being taken for analysis after 7, 30, 90 and 180 days.

Samples were frozen and stored for up to 40 weeks before analysis for oxathiapiprolin, IN-Q7H09, IN-RDG40, IN E8S72, IN-RZB20, IN RZD74, IN-SXS67, and IN WR791 using DuPont method 30422 Supplement 1. Mean recovery rates for each analyte ranged from 90% to 125% in samples spiked with 0.01–5.0 mg/kg. The LOQs were 0.01 mg/kg for all analytes.

Table 98 Residues in potatoes and processed commodities from supervised trials in USA involving exaggerated in-furrow and foliar applications of oxathiapiprolin and PHIs of 5 days

POTATO Country, year Location (Variety)	Application		Matrix	Residues (mg/kg)				Processing Factors
	method	kg ai/ha		oxathiapiprolin	IN-E8S72	IN-SXS67	IN-WR791	Oxathiapiprolin
USA, 2011	soil	1.4	Tubers [b]	0.037 [c]	ND [c]	ND [c]	< 0.01 [c]	
North Rose, NY	foliar	1.4	Washed tubers [b]	0.026	ND	ND	< 0.01	0.7
(Genesee)	4×foliar	0.175	Culls	0.026	ND	ND	< 0.01	0.7
			Steam-peeled tubers	ND	ND	ND	< 0.01	< 0.27
DP-31987-Trial 01			Abrasion-peeled tubers	ND	ND	ND	< 0.01	< 0.27
				0.13	ND	ND	< 0.01	3.5
			Abrasive waste	ND	< 0.01	ND	0.019	< 0.27
			Potato dried flakes	ND	< 0.01	ND	0.018	< 0.27
			Potato chips	ND	ND	ND	< 0.01	< 0.27
			French fries	< 0.01	ND	ND	0.011	< 0.27
			Unpeeled French fries	< 0.01	ND	ND	< 0.01	< 0.27
			Boiled [b]	ND	ND	ND	< 0.01	< 0.27
			Boiled peeled	0.016	ND	ND	< 0.01	0.43
			Microwaved [b]	0.034	ND	ND	< 0.01	
			Stored tubers [b]	0.046	ND	ND	< 0.01	
			7d	0.024	ND	ND	< 0.01	
			30d	0.028	ND	ND	< 0.01	
			90d	0.048	ND	ND	< 0.01	
			180d					

POTATO Country, year Location (Variety)	Application		Matrix	Residues (mg/kg)				Processing Factors
	method	kg ai/ha		oxathiapiprolin	IN-E8S72	IN-SXS67	IN-WR791	Oxathiapiprolin
USA, 2011 Gardner, ND (Gold Rush) DP-31987-Trial 02	seed foliar 4×foliar	1.6 1.4 0.175	Tubers [b] Washed tubers [b] Culls Steam-peeled tubers Abrasion-peeled tubers Abrasive waste Potato dried flakes Potato chips French fries Unpeeled French fries Boiled [b] Boiled peeled Microwaved [b] Stored tubers [b] 7d 30d 90d 180d	0.59 [c] 0.019 0.057 ND ND 0.15 ND ND 0.024 0.016 ND 0.03 0.37 0.49 0.12 1.3 0.23	ND [c] ND ND ND ND ND < 0.01 < 0.01 ND < 0.01 ND ND ND < 0.01 ND < 0.01 ND	ND [c] ND ND ND ND ND ND ND ND ND ND ND ND ND ND ND ND	< 0.01 [c] < 0.01 < 0.01 < 0.01 < 0.01 ND < 0.01 ND < 0.01 < 0.01 ND < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.03 0.1 < 0.02 < 0.02 0.25 < 0.02 < 0.02 < 0.02 0.04 0.03 < 0.02 0.05
USA, 2011 Jerome, ID (Gold Rush) DP-31987-Trial 03	soil foliar 4×foliar	1.4 1.4 0.172	Tubers [b] Washed tubers Culls Steam-peeled tubers Abrasion-peeled tubers Abrasive waste Potato dried flakes Potato chips French fries Unpeeled French fries Boiled [b] Boiled peeled Microwaved [b] Stored tubers [b] 30d 90d 180d	0.087 [c] < 0.01 0.011 ND ND 0.031 ND ND < 0.01 < 0.01 ND < 0.01 0.062 0.04 0.038 0.022	0.046 [c] 0.037 0.032 0.053 0.063 0.021 0.11 0.068 0.041 0.075 0.04 0.037 0.044 0.025 0.058 0.057 0.046	ND [c] ND ND ND ND ND ND ND ND < 0.01 ND ND ND ND ND ND ND	0.011 [c] < 0.01 < 0.01 0.013 0.011 < 0.01 0.025 0.013 < 0.01 0.02 0.011 < 0.01 0.01 < 0.01 0.012 0.015 0.012	< 0.11 0.13 < 0.11 < 0.11 0.36 < 0.11 < 0.11 < 0.11 < 0.11 < 0.11 < 0.11 < 0.11

[a] Residues of metabolites IN-Q7H09, IN-RDG40, IN-RZB20 and IN-RZD74, were ND in all samples

[b] Potatoes with peel

[c] Mean residues from 3 replicate samples (In Trial 02, oxathiapiprolin, individual values are 0.097, 1.5, 0.15 mg/kg)

Tomato

In a study reported by Shepard, 2012 [Ref: DP-31728], tomatoes were taken for processing from three field trials conducted in USA. In these field trials, two applications of 0.28 kg ai/ha (7 days apart) were made as directed sprays to the base of the tomato plants between 20–40 days before harvest and were followed by three foliar sprays of 0.175 kg ai/ha (with added adjuvant) applied at 5–6 day intervals until harvest.

Bulk samples of 100 kg ripe tomatoes were taken on the day of the last application, chilled to about 5–8 °C for 1–2 days before processing into juice, paste, puree, canned and dried tomatoes.

Tomatoes (without stems) were soaked in a dilute solution of sodium hydroxide for 3 minutes at 52–60 °C and rinsed in warm water.

For puree, paste and juice, the washed tomatoes were crushed and rapidly heated to 79–85 °C for 15–30 seconds and pressed to separate the hot-break juice and pomace, with the hot-break juice

being vacuum evaporated and adjusted to 12–13 °Bx (degrees Brix) (with added salt) to produce puree and to 24–33 °Bx to produce paste. Juice was prepared by diluting the paste to 4.5–5.5 °Bx and adding 0.5% salt. The puree, paste and juice were then heated to 82–88 °C, canned, immersed in a boiling water bath for about 15 minutes and cooled before labelling and freezing for subsequent analysis.

Canned tomatoes were prepared by boiling the tomatoes for about a minute (to crack the skins), manually removing the skins, cores and stems and placing the whole peeled tomatoes (with added salt) in cans. The canned tomatoes were then steam exhausted for about 10 minutes until a temperature of about 80 °C was reached, the cans were sealed and placed in boiling water for about 15 minutes before cooling , labelling and freezing for subsequent analysis.

Sun-dried tomatoes were prepared by manually removing the cores and stems, quartering the fruit and oven drying them until a moisture content of 16% had been achieved.

Samples were frozen and stored for up to 26 weeks before analysis for oxathiapiprolin, IN-Q7H09, IN-RDG40, IN E8S72, IN-RZB20, IN RZD74, IN-SXS67, and IN-WR791 using DuPont method 30422 Supplement 1. Mean recovery rates for each analyte ranged from 86% to 122% in samples spiked with 0.01–5.4 mg/kg. The LOQs were 0.01 mg/kg for all analytes.

Table 99 Residues in tomatoes and processed commodities from supervised trials in USA involving exaggerated foliar applications of oxathiapiprolin and PHIs of 0 days

TOMATO Country, year Location (Variety)	Application		Matrix	Residues (mg/kg) [a]		Processing factors
	method	kg ai/ha		oxathiapiprolin	IN-WR791	oxathiapiprolin
USA, 2011	2×directed basal	0.28	Tomatoes	0.42 [b]	ND [b]	
North Rose, NY	4×foliar	0.17-0.18	Washed	0.19	ND	0.45
(Early Girl)			Peeled	ND	ND	< 0.02
			Sun-dried	2.9	0.015	6.9
DP-31728-Trial 01			Canned (peeled)	ND	ND	< 0.02
			Juice	0.12	ND	0.29
			Wet pomace	4.8	ND	11
			Paste	0.44	< 0.01	1.1
			Puree	0.25	ND	0.6
USA, 2011	2×basal	0.28	Tomatoes	0.26[b]	ND[b)]	
Marysville, OH	4×foliar	0.175	Washed	0.12	ND	0.46
(La Roma II)			Peeled	ND	ND	< 0.04
			Sun-dried	0.76	< 0.01	2.9
DP-31728-Trial 02			Canned (peeled)	ND	ND	< 0.04
			Juice	0.042	ND	0.16
			Wet pomace	3.5	ND	13
			Paste	0.18	< 0.01	0.69
			Puree	0.067	ND	0.26
USA, 2011	2×basal	0.28	Tomatoes	0.23 [b]	ND [b]	
Corning, CA	4×foliar	0.177	Washed	0.087	ND	0.38
(Sun 6366)			Peeled	0.011	ND	0.05
			Sun-dried	1.7	0.022	7.4
DP-31728-Trial 03			Canned (peeled)	ND	< 0.01	< 0.04
			Juice	0.037	ND	0.16
			Wet pomace	3.0	< 0.01	13
			Paste	0.26	0.011	1.1
			Puree	0.15	< 0.01	0.65

[a] Residues of metabolites IN-Q7H09, IN-RDG40, IN-E8S72, IN-RZB20, IN-RZD74 and IN-SXS67 were ND in all samples

[b] Mean residues from 3 replicate samples

Summary of Processing Studies

In processing studies conducted on grapes, potatoes and tomatoes and simulating commercial practices, residues of oxathiapiprolin increased in commodities such as raisins, dried tomatoes and in

the grape and tomato pomaces and potato peel waste, where processing involves a reduction in moisture content. There was no concentration of oxathiapiprolin residues in the other processed commodities.

The more water-soluble IN-WR791 residues concentrated slightly or were conserved in juice, red wine and white wine, with median or average processing factors of 1.0–1.2 but also concentrated in raisins (median processing factor of 1.9). Residues were low (< 0.02 mg/kg) in other processed food commodities.

IN-Q7H09 and IN-E8S72 residues above the LOQ were measured in raisins (up to 0.015 mg/kg) and IN-E8S72 was also found in most processed potato commodities, up to 1.6 times the level measured in raw potato tubers (0.046 mg/kg) in peeled tubers, chips and unpeeled French fries.

Metabolites IN-RDG40, IN-RZB20, IN RZD74 and IN-E8S72 were either not detected or < 0.01 mg/kg in other processed commodities for human consumption.

Table 100 Summary of processing factors for oxathiapiprolin

RAC	Matrix	Oxathiapiprolin [a]	
		Calculated processing factors	PF median/best fit
Grape	Raw juice	0.26, 0.27, 0.28, 0.46	0.28
	Wet pomace [b]	1.0, 1.4, 1.5, 1.9, 2.0, 3.1, 4.0, 5.4	2.0
	Grape juice	0.13, 0.14, 0.18, 0.22	0.16
	Raisins	0.93, 1.3, 1.6, 4.1	1.4
	Dry pomace	3.5, 12, 16, 17	14
	AF wine	0.1, 0.18, 0.23, 0.34	0.2
	MF wine	0.09, 0.1, 0.19, 0.19	0.15
	Wine	0.08, 0.1, 0.17, 0.18	0.14
Potato	Tubers		
	Washed tubers [b]	0.03, < 0.11, 0.7	< 0.11
	Culls	0.1, 0.13, 0.7	0.13
	Steam-peeled tubers	< 0.02, < 0.11, < 0.27	< 0.11
	Abrasion-peeled tubers	< 0.02, < 0.11, < 0.27	< 0.11
	Abrasive waste	0.25, 0.36, 3.5	0.36
	Potato dried flakes	< 0.02, < 0.11, < 0.27	< 0.11
	Potato chips	< 0.02, < 0.11, < 0.27	< 0.11
	French fries	< 0.02, < 0.11, < 0.27	< 0.11
	Unpeeled French fries	0.04, < 0.11, < 0.27	< 0.11
	Boiled [b]	0.03, < 0.11, < 0.27	< 0.11
	Boiled peeled	< 0.02, < 0.11, < 0.27	< 0.11
	Microwaved[b]	0.05, < 0.11, 0.43	< 0.11
Tomato	Tomatoes		
	Washed	0.38, 0.45, 0.46	0.45
	Peeled	< 0.02, < 0.04, 0.05	< 0.04
	Sun-dried	2.9, 6.9, 7.4	6.9
	Canned (peeled)	< 0.02, < 0.04, < 0.04	< 0.04
	Juice	0.16, 0.16, 0.29	0.16
	Wet pomace	11, 13, 13	13
	Paste	0.69, 1.1, 1.1	1.1
	Puree	0.26, 0.6, 0.65	0.6

[a] Each value represents a separate study where residues were above the LOQ in the RAC. The factor is the ratio of oxathiapiprolin residues in the processed item divided by the residue of oxathiapiprolin in the RAC.

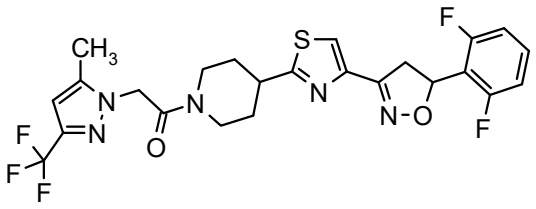
[b] Unpeeled tubers

APPRAISAL

Oxathiapiprolin is a systemic piperidinyl thiazole isoxazoline fungicide effective against oomycete pathogens, acting by inhibiting mycelial growth and zoospore release, encystment and mobility. It exhibits translaminar efficacy and gives systemic disease control following soil applications.

It was scheduled by the 47th Session of the CCPR as a new compound for consideration by the 2016 JMPR. The manufacturer submitted studies on metabolism, analytical methods, supervised field trials, processing, freezer storage stability and environmental fate in soil.

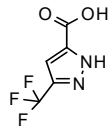
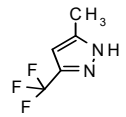
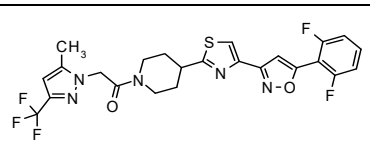
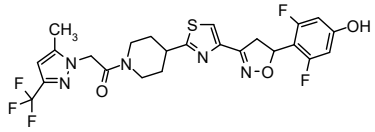
Authorisations exist for the use of oxathiapiprolin as foliar treatments or as soil treatments at planting (band/in-furrow or in transplant water) or via drip irrigation in a number of countries in Asia, the Pacific, Central and South America, and are pending in Europe and other countries. GAP information was available from Australia, China, New Zealand and North America.

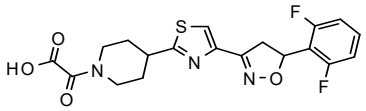
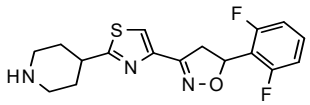
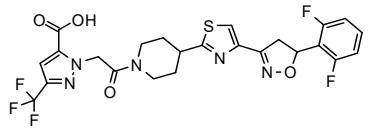
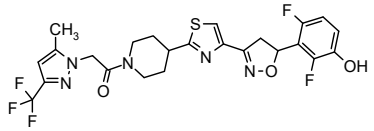
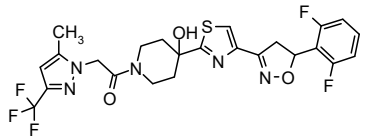
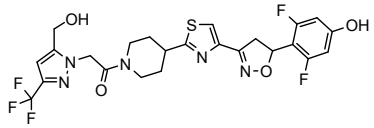
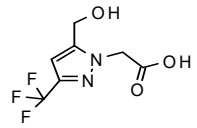
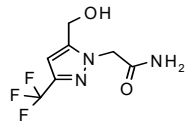
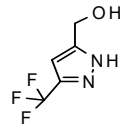
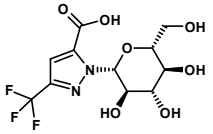


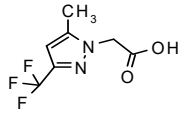
Oxathiapiprolin
(MW 539.53)

Oxathiapiprolin is a racemic mixture of (R)-oxathiapiprolin and (S)-oxathiapiprolin enantiomers with a low vapour pressure and water solubility (≈0.18 mg/L) that is not pH dependent. It is soluble in medium polarity organic solvents (e.g. dichloromethane, acetone or acetonitrile), but only slightly soluble in hexane. The octanol/water partition co-efficient (Log P_{OW} 3.6) is not pH dependent.

The following abbreviations are used for the major metabolites discussed below:

Code	Name and Matrix	Structure
IN-E8S72	5-(Trifluoromethyl)-1H-pyrazole-3-carboxylic acid Rat, plants, goat, soil	
IN-KJ552	3-Methyl-5-(trifluoromethyl)-1H-pyrazole Rat, plants	
IN-Q7D41	1-[4-[4-[5-(2,6-Difluorophenyl)-3-isoxazolyl]-2-thiazolyl]-1-piperidinyl]-2-[5-methyl-3-(trifluoromethyl)-1H-pyrazol-1-yl]ethanone Rat (transitory), goat, hen, soil, plants	
IN-Q7H09	1-[4-[4-[5-(2,6-Difluoro-4-hydroxyphenyl)-4,5-dihydro-3-isoxazolyl]-2-thiazolyl]-1-piperidinyl]-2-[5-methyl-3-(trifluoromethyl)-1H-pyrazol-1-yl]ethanone Rat, plants, hen, goat	

Code	Name and Matrix	Structure
IN-Q9L80	4-[4-[5-(2,6-Difluorophenyl)-4,5-dihydro-3-isoxazolyl)-2-thiazolyl]-α-oxo-1-piperidineactetic acid Rat, plants	
IN-QPS10	4-[4-[5-(2,6-Difluorophenyl)-4,5-dihydro-3-isoxazolyl)-2-thiazolyl]piperidine Plants, soil	
IN-RAB06	1-[2-[4-[4-[5-(2,6-Difluorophenyl)-4,5-dihydro-3-isoxazolyl]-2-thiazolyl]-1-piperidinyl]-2-oxoethyl]-3-(trifluoromethyl)-1H-pyrazole-5-carboxylic acid Rat, goat, hen, soil	
IN-RDG40	1-[4-[4-[5-(2,6-Difluoro-3-hydroxyphenyl)-4,5-dihydro-3-isoxazolyl]-2-thiazolyl]-1-piperidinyl]-2-[5-methyl-3-(trifluoromethyl)-1H-pyrazol-1-yl]ethanone Rat, plants, hen, goat	
IN-RDT31	1-[4-[4-[5-(2,6-Difluorophenyl)-4,5-dihydro-3-isoxazolyl]-2-thiazolyl]-4-hydroxy-1-piperidinyl]-2-[5-methyl-3-(trifluoromethyl)-1H-pyrazol-1-yl]ethanone Rat, soil	
IN-RLB67	1-[4-[4-[5-(2,6-Difluoro-4-hydrophenyl)-4,5-dihydro-3-isoxazolyl]-2-thiazolyl]-1-piperidinyl]-2-[5-(hydroxymethyl)-3-(trifluoromethyl)-1H-pyrazol-1-yl]ethanone Rat, goat	
IN-RZB20	5-(Hydroxymethyl)-3-(trifluoromethyl)-1H-pyrazole-1-acetic acid Rat, plants	
IN-RZB21	5-(Hydroxymethyl)-3-(trifluoromethyl)-1H-pyrazole-1-acetamide Plants	
IN-RZD74	3-(Trifluoromethyl)-1H-Pyrazole-5-methanol Plants, transient in goat, hen, rat	
IN-SXS67	1-ß-D-Glucopyranosyl-3-(-(trifluoromethyl)-1H-pyrazole-5-carboxylic acid Plants	

Code	Name and Matrix	Structure
IN-WR791	5-Methyl-3-(trifluoromethyl)-1H-pyrazole-1-acetic acid Plants, goat, rat, soil	

Plant metabolism

The Meeting received plant metabolism studies on potato, grape and lettuce following foliar applications of [^{14}C]oxathiapiprolin and on courgette, lettuce, wheat, turnips and potato grown in [^{14}C]oxathiapiprolin-treated soil.

Grapes-foliar applications

In a study on outdoor grapes, [^{14}C]oxathiapiprolin was applied to vines as three foliar treatments of 0.07 kg ai/ha, at flowering (BBCH 63–65), at BBCH 73 (cereal grain sized berries) and at BBCH 79 (pre-bunch-closure, adjacent berries on bunch beginning to touch). Foliage samples were taken at immediately after each treatment, 14 days after Treatments 2 and 3, and at grape maturity (76 DAT 3). Berry (grape) samples were taken 14 days after the second treatment, immediately after the third treatment, 14 days later and at grape maturity.

The majority of the radioactive residue in the berries and foliage was removed by surface washing and/or initial extraction (69–99% TRR). At maturity, 76 days after the third application, total radioactive residues were 0.30–0.32 mg eq/kg in berries and 1.1–1.38 mg eq/kg in foliage. Unextracted residues were about 10–16% TRR in foliage, 6.4–7.5% TRR in immature berries and about 15% TRR in mature berries.

Oxathiapiprolin was the major component identified in immature berries (36–74% TRR, 0.17–0.41 mg/kg) and accounted for 41% TRR (0.13 mg/kg) at maturity in [thiazole-^{14}C]-treated berries but was not the major residue in the [pyrazole-^{14}C]-treated berries, making up about 10% TRR (0.03 mg/kg). Chiral HPLC analysis showed no change in the isomer ratio over the study period.

In the [pyrazole-^{14}C]-treated mature berries, the polar pyrazole-specific metabolites IN-WR791 (19% TRR, 0.06 mg eq/kg) and IN-E8S72 (14% TRR, 0.04 mg eq/kg) were the predominant residues. Numerous minor metabolites were also identified or detected in berries, none of which were present at more than 6.2% TRR (0.02 mg eq/kg).

Oxathiapiprolin was also the predominant component identified in foliage, declining to 32–60% TRR (0.44–0.67 mg/kg) at grape maturity. Eleven minor identified metabolites and numerous unidentified metabolites were detected in foliage, the highest of which was IN-Q7H09, found at up to 5% TRR (0.36 mg eq/kg).

Lettuce-foliar applications

In a confined study on lettuce, three applications of 0.07 kg ai/ha [^{14}C]oxathiapiprolin were applied at 10 day intervals from BBCH 15 (5-leaf stage) to BBCH 19 (9-leaf stage) and leaves were sampled immediately after each application, 10 days after the first and second applications and 3, 7, 14 days after the last application.

The majority of the radioactive residue in the foliage was removed by surface washing and/or initial extraction (83–99.7% TRR). Unextracted residues in samples taken 7–14 days after treatment ranged from 7–14% TRR in the [pyrazole-^{14}C]-treated plants and from 14–17% TRR in the [thiazole-^{14}C]-treated plants.

Oxathiapiprolin was the major residue in all samples, making up 65% TRR (0.34 mg/kg) in the mature leaves (14 DAT 3) from the [pyrazole-^{14}C]-treated plants and 57% TRR (0.27 mg/kg) in

the leaves from the [thiazole-^{14}C]-treated plants. Chiral chromatographic analysis confirmed that the enantiomeric ratio remained unchanged over the study period.

The predominant metabolite in lettuce foliage was IN-Q7H09 (5.1% TRR–0.032 mg eq/kg). Other metabolites including multiple hydroxylated oxathiapiprolin compounds made up not more than 6% TRR (< 0.05 mg eq/kg).

Potato-foliar applications

In a confined study on potatoes, three applications of 0.07 kg ai/ha [^{14}C]oxathiapiprolin were applied just before flowering (BBCH 53), at first flowering (BBCH 59) and 14 days later (end of flowering, BBCH 69). Foliage samples (whole plants) were taken immediately after the first application, before and after the second and third (final) applications, and fourteen days after the final application. The final (maturity) harvest was taken 28 days after the final application. Potato tuber samples were taken before the third (final) treatment, 14 days after the final treatment (14 DAT 3) and at final harvest (28 DAT 3).

Radioactive residues in mature tubers were low (0.012 mg eq/kg [pyrazole-^{14}C] and 0.005 mg eq/kg [thiazole-^{14}C]) and were not investigated further. Unextracted tuber residues were < 0.01 mg eq/kg for both radiolabels. In foliage, the majority (75–96% TRR) of the radioactive residue was removed by surface washing and initial extraction (0.13–0.19 mg eq/kg extracted from mature foliage).

In foliage, oxathiapiprolin was the major residue component, accounting for 25–59% TRR, found in mature foliage samples at 0.04 mg/kg (pyrazole-label) and 0.11 mg/kg (thiazole-label). A range of minor metabolites were individually present at no greater than 8% TRR.

Potato-soil application

Seed potatoes were sown into a loam soil (3.1% organic matter, pH 5.3) immediately after a single soil application of [^{14}C]oxathiapiprolin at a rate equivalent to 0.6 kg ai/ha. Foliage and tubers were sampled 37 days after treatment (BBCH 65) and at maturity (BBCH 91), 72 days after treatment.

The total radioactive residues increased from 0.02–0.03 mg eq/kg (37DAT) to 0.05–0.11 mg eq/kg (72 DAT) in foliage and decreased from 0.01–0.02 mg eq/kg (37 DAT) to < 0.01 mg eq/kg (72 DAT) in tubers. More than 80% of the radioactive residue in tubers and foliage was extracted with acetonitrile, with unextracted residues being 0.003 mg eq/kg in tubers and up to 0.01 mg eq/kg in foliage. Extracts from tubers from the [isoxazoline ^{14}C]-oxathiapiprolin plots were low (up to 0.01 mg eq/kg) and were not investigated further.

Oxathiapiprolin was not a major residue, present at < 10% TRR and < 0.005 mg/kg in tubers and foliage. IN-WR791 was the major component identified in mature tubers (25% TRR and 0.003 mg eq/kg) with IN-E8S72 (14% TRR) and IN-RZB20 (12% TRR) also present at levels of 0.001–0.002 mg eq/kg. Unresolved IN-RZB21/IN-RZD74 and IN-RZB20 were the principal components identified in foliage (11–13% TRR, up to 0.015 mg eq/kg).

Lettuce-soil application

Lettuce seeds were sown into a loam soil (3.1% organic matter, pH 5.3) immediately after a single soil application [^{14}C]oxathiapiprolin at a rate equivalent to 0.6 kg ai/ha and leaves were sampled 44 days after treatment (BBCH 45) and at maturity, 57 days after treatment.

The total radioactive residues in lettuce samples grown in [isoxazoline-^{14}C]-oxathiapiprolin treated soil were less than the limit of quantification (0.01 mg eq/kg) and were not investigated further. TRR in lettuce samples grown in [pyrazole-^{14}C]-treated soil were < 0.02 mg eq/kg, with 88–91% TRR being removed in the initial acetonitrile extract and unextracted residues accounted for 9.5–12% TRR (0.002 mg eq/kg).

Oxathiapiprolin was not detected in any of the samples. Metabolites making up more than 10% TRR in immature and mature foliage samples were IN-WR791 (23% TRR and 30% TRR

respectively), IN-E8S72 (19–21%TRR) and IN RZB21/IN-RZD74 (19–21% TRR) but all individually found at 0.004 mg eq/kg or less.

Courgette-soil application

Courgette seeds were sown into a loam soil (3.1% organic matter, pH 5.3) immediately after a single soil application of either [^{14}C]oxathiapiprolin at a rate equivalent to 0.6 kg ai/ha with leaf and fruit samples being taken 44 days after treatment (BBCH 71) and at maturity, 79 days after treatment.

In samples taken from the plants grown in [pyrazole ^{14}C]-treated soil, total radioactive residues increased over time from 0.045 mg eq/kg (44DAT) to 0.17 mg eq/kg (79 DAT) in foliage and from 0.013 to 0.023 mg eq/kg in the corresponding fruit samples.

Conversely, the TRR in the foliage from [isoxazoline-^{14}C]-treated soil decreased over time from 0.028 mg eq/kg (44 DAT) to 0.008 mg eq/kg (79 DAT) and TRRs in fruit were below the limit of detection throughout the study (< 0.006 mg eq/kg). Residues in the mature fruit and leaf samples were < 0.01 mg eq/kg and not investigated further.

The majority (77–97%) of the radioactive residue in the foliage and fruit was extracted in the initial acetonitrile extracts. Unextracted residues were up to 23% TRR (0.006 mg eq/kg) in foliage (isoxazoline-label) and less than 10% TRR in fruit and in foliage from the pyrazole-labelled plants.

In fruit from plants grown in the [pyrazole-^{14}C]-treated soil, oxathiapiprolin was found at trace levels (0.5% TRR, < 0.001 mg/kg) in immature fruit but not in mature fruit.

IN-WR791 was the major component identified in fruit, found at 0.008 mg eq/kg (57% TRR) in immature fruit and 0.016 mg eq/kg (74% TRR) in mature fruit. Six other metabolites were identified at low levels (each < 5% TRR and up to 0.001 mg eq/kg).

In foliage, oxathiapiprolin was the predominant residue (24% TRR, 0.007 mg/kg) identified in the immature foliage grown in [isoxazoline-^{14}C]-treated soil and found in mature foliage from the [pyrazole-^{14}C]-treated soil at low levels (4.6% TRR, 0.008 mg/kg).

Major metabolites in immature foliage (44 DAT) were IN-WR791 (24% TRR, 0.011 mg eq/kg) and IN-E8S72 (24% TRR, 0.011 mg eq/kg). Other metabolites making up more than 10% TRR were IN Q7H09 (18.5% TRR), IN-RZB20 (17% TRR) and IN-RZB21/IN-RZD74 (13% TRR). None of these other metabolites exceeded 0.008 mg eq/kg. Unidentified components each did not exceed 2–3% TRR.

Major metabolites in mature foliage (79 DAT) were IN-WR791 (27.5% TRR, 0.047 mg eq/kg) and IN-E8S72 (21% TRR, 0.036 mg eq/kg). IN-RZB20 and IN-RZB21/IN-RZD74 were also present, each at about 0.02 mg eq/kg (11–12% TRR). Unidentified components each did not exceed 3% TRR.

In summary, when oxathiapiprolin was applied as a foliar treatment, parent compound was the major residue in lettuce, potato foliage, grape leaves and berries, making up 10–85% TRR. Chiral chromatographic analysis indicates that the enantiomeric ratio did not change over the study periods.

In grape berries, the significant metabolites were the cleavage products IN-WR791 (19% TRR, 0.6 mg eq/kg) and IN-E8S72 (14% TRR, 0.04 mg eq/kg). In grape leaves, lettuce and potato foliage, metabolites including IN Q7H09 (up to 0.36 mg eq/kg in grape leaves) were present in most samples, all individually present at less than 10% TRR.

Following soil applications, oxathiapiprolin is a minor component of the total residue in potato tubers, courgettes and foliage (lettuce, potato, grape, and courgette). Metabolites found above 10% TRR were pyrazole cleavage products (IN-WR791, IN-E8S72, IN-RZB21/IN-RZD74 and RZB20), each present at less than 0.05 mg eq/kg.

Animal metabolism

The Meeting received animal metabolism studies on rats, lactating goats and laying hens where animals were dosed with oxathiapiprolin radiolabelled in the pyrazole ring, the thiazole ring or the

isoxazoline ring (rat) and in lactating goats dosed with pyrazole-labelled IN-E8S72, a glucoside plant metabolite of oxathiapiprolin.

In rats, the metabolism of oxathiapiprolin was reviewed in the framework of the toxicological evaluation by the current Meeting.

Lactating goats were orally dosed with [^{14}C]oxathiapiprolin at rates equivalent to 14.2–14.3 ppm in the feed for 7 consecutive days and sacrificed 12 hours after the last dose.

The total recovery was 94–99.6% of the administered radioactivity (AR), with 84–86% being recovered from the urine, faeces, and cage wash, 8–12% found in the gastrointestinal tract, 0.1–0.2% in milk and 0.7–1% in edible tissues. Radioactivity plateaued in milk within 5 days.

Total radioactive residue levels were 0.01–0.02 mg eq/kg in milk, 0.75–0.97 mg eq/kg in liver, 0.07–0.08 mg eq/kg in kidney, 0.009–0.013 mg eq/kg in muscle and 0.025–0.03 mg eq/kg in fats.

Solvent extractions released 52–100% TRR from tissues and milk and subsequent enzymatic hydrolysis released another 26–29% TRR from liver.

Oxathiapiprolin accounted for 8.7–11% TRR (0.002 mg/kg) in milk, 6.4–12% TRR (0.05–0.11 mg/kg) in liver, 13–14% TRR (0.01 mg/kg) in kidney, 27–43% TRR (0.004 mg/kg) in muscle, and 36–58% TRR (< 0.016 mg/kg) in the fat fractions. Chiral chromatographic analysis confirmed that the oxathiapiprolin enantiomeric ratio remained unchanged over the study period.

The major metabolites in goat matrices were IN-E8S72 (maximum 24% TRR, 0.02 mg eq/kg in kidney) and the mono-hydroxy metabolites (including IN-RDG40 and IN-Q7H09) in liver (up to 0.13 mg/kg, 13% TRR) and in kidney (0.013 mg eq/kg, up to 21% TRR).

In a lactating goat study examining the fate of IN-SXS67 (the glucoside conjugate of IN-E8S72, not found in animals), one animal was dosed orally with 18.95 ppm [pyrazole-^{14}C]- IN-SXS67 in the diet for 7 days and the animal was sacrificed 6 hours after administration of the last dose.

Residues were rapidly excreted in urine and faeces (95–100% AR) and the total recovery of administered radiocarbon was 97.7%.

Unchanged IN-SXS67 made up 77% TRR (0.03 mg/kg) in liver and 58% TRR in kidney (0.28 mg/kg) while its aglycone metabolite, IN-E8S72 accounted for 16% TRR (0.006 mg eq/kg) in liver and 39% TRR (0.19 mg eq/kg) in kidney.

Laying hens were orally dosed with [^{14}C]oxathiapiprolin (pyrazole-label or thiazole-label) at doses equivalent to 17.4–17.8 ppm in the feed for 14 consecutive days and sacrificed 6 hours after the last dose.

More than 95% of the total administered dose was recovered from the excreta and cage wash. About 0.02% AR was recovered in edible tissues and another 0.01–0.02% AR found in the eggs. Radioactivity plateaued in whole eggs within 6 days.

TRRs in edible tissues and eggs from both the radiolabels were low (< 0.03 mg eq/kg) except for the liver which had higher residues (0.1 mg eq/kg).

Solvent extractions released 46–54% TRR in liver, 56–79% TRR in eggs and 87–94% TRR from fat and skin + fat. Subsequent protease treatments released another 21–54% TRR from liver and eggs.

While oxathiapiprolin was found in all tissues and eggs, levels were not more than 0.01 mg/kg. Oxathiapiprolin made up 10–22% TRR (0.002–0.003 mg/kg) in eggs, 4.0–8.0% TRR (0.004–0.008 mg/kg) in liver, 28–66% TRR (0.01 mg/kg) in abdominal fat, and 22–37% TRR (0.003–0.004) mg/kg in skin with fat.

IN-RAB06 was the only identified component present at more than 0.01 mg eq/kg, detected only in liver at up to 0.014 mg eq/kg (7.7–13.5% TRR).

IN-RDG40/IN-Q7H09 accounted for 15–33% TRR (0.003–0.005 mg eq/kg) in skin and fat, IN-Q7D41 was detected in eggs and fats (5–10% TRR, 0.001–0.002 mg eq/kg) but was not observed in liver.

Environmental fate

The Meeting received information on the environmental fate and behaviour of oxathiapiprolin, including hydrolytic stability, photochemical degradation in soils and aerobic metabolism studies.

Hydrolysis

Radiolabelled oxathiapiprolin incubated in the dark in sterile aqueous buffered solutions at pH 5, 7, and 9 for 5 days at 50 °C was stable with no major transformation products being observed.

Photochemical degradation in soil

In a 15-day photochemical degradation study in a sandy loam soil, 88% AR (moist soil) and 84–97% AR (dry soil) was able to be extracted, with no quantifiable levels of evolved $^{14}CO_2$ or non-specific volatile organic components.

Oxathiapiprolin accounted for 70% AR in moist soil and 80% AR in the dry soil after 15 days of continuous irradiation. Degradates IN-RDT31, IN-E8S72, IN-RAB06 and at least 15 minor transformation products were also found, all below 6% AR at two consecutive sampling intervals or 10% AR at a single sampling interval.

Calculated photodegradation DT_{50} values for oxathiapiprolin were 28.2 days (moist soil) and 36.3 days (dry soil) and the DT_{90} values were 93.5 days and 120.7 days respectively.

Aerobic soil metabolism

Aerobic degradation of radiolabelled oxathiapiprolin (0.2 mg/kg) was investigated in five different soils (120–134 days in the dark at 20 °C). Volatile organics were not produced in significant amounts and about 12% AR was evolved as $^{14}CO_2$. Chiral HPLC analysis in one study showed no change in the isomer ratio over the study period.

DT_{50} values for oxathiapiprolin ranged from 18 to 134 days at 20 ºC (geometric mean of 76 days). The predominant degradation products were IN-RAB06 (up to 13% AR), IN-RDT31, IN-QPS10 and IN-E8S72, each making up 7–9% AR.

The proposed degradation pathways include cleavage of the pyrazole ring to form IN-QPS10 and IN-E8S72; hydroxylation of the methyl group on the pyrazole ring followed by oxidation to form IN-RAB06 and hydroxylation at the 4-position of the piperidine ring to form IN-RDT31 (with subsequent cleavage to IN-WR791 and further degradation to IN-E8S72.

In ten field dissipation studies conducted in Europe and North America and involving bare soil treatments of 0.2–0.77 kg ai/ha oxathiapiprolin, DT_{50} values ranged from 5–205 days (geometric mean of 26 days). Residues of parent and degradates were found mostly in the upper soil segment (0–15 cm) with the highest concentration in the 0–5 cm layer and rarely found below 15 cm depth.

Laboratory degradation and field dissipation studies were also conducted with the four degradates where residues exceeded 5% AR at two or more consecutive sampling points. Geometric mean half-lives were 37 days (IN-RAB06), 48 days (IN-QPS10), 141 days (IN-RDT31) and 323 days (IN-E8S72).

Rotational crops

Two confined rotational crop studies using wheat, lettuce and turnip as rotational crops planted in bare sandy loam soil treated at rates equivalent to 0.21 kg ai/ha or 0.6 kg ai/ha. Plant-back intervals ranged from 30 to 365 days.

In samples from the rotational crops from soils treated with 0.21 kg ai/ha [isoxazoline^{14}C]- and [thiazole^{14}C]-oxathiapiprolin, TRRs were low (up to 0.013 mg eq/kg), and were higher in the samples from the soils treated with [pyrazole^{14}C]-oxathiapiprolin, attributed to the higher root uptake of polar metabolites derived from the pyrazole moiety.

Highest TRRs in food crops were 0.26 mg eq/kg (wheat grain), 0.02 mg eq/kg (mature lettuce, and turnip roots). In animal feed items, highest TRRs were 0.76 mg eq/kg (wheat straw), 0.27–0.3 mg eq/kg (wheat forage and hay) and 0.09–0.12 mg eq/kg (turnip tops).

In crop samples from soils treated with 0.6 kg ai/ha [isoxazoline^{14}C]-oxathiapiprolin, TRRs in food items ranged were also low, up to 0.09 mg eq/kg (wheat straw). In the pyrazole-label samples, highest TRRs in food crops were 0.19 mg eq/kg (wheat grain) and 0.02–0.04 mg eq/kg in lettuce and turnip roots). In animal feed items, highest TRRs were 0.7 mg eq/kg (wheat straw), 0.23–0.26 mg eq/kg (wheat forage and hay) and 0.09 mg eq/kg (turnip tops).

Oxathiapiprolin residues were not detectable in most rotational crops in both studies, and where present, residues were < 0.01 mg/kg and less than 15% TRR). Metabolites present at more than 0.01 mg eq/kg and above 10% TRR were IN-WR791, IN-E8S72, IN-SXS67, IN-RZB20 and IN-RZB21/IN-RZD74.

In the study approximating the maximum seasonal application rate (0.6 kg ai/ha), metabolites present in food commodities were IN-WR791 (23–37% TRR, 0.03–0.05 mg eq/kg), IN-RZB20 (up to 13% TRR, 0.015 mg eq/kg) and IN-E8S72 (up to 14% TRR, 0.02 mg eq/kg in wheat grain and up to 35% TRR, 0.01 mg eq/kg in immature lettuce.

In animal feed commodities, metabolites present above 10% TRR and 0.1 mg eq/kg were in wheat straw, IN-SXS67 at 26–39% TRR and up to 0.27 mg eq/kg, IN-RZB20 at 17–26% TRR and up to 0.15 mg eq/kg and IN-RZB21/IN-RZD74 at 12–15% TRR and up to 0.1 mg eq/kg.

Field rotational crop studies

In a series of rotational crop field trials involving total application rates of 0.12–0.56 kg ai/ha, residues of oxathiapiprolin and metabolites were measured in a number of representative root and tuber vegetables, leafy vegetables, stem vegetables, Brassica vegetables, bulb vegetables, legumes/pulses, cereals, oilseeds, and small berries at plant-back intervals ranging from 8 days to 365 days.

When scaled to a seasonal application rate of 0.56 kg ai/ha, residues of oxathiapiprolin in rotational crops at all plant-back intervals were ≤ 0.01 mg/kg in all commodities except legume feed commodities (≤ 0.04 mg/kg).

Residues of IN-WR791 were 0.01 mg/kg or less in all rotational food crops except leafy vegetables (< 0.02 mg/kg) and cereal forage and hay (< 0.03 mg/kg) when scaled to the maximum seasonal rate.

Scaled maximum residues of the metabolite IN-E8S72 in food commodities were < 0.01 mg/kg except in strawberries, green onions and oilseeds (< 0.02 mg/kg), legume vegetables and pulses (< 0.05 mg/kg) and were up to 0.35 mg/kg in leafy vegetables. In animal feed commodities, maximum IN-E8S72 residues above 0.1 mg/kg occurred in legume and cereal hays (up to 0.15 mg/kg).

Scaled maximum residues of IN-SXS67 (the glucose conjugate of IN-E8S72) in food commodities were ≤ 0.01 mg/kg except in strawberries (0.02 mg/kg), cereal grains and leafy vegetables (0.02 mg/kg), legume vegetables (0.04 mg/kg), pulses and root crops (0.07-0.08 mg/kg4 mg/kg) . In animal feed commodities, maximum residues above 0.1 mg/kg were in legume forage and hay (0.13–0.16 mg/kg) and cereal straw, forage and hay (0.26–0.9 mg/kg).

The Meeting concluded that since the application rates in the confined rotational crop studies and the field trials generally covered the range of GAP treatment rates for foliar or soil applications to annual crops, residues of parent are not expected in food commodities from rotational crops following

treatments according to the GAPs under consideration. In livestock feed commodities, parent residues could occur at low levels (up to 0.04 mg/kg in legume feed commodities).

Residues of metabolite IN-WR791 can be expected at low levels (up to 0.02–0.04 mg/kg) in leafy vegetables and cereal forage and hay.

Residues of < 0.05 mg/kg of IN-E8S72 can be expected in legume vegetables and, pulses, with expected levels of 0.15 mg/kg in legume and cereal hays and up to 0.35 mg/kg in leafy vegetables. Residues of the glucose conjugate (IN-SXS67) can be expected at levels of 0.02–0.08 mg/kg in food commodities and up to 0.9 mg/kg in legume and cereal feed commodities.

Methods of analysis

Analytical methods have been reported and validated for the analysis of oxathiapiprolin and metabolites in plant and animal commodities.

Data generation methods involved extraction with either formic acid:water:acetonitrile or formic acid:methanol, dilution with acetonitrile and water and separation of oxathiapiprolin and metabolites by reverse-phase LC-MS/MS. An additional SPE clean-up step was included for some matrices. Animal matrices were extracted with hexane:acetonitrile and acetonitrile:water. Quantitation was performed using mass transitions 540→500 and 540→163 for oxathiapiprolin. The LOQ for each analyte was 0.01 mg/kg.

For MRL-compliance, several multi-residue methods are available. Method DFG S 19 (LC-MS/MS module) is suitable for the analysis of oxathiapiprolin and metabolites IN-SXS67, IN-RZB20, IN-RZD74, IN-E8S72, IN-WR791, IN-RDG40, and IN-Q7H09 in representative samples with a high water content, high acid/water content, high acid content and high starch content with LOQs of 0.01 mg/kg for each analyte. However, in rape seed (high oil content), average recoveries were less than 70% in samples spiked with 0.01 mg/kg and 0.1 mg/kg.

Method DFG S 19 (LC-MS/MS module) is also suitable for the analysis of oxathiapiprolin and IN-Q7H09, IN-RDG40, IN-RLB67, and IN RAB06 in muscle, fat, liver, milk and eggs, with LOQs of 0.01 mg/kg for each analyte. However, for IN-Q7H09 in fat, the average recovery was 67% in the 0.01 mg/kg spiked samples, resulting in a higher LOQ of 0.1 mg/kg.

The QuEChERS multi-residue method was evaluated for measuring residues of oxathiapiprolin in commodities of plant origin (lettuce, wheat grain, orange, and maize grain) with LOQs of 0.01 mg/kg. However, the method did not adequately extract incurred oxathiapiprolin residues from dry crops such as wheat straw.

The Meeting concluded that suitable data generation methods are available to measure oxathiapiprolin and metabolites in plant and animal commodities and the multi-residue method DFG S 19 is suitable for monitoring residues of oxathiapiprolin and some metabolites in plant commodities with high water content, high acid content, dry crop commodities and also in animal commodities.

Stability of pesticide residues in stored analytical samples

Residues of oxathiapiprolin and metabolites IN-Q7H09, IN RDG40, IN E8S72, IN-RZB20, IN RZD74, IN SXS67, and IN WR791were stable in analytical samples stored frozen (-18 to -20 °C) for at least the storage intervals used in the supervised residue trials, with residues in the stored samples usually more than 80% of the spiked sample levels. In general, residue stability was shown for at least 18 months in representative commodities with high water content (wheat forage, and tomato), high starch content (potato, and wheat grain), high protein content (dry bean seed), high oil content (soya bean seed), high acid content (grape) and low moisture content (wheat straw, and dry grape pomace). The exception was for the metabolite IN-RDG40 in soya bean seed (high oil content), where residues were stable for up to 3 months.

Definition of the residue

Plant commodities

In the plant metabolism studies involving foliar applications, oxathiapiprolin was the predominant residue, accounting for 25–85% TRR in lettuce, potato and grape leaves and in most grape samples (10%, 0.03 mg/kg mature grapes from the pyrazole-label treatment).

When applied as soil treatments in the plant and rotational crop metabolism studies and in the rotational crop field trials, oxathiapiprolin, if detected, was a minor component of the total residue. However, in the supervised field trials involving soil treatments, oxathiapiprolin was the predominant residue but present at levels lower than those following foliar applications.

The Meeting considered that a suitable MRL-compliance residue definition for plant commodities would be oxathiapiprolin (parent only).

When considering a residue definition for dietary intake estimation, the Meeting noted that in food commodities, metabolites making up more than 10% TRR and > 0.01 mg eq/kg in the metabolism studies, detected in field trials or present in rotational crops above 0.01 mg/kg (after scaling to seasonal application rate of 0.56 kg ai/ha) were IN-WR791, IN-E8S72 and its glucose conjugate IN-SXS67.

IN-WR791 was detected occasionally in the supervised field trials, at concentrations of 0.01–0.02 mg/kg following foliar applications (up to 0.1 mg/kg in grape leaves). In the food commodities from the rotational crop field trials, after scaling to a seasonal application rate of 0.56 kg ai/ha, IN-WR791 residues were only found in legume vegetables (up to 0.015 mg/kg) and in leafy vegetables (0.02 mg/kg).

The toxicity of IN-WR791 is no greater than the parent, and as the residue contribution to the maximum long-term toxicological burden is minor (< 5%), the Meeting agreed that IN-WR791 need not be included in the residue definition for dietary intake risk assessment.

IN-E8S72 and its glucose conjugate (IN-SXS67) were found in crops following soil applications and in rotational crops, at levels of 0.02–0.03 mg/kg but higher(0.04–0.8 mg/kg) in legume vegetables, pulses and root crops and up to 0.35 mg/kg in leafy vegetables. To estimate the long-term toxicological burden of these metabolites, the Meeting used the data from the crop rotation field studies (after scaling to the maximum seasonal rate of 0.56 kg ai/season) to calculate mean residues (expressed as oxathiapiprolin equivalents) for the relevant crop groups where significant levels of these metabolites could be expected. These mean values are summarized below.

Crop group [A]	Mean in rotational crops after residues scaled to 560 g ai/ha (mg parent eq/kg)[B]			
	Oxathiapiprolin	IN-E8S72	IN-SXS67	Total
Leafy vegetables [C]	< 0.01	0.30	< 0.016	0.33
Legume vegetables	< 0.01	0.047	0.026	0.083
	< 0.01	0.065	0.043	0.12
Pulses	0.004 (liver)[E]	0.006 (kidney [D]	0.009 (kidney)[D]	0.015 (kidney) 0.004 (liver)
Edible offal	93.8F	22.5	11.1	
IEDI (ųg/person /day)	< 0.01	0.30	< 0.016	0.33

[A] Median residues of IN-E8S72 and IN-SXS67 were < LOD in representative root and tuber vegetables, stem vegetables, head and flowerhead Brassica vegetables, bulb vegetables, cereals, oilseeds, and small berries

[B] Based on metabolite:oxathiapiprolin conversion factors of 2.99 (IN-E8S72) and 1.58 (IN-SXS67)

[C] Mature leaves, including leafy Brassicas

[D] From consumption of rotational crop feed commodities

[E] For total dietary exposure

IN-E8S72 was identified in the rat metabolism studies and its toxicity is no greater than the parent. Its IN-SXS67 conjugate is a plant metabolite and following consideration of its structural characteristics, the WHO experts' panel were of the opinion that it would be of no greater toxicity than the parent.

As the combined residue contribution of these metabolites to the long-term toxicological burden for oxathiapiprolin is significant (about 36% that of parent), the Meeting considered that IN-E8S72 and IN-SXS67 should be included in the residue definition for estimation of dietary intake for plant commodities.

Animal commodities

In the goat and hen metabolism studies, only about 1–2% of the applied doses remained in goat tissues and about 0.02% remained in hen tissues, with 0.1–0.2% of the dose being eliminated in eggs and milk respectively.

Oxathiapiprolin was found in most matrices, mostly present at more than 10% TRR and above 0.01 mg/kg in goat liver (up to 0.11 mg/kg), fats (0.01–0.016 mg/kg) and kidney (0.01 mg/kg).

Since oxathiapiprolin was the predominant residue in eggs, muscle and fat and a major component of the residue in liver and kidney, the Meeting considered that a suitable MRL-compliance residue definition for animal commodities would be oxathiapiprolin (parent only).

For dietary intake estimation, metabolites exceeding 10% TRR and more than 0.01 mg/kg in animal commodities were IN-RAB06 in poultry liver (8–14%TRR), IN-E8S72 in goat kidney (24% TRR) and the combined hydroxy metabolites including IN-RDG40 and IN-Q7H09 in goat liver (11–13% TRR) and kidney (16–20% TRR).

Noting that the toxicity of IN-RAB06, IN-RDG40 and IN-Q7H09 are each no greater than the parent and since their concentrations in animal commodities are low, the Meeting concluded that their contribution to the long-term toxicological burden would be insignificant and that they need not be included in the residue definition for dietary intake risk assessment for animal commodities.

Since the overall intake of IN-E8S72 contributes significantly to the toxicological burden, the Meeting agreed that IN-E8S72, together with its glucose conjugate (IN-SXS67) should be included in the residue definition for dietary intake risk assessment for animal commodities.

The Meeting therefore considered that a suitable residue definition for estimation of dietary intake for animal commodities would be the sum of oxathiapiprolin, IN-E8S72 and IN-SXS67, expressed as parent.

The Meeting noted that multi-residue methods exists to measure oxathiapiprolin residues in plant and animal commodities and agreed that for MRL-compliance and dietary intake estimation for plant and animal commodities the residue definitions should be oxathiapiprolin.

The Meeting noted that the octanol/water partition coefficient (Log P_{ow}) for oxathiapiprolin was 3.6, suggesting a potential for the residue to be fat soluble. Information on relative concentrations in muscle and fat were in the animal metabolism studies inconclusive on the relative distribution of residues in muscle and fat and the Meeting concluded that the residue is not fat soluble.

Proposed definition of the residue for compliance with the MRL: *oxathiapiprolin.*

Proposed definition of the residue for estimation of dietary intake for plant and animal commodities): *Sum of: oxathiapiprolin, 5-(Trifluoromethyl)-1H-pyrazole-3-carboxylic acid and 1-ß-D-Glucopyranosyl-3-(-(trifluoromethyl)-1H-pyrazole-5-carboxylic acid*, expressed as parent.

The residue is not fat soluble.

Results of supervised residue trials on crops

The Meeting received supervised trial data for oxathiapiprolin applied as foliar or soil treatments on grapes, indoor tomatoes and cucumbers and on a range of vegetable field crops. These trials were

conducted in China, Europe and North America. GAP information was available from Australia, Canada, China, New Zealand and the USA.

Where residues have been reported in the studies as being not detected or not quantifiable, the values have been considered as < LOQ for the purposes of MRL setting.

Residues in rotational crops

The Meeting noted that in rotational crops, significant residues of IN-E8S72 and/or its glucose conjugate (IN-SXS67) can be expected in legumes, pulses, leafy vegetables and cereal crops.

For the purposes of dietary exposure estimation, the Meeting agreed to include the residues of IN-E8S72 and IN-SXS67 (expressed as parent equivalents) in the long-term dietary intake estimate for oxathiapiprolin, and to estimate mean residue values of 0.33 mg/kg for leafy vegetables; 0.083 mg/kg for legume vegetables; and 0.12 mg/kg for pulses to account for the presence of these metabolites in rotational food crops. Their presence in non-rotational crops was below LOQ.

Grapes

Results from supervised field trials on grapes conducted in China and Europe were provided to the Meeting. The GAP in China is for a maximum of two foliar applications of 4–5 g ai/hL, with a 14-day PHI.

In four independent trials on grapes conducted in China and matching the Chinese GAP, oxathiapiprolin residues were 0.17, 0.37, 0.38 and 0.5 mg/kg.

Residues in five trials from Europe matching the GAP in China were 0.02, 0.2, 0.2, 0.21 and 0.23 mg/kg.

Since the Mann-Whitney test indicated that the populations from the trials in China and Europe were not statistically different, the Meeting agreed to use the global data set approach to estimate a maximum residue level based on the combined data set of: 0.02, 0.17, 0.2, 0.2, 0.21, 0.23, 0.37, 0.38 and 0.5 mg/kg.

The Meeting estimated an STMR of 0.21 mg/kg and a maximum residue level of 0.9 mg/kg for oxathiapiprolin on grapes.

Bulb vegetables

Results from supervised trials on dry bulb onions and spring onions conducted in North America were provided to the Meeting.

The critical GAP for bulb vegetables in the USA is for a maximum of four foliar applications of 35 g ai/ha, with a maximum seasonal rate of 140 g ai/ha and with a PHI of 0 days.

Onion, Bulb

In ten independent trials on bulb onions conducted in North America and matching the USA GAP, oxathiapiprolin residues were < 0.01, < 0.01, < 0.01, 0.01, 0.01, 0.01, 0.01, 0.02, 0.02 and 0.03 mg/kg.

The Meeting estimated an STMR of 0.01 mg/kg and a maximum residue level of 0.04 mg/kg for oxathiapiprolin on onion, bulb.

Noting that the GAP in the USA included other crops in their onion bulb subgroup, the Meeting agreed to extrapolate these recommendations to garlic, shallots and great-headed garlic.

Spring onion

In four independent trials on spring onions conducted in North America and matching the USA GAP, oxathiapiprolin residues were 0.45, 0.57, 0.63 and 0.85 mg/kg.

The Meeting estimated an STMR of 0.6 mg/kg and a maximum residue level of 2.0 mg/kg for oxathiapiprolin on spring onion.

Noting that the GAP in the USA was for bulb vegetables, the Meeting agreed to extrapolate these recommendations to leek and Welsh onion.

Brassica (cole or cabbage) vegetables, Head cabbages, Flowerhead brassicas

Results from supervised trials on broccoli, cauliflower and head cabbages conducted in North America were provided to the Meeting.

The critical GAP for Head and Stem Brassica vegetables in the USA is for a maximum of four foliar applications of 35 g ai/ha, with a maximum seasonal rate of 140 g ai/ha and with a PHI of 0 days

Broccoli

In five independent trials on broccoli conducted in North America and matching the USA GAP, oxathiapiprolin residues were 0.08, 0.17, 0.22, 0.23 and 0.81 mg/kg.

The Meeting estimated an STMR of 0.22 mg/kg and a maximum residue level of 1.5 mg/kg for oxathiapiprolin on broccoli.

Cabbages, head

In ten independent trials on head cabbage conducted in North America and matching the USA GAP, oxathiapiprolin residues were 0.04, 0.06, 0.06, 0.12, 0.12, 0.16, 0.22, 0.29, 0.32 and 0.42 mg/kg.

The Meeting estimated an STMR of 0.14 mg/kg, a highest residue of 0.46 mg/kg (for livestock dietary burden estimation) and a maximum residue level of 0.7 mg/kg for oxathiapiprolin on cabbages, head.

Cauliflower

In five independent trials on cauliflower conducted in North America and matching the USA GAP, oxathiapiprolin residues were 0.08, 0.08, 0.08, 0.09 and 0.14 mg/kg.

The Meeting estimated an STMR of 0.08 mg/kg and a maximum residue level of 0.3 mg/kg for oxathiapiprolin on cauliflower.

Fruiting vegetables, Cucurbits

Results from supervised trials on protected and field cucumbers, summer squash (courgettes, squash) and melons (cantaloupes) conducted in Europe and North America were provided to the Meeting.

The critical GAP for cucurbit vegetables in the USA is for either a maximum of four foliar applications of 35 g ai/ha, with a maximum seasonal rate of 140 g ai/ha and with a PHI of 0 days or 2–4 soil drench or drip irrigation treatments of up to 280 g ai/ha up to 0 days before harvest and with a maximum seasonal rate of 560 g ai/ha. The label also specifies the use of either foliar or soil treatments, but not both.

Cucumber

In four independent trials on protected cucumbers conducted in North America and matching the USA GAP for foliar applications (max 4 × 35 g ai/ha, 0-day PHI), oxathiapiprolin residues were 0.02, 0.04, 0.04, and 0.04 mg/kg.

In 11 independent trials on field cucumbers conducted in North America and matching the USA GAP for foliar applications, oxathiapiprolin residues were < 0.01, < 0.01, 0.01, 0.01, 0.02, 0.03, 0.03, 0.03, 0.04, 0.07 and 0.09 mg/kg.

In 10 independent trials matching the soil treatment GAP in the USA, residues of oxathiapiprolin were < 0.01 (9) and 0.01 mg/kg.

Summer squash

In 10 independent trials on field squash conducted in North America and matching the USA GAP for foliar applications, oxathiapiprolin residues were 0.01, 0.02, 0.02, 0.03, 0.03, 0.03, 0.04, 0.04, 0.08 and 0.12 mg/kg.

In 14 independent trials matching the soil treatment GAP in the USA, residues of oxathiapiprolin were < 0.01 (10), 0.01, 0.02, 0.03 and 0.08 mg/kg following pre-harvest treatments (0-day PHI).

Melons

In 11 independent trials on field melons (conducted in North America and matching the USA GAP for foliar applications, oxathiapiprolin residues were 0.01, 0.02, 0.03, 0.03, 0.04, 0.04, 0.05, 0.06, 0.07, 0.09 and 0.12 mg/kg.

In 11 independent trials matching the soil treatment GAP in the USA, residues of oxathiapiprolin were < 0.01 (7), 0.02, 0.02, 0.02 and 0.03 mg/kg.

The Meeting noted that the median residues in cucumbers, summer squash and melons from the foliar treatment field trials were within a 5-fold range (0.03–0.045 mg/kg) and that the Kruskall-Wallis test indicated that the populations were not statistically different. The Meeting therefore agreed to combine the data sets to estimate a group MRL for cucurbits. The combined data set is < 0.01, < 0.01, 0.01 (4), 0.02 (4), 0.03 (8), 0.04 (5), 0.05, 0.06, 0.07, 0.07, 0.08, 0.09, 0.09, 0.12 and 0.12 mg/kg.

The Meeting estimated an STMR of 0.03 mg/kg and a maximum residue level of 0.2 mg/kg for oxathiapiprolin on fruiting vegetables, cucurbits.

The Meeting also noted that oxathiapiprolin residues following soil applications were lower than the associated foliar treatments, and would be accommodated by the proposed group maximum residue level.

Fruiting vegetables, other than Cucurbits

Results from supervised trials on protected and field peppers and tomatoes conducted in Europe and North America were provided to the Meeting.

The critical GAP for fruiting vegetables (other than cucurbits) in the USA is for either a maximum of four foliar applications of 35 g ai/ha, with a maximum seasonal rate of 140 g ai/ha and with a PHI of 0 days or 2–4 soil drench or drip irrigation treatments of up to 280 g ai/ha up to 0 days before harvest and with a maximum seasonal rate of 560 g ai/ha. The label also specifies the use of either foliar or soil treatments, but not both.

Peppers

In two independent trials on protected bell peppers conducted in North America and matching the USA GAP for foliar applications (max 4 × 35 g ai/ha, 0-day PHI), oxathiapiprolin residues were 0.06 and 0.12 mg/kg.

In 10 independent trials on field bell peppers conducted in North America and matching the USA GAP for foliar applications, oxathiapiprolin residues were 0.02, 0.02, 0.02, 0.03, 0.03, 0.04, 0.04, 0.05, 0.05 and 0.12 mg/kg.

In five independent trials on field non-bell peppers conducted in North America and matching the USA GAP for foliar applications, oxathiapiprolin residues were 0.03, 0.06, 0.06, 0.08 and 0.12 mg/kg.

In 11 independent trials on bell and non-bell peppers matching the soil treatment GAP in the USA, residues of oxathiapiprolin were < 0.01 (14) and 0.02 mg/kg.

For dried chilli peppers, applying the default processing factor of 10 to the STMR and the maximum residue level estimated for fruiting vegetables, other than cucurbits, the Meeting estimated an STMR of 0.4 mg/kg and a maximum residue level of 4 mg/kg for oxathiapiprolin on chilli peppers, dried.

Tomatoes

In four independent trials on protected tomatoes conducted in North America and matching the USA GAP for foliar applications (max 4 × 35 g ai/ha, 0-day PHI), oxathiapiprolin residues were < 0.01, < 0.01, 0.03 and 0.08 mg/kg in tomatoes.

In 19 independent trials on field tomatoes, conducted in North America and matching the USA GAP for foliar applications, oxathiapiprolin residues were < 0.01, 0.02 (3), 0.03 (4), 0.04 (3), 0.05, 0.05, 0.08, 0.08, 0.1, 0.12, 0.14 and 0.31 mg/kg in tomatoes.

In 21 independent trials on field tomatoes matching the soil treatment GAP in the USA, residues of oxathiapiprolin were < 0.01 (18), 0.03, 0.05 and 0.24 mg/kg.

The Meeting noted that the median residues in bell peppers, non-bell peppers and tomatoes from the foliar treatment trials were within a 5-fold range and that the Kruskall-Wallis test indicated that the populations were not statistically different. The Meeting therefore agreed to combine the data sets to estimate a group MRL for fruiting vegetables other than cucurbits. The combined data set is < 0.01, 0.02 (6), 0.03 (7), 0.04 (5), 0.05 (4), 0.06, 0.06, 0.08 (3), 0.1, 0.12 (3), 0.14 and 0.31 mg/kg.

The Meeting estimated an STMR of 0.04 mg/kg and a maximum residue level of 0.4 mg/kg for oxathiapiprolin on fruiting vegetables, other than cucurbits (except mushrooms and sweetcorn).

The Meeting also noted that oxathiapiprolin residues in peppers and tomatoes following soil applications were lower than the associated foliar treatments, and would be accommodated by the estimated group maximum residue level.

Leafy vegetables

Results from supervised trials on field lettuce and spinach conducted in Europe and North America were provided to the Meeting.

The critical GAP for leafy greens (including lettuce and spinach) in the USA is for either a maximum of four foliar applications of 35 g ai/ha, with a maximum seasonal rate of 140 g ai/ha and with a PHI of 0 days or a maximum of four soil drench or drip irrigation treatments of up to 280 g ai/ha up to 0 days before harvest and with a maximum seasonal rate of 560 g ai/ha. The label also specifies the use of either foliar or soil treatments, but not both.

Lettuce

In 10 independent trials on field head lettuce conducted in North America and matching the USA GAP for foliar applications, oxathiapiprolin residues were 0.23, 0.28, 0.3, 0.5, 0.57, 0.7, 0.82, 0.83, 1.3 and 1.4 mg/kg.

In these trials, residues of oxathiapiprolin in head lettuce from plots treated according to the soil treatment GAP in the USA, were < 0.01 (8), 0.37 and 0.43 mg/kg.

The Meeting estimated a median residue of 0.64 mg/kg and a maximum residue level of 3.0 mg/kg for oxathiapiprolin on lettuce, head.

In field studies on rotational crops, mean residues of IN-E8S72 and IN-SXS67 (expressed as parent equivalents were 0.33 mg eq/kg in leafy vegetables. The Meeting decided to add the mean residue found in the leafy vegetable rotational crop studies to the median residue from the lettuce (foliar application) field trials to estimate an overall STMR of 0.97 mg eq/kg for lettuce, head.

In 10 independent trials on field open-head lettuce conducted in North America and matching the USA GAP for foliar applications, oxathiapiprolin residues were 0.54, 0.8, 0.81, 1.2, 1.8, 1.9, 1.9, 1.9, 2.0 and 3.0 mg/kg.

In these trials, residues of oxathiapiprolin in open-head lettuce from plots treated according to the soil treatment GAP in the USA, were < 0.01 (4), 0.01, 0.02, 0.07, 0.09 and 0.37 mg/kg.

The Meeting estimated a median residue of 1.85 mg/kg and a maximum residue level of 5.0 mg/kg for oxathiapiprolin on lettuce, leaf.

In field studies on rotational crops, mean residues of IN-E8S72 and IN-SXS67 (expressed as parent equivalents were 0.33 mg eq/kg in leafy vegetables. The Meeting decided to add the mean residue found in the leafy vegetable rotational crop studies to the median residue from the lettuce (foliar application) field trials to estimate an overall STMR of 2.2 mg eq/kg for lettuce, leaf.

Spinach

In 10 independent trials on field spinach conducted in North America and matching the USA GAP for foliar applications, oxathiapiprolin residues were 1.4, 1.6, 2.2, 2.4, 3.2, 3.5, 4.0, 5.7, 6.4 and 6.5 mg/kg.

In these trials, residues of oxathiapiprolin in spinach from plots treated according to the soil treatment GAP in the USA, were < 0.01 (3), 0.01, 0.11, 0.12, 1.6, 1.8, 2.0 and 2.1 mg/kg.

The Meeting estimated a median residue of 3.35 mg/kg and a maximum residue level of 15 mg/kg for oxathiapiprolin on spinach.

In field studies on rotational crops, mean residues of IN-E8S72 and IN-SXS67 (expressed as parent equivalents were 0.33 mg eq/kg in leafy vegetables. The Meeting decided to add the mean residue found in the leafy vegetable rotational crop studies to the median residue from the spinach (foliar application) field trials to estimate an overall STMR of 3.7 mg eq/kg for spinach.

The Meeting also noted that oxathiapiprolin residues in lettuce and spinach following soil applications would be accommodated by the estimated maximum residue levels.

Legume vegetables

Results from supervised trials on peas conducted in North America were provided to the Meeting.

The critical GAP for succulent shelled and edible-podded peas in the USA is for up to four foliar applications of 35 g ai/ha, with a maximum seasonal rate of 140 g ai/ha and with a PHI of 0 days.

Peas, shelled (succulent seeds)

In five independent trials matching the GAP in the USA, residues of oxathiapiprolin in succulent peas (without pods) were < 0.01, < 0.01, 0.01, 0.03 and 0.03 mg/kg.

The Meeting estimated a median residue of 0.01 mg/kg and a maximum residue level of 0.05 mg/kg for oxathiapiprolin on peas, shelled.
In field studies on rotational crops, mean residues of IN-E8S72 and IN-SXS67 (expressed as parent equivalents were 0.083 mg eq/kg in legume vegetables. The Meeting decided to add the mean residue found in the legume vegetable rotational crop studies to the median residue from the pea (foliar application) field trials to estimate an overall STMR of 0.09 mg eq/kg for peas, shelled.

Peas (pods and succulent = immature seeds)

In five independent trials matching the GAP in the USA, residues of oxathiapiprolin in succulent peas (with pods) were 0.2, 0.3, 0.3, 0.28 and 0.55 mg/kg.

The Meeting estimated a median residue of 0.3 mg/kg and a maximum residue level of 1.0 mg/kg for oxathiapiprolin on peas (pods and succulent seeds).
In field studies on rotational crops, mean residues of IN-E8S72 and IN-SXS67 (expressed as parent equivalents were 0.083 mg eq/kg in legume vegetables. The Meeting decided to add the mean residue found in the legume vegetable rotational crop studies to the median residue from the pea (foliar application) field trials to estimate an overall STMR of 0.38 mg eq/kg for peas (pods and succulent seeds).

Root and tuber vegetables

Results from supervised trials on potatoes and ginseng conducted in North America were provided to the Meeting.

Potato

The critical GAP for tuberous and corm vegetables (including potato) in the USA is for a maximum of four foliar applications of 35 g ai/ha, with a PHI of 5 days. In addition, there is an option for two foliar sprays of 50 g ai/ha over flowering, and a maximum seasonal rate of 200 g ai/ha.

In 18 independent trials matching the close-to-harvest GAP in the USA but using a higher application rate of 50 g ai/ha, oxathiapiprolin residues in tubers were all < 0.01 mg/kg.
The Meeting estimated an STMR of 0 mg/kg and a maximum residue level of 0.01* mg/kg for oxathiapiprolin on potato and agreed to extrapolate these estimates to sweet potato.

Ginseng

The critical GAP for ginseng in the USA is for a maximum of four foliar applications per year of 35–280 g ai/ha, with a PHI of 14 days and a maximum total rate of 560 g ai/ha/year.

In four independent trials conducted in the USA and involving two close-to-harvest foliar applications of 280 g ai/ha, oxathiapiprolin residues in dried ginseng roots (10–30% moisture content) were 0.04, 0.04, 0.04 and 0.05 mg/kg.

The Meeting estimated an STMR of 0.04 mg/kg and a maximum residue level of 0.15 mg/kg for oxathiapiprolin on ginseng dried (including red ginseng).

Fate of residues during processing

Oxathiapiprolin is stable to hydrolysis in aqueous media within a pH range of 4 to 9 (< 10% degradation after 5 days at 50 ºC), and is also stable under conditions simulating pasteurisation, baking, brewing, boiling and sterilisation, with recovery rates of 95–97%.

The fate of oxathiapiprolin residues has been examined in a number of studies simulating household processing of melons (peeling), lettuce and Brassica vegetables (washing), and commercial processing of grapes, potatoes and tomatoes.

Residues of oxathiapiprolin increased in commodities such as raisins, dried tomatoes and in the grape and tomato pomaces and potato peel waste, where processing involves a reduction in moisture content, with no residue concentration in the other processed commodities.

For the commodities considered (grapes, potatoes and tomatoes) the Meeting estimated processing factors and STMR-Ps for their processed food or feed commodities are summarized below.

Summary of selected processing factors and STMR-P values for oxathiapiprolin

RAC (STMR)	Matrix	Oxathiapiprolin [a]		STMR-P (mg/kg)
		Calculated processing factors	PF median	
Grape (0.21 mg/kg)	Raw juice	0.26, 0.27, 0.28, 0.46	0.28	0.059
	Wet pomace	1.0, 1.4, 1.5, 1.9, 2.0, 3.1, 4.0, 5.4	2.0	0.42
	Juice	0.13, 0.14, 0.18, 0.22	0.16	0.034
	Raisins	0.93, 1.3, 1.6, 4.1	1.4	0.29
	Wine	00.08, 0.1, 0.17, 0.18	0.14	0.029
	Must	0.58, 62, 1.9	0.62	0.13
Potato (0 mg/kg)	Tubers			
	Culls	0.1, 0.13, 0.7	0.13	0
Tomato (0.04 mg/kg)	Tomatoes			
	Sun-dried	2.9, 6.9, 7.4	6.9	0.28
	Canned (peeled)	< 0.02, < 0.04, < 0.04	< 0.04	0.0016
	Juice	0.16, 0.16, 0.29	0.16	0.006
	Wet pomace	11, 13, 13	13	0.52
	Paste	0.69, 1.1, 1.1	1.1	0.044

[a] Each value represents a separate study where residues were above the LOQ in the RAC. The factor is the ratio of oxathiapiprolin residues in the processed item divided by the residue of oxathiapiprolin in the RAC.

For oxathiapiprolin in processed tomato commodities, based on a processing factor of 6.9 for sun-dried tomatoes and the estimated maximum residue level of 0.4 mg/kg for fruiting vegetables, other than cucurbits, the Meeting estimated an STMR-P of 0.28 mg/kg and a maximum residue level of 3 mg/kg for oxathiapiprolin on for tomato, dried.

For dried grapes, based on a processing factor of 1.4 and the estimated maximum residue level of 0.9 mg/kg for grapes, the Meeting estimated an STMR-P of 0.29 mg/kg and a maximum residue level of 1.3 mg/kg for oxathiapiprolin on for dried grapes.

Residues in animal commodities

Farm animal feeding studies

No lactating cow feeding studies were provided. In the lactating goat metabolism studies, goats were dosed with approximately 14 ppm oxathiapiprolin in the feed for 7 days. The highest residue of oxathiapiprolin or a metabolite in tissues or milk was 0.11 mg/kg for oxathiapiprolin in liver.

No poultry feeding studies were provided. In the poultry metabolism study, laying hens were dosed with approximately 17.6 mg oxathiapiprolin/kg feed for 14 days. Residues of oxathiapiprolin were all not more than 0.01 mg/kg in tissues and eggs.

In a supplementary lactating goat metabolism study, goats were dosed with approximately 19 ppm IN-SXS67 in the feed for 7 days. The highest residues in tissues or milk were in kidney, 0.28 mg/kg for IN-SXS67 and 0.19 mg eq/kg for IN-E8S72 and residues in liver were 0.03 mg/kg and 0.006 mg eq/kg respectively. TRRs in milk, muscle and fat were lower and not investigated further.

Farm animal dietary burden

The Meeting estimated the dietary burden of oxathiapiprolin in farm animals on the basis of the diets listed in Appendix IX of the 2009 edition of the JMPR Manual. Dietary burden calculations for beef cattle, dairy cattle, broilers and laying poultry are presented in Annex X. Livestock feed commodities considered by the Meeting were grape pomace, tomato pomace, potato culls and waste and cabbage heads/leaves.

Estimated maximum and mean dietary burdens of farm animals

	Animal dietary burden, oxathiapiprolin, ppm of dry matter diet							
	US-Canada		EU		Australia		Japan	
	max	mean	max	mean	max	mean	max	mean
Beef cattle	0	0	0.19	0.19	0.56 [A]	0.56 [C]	0	0
Dairy cattle	0	0	0.19	0.19	0.56 [B]	0.56 [D]	0	0

[A] Highest maximum beef or dairy cattle dietary burden suitable for MRL estimates for mammalian tissues

[B] Highest maximum dairy cattle dietary burden suitable for MRL estimates for mammalian milk

[C] Highest mean beef or dairy cattle dietary burden suitable for STMR estimates for mammalian tissues.

[D] Highest mean dairy cattle dietary burden suitable for STMR estimates for milk.

The Meeting also estimated a mean dietary burden of 0.4 ppm for IN-SXS67 in farm animals exposed to residues of this metabolite in rotational crop feed and forage items.

Animal commodity maximum residue levels

In the ruminant metabolism study, lactating goats were dosed with approximately 14 mg oxathiapiprolin/kg feed for 7 days. The highest residues of oxathiapiprolin in tissues or milk were seen in liver at 0.11 mg/kg.

The 14 ppm dose rate used in the goat metabolism studies is about 25 × the highest estimated cattle dietary burden of 0.56 ppm/day and the Meeting estimated that oxathiapiprolin residues in tissues and milk from cattle exposed to the maximum dietary burden would be not more than 0.004 mg/kg (in liver).

The Meeting estimated maximum residue levels of 0.01* mg/kg for oxathiapiprolin in meat (from mammals other than marine mammals), edible offal (mammalian), mammalian fat and for milks.

Estimated STMRs for dietary intake estimation are 0 mg/kg for meat, 0 mg/kg for fat and 0 mg/kg for milk.

For edible offal, the Meeting estimated a mean residue of 0.004 mg/kg (liver) to accommodate exposure from parent residues in feed items.

Based on the mean dietary burden of 0.4 ppm for IN-SXS67 from rotational crop feed items the Meeting estimated mean residues of 0.006 mg (IN-SXS67) and 0.004 mg eq/kg (IN-E8S72) for kidney to accommodate exposure to residues of IN-E8S72 in rotational crops.

When expressed as oxathiapiprolin equivalents, the mean residues in kidney are 0.009 mg eq/kg for IN-SXS67 and 0.006 mg eq/kg for IN-E8S72 and the Meeting estimated an overall STMR of 0.015 mg/kg for edible offal (mammalian).

As no poultry feed items were identified, the Meeting estimated maximum residue levels of 0.01* mg/kg for oxathiapiprolin in poultry meat, poultry offal, poultry fat and eggs. Estimated STMRs for dietary intake estimation are 0 mg/kg for meat, 0 mg/kg for edible offal, 0 mg/kg for fat and 0 mg/kg for milk.

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 assessment.

Definition of the residue for compliance with the MRL: *oxathiapiprolin.*

Definition of the residue for estimation of dietary intake: *Sum of: oxathiapiprolin, 5-(Trifluoromethyl)-1H-pyrazole-3-carboxylic acid and 1-ß-D-Glucopyranosyl-3-(-(trifluoromethyl)-1H-pyrazole-5-carboxylic acid, expressed as parent.*

The residue is not fat soluble.

	Commodity	MRL	STMR or	HR or
CCN	Name	New	STMR-P	HR-P
VB 400	Broccoli	1.5	0.22	
VB 0041	Cabbages, Head	0.7	0.14	0.46
VB 404	Cauliflower	0.3	0.08	
MO 0105	Edible offal (Mammalian)	0.01 *	0.015	
PE 0112	Eggs	0.01 *	0	
VC 0045	Fruiting vegetables, Cucurbits	0.2	0.03	
VO 0050	Fruiting vegetables, other than Cucurbits (except sweetcorn and mushrooms)	0.4	0.04	
VA 381	Garlic	0.04	0.01	
VA 382	Garlic, Great-headed	0.04	0.01	
DR 0604	Ginseng, dried including red ginseng	0.15	0.04	
FB 0629	Grapes	0.9	0.21	
VA 384	Leek	2.0	0.6	

	Commodity	MRL	STMR or	HR or
CCN	Name	New	STMR-P	HR-P
VL 482	Lettuce, Head	3.0	0.97	
VL 483	Lettuce, Leaf	5.0	2.2	
MM 0100	Mammalian fats (except milk fats)	0.01 *	0	
MM 0095	Meat (from mammals other than marine mammals)	0.01 *	0	
ML 0106	Milks	0.01 *	0	
VA 0385	Onion, Bulb	0.04	0.01	
VP 0063	Peas (pods and succulent = immature seeds)	1.0	0.38	
VP 0064	Peas, shelled	0.05	0.09	
HS 0444	Peppers Chili, dried	4	0.4	
VR 0589	Potato	0.01 *	0	
PF 0111	Poultry fat	0.01 *	0	
PM 0110	Poultry meat	0.01 *	0	
PO 0111	Poultry, Edible offal of	0.01 *	0	
VD 0070	Pulses		0.12	
VA 388	Shallots	0.04	0.01	
VA 389	Spring onion	2.0	0.6	
VL 502	Spinach	15	3.7	
VR 0508	Sweet potato	0.01 *	0	
DV 0448	Tomato, dried	3.0	0.28	
	Tomato, canned (and peeled)		0.0016	
	Tomato paste		0.044	
	Tomato puree		0.024	
	Tomato juice		0.006	
VA 387	Onion, Welsh	2.0	0.6	
	Grape juice		0.034	
DF 0269	Dried grapes	1.3	0.29	
	Wine		0.029	
	Grape must		0.13	

For calculating dietary exposure

	Tomato pomace (wet)		0.52	
	Wet grape pomace		0.42	

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The International Estimated Daily Intake (IEDI) for oxathiapiprolin was calculated for the food commodities for which STMRs were estimated and for which consumption data were available. The results are shown in Annex 3.

The International Estimated Daily Intakes of oxathiapiprolin for the 17 GEMS/Food cluster diets, based on estimated STMRs were 0–0% of the maximum ADI of 4 mg/kg bw (Annex 3). The Meeting concluded that the long-term dietary exposure of residues of oxathiapiprolin from uses that have been considered by the JMPR is unlikely to present a public health concern.

Short-term dietary exposure

The Meeting decided that an ARfD is unnecessary and concluded that the short-term dietary exposure to residues of oxathiapiprolin, from uses considered by the current Meeting, are unlikely to present a public health concern.

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