CHLORANTRANILIPROLE (230)

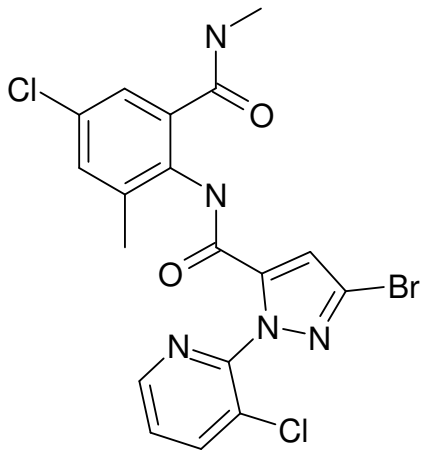
The first draft was prepared by Dr Dugald MacLachlan, Australian Quarantine and Inspection Service, Canberra, Australia

EXPLANATION

Chlorantraniliprole was considered for the first time by the present Meeting. The Meeting received information on chlorantraniliprole metabolism and environmental fate, methods of residue analysis, freezer storage stability, national registered use patterns, supervised residue trials, farm animal feeding studies and fate of residues in processing.

The 2008 JMPR established an ADI and ARfD for chlorantraniliprole of 0-2 mg/kg bw/day and not required respectively.

IDENTITY

ISO common name	Chlorantraniliprole
Synonyms:	DPX-E2Y45
IUPAC name	3-Bromo-N-[4-chloro-2-methyl-6-(methylcarbamoyl)phenyl]-1-(3-chloropyridin-2-yl)-1H-pyrazole-5-carboxamide
Chemical Abstracts name	3-Bromo-N-[4-chloro-2-methyl-6-[(methylamino)carbonyl]phenyl]-1-(3-chloro-2-pyridinyl)-1H-pyrazole-5-carboxamide
CAS Number	500008-45-7
CIPAC Number	794
Molecular formula	C18H14BrCl2N5O2
Molecular mass	483.15 g/mol
Structural formula	

PHYSICAL AND CHEMICAL PROPERTIES

Pure active ingredient

Property	Results	Reference
Appearance	Pure analytical grade: fine crystalline powder Technical grade: fine powder	Craig & Ramsay 2004c 13180
Physical state, colour	Pure analytical grade: off white (Munsell colour N9.5 90%R) Technical grade: brown (Munsell colour 7.5 YR 8/4)	Craig & Ramsay 2004c 13180
Odour	Pure analytical grade: No odour Technical grade: No odour	Craig & Ramsay 2004c 13180
Melting point	Pure analytical grade: 208–210 °C Technical grade: 200–202 °C	Craig & Ramsay 2004c 13180
Relative density	pure active ingredient: 1.5070 technical grade: 1.5189 at 20 °C.	Craig & Ramsay 2004c 13180
pH	5.77 ± 0.087 at 20 °C. The pH measured is a function of the pH of the water used to make the measurement.	Craig & Ramsay 2004b 13176
Vapour pressure	Pure analytical grade: 6.3×10^{-12} Pa at 20 °C and 2.1×10^{-11} Pa at 25 °C.	Hatzenbeler & Peterson 2006 16517
Volatility	Henry's law constant at 20 °C (calculated) 3.1×10^{-14} atmosphere.m^3.$mole^{-1}$ or 3.2×10^{-9} Pa.m^3.$mole^{-1}$	Hirata 2007 13174 Revision 1
Solubility in water including effect of pH	Unbuffered distilled water 1.023 mg/L pH 4: 0.972 mg/L pH 7: 0.880 mg/L pH 9: 0.971 mg/L	Craig & Ramsay 2004a 13169
Solubility in organic solvents (at 20 °C)	acetone 3.446 ± 0.172 acetonitrile 0.711 ± 0.072 dichloromethane 2.476 ± 0.058 dimethylformamide 124 ± 4 ethyl acetate 1.144 ± 0.046 n-hexane < 0.0001 methanol 1.714 ± 0.057 n-octanol 0.386 ± 0.010 o-xylene 0.162 ± 0.010	Craig 2004b 13173

Property	Results	Reference
Partition coefficient n-octanol/water (at 20 °C)	pH 4.0 log Kow = 2.77 ± 0.067 pH 7.0 log Kow = 2.86 ± 0.010 pH 9.0 log Kow = 2.80 ± 0.116 distilled water log Kow = 2.76 ± 0.104	Craig 2004c 13177
Hydrolysis	Hydrolysis of chlorantraniliprole at 25 °C was studied at pH 4, 7, and 9, at a concentration of 0.6 mg/L. Chlorantraniliprole was stable at pH 4 and 7. At pH 9, chlorantraniliprole hydrolysed with a half-life of ~10 days.	Chapleo *et al*. 2004 12782
Photolysis	The photolytic half-life of chlorantraniliprole in sterile aqueous buffer solution (pH 7.0) under continuous irradiation was 0.37 days. Conversion to 12 hour sunlight days (Tranent, UK, 55°57'N 2°58'W) results in a half-life of 0.7 days.	MacDonald, et al. 2005 12783
Dissociation constant	pKa = 10.88 ± 0.71 at 20 °C.	Craig & Clipston 2005 13254

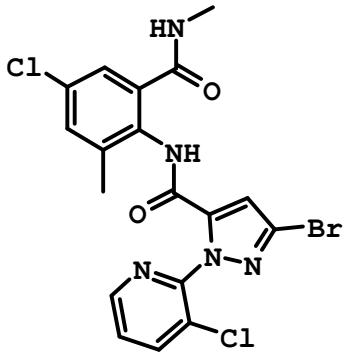
Formulations

Formulations	Active ingredient content
Suspension concentrate (SC)	Chlorantraniliprole 50 g/L and 200 g/L
Water dispersible granules (WG)	Chlorantraniliprole 350 g/kg

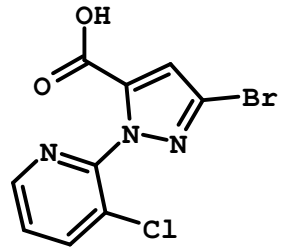
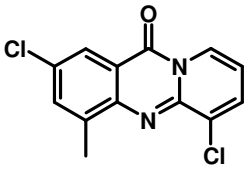
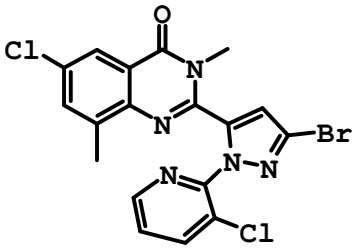
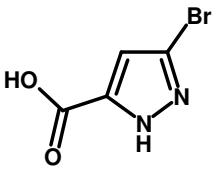
METABOLISM AND ENVIRONMENTAL FATE

Metabolites are given various abbreviations and code numbers in the studies. Structures and abbreviations and codes are shown below.

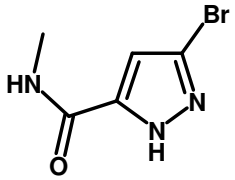
Chlorantraniliprole (DPX-E2Y45) CAS name: 3-Bromo-1-(3-chloro-2-pyridinyl)-N-[4-chloro-2-methyl-6-[(methylamino)carbonyl]phenyl]-1H-pyrazole-5-carboxamide



CAS number:	500008-45-7	Molecular Weight:	483.15
Structural formula:	$C_{18}H_{14}BrCl_2N_5O_2$	Observed in:	Water, soil, goat, rat, hen, plants

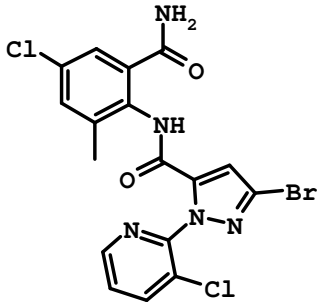
IN-DBC80	CAS name:	3-Bromo-1-(3-chloro-2-pyridinyl)-1H-pyrazole-5-carboxylic acid	
			
CAS number:	500011-86-9	Molecular Weight:	302.52
Structural formula:	$C_9H_5BrClN_3O_2$	Observed in:	Goat, hen, rat, rice
IN-ECD73	CAS name:	2,6-dichloro-4-methyl-11H-pyrido[2,1-b]quinazolin-11-one	
			
CAS number:	Not available	Molecular Weight:	279.13
Structural formula:	$C_{13}H_8Cl_2N_2O$	Observed in:	Soil, high temperature hydrolysis
IN-EQW78	CAS name:	2-[3-Bromo-1-(3-chloro-2-pyridinyl)-1H-pyrazol-5-yl]-6-chloro-3, 8-dimethyl-4(3H)-quinazolinone	
			
CAS number:	Not available	Molecular Weight:	465.14
Structural formula:	$C_{18}H_{12}BrCl_2N_5O$	Observed in:	Goat, hen, rat, plants, confined rotational crops, soil, water, high temperature hydrolysis
IN-EVK64	CAS name:	5-Bromo-1H-pyrazole-3-carboxylic acid	
			
CAS number:	Not available	Molecular Weight:	190.98
Structural formula:	$C_4H_3BrN_2O_2$	Observed in:	Soil (high temperature only)

IN-F6L99 CAS name: 5-Bromo-N-methyl-1H-pyrazole-3-carboxamide



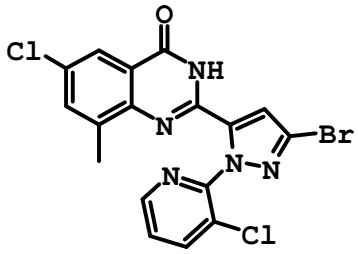
CAS number:	Not available	Molecular Weight:	204.03
Structural formula:	$C_5H_6BrN_3O$	Observed in:	Rice, confined rotational crops, soil, water, high temperature hydrolysis

IN-F9N04 CAS name: N-[2-(Aminocarbonyl)-4-chloro-6-methylphenyl]-3-bromo-1-(3-chloro-2-pyridinyl)1H-pyrazole-5-carboxamide



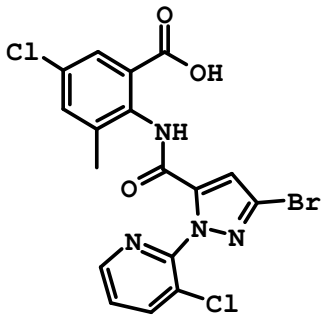
CAS number:	Not available	Molecular Weight:	469.13
Structural formula:	$C_{17}H_{12}BrCl_2N_5O_2$	Observed in:	Goat, hen, rat, rice, soil

IN-GAZ70 CAS name: 2-[3-Bromo-1-(3-chloro-2-pyridinyl)-1H-pyrazol-5-yl]-6-chloro-8-methyl-4(3H)-quinazolinone



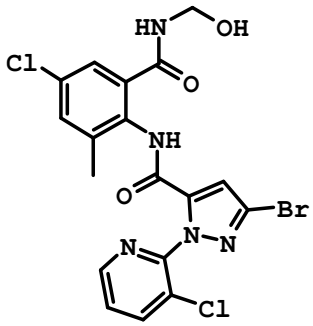
CAS number:	Not available	Molecular Weight:	451.11
Structural formula:	$C_{17}H_{10}BrCl_2N_5O$	Observed in:	Goat, hen, rat, rice, confined rotational crops, soil

IN-GKQ52 CAS name: 2-[[[3-Bromo-1-(3-chloro-2-pyridinyl)-1H-pyrazol-5-yl]carbonyl]amino]-5-chloro-3-methylbenzoic acid



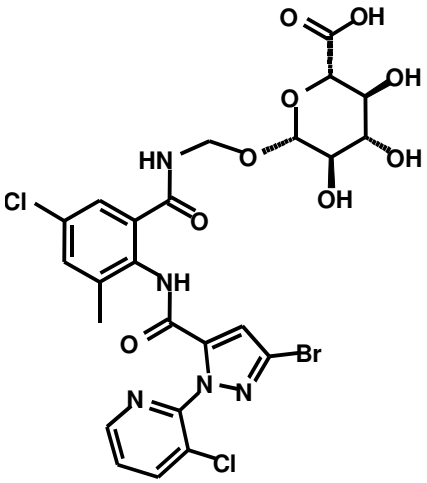
CAS number:	Not available	Molecular Weight:	470.11
Structural formula:	$C_{17}H_{11}BrCl_2N_4O_3$	Observed in:	Goat, hen, rat

IN-H2H20 CAS name: 3-Bromo-N-[4-chloro-2-[[(hydroxymethyl)amino]carbonyl]-6-methylphenyl]-1-(3-chloro-2-pyridinyl)-1H-pyrazole-5-carboxamide



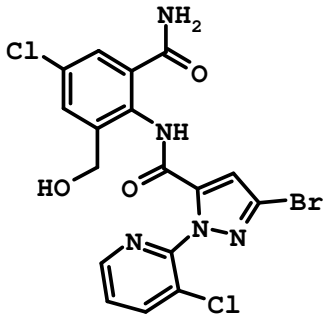
CAS number:	Not available	Molecular Weight:	499.15
Structural formula:	$C_{18}H_{14}BrCl_2N_5O_3$	Observed in:	Goat, hen, rat, rice, confined rotational crops

IN-H2H20-O-glucuronide CAS name: [2-[[[3-Bromo-1-(3-chloro-2-pyridinyl)-1H-pyrazol-5-yl)carbonyl]amino]-5-chloro-3-methylbenzoyl]amino]methyl β-D-glucopyranosiduronic acid



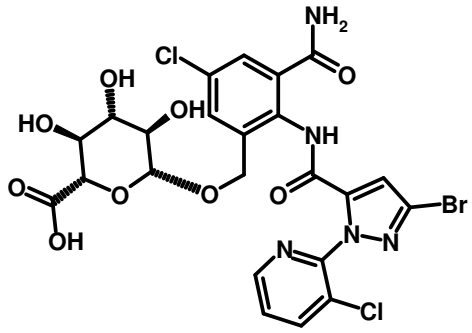
CAS number:	Not available	Molecular Weight:	675.28
Structural formula:	$C_{24}H_{22}BrCl_2N_5O_9$	Observed in:	Goat, rat

IN-HXH40 CAS name: N-[2-Aminocarbonyl]-4-chloro-6-(hydroxymethyl)phenyl]-3-bromo-1-(3-chloro-2-pyridinyl)-1H-pyrazole-5-carboxamide



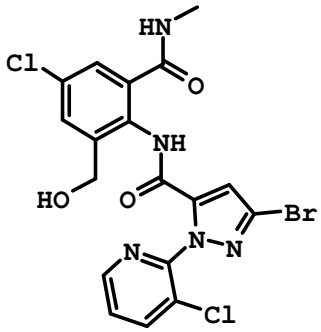
CAS number:	Not available	Molecular Weight:	485.13
Structural formula:	$C_{17}H_{12}BrCl_2N_5O_3$	Observed in:	Goat, hen, rat, rice, confined rotational crops

IN-HXH40-O-glucuronide	CAS name:	[3-(Aminocarbonyl)-2-[[[3-bromo-1-(3-chloro-2-pyridinyl)-1H-pyrazol-5-yl]carbonyl]amino]-5-chlorophenyl]methyl β-D-glucopyranosiduronic acid



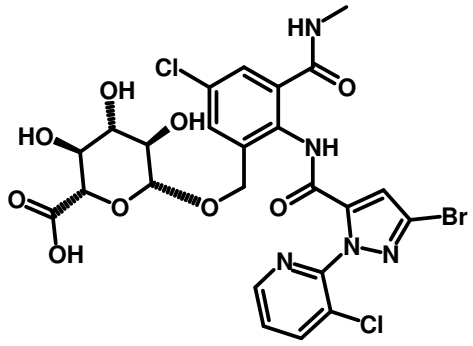
CAS number:	Not available	Molecular Weight:	661.25
Structural formula:	$C_{23}H_{20}BrC_{l2}N_5O_9$	Observed in:	Rat

IN-HXH44	CAS name:	3-Bromo-N-[4-chloro-2-(hydroxymethyl)-6-[(methylamino)carbonyl]phenyl]-1-(3-chloro-2-pyridinyl)-1H-pyrazole-5-carboxamide



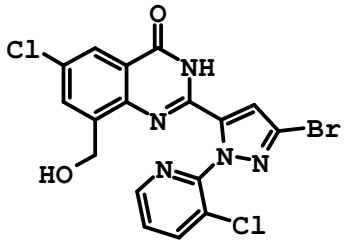
CAS number:	Not available	Molecular Weight:	499.15
Structural formula:	$C_{18}H_{14}BrCl_2N_5O_3$	Observed in:	Goat, hen, rat, rice, confined rotational crops

IN-HXH44-O-glucuronide	CAS name:	[2-[[[3-Bromo-1-(3-chloro-2-pyridinyl)-1H-pyrazol-5-yl]carbonyl]amino]-5-chloro-3-[(methylamino)carbonyl]phenyl]methyl β-D-glucopyranosiduronic acid



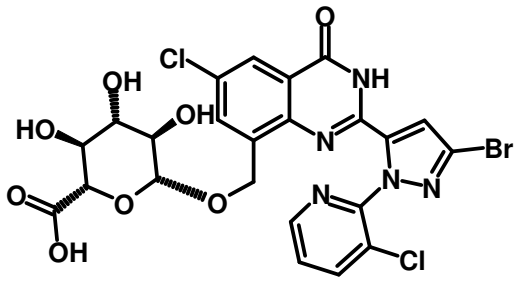
CAS number:	Not available	Molecular Weight:	675.28
Structural formula:	$C_{24}H_{22}BrCl_2N_5O_9$	Observed in:	Goat, hen, rat

IN-K7H29 CAS name: 2-[3-Bromo-1-(3-chloro-2-pyridinyl)-1H-pyrazol-5-yl]-6-chloro-8-(hydroxymethyl)-4(3H)-quinazolinone



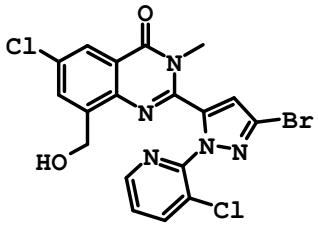
CAS number:	Not available	Molecular Weight:	467.11
Structural formula:	$C_{17}H_{10}BrCl_2N_5O_2$	Observed in:	Goat, hen, rat, confined rotational crops

IN-K7H29-O-glucuronide CAS name: 2-[3-bromo-1-(3-chloro-2-pyridinyl)-1H-pyrazol-5-yl]-6-chloro-1,4-dihydro-4-oxo-8-quinazolinyl]methyl β-D-glucopyranosiduronic acid



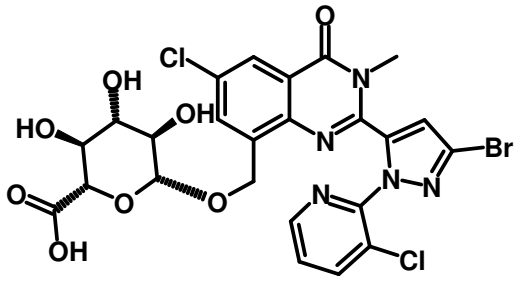
CAS number:	Not available	Molecular Weight:	643.24
Structural formula:	$C_{23}H_{18}BrCl_2N_5O_8$	Observed in:	Goat, hen, rat

IN-K3X21 CAS name: 2-[3-Bromo-1-(3-chloro-2-pyridinyl)-1H-pyrazol-5-yl]-6-chloro-8-(hydroxymethyl)-3-methyl-4(3H)-quinazolinone



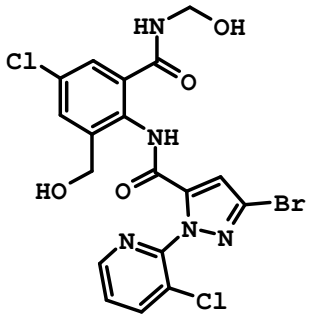
CAS number:	Not available	Molecular Weight:	481.14
Structural formula:	$C_{18}H_{12}BrCl_2N_5O_2$	Observed in:	Goat, hen, rat

IN-K3X21-O-glucuronide CAS name: 2-[3-bromo-1-(3-chloro-2-pyridinyl)-1H-pyrazol-5-yl]-6-chloro-3,4-dihydro-3-methyl-4-oxo-8-quinazolinyl]methyl β-D-glucopyranosiduronic acid



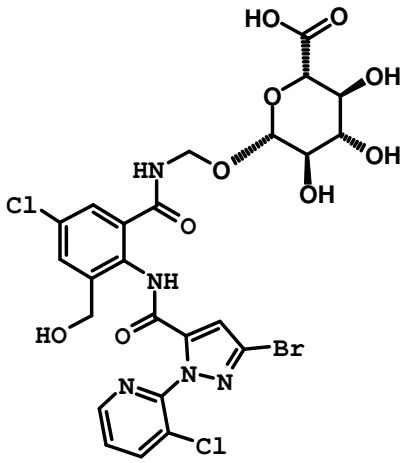
CAS number:	Not available	Molecular Weight:	657.27
Structural formula:	$C_{24}H_{20}BrCl_2N_5O_8$	Observed in:	Goat, hen

IN-K9T00 CAS name: 3-Bromo-N-[4-chloro-2-(hydroxymethyl)-6-[[(hydroxymethyl)amino)carbonyl]phenyl]-1-(3-chloro-2-pyridinyl)-1H-pyrazole-5-carboxamide



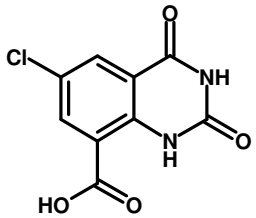
CAS number:	Not available	Molecular Weight:	515.15
Structural formula:	$C_{18}H_{14}BrCl_2N_5O_4$	Observed in:	Confined rotational crops, goat, hen, rat

IN-K9T00-O-glucuronide CAS name: [[2-[[[3-Bromo-1-(3-chloro-2-pyridinyl)-1H-pyrazol-5-yl]carbonyl]amino]-5-chloro-3-(hydroxymethyl)benzoyl]amino]methyl β-D-glucopyranosiduronic acid



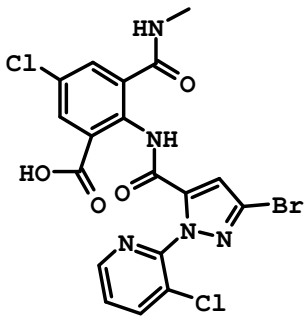
CAS number:	Not available	Molecular Weight:	691.28
Structural formula:	$C_{24}H_{22}BrCl_2N_5O_{10}$	Observed in:	Rat

IN-K9X71 CAS name: 6-Chloro-1, 2, 3, 4-tetrahydro-2, 4-dioxo-8-quinazolinecarboxylic acid



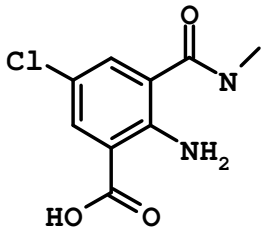
CAS number:	Not available	Molecular Weight:	240.60
Structural formula:	$C_9H_5ClN_2O_4$	Observed in:	Goat, hen, rat, confined rotational crops

IN-KAA24 CAS name: 2-[[[3-Bromo-1-(3-chloro-2-pyridinyl)-1*H*-pyrazol-5-yl]carbonyl]amino]-5-chloro-3-[(methylamino)carbonyl]benzoic acid



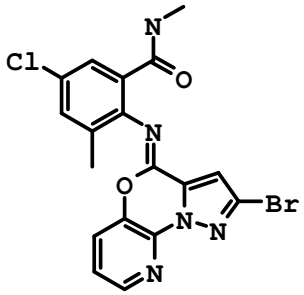
CAS number:	Not available	Molecular Weight:	513.14
Structural formula:	$C_{18}H_{12}BrCl_2N_5O_4$	Observed in:	Goat, hen, rat, rice, confined rotational crops

IN-L8F56 CAS name: 2-Amino-5-chloro-3-[(methylamino)carbonyl]benzoic acid



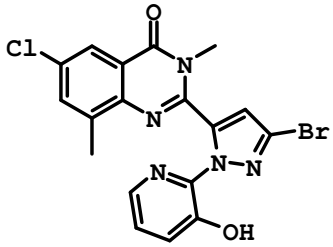
CAS number:	Not available	Molecular Weight:	228.64
Structural formula:	$C_9H_9ClN_2O_3$	Observed in:	Goat, hen, rat, confined rotational crops

IN-LBA22 CAS name: 2-[(2-Bromo-4*H*-pyrazolo[*1,5-d*]pyrido[*3,2-b*] [1.4]oxazin-4-ylidene)amino]-5-chloro-*N*,3-dimethylbenzamide



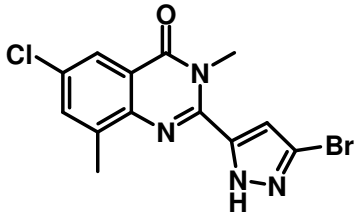
CAS number:	Not available	Molecular Weight:	446.69
Structural formula:	$C_{18}H_{13}BrClN_5O_2$	Observed in:	Aqueous photolysis

IN-LBA23 CAS name: 2-[3-Bromo-1-(3-hydroxy-2-pyridinyl)-1*H*-pyrazol-5-yl]-6-chloro-3,8-dimethyl-4(3*H*)-quinazolinone

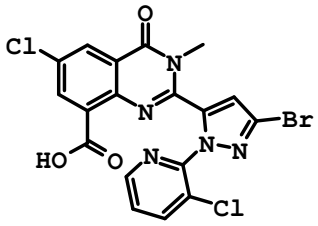
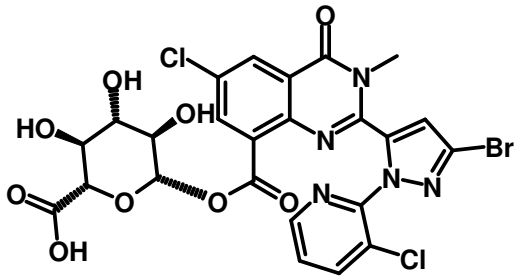
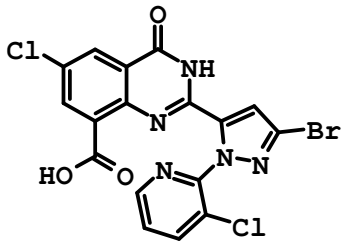


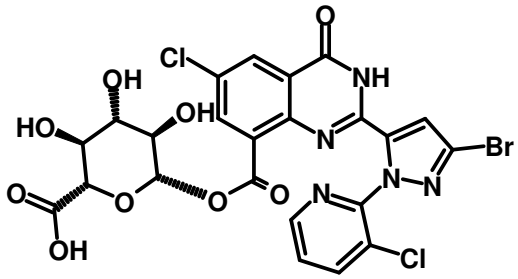
CAS number:	Not available	Molecular Weight:	446.69
Structural formula:	$C_{18}H_{13}BrClN_5O_2$	Observed in:	Aqueous photolysis

IN-LBA24 CAS name: 2-(5-Bromo-1*H*-pyrazol-3-yl)-6-chloro-3,8-dimethyl-4(3*H*)-quinazolinone



CAS number:	Not available	Molecular Weight:	353.61
Structural formula:	$C_{13}H_{10}BrClN_4O$	Observed in:	Aqueous photolysis

IN-LEM10	CAS name:	2-[5-Bromo-2-(3-chloro-pyridin-2-yl)-2*H* pyrazol-3-yl]-6-chloro-3,4-dihydro-3-methyl-4-oxo-8-quinazolinecarboxylic acid	
			
CAS number:	Not available	Molecular Weight:	495.12
Structural formula:	$C_{18}H_{10}BrCl_2N_5O_3$	Observed in:	Goat, hen, rat, confined rotational crops
IN-LEM10 glucuronide	CAS name:	β-D-Glucopyranuronic acid 1-[2-[3-bromo-1-(3-chloro-2-pyridinyl)-1*H*-pyrazol-5-yl]-6-chloro-3,4-dihydro-3-methyl-4-oxo-8-quinazolinecarboxylate	
			
CAS number:	Not available	Molecular Weight:	671.25
Structural formula:	$C_{24}H_{18}BrCl_2N_5O_9$	Observed in:	Goat
IN-LQX30	CAS name:	2-[3-Bromo-1-(3-chloro-2-pyridyl)-1*H*-pyrazol-5-yl]-6-chloro-1,4-dihydro-4-oxo-8-quinazolinecarboxylic acid	
			
CAS number:	Not available	Molecular Weight:	481.10
Structural formula:	$C_{17}H_8BrCl_2N_5O_3$	Observed in:	Hen, rat
IN-LQX30-O-glucuronide	CAS name:	β-D-Glucopyranuronic acid 1-[2-[3-bromo-1-(3-chloro-2-pyridinyl)-1*H*-pyrazol-5-yl]-6-chloro-1,4-dihydro-4-oxo-8-quinazolinecarboxylate	

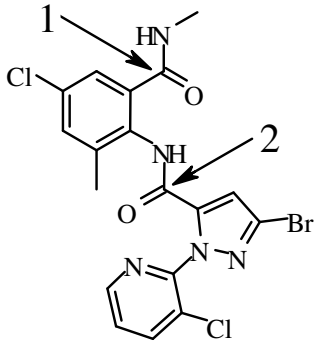


CAS number:	Not available	Molecular Weight:	657.22
Structural formula:	$C_{23}H_{16}BrCl_2N_5O_9$	Observed in:	Goat, hen, rat

Animal Metabolism

The Meeting received studies on the metabolism of chlorantraniliprole in rats, lactating goats and laying hens. The studies on the metabolism of chlorantraniliprole in animals using radioactive material were conducted with chlorantraniliprole labelled with ^{14}C at the benzamide carbonyl (1) and the pyrazole carbonyl (2). The studies on rats were evaluated by the WHO Core Assessment Group.

1. [Benzamide carbonyl–^{14}C]-chlorantraniliprole
2. [Pyrazole carbonyl–^{14}C]-chlorantraniliprole:



The studies on rats showed chlorantraniliprole is well absorbed and rapidly eliminated from the body. Absorption was dose dependent with 73–85% of the dose absorbed at "low dose (10 mg/kg bw) compared with 12–13% at the high dose (200 mg/kg bw). The plasma elimination half-lives ranged from 38–82 h. Most of the administered dose (88–97%) was eliminated in the excreta. Faecal excretion was the primary route of elimination followed by the urine with no significant excretion occurring by exhalation. Metabolism of the absorbed dose was extensive and involved sex differences primarily in initial methylphenyl and N-methyl carbon hydroxylation. Further metabolism of the hydroxylated metabolites included N-demethylation, nitrogen-to-carbon cyclisation with loss of a water molecule, oxidation of alcohols to carboxylic acids, amide bridge cleavage, amine hydrolysis, and O-glucuronidation.

Lactating goat

McLellan *et al.* (2006 14377) dosed orally by gelatine capsule a lactating goat (British Saanen, 42 kg bw) with ^{14}C-chlorantraniliprole at 0.36 mg/kg bw/day for 7 consecutive days (8.5–11.5 ppm in the diet based on feed consumption of 1.3–1.8 kg dry matter/day). The mean milk yield during the dosing period was 1.8 kg/day.

Urine and faeces were collected once daily and milk twice daily (composite afternoon and morning milk from the next day). The animals were slaughtered approximately 23 h after the last dose and tissue (liver, kidney, composite muscle from loin, hind and forequarters, omental fat, renal fat, and subcutaneous fat) and bile samples collected. Radioactivity in all samples was quantified using combustion analysis and LSC and residues characterized by HPLC and LC/MS. Radioactive residues were extracted from milk and tissues using 9:1 acetonitrile:water. Extracts from milk samples were cleaned up using solid phase extraction cartridges which were eluted with ethyl acetate, the extract evaporated to dryness and the residue taken up in acetonitrile:water (3:1 v/v) for analysis by LSC and HPLC. Tissue samples were homogenised and extracted three times with acetonitrile/water (9:1 v/v), centrifuged, the supernatant decanted and evaporated to dryness and the residue dissolved in acetonitrile for analysis by LSC and HPLC. Liver samples were additionally subjected to enzyme digestion (pepsin, protease) and acid as well as base hydrolysis to release more radioactivity. Identification of metabolites was by comparison with retention times and mass spectra of authentic standards. Extraction and initial analysis were conducted within 1 month of sacrifice while primary analyses of ^{14}C residues were completed within 3 months.

The majority of the administered dose was recovered in excreta (79% in faeces, 11% in urine) with an additional 3.9% recovered from the cage wash. Radioactivity retained in tissues, bile or secreted in milk accounted for approximately 1.3% of the administered dose. Overall 95% of administered radioactivity was accounted for.

Radiocarbon content in various tissues were highest in liver (0.64 mg/kg) followed by kidney (0.076 mg/kg), fat (0.07 mg/kg) and muscle (0.016 mg/kg). With the exception of liver, the majority of radioactive residues in tissues and milk were extracted with the organic solvent used, with parent compound accounting for $\geq 80\%$ of the residue in muscle and fat but only 24% in milk, 19% in kidney and 4.0% in liver.

In addition to parent compound, 26 and 27% of the radioactivity in milk was identified as IN-K9T00 and IN-HXH44 respectively. Five minor unidentified components accounted for 9.5% TRR in milk with no individual component > 2.4% TRR.

A number of metabolites, all individually present at < 10% TRR were detected in kidney (IN-L8F56, IN-K9T00, IN-H2H20 and IN-LEM10). A polar component (18% TRR) and five minor components (each < 12% TRR) remained unidentified in kidney.

No individual compound accounted for more than 10% TRR in liver. Major components identified in liver were IN-L8F56 (7.5% TRR) and unchanged parent compound (4.0% TRR). Six compounds present at low levels remained unidentified. Treatment of liver post-extraction solids with pepsin, protease and HCl liberated an additional 32, 5.0 and 21% of the radioactivity respectively. Forty-one components were detected in the protease and 25 in the pepsin digests however most were unresolved in the chromatograms. Unchanged chlorantraniliprole was detected in all sample extracts (solvent, pepsin, protease and base). A total of 13 metabolites were identified in liver at levels corresponding to 0.1–8.2% TRR. Metabolites IN-L8F56, IN-HXH40, IN-DBC80, IN-HXH44, IN-KAA24, IN-LEM10, IN-GAZ70, and IN-EQW78 were all present in at least one liver extract fraction at concentrations > 0.01 mg/kg. Base hydrolysis liberated a number of metabolites/degradation products that were not observed in the other fractions, but these were present at levels ≤ 0.04 mg/kg.

The HPLC profile for bile contained at least 25 radiolabelled components. Unchanged chlorantraniliprole accounted for 1.2% TRR however the major component (58% TRR) was assigned as the glucuronide conjugate of IN-HXH44 by LC/MS. A glucuronide conjugate of IN-K7H29 accounting for 2.5% TRR was also identified. Other minor components in bile were IN-K9X71, IN-K9T00, IN-HXH40, IN-HXH44, IN-K3X21, IN-GKQ52, IN-LEM10, IN-GAZ70, and IN-EQW78. Thirteen unidentified components were detected.

Table 1 Distribution of total radioactive residue (chlorantraniliprole equivalents) and identification of metabolites in milk, liver and kidney after oral dosing of lactating goats with of ^{14}C-chlorantraniliprole (values are given in % of total radioactivity)

	Milk [a]	Kidney	Liver
TRR (mg/kg as chlorantraniliprole)	0.067	0.076	0.64
		%TRR	
Total Extracted	94	84	26
chlorantraniliprole	24	19	4.0
IN-K9X71	ND	ND	ND
IN-L8F56	ND	1.4	7.5
IN-K9T00	26	2.8	ND
IN-HXH40	5.9	ND	ND
IN-DBC80	ND	ND	ND
IN-HXH44	27	3.4	0.85
IN-KAA24	ND	ND	0.64
IN-H2H20	ND	2.5	0.89
IN-K7H29	ND	ND	ND
IN-F9N04	ND	ND	ND
IN-K3X21	ND	ND	ND
IN-GKQ52	ND	ND	ND
IN-LEM10	ND	5.2	ND
IN-GAZ70	ND	ND	ND
IN-EQW78	ND	ND	ND
Unextracted	0	23	77
Sample preparation losses	5.8	-6.5	2.4
Accountability	100	100	100

[a] Composite of milk from days 1–7.

Table 2 Distribution of total radioactive residue (chlorantraniliprole equivalents) and identification of metabolites in muscle and fat after oral dosing of lactating goats with of ^{14}C-chlorantraniliprole (values are given in % of total radioactivity)

	Muscle	Omental Fat	Renal Fat	Subcutaneous Fat
TRR (mg/kg as chlorantraniliprole)	0.016	0.07	0.065	0.068
		%TRR		
Extracted	81	100	97	100
chlorantraniliprole	41	35	67	75
IN-K9X71	ND	1.1		
IN-L8F56	ND	0.96		
IN-K9T00	ND			
IN-HXH40	ND	1.2		
IN-DBC80	ND	0.32		
IN-HXH44	11		1.4	1.8

	Muscle	Omental Fat	Renal Fat	Subcutaneous Fat
TRR (mg/kg as chlorantraniliprole)	0.016	0.07	0.065	0.068
		%TRR		
IN-KAA24	ND	0.60		
IN-H2H20	5.8		1.2	
IN-K7H29	ND	1.8	1.5	
IN-F9N04	ND			
IN-K3X21	ND	0.80		
IN-GKQ52	ND	0.73		
IN-LEM10	ND	1.2	6.9	6.8
IN-GAZ70	ND	4.9		
IN-EQW78	2.0	6.4	11	7.4
Unextracted	13	0.2	0	0.10
Sample preparation losses	6.5	0.12	2.6	0.34
Accountability	100	100	100	100

Metabolites IN-K9T00, IN-HXH44, and unchanged chlorantraniliprole, were the main components detected in faeces, accounting for 11% TRR, 34% TRR and 30% TRR, respectively. The HPLC profile of the urine sample contained at least 16 radiolabelled components. Two major components were identified as IN-K7H29 (16% TRR) and the glucuronide conjugate of IN-HXH44 (29% TRR). Other minor components detected were IN-K9X71, IN-K9T00, IN-HXH40, IN-DBC80, IN-HXH44, IN-GKQ52, and IN-LEM10. Glucuronide conjugates of IN-K7H29 and IN-LQX30 were identified by LC/MS analysis.

A proposed metabolic pathway for chlorantraniliprole in lactating goats is presented in Figure 1.

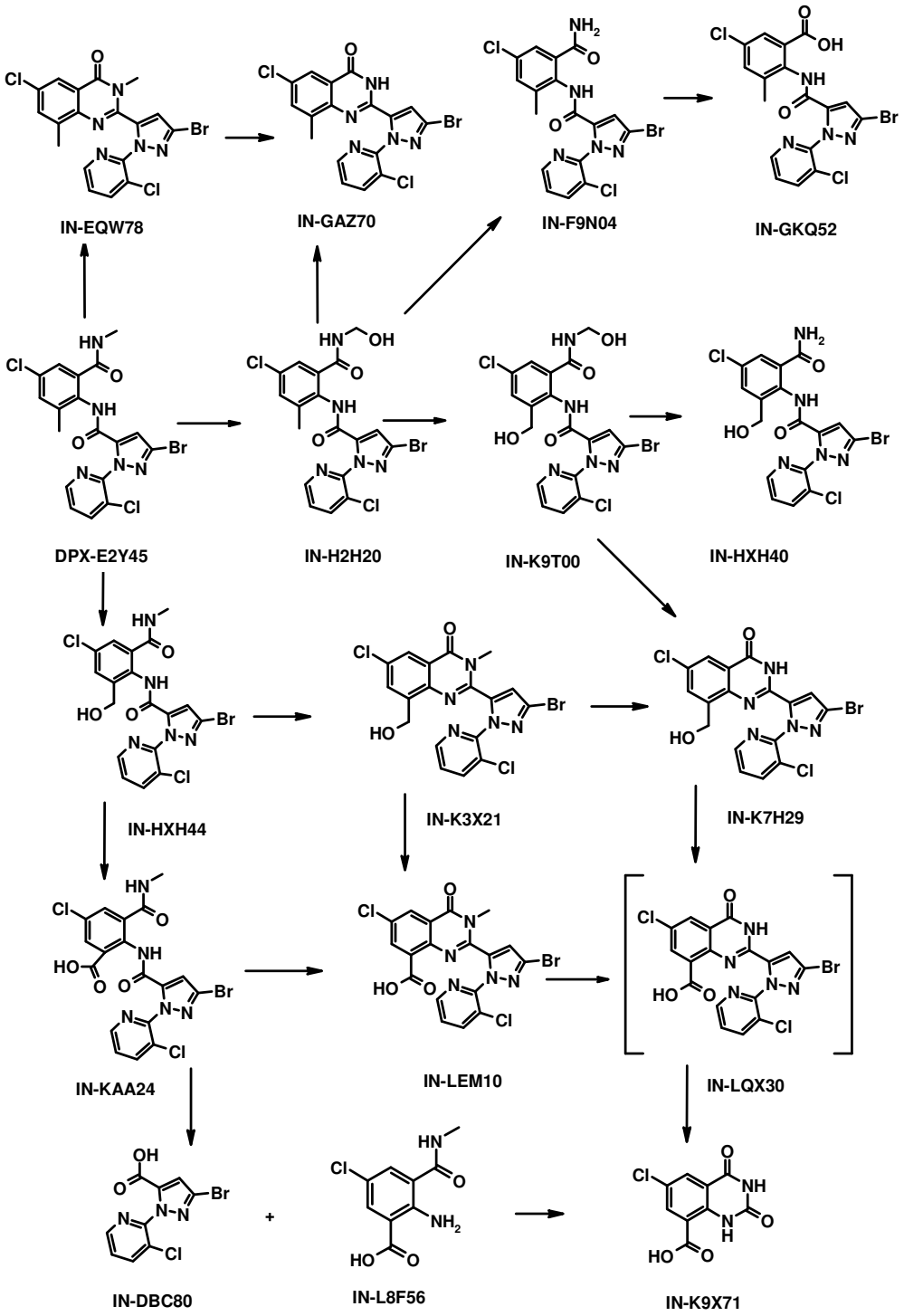


Figure 1 Proposed metabolic pathway for chlorantraniliprole in goats

In summary, when ^{14}C-chlorantraniliprole was orally administered to a lactating goat at 10 ppm in the feed, chlorantraniliprole was the major component of the extracted radioactivity identified in kidney, muscle, and fat samples and was also present in liver. IN-L8F56 was the major component of the ^{14}C identified in liver. Major components identified in milk in addition to chlorantraniliprole (24% TRR) were: IN-K9T00 (26% TRR) and IN-HXH44 (27% TRR).

Laying hen

ISA Brown laying hens (1.6–1.9 kg) were orally dosed at 0.8 mg/kg bw with ^{14}C-chlorantraniliprole by gelatine capsule for 14 consecutive days (MacPherson *et al.,* 2006 14776). Based on pre-dosing daily feed intakes of 93–205 g/day the daily dose would be equivalent to 10 ppm in the diet. Egg yield was 95% for the dose period (100% = 1 egg per hen per day). Eggs, excreta and cage wash were collected daily. Eggs from a single day's collection were separated into whites and yolks. The animals were sacrificed 23 h after the last dose and tissues collected (liver, muscle (thigh and breast), skin with adhering fat, abdominal fat pad and undeveloped eggs).

Radioactivity in egg white (composite days 5–8 and 9-14) and egg yolk (composite days 5–8 and 9-14) samples was extracted several times with hexane and acetonitrile:water (80:20, v/v) and the corresponding extracts combined. Aliquots were assayed by LSC to determine the extracted radioactivity. Muscle and liver samples were homogenised and extracted with a mixture of CH_3CN:water (80:20, v/v) and hexane. The solvent system for extraction of abdominal fat and skin with fat attached was CH_3CN:water (90:10, v/v). For each tissue the pooled extracts were concentrated under a stream of nitrogen and the radioactive residues determined by assaying aliquots of each extract using LSC. A composite excreta sample was extracted with CH_3CN:water (80:20, v/v) and hexane. The combined extracts were concentrated and radioactivity determined by LSC. Known amounts of duplicate sub-samples of the post-extraction solids (PES) were assayed by combustion followed by LSC analysis. Several additional extraction techniques were applied to liver including treatment with protease or pepsin. All the extracts from tissues, eggs and excreta were analysed using reversed phase HPLC. Metabolites were identified by comparing the retention times and confirmed by mass spectrum comparison with authentic references standards. Tissue samples were extracted and initial analysis conducted by HPLC within 1 month of sacrifice. All primary analyses of ^{14}C residues were completed within 3 months, except for the muscle extract, which was stored for up to 13 months.

The majority of the administered radioactivity is excreted (98%), with 5% recovered from cage wash and approximately 3% in eggs (white and yolks). In tissues, the highest concentrations of radioactivity are in liver, followed by fat and muscle. The ratio of residues of chlorantraniliprole (parent compound) in skin with fat and muscle is 12:1.

Chlorantraniliprole and IN-GAZ70 were the major components of the radioactivity in eggs with a large number of metabolites individually present at < 10% TRR, principally IN-K7H29, IN-H2H20, IN-EQW78 and IN-F9N04.

Table 3 Distribution of total radioactive residues (chlorantraniliprole equivalents) and identification of metabolites in eggs after dosing laying hens with ^{14}C-chlorantraniliprole

	Egg yolk (day 5-8)	Egg yolk (day 9-14)	Egg white (day 5-8)	Egg white (day 9-14)	Whole egg (mg/kg) [a] (day 5-8)	(day 9-14)
TRR (mg/kg as chlorantraniliprole)	0.468	0.502	1.294	1.356	1.019	1.071
	%TRR				Residue (mg/kg)	
Total Extracted	80	87	94	82	0.937	0.888
chlorantraniliprole	23	12	32	26	0.308	0.256
IN-GAZ70	4.2	6.6	33	40	0.287	0.377
IN-EQW78	ND	0.85	3.2	6.4	0.028	0.059
IN-K7H29	24	13	3.5	3.1	0.068	0.050
IN-H2H20	17	11	3.5	ND	0.056	0.018
IN-HXH44	ND	2.0	2.9	ND	0.025	0.004
IN-KAA24	ND	1.9	ND	ND	ND	0.003
IN-F9N04	ND	ND	9.2	4.4	0.079	0.037
IN-GKQ52	ND	3.7	ND	ND	ND	0.006
IN-K3X21	ND	0.43	2.1	ND	0.018	0.001

	Egg yolk (day 5-8)	Egg yolk (day 9-14)	Egg white (day 5-8)	Egg white (day 9-14)	Whole egg (day 5-8)	(mg/kg) [a] (day 9-14)
TRR (mg/kg as chlorantraniliprole)	0.468	0.502	1.294	1.356	1.019	1.071
			%TRR		Residue	(mg/kg)
IN-L8F56	ND	0.55	ND	ND	ND	< 0.001
IN-DBC80	ND	4.0	2.6	ND	0.022	0.007
Unextracted	8.5	9.0	1.5	4.0		
Sample preparation losses	11	3.6	4.4	14		
Accountability	100	100	100	100		

ND = not detected

[a] assumed a whole egg is made up of ⅓ yolk and ⅔ egg white

Table 4 Distribution of total radioactive residue (chlorantraniliprole equivalents) and identification of metabolites in different liver, muscle and skin with fat after dosing laying hens with ^{14}C-chlorantraniliprole

	Liver (Solvent)	Liver (Protease)	Liver (Pepsin)	Muscle	Skin with Fat
TRR (mg/kg as chlorantraniliprole)	0.515	0.515	0.515	0.022	0.052
			%TRR		
Total Extracted	37	47	62	54	75
chlorantraniliprole	3.8	2.2	3.3	3.5	18
IN-K9X71	ND	ND	3.7	ND	ND
IN-GAZ70	ND	ND	ND	ND	1.1
IN-EQW78	ND	ND	ND	6.8	3.1
IN-K7H29	2.3	ND	ND	1.0	3.2
IN-H2H20	0.50	ND	ND	ND	ND
IN-HXH44	1.6	2.0	ND	0.88	ND
IN-KAA24	ND	ND	0.77	ND	ND
IN-F9N04	ND	1.2	5.4	ND	8.8
IN-GKQ52	4.0	1.8	5.0	ND	ND
IN-K3X21	ND	ND	ND	1.5	5.9
IN-HXH40	3.2	ND	2.9	1.1	1.3
IN-L8F56	0.41	ND	1.2	0.88	ND
IN-DBC80	ND	ND	1.6	ND	ND
IN-LEM10	ND	ND	ND	ND	ND
Unextracted	48	25	19	31	24
Sample preparation losses	15	27	19	14	0.90
Accountability	100	100	100	100	100

ND = not detected

Unchanged parent compound and metabolites individually accounted for < 10% TRR in liver and muscle with chlorantraniliprole present at only 2.2–3.7% TRR. Residues of chlorantraniliprole formed the major component of the residue in skin with fat at 18% TRR. No other metabolite exceeded 9% TRR.

Chlorantraniliprole is metabolized in the hen primarily by three major pathways:

- hydroxylation of N-methyl and methyl-phenyl carbons to yield IN-H2H20 and IN-HXH44 respectively;
- condensation with a loss of water from chlorantraniliprole to yield a quinazolinone derivative, IN-EQW78. Similar condensation of the oxidative metabolites (IN-HXH44, IN-H2H20, IN-KAA24, and IN-K9T00) generates corresponding quinazolinone derivatives (IN-K3X21, IN-GAZ70, IN-LEM10, and IN-K7H29);
- N-demethylation of hydroxymethylamide group in IN-H2H20 to IN-F9N04 and amidic bridge cleavage between phenyl and heterocyclic rings yields IN-L8F56 and IN-DBC80.

See Figure 2 for the proposed metabolic pathway for chlorantraniliprole in laying hens.

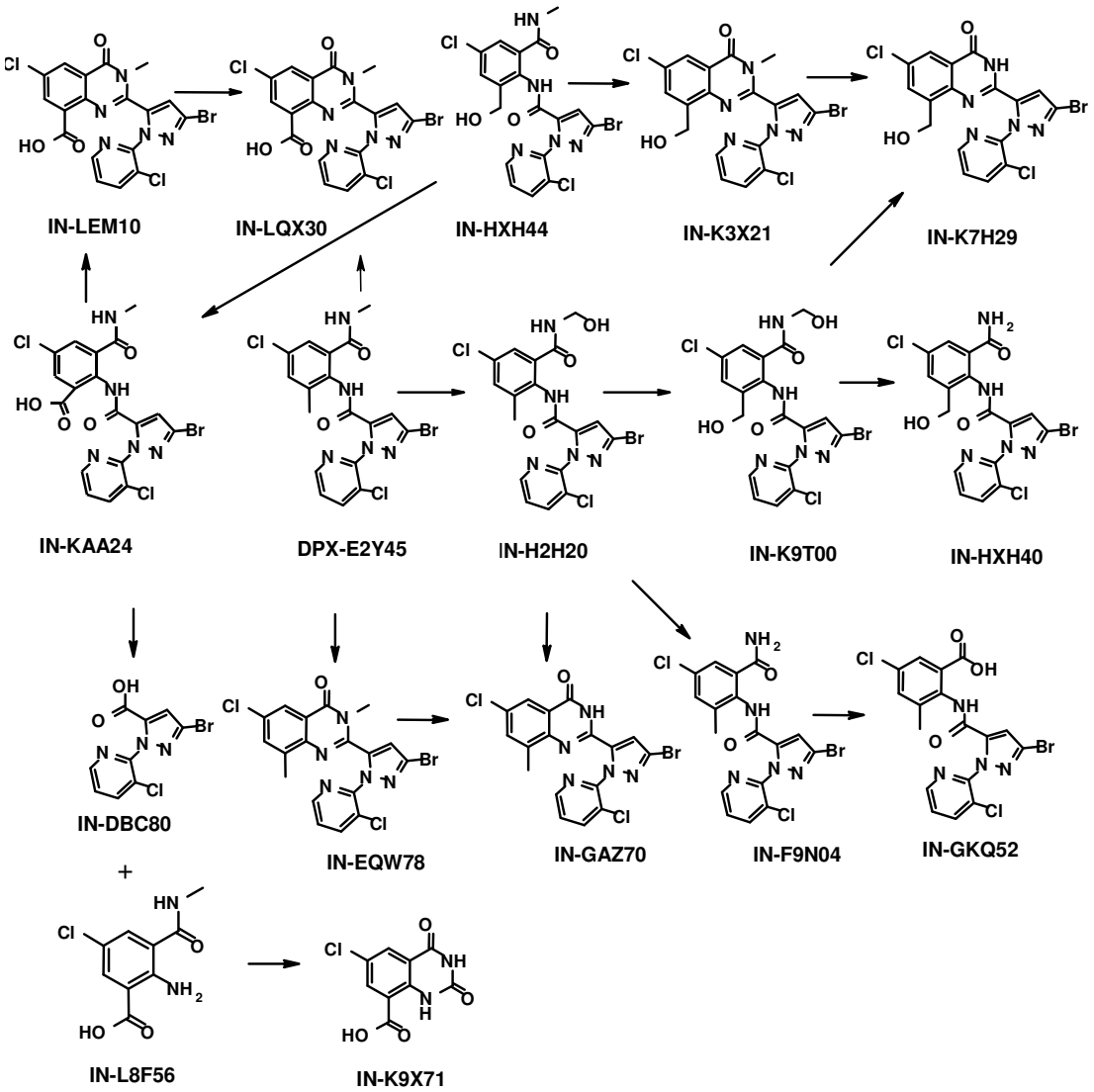


Figure 2 Proposed metabolic pathway for chlorantraniliprole in laying hens

Summary of metabolism of chlorantraniliprole in animals

Radiolabelled chlorantraniliprole separately ^{14}C-labelled at the benzamide-carbonyl and pyrazole-carbonyl positions, was used in the metabolism and environmental studies. The metabolism of laboratory animals was qualitatively the same as for farm animals though some species related differences were noted. The major route of chlorantraniliprole metabolism in livestock is via (i)

hydroxylation of the N-methyl group (to IN-H2H20) or hydroxylation of the tolyl methyl group (to IN-HXH44); (ii) cyclization with loss of water to a quinazolinone derivative (IN-EQW78); and (iii) N-demethylation via IN-H2H20 to IN-F9N04.

Plant metabolism

Metabolism studies on apples, tomato, lettuce, rice, and cotton were made available to the Meeting.

Apples

MacDonald et al (2005 12264), applied radiolabelled ^{14}C-chlorantraniliprole (20% SC formulations of benzamide carbonyl-^{14}C and pyrazole carbonyl-^{14}C-chlorantraniliprole individually) to apple trees (cv Braeburn) maintained in a glasshouse as three foliar applications of 100 g ai/ha at varying stages of fruit formation and maturity: application 1 when fruit had reached 10% of final size (early fruiting stage; BBCH 71), application 2 when fruit had reached 50% of final size (mid-fruit size; BBCH 75), and application 3 (BBCH 77) at 30 days prior to maturity/harvest. Samples of immature apple leaves and fruit were taken immediately after the first application (Sample 1), before (Sample 2) and after the second (Sample 3) and third (Sample 4, 5) applications and at 15 days after the third application (Sample 6). The final (maturity) harvest samples were taken at 30 days after the last application (Sample 7). The apple leaves and fruit were rinsed with acetonitrile (CH_3CN) and the surface residues determined by LSC. All samples were stored at –20 °C after surface rinsing.

Rinsed fruit was homogenized and extracted twice with CH_3CN, followed by two extractions with CH_3CN:H_2O, 1:1 v/v. The radioactivity in the extracts was determined using LSC. The remaining radioactivity in the post extraction solids (PES) was determined by combustion analysis. Total radioactive residues in each sample were determined by summing the radioactivity in the surface rinses, the extracts, and post-extraction solids. All samples were analysed within 1 month of collection. The surface rinse from the benzamide carbonyl-^{14}C-chlorantraniliprole apple sample at 30 days after the final application was subjected to further characterization and analysis using LC/MS.

TRR in the leaf and apple samples, including surfaces rinses are shown in Tables 5 and 6 below for sampling points 1, 2, 4, 6 and 7.

Table 5 Distribution of total radioactive residues (mg/kg as chlorantraniliprole) in apple leaves and fruit

	[benzamide carbonyl-^{14}C]-chlorantraniliprole			[pyrazole carbonyl-^{14}C]- chlorantraniliprole		
	TRR	Surface rinse (% TRR)	Rinsed sample (% TRR)	TRR	Surface rinse (% TRR)	Rinsed sample (% TRR)
Apple leaves						
Sample 1	9.646	76	24	9.347	77	23
Sample2	2.603	79	21	4.225	37	63
Sample3	9.971	69	31	7.594	70	30
Sample4	5.188	76	24	3.723	73	27
Sample5	14.733	86	13	9.729	91	9.3
Sample6	6.457	86	14	4.991	84	16
Sample7	4.153	66	34	4.280	75	25
Apple fruit						
Sample1	0.672	92	8.5	0.626	96	4.3
Sample2	0.088	72	28	0.032	68	32
Sample3	0.405	96	4.5	0.298	92	8.1
Sample4	0.110	88	12	0.055	78	22
Sample5	0.163	96	3.5	0.213	95	5.1
Sample6	0.138	93	7.4	0.104	92	8.2
Sample7	0.107	79	21	0.092	75	25

Sample1 = samples collected immediately after application 1
Sample2 = samples collected immediately prior to application 2
Sample 3 = samples collected immediately after application 2
Sample4 = samples collected immediately prior to application 3
Sample 5 = samples collected immediately after application 3
Sample 6 = samples collected 15 days after application 3
Sample 7 = samples collected 30 days after application 3

Table 6 Nature of the radioactive residues in apple leaves following foliar applications of ^{14}C-chlorantraniliprole

	Sample 1	Sample 2	Sample 4	Sample 6	Sample 7
[benzamide carbonyl-^{14}C]-chlorantraniliprole					
Total TRR (mg equiv/kg) [a]	9.521	2.645	5.204	6.372	3.966
			%TRR		
CH_3CN surface rinse	76	80	76	86	66
chlorantraniliprole	75	77	75	83	64
Unidentified	1.0	1.9	1.9	2.2	1.6
CH_3CN leaf extract	20	15	20	12	26
chlorantraniliprole	20	15	19	11	25
Unidentified	0.2	0.3	0.9	0.1	0.4
CH_3CN/water leaf extract	2.1	6.0	2.9	0.6	3.3
chlorantraniliprole	< 0.1	5.4	2.7	0.6	2.9
Unidentified	2.1	0.5	0.1	< 0.	0.1
Total rinse + extracted	99	101	99	98	95
Unextracted	0.1	0.5	0.9	0.3	0.7
Total chlorantraniliprole [b]	95	98	96	96	92
[pyrazole carbonyl-^{14}C]-chlorantraniliprole					
Total TRR (mg equiv/kg) [a]	9.244	3.950	3.682	5.026	4.216
			%TRR		
CH_3CN surface rinse	77	36	73	84	75
chlorantraniliprole	75	35	71	82	72
Unidentified	1.0	1.4	1.4	1.5	3.1
CH_3CN leaf extract	18	55	25	15	20
chlorantraniliprole	18	53	23	13	20
Unidentified	ND	1.7	1.3	0.9	0.2
CH_3CN/water leaf extract	3.5	1.4	2.2	0.8	2.2
chlorantraniliprole	< 0.1	1.2	1.9	0.5	2.0
Unidentified	3.5	0.1	0.1	0.1	0.1
Total rinse + extracted	99	93	100	100	98
Unextracted	0.2	0.8	0.8	0.7	0.9
Total chlorantraniliprole [b]	94	89	97	96	94

[a] Total TRR = Total extracted + total unextracted.

[b] Total chlorantraniliprole identified = sum of chlorantraniliprole in the surface wash, acetonitrile extract and aqueous acetonitrile extract

ND = Not detectable

Sample 1 = samples collected immediately following application 1

Sample 2 = samples collected immediately prior to application 2
Sample 4 = samples collected immediately prior to application 3
Sample 6 = samples collected 15 days after application 3
Sample 7 = samples collected 30 days after application 3

The surface rinse and rinsed fruit extracts showed one major component in all samples analysed that was identified as chlorantraniliprole.

The majority of radioactivity on leaves is removed by surface rinses. There appears to be a small amount of transfer from the leaf surface to the leaf itself. Apple leaves collected 15 days after the third 100 g ai/ha application with benzamide carbonyl-^{14}C and pyrazole carbonyl-^{14}C chlorantraniliprole contained total residues of 6.46 and 4.99 mg/kg equivalents, respectively. The majority of the residue was rinsed from the surface of the leaves (84.5–86.1% TRR).

At maturity, most of the radioactivity in apple leaves collected 30 days after the third 100 g ai/ha application was removed by surface rinsing (65.9–75.3% TRR).

Table 7 Nature of the radioactive residues in apple fruit following foliar application of ^{14}C-chlorantraniliprole

	Sample 1	Sample 2	Sample 4	Sample 6	Sample 7
[benzamide carbonyl-^{14}C]-chlorantraniliprole					
Total TRR (mg equiv/kg) [a]	0.672	0.083	0.110	0.131	0.100
			%TRR		
CH_3CN surface rinse	92	71	86	89	79
chlorantraniliprole	90	67	84	87	76
Unidentified	0.6	2.7	1.7	1.4	1.6
CH_3CN fruit extract	7.9	18	12	4.9	10
chlorantraniliprole	< 0.1	16	6.3	4.3	7.3
Unidentified	7.9	1.4	4.6	0.4	3.1
CH_3CN/water fruit extract	0.5	3.7	0.9	0.6	2.7
chlorantraniliprole	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
Unidentified	0.5	3.7	0.9	0.6	2.7
Total rinse + extracted	100	93	99	94	92
Unextracted	0.1	1.8	1.3	0.9	1.7
Total chlorantraniliprole [b]	90	84	91	92	84
[pyrazole carbonyl-^{14}C]- chlorantraniliprole					
Total TRR (mg equiv/kg) [a]	0.625	0.030	0.055	0.094	0.090
			%TRR		
CH_3CN surface rinse	96	65	74	85	75
chlorantraniliprole	94	62	72	84	74
Unidentified	< 0.1	2.4	1.4	0.9	0.5
CH_3CN fruit extract	3.8	26	26	4.3	18
chlorantraniliprole	< 0.1	23	19	2.6	9.3
Unidentified	3.8	2.5	7.3	0.8	8.3
CH_3CN /water fruit extract	0.5	1.5	1.7	0.6	4.3
chlorantraniliprole	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
Unidentified	0.5	1.5	1.7	0.6	4.3
Total rinse + extracted	100	93	102	90	97
Unextracted	< 0.1	2.1	1.3	1.1	1.3
Total chlorantraniliprole [b]	95	85	91	86	83

[a] Total TRR = Total extracted + total unextracted.

[b] Total chlorantraniliprole identified = sum of chlorantraniliprole in the surface wash, acetonitrile extract and aqueous acetonitrile extract

Sample 1 = samples collected immediately following application 1

Sample 2 = samples collected immediately prior to application 2

Sample 4 = samples collected immediately prior to application 3

Sample 6 = samples collected 15 days after application 3

Sample 7 = samples collected 30 days after application 3

Apple fruit collected 15 days after the third application of benzamide carbonyl-^{14}C or pyrazole carbonyl-^{14}C chlorantraniliprole, contained total residues of 0.137 or 0.104 mg/kg equivalents, respectively. The majority of the residue was rinsed from the surface of the fruit (91.8–92.6% TRR). At maturity (30 days after the last application), the TRR in apple fruit was 0.107 and 0.092 mg/kg equivalents in the benzamide carbonyl-^{14}C and pyrazole carbonyl-^{14}C chlorantraniliprole treated samples, respectively. Once more most of the radioactive residues were recovered in the surface rinses (74.8–78.8% TRR). With both leaf and apples samples there appear to be no significant differences between the two labels.

The major component of the radioactivity in surface rinses and solvent extracts of rinsed fruit and leaves was parent chlorantraniliprole, comprising 83–92% of the extracted TRR.

When chlorantraniliprole is applied to apple trees in a treatment regime that reflects the proposed use pattern, most of the applied radioactivity is present on the surface of the fruit and leaves. A large proportion of the applied radioactivity is readily extracted into CH_3CN and CH_3CN: H_2O and the majority of the extracted radioactivity from leaves and fruit is chlorantraniliprole.

Tomato

MacDonald and Gray (2005 12266) studied the metabolism of chlorantraniliprole on tomatoes. Greenhouse grown tomato plants were treated with a 1:1 mixture of [benzamide carbonyl–^{14}C]- and [pyrazole carbonyl–^{14}C]-chlorantraniliprole as a 20% SC formulation. The chlorantraniliprole mixture was applied as three foliar applications at rates equivalent to 100 g ai/ha to the tomato plants (cv. Moneymaker) at varying stages of plant growth and fruit formation: application 1 at stages ranging from 62 days post-emergence to late bloom early fruiting (BBCH 19 - 61), application 2 at 23 days after the 1st application (BBCH 19–73), and application 3 at 27 days after the 2nd application (BBCH 19–81). Samples of immature tomato leaf and fruit were taken immediately after the first application. Immature treated tomato leaf and fruit samples were taken before and after the second and third (final) applications and at 7 days after the final application. Final (maturity) harvest was 15 days after the last application.

Each sample was rinsed with CH_3CN before being homogenised and the total radioactive residue (TRR) determined by liquid scintillation counting (LSC). All samples were stored at –20 °C after surface rinsing and prior to homogenisation. Washed fruit and leaves were homogenized and extracted twice with CH_3CN, followed by two extractions with CH_3CN:H_2O 1:1, v/v. Each of the extracts was separated from the remaining solids by centrifugation. The radioactivity in the extracts was determined using LSC. The remaining radioactivity in the post-extracted solids (PES) was determined by LSC and/or combustion analysis. Total radioactive residues (TRR) in each sample were determined by summing the radioactivity in the surface washes, the extracts, and post-extraction solids. The surface wash from the tomato fruit samples at 15 days after the final application was subjected to further characterization by analysis using LC/MS. All samples were analysed within 3 months of sample collection.

In Table 8, the TRR in surface rinses, leaf and fruit taken at all sampling points are shown.

Table 8 Distribution of total radioactive residues in tomato leaves and fruit sampled at various intervals following foliar application of ^{14}C-chlorantraniliprole

	Leaves			Fruit		
	TRR mg/kg equivalents	Surface rinse (%TRR)	Rinsed leaves (%TRR)	TRR mg/kg equivalents	Surface rinse (%TRR)	Rinsed fruit (%TRR)
Sample 1	0.856	93	7.2	NA	NA	NA
Sample 2	0.318	79	21	0.001	85	15*
Sample 3	1.313	76	24	0.073	88	12
Sample 4	0.926	52	48	0.012	65	35
Sample 5	2.348	71	29	0.032	79	21
Sample 6	2.451	73	27	0.056	79	21
Sample 7	1.365	83	17	0.013	83	17

NA = Sample not collected

Sample 1 = samples collected immediately following application 1

Sample 2 = samples collected immediately prior to application 2

Sample 3 = samples collected immediately following application 2

Sample 4 = samples collected immediately prior to application 3

Sample 5 = samples collected immediately following application 3

Sample 6 = samples collected 7 days after application 3 (7-day PHI)

Sample 7 = samples collected 15 days after following application 3 (15-day PHI)

^{14}C residues are highest in tomato fruit and leaf samples taken immediately after each application. The total radioactive residues in the tomato fruit sampled 7 and 15 days following the last application were 0.056 and 0.013 mg/kg equivalents, respectively. The total radioactive residues in the tomato leaf sampled 7 and 15 days following the last application were 2.45 and 1.36 mg/kg equivalents respectively.

The wash and extract samples that were subjected to HPLC analysis showed the same major component in all samples collected at various intervals that was identified as chlorantraniliprole.

TRR in the leaf and tomato samples, including surfaces rinses, are tabulated below for samples taken at harvests 1, 2, 4, 6 and 7. The proportion of parent chlorantraniliprole in each of the extracts as identified by HPLC is also shown.

Table 9 Nature of the radioactive residues in tomato leaves following foliar application of ^{14}C-chlorantraniliprole

	% TRR in each component of tomato leaves (mg/kg)				
	Sample 1	Sample 2	Sample 4	Sample 6	Sample 7
Total TRR [a]	0.796	0.311	0.923	2.458	1.374
	%TRR				
CH_3CN surface rinse	86	78	52	73	83
chlorantraniliprole	82	77	52	72	82
Unidentified	4.3	1.6	0.3	0.9	0.5
CH_3CN leaf extract	5.9	15	44	24	16
chlorantraniliprole	5.4	14	42	23	15
Unidentified	0.6	0.9	0.7	0.9	0.9
CH_3CN/water leaf extract	0.5	3.2	3.3	2.1	1.3
chlorantraniliprole	NP	3.0	1.9	2.0	1.1
Unidentified	NP	< 0.1	1.2	< 0.1	< 0.1

	% TRR in each component of tomato leaves (mg/kg)				
	Sample 1	Sample 2	Sample 4	Sample 6	Sample 7
Total rinse + extracted	923	96	99	100	100
Total chlorantraniliprole [b]	87	93	96	97	98
Unextracted	0.1	1.3	0.7	0.6	0.4

[a] Total TRR was derived by summing up identified chlorantraniliprole, characterized, unidentified, and unextracted residues.

[b] Total chlorantraniliprole identified = sum of chlorantraniliprole in the surface wash, acetonitrile extract and aqueous acetonitrile extract

NP = Not profiled

Sample 1 = samples collected immediately following application 1

Sample 2 = samples collected immediately prior to application 2

Sample 4 = samples collected immediately prior to application 3

Sample 6 = samples collected 7 days after application 3 (7-day PHI)

Sample 7 = samples collected 15 days after following application 3 (15-day PHI)

Table 10 Nature of the radioactive residues in tomato fruit following foliar application of ^{14}C-chlorantraniliprole

Sample Point	% TRR in each component of tomato fruit (mg/kg)				
	Sample 1	Sample 2	Sample 4	Sample 6	Sample 7
Total TRR [a]	NS	0.001	0.011	0.053	0.012
			%TRR		
CH_3CN surface rinse	-	87	60	78	79
chlorantraniliprole	-	85	59	76	79
Unidentified	-	ND	0.5	1.0	0.4
CH_3CN fruit extract	-	ND	32	17	15
chlorantraniliprole	-	ND	26	16	13
Unidentified	-	ND	3.1	0.4	ND
CH_3CN/water fruit extract:	-	ND	ND	ND	ND
chlorantraniliprole	-	ND	ND	ND	ND
Unidentified	-	ND	ND	ND	ND
Total rinse + extracted	-	87	92	95	95
Total chlorantraniliprole [b]	-	85	85	93	92
Unextracted	-	15*	0.9**	0.3 **	1.1

[a] Total TRR was derived by summing up identified chlorantraniliprole, characterized, unidentified, and unextracted residues.

[b] Total chlorantraniliprole identified = sum of chlorantraniliprole in the surface rinse, acetonitrile extract and aqueous acetonitrile extract

ND = Not detectable; < 0.001 mg/kg equivalents.

NS = No fruit sample available at this sampling interval

Sample 1 = samples collected immediately following application 1

Sample 2 = samples collected immediately prior to application 2

Sample 4 = samples collected immediately prior to application 3

Sample 6 = samples collected 7 days after application 3 (7-day PHI)

Sample 7 = samples collected 15 days after following application 3 (15-day PHI)

The majority of residue in TRR of tomato leaves collected 7 days after the third application was chlorantraniliprole (97.1% TRR; surface rinse 72.1% and CH_3CN extracts 25%). At maturity,

surface rinses accounted for 82.7% TRR levels in the tomato leaves (collected 15 days after the third application of 100 g ai/ha).

TRR levels in tomato fruit collected 7 days after the third application at 100 g ai/ha were 0.056 mg/kg equivalents. The majority of the radioactivity was removed by surface rinses (78% TRR). Chlorantraniliprole accounted for 93% of the TRR.

Tomato fruit at maturity, collected 15 days after the third application, contained radioactive residues of 0.013 mg/kg. The majority of residues were removed by rinsing with acetonitrile (79% TRR) and is present on the surface of the fruit.

Parent chlorantraniliprole comprised the majority of the radioactive residues in day 7 and day 15 tomato samples, 92–93% of the TRR.

When chlorantraniliprole is applied to tomato plants in a treatment regime that reflects the proposed use pattern, most of the applied radioactivity is present on the surface of the fruit and leaves. A large proportion of the applied radioactivity is easily extracted into CH_3CN and CH_3CN: H_2O and the largest component of the radioactivity from leaves and fruit is parent chlorantraniliprole.

Lettuce

Lettuce plants grown in an outdoor test plot were treated with a mixture of the two radiolabelled forms of chlorantraniliprole (1:1 [benzamide carbonyl-^{14}C]- and [pyrazole carbonyl-^{14}C]-chlorantraniliprole), applied as a 20% SC formulation (MacDonald et al. 2007 12265). The chlorantraniliprole mixture was applied as three foliar applications equivalent to 100 g ai/ha to the lettuce (cv. Green salad bowl) at varying stages of plant growth: application 1 at 29 days post emergence (BBCH 13), application 2 at 13 days after the 1st application (BBCH 19), and application 3 at 10 days after the 2nd application (BBCH 19). Samples of lettuce were harvested by cutting at the soil surface. Samples of immature lettuce samples (whole plants) were taken immediately after the first application, before and after the second and third (final) applications and at 7 days after the final application. The final (maturity) harvest was taken at 15 days after the last application. The samples of lettuce were rinsed with CH_3CN and the surface residues were determined using LSC. The rinsed lettuce samples were homogenized and then extracted using CH_3CN followed by CH_3CN:H_2O (acetonitrile: water 1:1, v/v). The unextracted radioactivity in the post-extraction solids was determined by combustion analysis. Total radioactive residues (TRR) in each sample were determined by summing the radioactivity in the surface rinses, extracts and post-extraction solids. Further characterization was by HPLC with identification of components by comparison with an authentic standard. Radioactive residues in lettuce were extracted and initially analysed by HPLC within 30 days of harvest. All analyses of ^{14}C residues were completed within 3 months.

The TRR in the surface rinse and from the lettuce samples collected pre- and post-application 3, and 7 and 15 days after application 3 are shown in Table 11.

Table 11 Distribution of ^{14}C residues in lettuce following foliar application of ^{14}C-chlorantraniliprole

	TRR mg/kg	Surface rinse mg/kg (% TRR)	Rinsed leaves mg/kg (% TRR)
Sample 1	1.864	67	33
Sample 2	0.190	37	63
Sample 3	2.860	92	7.9
Sample 4	0.088	71	29
Sample 5	1.339	84	16
Sample 6	0.372	61	39
Sample 7	0.301	44	56

Sample 1 = samples collected immediately following application 1

Sample 2 = samples collected immediately prior to application 2

Sample 3 = samples collected immediately following application 2

Sample 4 = samples collected immediately prior to application 3

Sample 5 = samples collected immediately following application 3

Sample 6 = samples collected 7 days after application 3 (7-day PHI)

Sample 7 = samples collected 15 days after application 3 (15-day PHI)

Lettuce samples collected 7 days after the third application at 100 g ai/ha contained ^{14}C residues of 0.372 mg/kg equivalents. The majority of this residue was rinsed from the lettuce surface (61% TRR). At maturity the TRR in lettuce collected 15 days after the third application was 0.301 mg/kg equivalents. Most of the radioactivity was removed by surface rinses (44% TRR) and extraction with CH_3CN (50% TRR).

Table 12 Nature of the radioactive residues in lettuce following foliar application of ^{14}C-chlorantraniliprole

	Sample 1	Sample 2	Sample 4	Sample 6	Sample 7
Total TRR [a]	1.791	0.186	0.086	[c] 0.372	0.276
			%TRR		
CH_3CN surface rinse	67	35	66	64	39
Chlorantraniliprole	66	34	64	63	39
Unidentified	0.2	0.4	2.2	0.6	0.2
CH_3CN extract	29	59	30	38	46
Chlorantraniliprole	28	58	29	36	45
Unidentified	0.3	0.5	0.8	1.2	0.5
CH_3CN/water extract:	0.5	2.7	ND	2.2	4.6
Chlorantraniliprole	NA	NA	ND	NA	4.2
Unidentified	0.5	2.7	ND	2.2	0.1
Total rinse + extracted	96	97	97	104	90
Unextracted	0.1	0.8	0.6	1.1	1.8
Total chlorantraniliprole [b]	94	92	93	99	89

[a] Total TRR = Total extracted + total unextracted.

[b] Total chlorantraniliprole identified = sum of chlorantraniliprole in the surface rinse, acetonitrile extract and aqueous acetonitrile extract

[c] Total TRR = (Total extracted + total unextracted) – concentration differences extract #1 = (104 + 1.1) – 5.1 = 100%.

ND = Not detectable

NA = Not analysed

Sample 1 = samples collected immediately following application 1

Sample 2 = samples collected immediately prior to application 2

Sample 4 = samples collected immediately prior to application 3

Sample 6 = samples collected 7 days after application 3 (7-day PHI)

Sample 7 = samples collected 15 days after application 3 (15-day PHI)

At each of the sampling intervals > 88% of the total radioactive residue (TRR) was identified as chlorantraniliprole. Several other minor radioactive components, individually not exceeding 2.7% TRR or 0.005 mg/kg were also observed at other sampling intervals, but not further identified due to low levels of radioactivity.

In summary, when chlorantraniliprole is applied to lettuce in a treatment regime that reflects the proposed use pattern, most of the applied radioactivity is present on the leaf surface. A large proportion of the radioactivity present in leaves is extracted into CH_3CN and CH_3CN: H_2O. The majority of the extracted radioactivity is present as parent chlorantraniliprole.

Rice

Chlorantraniliprole (a 1:1 mixture [benzamide carbonyl-^{14}C]- and [pyrazole carbonyl-^{14}C] chlorantraniliprole), was applied as a soil drench to rice plants (cv. Montsianell). The test material was formulated as a 20% SC and applied at a rate equivalent to 300 g ai/ha. The plants were at the 1–2 leaf stage of growth (BBCH 11–12) at the time of application and were grown outdoors under protective netting. Two days after application of the ^{14}C-chlorantraniliprole, the plants were flooded. The rice plants were maintained in water until 4 days prior to crop maturity/harvest when the flood water was drained to allow the plants to dry (Chapleo & Gray 2006 16967). Immature plant samples (whole plants) were collected at 14, 28 and 56 days after application, with the final harvest at 132 days after application (BBCH 87). Whole plants sampled at 14, 28 and 56 days were separated into leaf, sheath, root, and whole panicle fractions. The whole panicles sampled at crop maturity were separated into grain (with bran) and hulls. Samples were stored frozen at −20 °C prior or preparation for analysis.

Soil core samples were taken immediately after application. After flooding, samples of flood water and sediment were taken at 14, 28, 56 and 128 days after application.

Soil sediment samples were shaken with CH_3CN:H_2O (9:1 v/v) and the solvent extracts separated from the soil by centrifugation. This was repeated and the extracts combined (extract #1). The soil was then shaken with a mixture of CH_3CN:HCHOOH (4:1 v/v) and separated. This was repeated twice and the extracts combined to form extract #2. Radioactivity in the extracts was determined by LSC and in the post extraction solids using oxidative combustion followed by LSC.

Each of the rice fractions was extracted twice with CH_3CN (extract #1), followed by two extractions with CH_3CN:H_2O (1:1 v/v) (extract #2). Where the TRR in each of the extracts was considered significant, equivalent volumes of extracts #1 and #2 were combined, evaporated to dryness and taken up in CH_3CN or CH_3CN:H_2O prior to analysis using HPLC. In some cases, the extracts separated into organosoluble and aqueous fractions prior to further analysis. For example the leaf and sheath extracts were partitioned against CH_2Cl_2 reduced and the organosoluble phase taken up in CH_3CN:H_2O prior to analysis by LSC and HPLC. The aqueous phase was analysed directly by LSC and HPLC.

Radioactive residues greater than 0.05 mg/kg equivalents which remained in the PES of samples of leaf, hull and grain (with bran) at harvest were subjected to further extraction.

Additional extraction procedures included enzyme hydrolysis (using driselase: a mixture of exo-hydrolases, including galactosidases, glucosidases and mannosidases and endohydrolases, including cellulase and pectinase) and acid and base hydrolysis. The radioactivity in selected driselase, acid, and base extracts was further characterized by partitioning with ethyl acetate. Each sample was shaken with ethyl acetate to produce an aqueous and an organic fraction. Where appropriate, the organosoluble fractions were reduced to dryness and reconstituted in CH_3CN:H_2O (1:1, v/v) prior to chromatography. Selected aqueous fractions of driselase, acid, and base extracts of leaves (taken at maturity) were also analysed by HPLC.

HPLC-MS in electrospray positive ion mode was conducted to confirm the presence of chlorantraniliprole, IN-H2H20 and IN-GAZ70. The identity of selected metabolites (IN-EQW78, IN-F9N04, IN-KAA24, IN-DBC80, IN-HXH40, IN-HXH44, IN-E5F18, IN-L8F56 and IN-F6L99) was confirmed using a contrasting HPLC liquid phase (Hamilton PRP-1) eluted with several gradients of acetonitrile and water containing 0.1% formic acid and by normal phase TLC using authenticated reference standards.

Table 13 Distribution of ^{14}C in rice, soil, sediment and surface water sampled at various intervals following a single 300 g ai/ha soil application of ^{14}C- chlorantraniliprole

Sample point [a, b]	TRR in sample (mg/kg as chlorantraniliprole)							
	Grain	Hulls	Leaves	Sheaths	Straw	Roots	Water	Sediment
0 DAT	NA	NA	NS	NS	NA	NS	NS	0.404 [c]
14 DAT	NA	NA	0.338	0.174	NA	0.065	0.053	0.208
56 DAT	NS	NS	1.269	0.081	NA	0.207	0.004	0.154
Maturity [d]	0.155	0.174	4.056	0.133	0.903	0.279	0.004	0.040

[a] DAT = Days after treatment

[b] Samples collected 28 days after treatment were not analysed

[c] Value is for soil prior to flooding

[d] Crop maturity was 132 DAT; final water and sediment samples were taken 128 DAT

NA = Not applicable

NS = Not sampled

There is a steady and noticeable increase in radioactivity in leaf and root samples after application, leading to detectable levels in grain, hulls and straw at maturity.

The TRR in soil on the day of application was 0.404 mg/kg equivalents. The levels of radioactivity in the sediment declined after flooding to 0.208, 0.154 and 0.040 mg/kg equivalents at 14, 56 and 128 days after application, respectively. The levels in the surface water declined from 0.053 mg/L at 14 days to 0.004 mg/L at crop maturity (128 days).

The results suggest that there is some uptake of ^{14}C-chlorantraniliprole from water and sediment to the rice crop and this is evident in the increasing levels in rice leaf with decreasing levels in sediment/soil and water at comparable sampling intervals.

Total radioactive residues in grain (including bran) were 0.155 mg/kg equivalents, of which 76.6% was extracted with the solvent system used. The major component in the extracts was parent chlorantraniliprole accounting for 51.4% TRR or 0.080 mg/kg equivalents.

Table 14 shows all of the identified components of the extractable radioactivity in grain, straw, leaf, sheath and hulls.

Table 14 Nature of residues in rice plant fractions collected at crop maturity following a single 300 g ai/ha soil application of ^{14}C-chlorantraniliprole

	Grain	Leaves	Sheaths	Hulls	Straw [a]
TRR (mg equiv/kg)	0.155	4.056	0.133	0.174	0.903
			%TRR		
Extracted	77	103	88	94	101
chlorantraniliprole	51	52	65	66	54
IN-F6L99	1.5	2.7 [b]	1.2	ND	2.5
IN-L8F56	1.8	3.3	ND	ND	2.9
IN-HXH40	ND	3.7 [c]	1.3	0.5	3.4
IN-DBC80	ND	0.9	‘””	0.7	0.8
IN-HXH44	ND	2.3 [d]	ND	ND	2.0
IN-KAA24	0.3	4.3	0.4	ND	3.9
IN-H2H20	ND	2.5	ND	ND	2.2
IN-E5F18	ND	1.2	ND	ND	1.1
IN-F9N04	ND	3.2	ND	ND	2.8
IN-GAZ70	ND	6.1	ND	ND	5.4

	Grain	Leaves	Sheaths	Hulls	Straw [a]
TRR (mg equiv/kg)	0.155	4.056	0.133	0.174	0.903
			%TRR		
IN-EQW78	1.3	4.2	5.3	3.2	4.3
IN-K7H29	1	0.3			0.2
IN-K9T00		ND		0.9	
IN-K9X71		0.2	0.4		0.2
Unextracted [e]	9.1	3.2	20	11	5.2
Losses	14	11	11	4.2	6.1

ND = Not detected

[a] Calculated from leaves and sheath data as fresh weight straw.

[b] In some extracts, < 1.1% TRR eluted with IN-EVK64.

[c] Quantitation from TLC analysis of an isolate containing IN-HXH40, IN-DBC80 and other components (individually < 0.05 mg/kg).

[d] Quantitation from TLC analysis of an isolate containing IN-HXH44, IN-KAA24 and other components (individually< 0.05 mg/kg).

e Value is for the final unextracted residue

Metabolites in grain included IN-F6L99, IN-L8F56, IN-KAA24, IN-K7H29, and IN-EQW78, none of which exceeded 1.8% TRR (0.003 mg/kg, IN-L8F56). Two unidentified metabolites accounted for 1.2 and 7.6% TRR (0.002 and 0.011 mg/kg, respectively).

TRR in hulls was 0.174 mg/kg equivalents of which 94% was extracted. Chlorantraniliprole was the main component of the extracted radioactivity at 66% TRR (0.117 mg/kg). Minor components included IN-EQW78, IN-DBC80 and IN-HXH40, individually present at ≤ 3.2% TRR (< 0.001–0.006 mg/kg equivalents).

The majority of the TRR in leaves was extracted (103% TRR). Chlorantraniliprole (2.12 mg/kg, 52% TRR) was the major component in the leaf extracts. Minor components including IN-EQW78, IN-GAZ70, IN-F9N04, IN-E5F18, IN-H2H20, IN-KAA24, IN-HXH44, IN-DBC80, IN-HXH40, IN-L8F56 and IN-F6L99 which were individually present at (0.9–6.1% TRR). At crop maturity the solvent extracted residue of leaves also contained up to 7 unidentified components totalling 11% TRR.

In sheaths, the majority of the TRR was extracted (88% TRR). Parent chlorantraniliprole at 65% TRR 0.086 mg/kg, was the major component. Minor components were individually present at ≤ 5.3% TRR and included IN-EQW78, IN-DBC80, IN-HXH40, IN-KAA24, and IN-F6L99. Up to 3 unidentified components 0.008 mg/kg equivalents (≤ 6.3% TRR) were also observed in sheaths of rice harvested at maturity.

The TRR reported for straw was calculated from data generated from leaves and sheaths. The calculated values are comparable to the data reported for leaves. The proportion of parent chlorantraniliprole in straw was calculated as 54% TRR.

The distribution and identification of components in the soil and sediment samples taken at 0, 14, 56 and 128 days after application is shown in table 15.

Table 15 Nature of residues in soil (0 DAT) and sediment sampled at various intervals following a single 300 g ai/ha application of ^{14}C-chlorantraniliprole

	Days after soil treatment			
	0	14	56	128
Total TRR [a]	0.393	0.225	0.149	0.037
%TRR				
Total extracted	97	95	90	77
chlorantraniliprole	91	80	52	54
IN-F6L99	0.9	1.8	ND	ND
IN-DBC80	ND	ND	0.2	ND
IN-HXH44	ND	ND	1.0	ND
IN-H2H20	ND	ND	0.5	ND
IN-K9X71	ND	ND	ND	0.4
IN-E5F18	ND	ND	ND	0.3
IN-F9N04	ND	0.4	1.3	0.2
IN-GAZ70	0.6	2.6	6.5	3.2
IN-EQW78	0.3	5.7	19	15
Unextracted [b]	< 1.0	3.3	6.9	16
Uncharacterized extract [c]	NA	NA	NA	< 1.0

ND = Not detected.

NA = Not applicable, Extracts 1 and 2 combined prior to analysis

[a] Total TRR = total extracted + unextracted ^{14}C

[b] Value is for the final unextracted residue

[c] Extracted with acetonitrile: 1N formic acid (8:2, v/v), The sum of individual unidentified metabolites retained on the HPLC column, none exceeding 6.3% TRR.

The majority of the residues in soil and sediment were extracted. Residues extracted from sediment samples collected at 14, 56, and 128 days after application represented 95% TRR (0.198 mg/kg), 90% TRR (0.138 mg/kg), and 77% TRR (0.031 mg/kg), respectively.

Levels of chlorantraniliprole in soil/sediment steadily decreased from 91% TRR (0.368 mg/kg) in soil at 0 DAT to 52% and 54% TRR (0.079 mg/kg and 0.022 mg/kg) in the 56 and 128 DAT samples, respectively. Concentrations of IN-EQW78 in the sediment increased from 0.3% TRR (0.001 mg/kg) in soil immediately after application to 19% TRR (0.029 mg/kg) at 56 DAT and 15% TRR (0.006 mg/kg) at 128 DAT. Other metabolites, including IN-F9N04, IN-GAZ70, IN-H2H20 and IN-HXH44 were present at low concentrations (0.001–0.01 mg/kg, ≤ 6.5% TRR) particularly in the samples taken at 56 and 128 DAT. Minor metabolites (IN-F6L99, IN-F9N04, IN-E5F18 and IN-DBC80) were only detected at < 2% TRR.

Chlorantraniliprole was the major component of the radioactivity in flood water at 14 DAT; 80% TRR. The main metabolite was IN-EQW78, which accounted for 9.6% TRR (0.005 mg/L). Other metabolites, including IN-F9N04, IN-KAA24, IN-HXH40, IN-K9X71 and IN-F6L99, were present at low concentrations < 2% TRR (< 0.001 mg/L). Characterisation of the radioactivity in other flood water samples was not conducted.

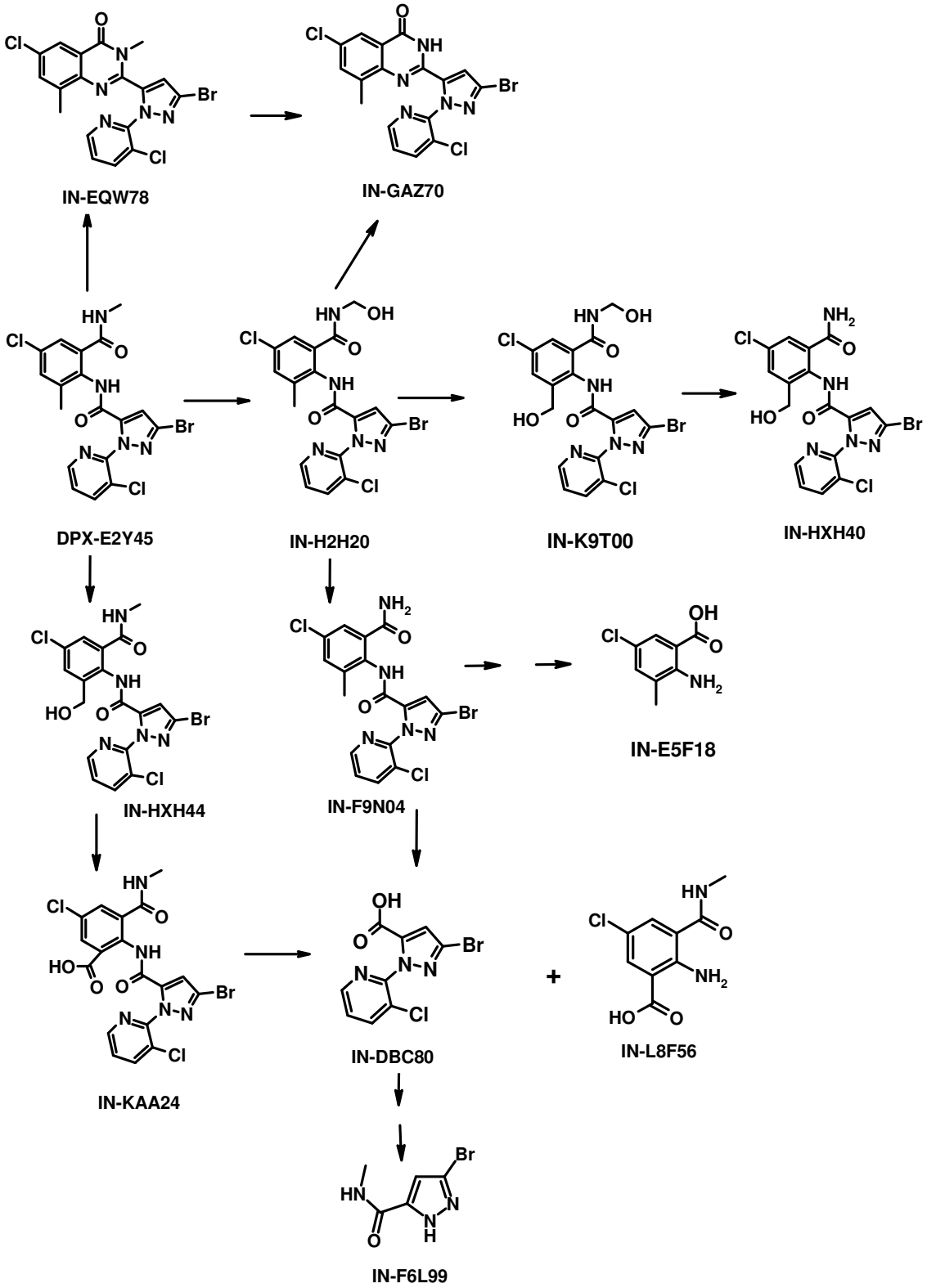


Figure 3 The proposed metabolic pathway for chlorantraniliprole in rice

Numerous metabolites were formed primarily through three major pathways:

hydroxylation of the N-methyl group to IN-H2H20 or hydroxylation of the methyl-phenyl carbon to yield IN-HXH44; condensation with the loss of water from chlorantraniliprole to yield a

quinazolinone derivative, IN-EQW78; and similar condensations of IN-H2H20 with an additional loss of $–CH_2OH$ giving rise to IN-GAZ70; and further metabolism such as N-demethylation of the hydroxymethylamide group in IN-H2H20 to IN-F9N04. The amide bridge cleavage between the phenyl and heterocyclic rings was a minor pathway yielding IN-L8F56 and IN-DBC80, with further metabolism forming minor amounts of IN-F6L99.

In summary, the metabolic fate of chlorantraniliprole in rice is complex with the formation of over 20 minor metabolites in the different crop and soil/sediment matrices. Chlorantraniliprole is either metabolised in the sediment and taken up by the roots, or in part metabolised in the rice plants. Parent chlorantraniliprole is the major component of the radioactive residues in rice grain, straw and leaves at > 50% TRR at crop maturity. Other metabolite components of the extracted radioactivity in rice grain were < 2% TRR with 9% TRR unextracted.

Cotton

Brown *et al*. (2004 12698) studied the uptake of ^{14}C-chlorantraniliprole in excised or cut stems of cotton seedlings as well as the distribution of radioactivity following foliar application to whole cotton plants. In the experiments using cut seedlings, cotton plants (cv. Delta Pine 50) grown for 18 days were cut and placed in beakers of water. The plants were cut again under water to remove any air bubbles then placed in two uptake solutions, each containing one labelled form of the ^{14}C-chloroantraniliprole, ([benzamide carbonyl–^{14}C]-chlorantraniliprole and [pyrazole carbonyl–^{14}C]-chlorantraniliprole). The plants were removed from the solutions after 4 days. The cotton foliage was homogenised with dry ice and samples were stored frozen until combustion analysis and/or extraction. Part of the homogenised foliage was combusted; evolved CO_2 was collected and total radioactivity determined by LSC. Cotton foliage was extracted with CH_3CN:H_2O (9:1, v:v once), and CH_3CN:H_2O (7:3, v:v twice). The sample was extracted by shaking, centrifuged at 4-9 °C then filtered. All extracts were pooled and assayed by LSC. The combined extract was concentrated, until only an aqueous solution remained. The extract was adjusted to 50% aqueous CH_3CN and analysed by LSC to ensure that the majority of the radioactivity was recovered upon concentration (> 90%). In general, extracts were centrifuged/filtered but were not submitted to any other clean-up prior to HPLC analysis; concentrated extracts were analysed directly by HPLC. Post-extraction solids were combusted to determine the amount of unextracted radioactivity. The uptake solutions from the plant incubations were also analysed for remaining radioactivity. The distribution of the radioactivity in the excised plants is shown in Table 16.

HPLC analysis of the uptake solutions showed that 100% and 96% of the remaining radioactivity from the [benzamide carbonyl–^{14}C]-chlorantraniliprole and the [pyrazole carbonyl–^{14}C]-chlorantraniliprole solutions, respectively, comprised parent chlorantraniliprole. In the [pyrazole carbonyl–^{14}C]-chlorantraniliprole solution, an additional 1.1% of the radioactivity was ascribed to metabolite IN-EQW78.

In the plant extracts, parent chlorantraniliprole comprised 98% and 95% of the extracted radioactivity from [benzamide carbonyl–^{14}C]-chlorantraniliprole and the [pyrazole carbonyl–^{14}C]-chlorantraniliprole treated plants, respectively. In the extract from the [benzamide carbonyl–^{14}C]-chlorantraniliprole treated plants, 0.9% of the extracted radioactivity was identified as metabolite IN-GAZ70. In the extract from [pyrazole carbonyl–^{14}C]-chlorantraniliprole treated plants, 0.6% and 0.3% of the extracted radioactivity was identified as metabolites IN-GAZ70 and IN-EQW78, respectively.

In the whole plant experiment, cotton plants were grown in a greenhouse. In experiment A, 41-day old plants were sprayed with either [benzamide carbonyl–^{14}C] or [pyrazole carbonyl–^{14}C]chlorantraniliprole at the equivalent of 150 g ai/ha with 0.5% surfactant added to the spray solutions. The test materials were applied as 20% SC. In the experiment B, 57-day old plants were sprayed with a solution of [benzamide carbonyl–^{14}C]chlorantraniliprole (20% SC formulation) at a rate equivalent to 150 g ai/ha.

Samples of cotton foliage were collected by cutting the plants above the soil surface. Foliage samples from the [benzamide carbonyl–^{14}C]chlorantraniliprole spray without surfactant were collected at 8, 21 and 48 days after application. Bolls were collected from the [benzamide carbonyl–

^{14}C]chlorantraniliprole treated plants (Experiment B) on day 86 and from Experiment A plants ([benzamide carbonyl-^{14}C] and [pyrazole carbonyl–^{14}C]chlorantraniliprole, + surfactant) at harvest on day 126. Cotton bolls were separated into hulls, lint and seed. Cotton seed was manually removed from the lint where possible.

The extraction of the various cotton matrices was conducted in the same manner as described above for cotton foliage. Concentrated extracts were adjusted to 70:30 CH_3OH:H_2O and stored in the freezer overnight to remove chlorophyll. The extracts were centrifuged and the supernatants concentrated prior to analysis using LSC and HPLC. All extraction and primary analyses of ^{14}C residues were completed within 4 months of sample collection. No storage stability analyses were conducted.

The distribution of the radioactivity in various fractions and extracts is shown in Tables 16 and 17.

Table 16: Distribution of ^{14}C in cotton plants after foliar application of ^{14}C-chlorantraniliprole

Sample	PHI (days)	TRR (mg/kg as chlorantraniliprole)	
		[benzamide carbonyl–^{14}C]-label	[pyrazole carbonyl–^{14}C]-label
Experiment A: +surfactant			
foliage	8	2.2	1.80
	15	1.54	1.26
	22	0.66	2.0
	86	0.07	–
hulls	86	0.01	–
lint/seed	86	0.01	–
foliage/hulls	126	0.06	0.06
lint	126	(0.01)	< 0.01
seed	126	< 0.01	< 0.01
Experiment B			
Foliage	8	0.66	
	21	3.68	
	48	1.45	

Table 17 Nature of ^{14}C residues in cotton plants after foliar application of ^{14}C-chlorantraniliprole

		I			II			III		
fraction	DAT	TRR	Extracted	Parent	TRR	Extracted	Parent	TRR	Extracted	Parent
Foliage	8	2.2	96	91	0.66	93	90	1.80	98	92
	15	1.54	98	93				1.26	97	90
	22	0.66	97	89	3.68	97	95	2.0	97	92
	48				1.45	92	91			
	86	0.07	94	68						
Hulls	86	0.01	94	43						
lint/seed	86	0.01	100	57						
foliage/hulls	126	0.06	88	25				0.06	86	53

I = Experiment A: [benzamide carbonyl-^{14}C]-chlorantraniliprole + 0.5% surfactant

II = Experiment B: [benzamide carbonyl-^{14}C]-chlorantraniliprole

III = Experiment A: [pyrazole carbonyl-^{14}C]-chlorantraniliprole + 0.5% surfactant

TRR = mg equiv/kg

Parent = chlorantraniliprole

The majority of the radioactivity was extracted from the Experiment A (+0.5% surfactant) samples: cotton foliage (86–98% TRR), hulls (93% TRR; 86 DAT), and undelinted seeds (94–100% TRR). The major component of the residue in foliage (25–93% TRR), hulls (86 DAT, 43% TRR) and undelinted seeds (86 DAT, 57% TRR) was parent chlorantraniliprole.

Similarly, for Experiment B (no surfactant) the majority of the foliage residues were extracted (≥ 92% TRR) and chlorantraniliprole comprised the majority of the ^{14}C-residue (≥ 90% TRR).

In summary, chlorantraniliprole was not metabolised to an appreciable extent in cotton foliage, hulls or undelinted seed. The majority of the extracted radioactivity in cotton samples (foliage, hulls and seed) at all sampling intervals was chlorantraniliprole.

Summary of plant metabolism studies

The metabolic fate of chlorantraniliprole in plants was investigated by the conduct of radiolabelled studies in apple, cotton, lettuce, rice, and tomato. Chlorantraniliprole is not metabolised to any great extent when applied as a foliar spray. With up to three consecutive foliar applications of chlorantraniliprole to apples, tomatoes and lettuce, and following a single application to cotton, parent compound was the major component of the extracted radioactivity.

However, when applied as a soil drench to rice crops, metabolism was complex due to uptake of degradates in water through roots, with numerous metabolites identified in addition to parent compound. IN-GAZ70 (0.049 mg/kg) and IN-EQW78 (0.039 mg/kg) were two major metabolites in the rice straw but were present at less than 7% of the TRR. Other minor metabolites (< 0.035 mg/kg) identified in rice straw included IN-KAA24, IN HXH40, IN H2H20, IN-HXH44, and IN-F6L99.

Environmental Fate in soil

The Meeting received information on the confined rotational crops, field crop rotation, aerobic and anaerobic soil metabolism and soil photolysis. The fate and behaviour of chlorantraniliprole in soils was investigated with [benzamide carbonyl-^{14}C]- or [pyrazole carbonyl-^{14}C]-labelled chlorantraniliprole. Only those data relevant to the current evaluation are reported below.

Soil degradation

Aerobic degradation

The biotransformation of radiolabelled chlorantraniliprole was studied in Marietta sandy loam soil (USA) under aerobic conditions in the dark at approximately 45% of the maximum water-holding capacity for 365 days at 25 °C and for 240 days at 35 °C (McCorquodale and Addison, 2007 12779). The biotransformation was also studied in 3 additional soils incubated under similar conditions for up to 120 days: Tama silty clay loam (USA), Sassafras loam (USA) and Lleida clay loam (Spain) (McCorquodale and Mackie, 2005 12780). Two radiolabelled forms of the test substance were used separately, radiolabelled with ^{14}C either in the benzamide carbonyl (BC) carbon or the pyrazole carbonyl (PC) carbon. Each form was applied to the soil to provide a nominal concentration of 0.3 mg/kg soil dry weight. The composition of radioactivity in the different extracts was determined by reversed-phase HPLC with radiochemical detection. Identification of metabolites was performed by co-chromatography with reversed-phase HPLC and by either normal phase TLC or LC/MS. Non-extracted residues were quantified by combustion analysis.

In all four soils tested, the route of degradation was the same at 25 and 35 °C, with generally higher concentrations of all metabolites in the 35 °C samples (increased degradation). The principal metabolite in all soils was IN-EQW78 (maximum of 33% applied radioactivity at day 120, Lleida soil, 35 °C). Minor metabolites (> 5% applied radioactivity) were IN-F6L99 (maximum of 5.2% applied radioactivity at day 240, Marietta soil, 35 °C), IN-ECD73 (maximum of 8.2% applied radioactivity at day 180, Marietta soil, 35 °C) and IN-GAZ70 (maximum of 7.4% applied radioactivity at day 120, Lleida soil, 35 °C). IN-F9N04 was not significant in any soil, with maximum concentrations of less

than 5% applied radioactivity. In addition, IN-F9N04 was an impurity in ^{14}C-chlorantraniliprole and was found at concentrations of up to 1.9% applied radioactivity at day 0 in some studies. The maximum unextracted radioactivity ranged between 5 to 10% applied radioactivity. The maximum amount of $^{14}CO_2$ generated was 7.3% applied radioactivity. The average mass balance in each soil was greater than 99% of applied radioactivity.

In all soils the major transformation pathways of chlorantraniliprole was via abiotic transformations: cyclization followed by dehydration to form IN-EQW78 or rearrangement followed by cleavage to form IN-F6L99 and IN-ECD73. Minor biotic transformation pathways were N-demethylation reactions leading to formation of IN-F9N04 or IN-GAZ70. In the Lleida soil, the biotic transformation to yield IN-GAZ70 was more significant than in the other soils.

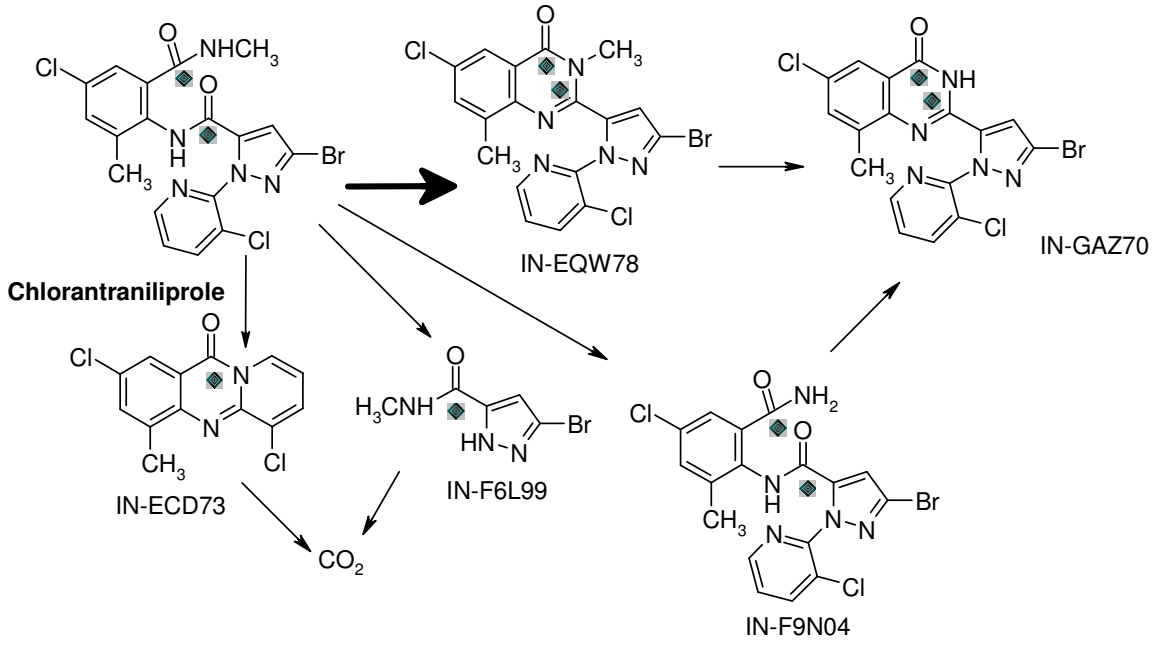


Figure 4 Proposed pathways for the degradation of chlorantraniliprole in non-sterile soil under aerobic and anaerobic conditions

Chlorantraniliprole degrades in soil; however, the degradation is sometimes limited by sequestration (or aging) of the compound in soil. The sequestration of chlorantraniliprole in soil makes the compound more difficult to extract and protects the compound from degradation, while limiting mobility. Sequestration may in part be explained by sorption/intercalation into smectite clays (2:1 expandable clays) and interaction with soil organic matter (humic acids). Whatever the mechanism, sequestration results in loss of extractability on aging and a concomitant decrease in degradation rate, and decreased mobility of aged residues. Differential extraction was used in some studies to distinguish between readily extracted and total extracted residues.

The rate of degradation of chlorantraniliprole was measured at multiple temperatures in several soils from the United States and Europe (McCorquodale and Addison, 2007 12779; McCorquodale and Mackie, 2005 12780; Singles, 2006b 12779a and Singles, 2006c 12780a). These data demonstrate that the readily extracted residues of chlorantraniliprole are more inherently degradable than the sequestered residues.

The average laboratory DT_{50} value (25 °C) for the readily extractable chlorantraniliprole is 369 days (n = 4) compared to 510 days (n = 4) for the total extracted residues in the same soils. At

35 °C, the average DT_{50} values are 184 days (n = 4) and 312 days (n = 4) for the readily extractable and total extracted residues, respectively. In soils where the fastest degradation occurs, the readily extractable residues account for a greater percentage of the chlorantraniliprole than in soils with slower degradation. As more of the applied chlorantraniliprole is sequestered in the soils, the DT_{50} values increase.

In addition to sequestration and temperature, anaerobic conditions and the presence of sunlight also impacted the rate of degradation of chlorantraniliprole under laboratory conditions. Under anaerobic conditions, the DT_{50} of chlorantraniliprole was 208 days at 25 °C versus a DT_{50} of 886 days in the same soil (Marietta) incubated under aerobic conditions.

The rate of degradation of four metabolites of chlorantraniliprole was measured in five soils from the United States and Europe. The rate of degradation of radiolabelled IN-EQW78 (Lowrie and Coyle, 2005 14621), IN-ECD73 (Morris and Coyle, 2005a 14620) and IN-GAZ70 (McCorquodale and Wardrope, 2006 17046) was measured in Sassafras sandy loam (USA), Speyer loamy sand (Germany), Lleida silty clay loam (Spain), Cajon sandy loam (USA) and Tama silt loam (USA). The degradation of radiolabelled IN-F6L99 was measured in the same soils except Hidalgo sandy clay loam (USA) was used instead of the Cajon soil (Lowrie and McCorquodale, 2005 14623). The moisture content of the soils was adjusted to between 40 and 60% maximum water holding capacity. The test substance was radiolabelled on the carbonyl group and was applied to give a concentration of 0.5 mg/kg soil.

Table 18 Chlorantraniliprole metabolites as %applied residue at *x* days after application to soil

Soil Name	Soil type	IN-EQW78	IN-ECD73	IN-F6L99	IN-GAZ70
Sassafras	Sandy Loam	91% AR @120D	88% AR @ 120D	6.8% AR@90D	99% AR@120D
Speyer	Loamy Sand	91% AR @ 120D	87% AR @120D	9.4% AR@90D	94% AR@120D
Lleida	Silty Clay Loam	94% AR @ 120D	85% AR @120D	6.3% AR@90D	91% AR@120D
Cajon	Sandy Loam	89% AR @ 120D	92% AR @120D	*11.7%AR @120D	104%AR@120D
Tama	Silt Loam	91% AR @ 120D	90% AR @120D	18% AR@120D	93% AD@120D

* Hidalgo sandy clay loam instead of Cajon

The average DT_{50} values of IN-EQW78, IN-ECD73 and IN-GAZ70 in aerobic soils were generally greater than 1 year in laboratory studies. The average DT_{50} value for IN-F6L99 was approximately 23 days. The DT_{50} values (days) for degradation products calculated using simple first order kinetics are summarized below.

Table 19 DT_{50} values for chlorantraniliprole metabolites in various soils (laboratory studies)

Soil Name	Soil type	IN-EQW78	IN-ECD73	IN-F6L99	IN-GAZ70
Sassafras	Sandy Loam	651	> 1000	12	> 1000
Speyer	Loamy Sand	646	> 1000	11	> 1000
Lleida	Silty Clay Loam	763	752	14	741
Cajon	Sandy Loam	671	> 1000	37*	> 1000
Tama	Silt Loam	785	2580	40	> 1000

* Hidalgo sandy clay loam instead of Cajon

Field studies

A total of 22 field studies were carried out in the United States, Canada, and Europe to evaluate the behaviour of chlorantraniliprole under field conditions: Two soil dissipation trials in the USA using radiolabelled chlorantraniliprole, 8 bare soil dissipation trials in the USA and Canada, 8 bare soil dissipation trials in Europe and 4 dissipation trials in the presence of a cover crop in the USA. The

bare soil dissipation trials were approximately 18 months in duration and the trials with crop cover were approximately 6 months in duration.

The major transformation products detected in the ^{14}C studies were IN-EQW78 IN-F6L99, IN-ECD73 and IN-GAZ70. Multiple minor unidentified radioactivity components (combined < 10% applied radioactivity) were observed at later sampling intervals.

Results from the field dissipation studies are summarised in Table 20. It was noted that the duration of the field studies was such that seasonal dependencies in degradation were observed (faster degradation in summer compared to winter). Degradation of total extracted chlorantraniliprole residues was slower in soils with higher clay contents.

Table 20 Summary of results of field dissipation studies for chlorantraniliprole applied to bare soil

	% sand	% silt	% clay	%OM	pH	%remaining		DT_{50} (days)	Reference
California, USA	52	42	6.0	0.27	8.4	23-29% @540D	Readily[a]	108	12785 (^{14}C)
							Total[b]	181	
Texas USA Application 1	39	21	40	1.1	8.2	21-34% @379D	Readily[a]	184	12784 (^{14}C)
							Total[b]	239	
Application 2						20-26% @741D	Readily[a]	188	
							Total[b]	222	
California, USA	38.0	58.0	4.0	1.2	8.1	15%@540D	Readily[a]	34	12788
							Total[b]	45	
Texas, USA	39	21	40	1.1	8.2	16%@540D	Total[b]	206	12786
New Jersey, USA	26	56	18	1.7	6.6	64%@541D	Readily[a]	292	12790
							Total[b]	697	
Georgia, USA	88.0	8.0	4.0	0.7	6.5	71%@540D	Readily[a]	444	12789
							Total[b]	1130	
Washington, USA	62	34	4.0	1.1	7.6	48%@540D	Total	411	14439
Ohio, USA	15	51	34	2.8	7.2	38%@521D	Total	335	14553
Minnesota, USA	35	46	18	4.3	6.7	48%@539	Total	210	14440
Prince Edward Island, Canada	76	16	8	3.7	6.0	60%@93D	Total	274	16518
Los Palacios y Villafranca, Spain	76	12	12	1.1	8.1	24%@549D	Readily[a]	121	12787
							Total[b]	227	
Nuits-St-Georges, France	10	64	26	4.2	7.7	39%@551D	Readily	247	12791
							Total[b].	362	
Nambsheim, France	16	62	22	2.7	7.9	32%@543D	Readily	158	12792
							Total	229	
Crespelano, Italy	26	50	24	2.0	8.1	42%@553D	Readily	196	12793
							Total	435	
Lleida, Alpicat, Spain	14	54	32	4.5	8.0	26%@545D	Total[a]	163	14441
Vittoria, Italy	81	6.0	13	1.1	8.3	52%@539D	Total	611[b]	14442
Suchożebry, Poland[c]	70	20	10	1.4	5.5	49%@481D	Total	361	14443
Goch, Germany	29	60	10	2.8	6.4	60%@537D	Total	504	14444

[a] chlorantraniliprole extracted by conventional extraction method

[b] Total chlorantraniliprole is the sum of the conventional and exhaustive extraction methods for California, for all other

sites it is the exhaustive extraction.

Studies were also performed in the presence of a crop cover. Two terrestrial turf dissipation studies were conducted in the United States (Huang *et al*., 2006a,b 16521, 16522). Chlorantraniliprole (SC formulation) was applied to turf at a nominal concentration of 560 g ai/ha. The DT_{50} values ranged from 150 to 258 days in the turf dissipation trials for decline of total chlorantraniliprole in all matrices (sum of grass, thatch and soil).

Two additional dissipation studies were performed in the presence of crops in the United States. One trial was designated as a pre-emergent grass trial (application to bare soil that had been pre-seeded with grass, 16519) and the other a post-emergent pepper trial (application to a field containing pepper plants 16520). For the pre-emergent grass trial, the soil was pre-seeded with grass and then treated at a nominal concentration of 300 g chlorantraniliprole per hectare (SC formulation). For the post emergent pepper study, chlorantraniliprole was applied as a broadcast spray to both soil and plants in two applications at target concentration of 150 g active ingredient/ha at each application, with a 5 day interval between applications. In the presence of plants, DT_{50} values for readily extractable chlorantraniliprole ranged from 59 to 114 days and DT_{50} values for the total extracted residues ranged from 85 to 232 days, shorter than observed following application to bare soil.

Aqueous hydrolysis

Hydrolysis of radiolabelled chlorantraniliprole was studied in the dark at 25 °C in sterile aqueous buffered solutions at pH 4 (citrate buffer), pH 7 (TRIS-maleic acid buffer) and pH 9 (borate buffer) for 30 days (Chapleo *et al.*, 2007 12782). Two radiolabelled forms of chlorantraniliprole were used in this study: [benzamide carbonyl-^{14}C] and [pyrazole carbonyl-^{14}C]. The concentration of chlorantraniliprole in the buffer solutions was approximately 0.6 µg/mL and acetonitrile (1%) was used as a co-solvent.

At pH 4 and 7, no significant decline of chlorantraniliprole was observed during the incubation period. At pH 4 and pH 7, no major transformation products (> 10% applied radioactivity) were detected. Chlorantraniliprole was unstable at pH 9. Chlorantraniliprole underwent cyclization followed by dehydration to form IN-EQW78 which accounted for 86.7% AR at day 30. The first order DT_{50} for chlorantraniliprole at pH 9 is 10 days.

IN-EQW78 did not hydrolyse further at pH 9. IN-EQW78 was stable in strongly acidic conditions used in soil extraction studies and it is not expected to convert back to the parent molecule.

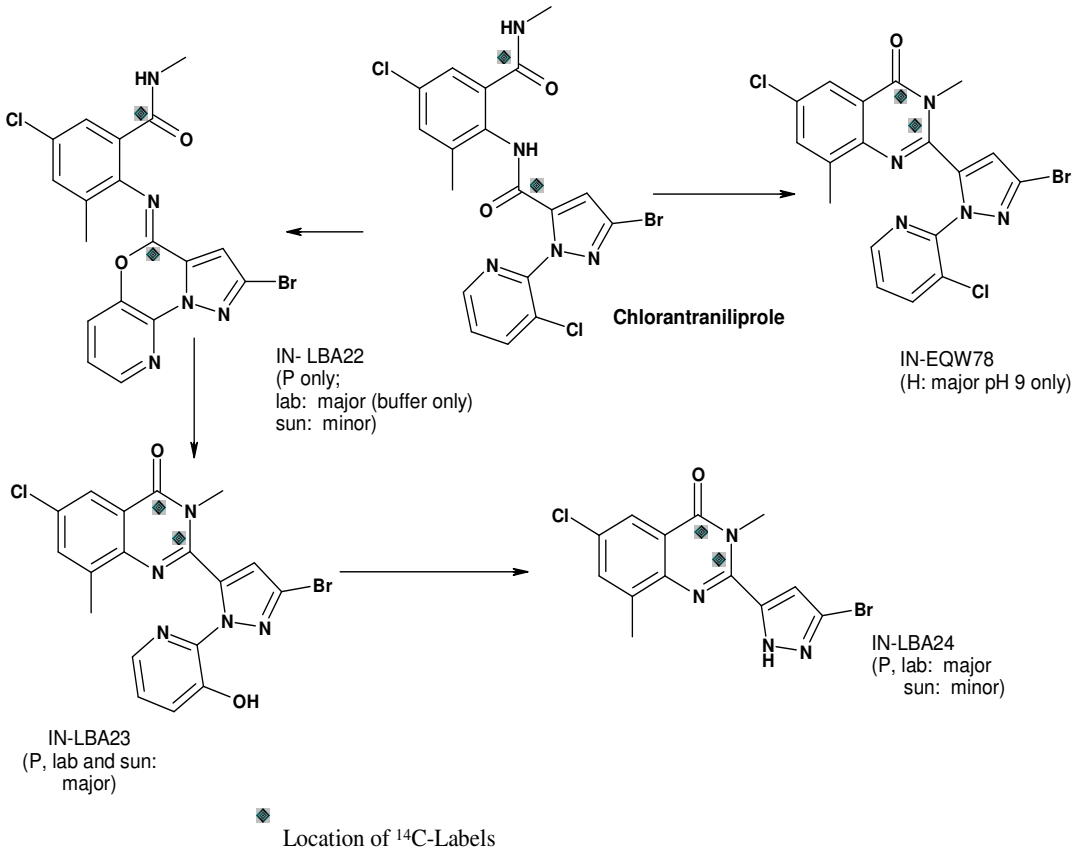


Figure 5 Proposed pathway for the degradation of chlorantraniliprole in irradiated and non-irradiated sterile buffer

Photolysis

The aqueous phototransformation of radiolabelled ^{14}C-chlorantraniliprole was studied at 25 °C in sterile aqueous tris maleic acid buffer at pH 7 and in natural water (MacDonald *et al.*, 2007 12783). The photolysis half-life of chlorantraniliprole in sterile pH 7 buffer was 0.37 days under continuous irradiation (Xe arc lamp, 300–800 nm, UV filter). In pH 7 buffer, 3 major degradation products were formed: IN-LBA22 (maximum of 53% AR), IN-LBA23 (maximum of 41% AR) and IN-LBA24 (maximum of 90% AR). IN-LBA22 rapidly hydrolysed to form IN-LBA23, which then photolysed to yield IN-LBA24. The DT_{50} values for IN-LBA22 and IN-LBA23 in irradiated buffer were 0.9 and 1.5 days, respectively. IN-LBA24 was stable under these conditions. In sterile natural water, the photolytic half-life of chlorantraniliprole was 0.31 days under continuous irradiation. In natural water, 2 major degradation products were formed: IN-LBA23 (maximum of 51% AR) and IN-LBA24 (maximum of 94% AR).

Confined rotational crop studies

The uptake and metabolism of ^{14}C-chlorantraniliprole in succeeding crops (or follow crops) of spring wheat, red beet and lettuce was studied by Chapleo (2006 12314). [^{14}C]chlorantraniliprole (either [benzamide carbonyl-^{14}C]- or [pyrazole carbonyl-^{14}C] chlorantraniliprole) was applied to sandy loam soil (pH 6; OC 1.8%; 72% sand, 15% silt, 13% clay; moisture holding capacity 54%; CEC 11.5 meq/100 g) as a 20% SC formulation at application rates equivalent to 300 or 900 g ai/ha. The soil was aged for various intervals after application and then sown with lettuce, red beet and wheat. The

experimental design is shown in Table 21. Indoor plots for each crop (glasshouse) were 0.71 × 0.72 × 0.5 m for the 300 g ai/ha applications and 0.47 × 0.46 × 0.5 m for the 900 g ai/ha applications. Each plot was a free-standing wooden crate placed on layers of plastic mesh.

Table 21 Experimental design for laboratory succeeding crop study

Radiolabel	Application rate (g ai/ha)	Sowing Interval (DAT) [a]	Crops sown
[benzamide carbonyl-^{14}C] chlorantraniliprole	300	30	Lettuce, red beet, wheat
[pyrazole carbonyl-^{14}C] chlorantraniliprole	300	0, 30, 120 and 365	Lettuce, red beet, wheat
[pyrazole carbonyl-^{14}C] chlorantraniliprole	900 [b]	0 and 365	Wheat

[a] Days after soil treatment.

[b] Samples from this treatment were used for metabolite identification.

Core samples of soil were taken on day 0 (0 days after application), immediately prior to sowing and at crop maturity (365 days after application). Samples of spring wheat were collected at stages that reflected early forage, hay and at crop maturity (harvest). From each sowing interval (0, 30, 120 and 365 days after application), samples of forage, hay, straw and grain were collected. Hay samples were collected by cutting whole plants above the soil surface and drying in the glasshouse at ambient temperature for 8, 4, 9 and 9 days from the 0, 30, 120 and 365 days sowings, respectively. The moisture contents of the hay were 20, 15.3, 15 and 15%, respectively. Immediately after harvest, grain was separated from the heads using a laboratory thresher and also by hand, and the chaff was combined with the straw for analysis. For the red beets, whole root and foliage were collected at maturity (BBCH 49). Lettuce foliage was collected at maturity (BBCH 49) and combined to form a single sample for each sowing. All samples were kept frozen at –20 °C as soon as possible after collection. The crop samples were homogenized and extracted within 4 weeks of harvest, with the exception of 0 day wheat grain which was extracted after 37 days; all extracts were stored and analysed within 37 days. Soil samples were extracted and analysed within 42 days.

The characterisation and identification of the radioactivity in soil treated with [pyrazole carbonyl-^{14}C]-chlorantraniliprole is shown in Table 22. The proportion of chlorantraniliprole in soil steadily decreased from 96% TRR following application (day 0) to 71% TRR at 365 days and 48% TRR at 479 days. Identified metabolites, including IN-EQW78, IN-F9N04, IN-GAZ70, IN-LEM10, IN-HXH40, and IN-KAA24, were present at ≤ 5.4% TRR in the soil samples taken at longer time intervals after application. IN-F6L99 was only detected occasionally and at < 5% TRR. It is noted that the concentrations of IN-F9N04 steadily increase at each sample taken at sowing and show slight decreases following crop harvest.

Table 22 Identification of ^{14}C residues in soil sampled at various intervals following a single 300 g ai/ha application of [pyrazole carbonyl-^{14}C]chlorantraniliprole

Soil (0-15 cm)	Days after soil treatment							
	0[a]	30[a]	108[b]	165[b]	120[a]	249[b]	365[a]	479[b]
TRR	0.252	0.377	0.059	0.054	0.125	0.017	0.068	0.053
%TRR								
chlorantraniliprole	96	95	80	86	93	80	71	48
IN-F6L99	1.0	ND	ND	ND	ND	ND	ND	4.1
IN-HXH40	ND	ND	ND	ND	ND	ND	ND	5.0
IN-KAA24	ND	ND	ND	ND	ND	ND	ND	2.0
IN-F9N04	ND	1.1	2.0	2.9	4.4	2.4	6.1	5.4
IN-LEM10	ND	ND	ND	ND	ND	ND	ND	3.2

Soil (0-15 cm)	Days after soil treatment							
	0[a]	30[a]	108[b]	165[b]	120[a]	249[b]	365[a]	479[b]
IN-GAZ70	ND	ND	ND	ND	ND	ND	ND	2.7
IN-EQW78	ND	ND	4.0	ND	2.5	12.8	4.2	3.5
Unidentified	< 0.010	< 0.010	< 0.010	< 0.010	< 0.010	< 0.010	5.8	7.6

[a] Soil sample taken at time of crop sowing.

[b] Soil sample taken at time of crop harvest or maturity.

ND = Not detected

The sum of individual unidentified metabolites retained on the HPLC column, none exceeding 3.7% TRR

Extracted with acetonitrile:1N formic acid (4:1, v/v)

Value is for the final unextracted residue

Total recovery is the total extractable + remaining unextracted ^{14}C-residues

Much of the radioactivity in crop fractions was extracted ranging from 40 to 100% TRR; unextracted radioactivity ranged from 2.5% in lettuce to 51% in wheat grain (300 g ai/ha treatment). In all samples, with the exception of red beets (roots and foliage), parent compound comprises a large proportion of the extracted radioactivity, ranging from 36 to 85% of the TRR.

Table 23 Distribution and nature of ^{14}C residues in samples from crops sown at various intervals following a single 300 g ai/ha application of [pyrazole carbonyl-^{14}C]chlorantraniliprole to soil and harvested at maturity.

	Interval (DAT[a])	TRR mg/kg	Extracted (% TRR)	Extracted Chlorantraniliprole (% TRR)	Unextracted (%TRR)
Wheat Grain	0	0.01	40	NA	51
	30	< 0.010	NA	NA	NA
	120	0.041	99	48	4.2
	365	0.008	NA	NA	NA
Wheat Early Forage	0	0.082	92	84	8.8
	30	0.110	92	54	7.0
	120[b]	0.261	91	76	4.5
	365	0.062	93	77	8.6
Wheat Hay	0	0.431	86	61	4.5
	30	0.572	87	73	7.1
	120[b]	1.573	101	51	8.2
	365	0.332	92	67	7.1
Wheat Straw	0	0.360	95	69	8.2
	30	0.355	89	63	6.7
	120[b]	2.085	84	64	9.4
	365	0.485	88	37	8.3
Lettuce	0	0.023	91	85	2.5
	30	0.046	105	70	3.8
	120	0.032	83	72	3.2
	365	0.031	92	64	NQ
Red beet Roots	0	0.007	83	NA	14
	30	0.011	69	NA	14
	120	0.009	78	NA	26
	365	0.006	NA	NA	NA

	Interval (DAT[a])	TRR mg/kg	Extracted (% TRR)	Chlorantraniliprole (% TRR)	Unextracted (%TRR)
Red beet Foliage	0	0.063	83	4.7	6.0
	30	0.118	97	4.8	6.7
	120	0.065	89	0.9	7.3
	365	0.113	85	1.8	7.1

[a] DAT = days after application of chlorantraniliprole to soil

[b] The 120 day aged soil sample was from a different initial soil sample

The composition of the radioactivity in mature lettuce, following sowing at various intervals after application of [pyrazole carbonyl-^{14}C]chlorantraniliprole at 300 g ai/ha is shown in Table 24. The TRRs range from 0.023 mg/kg equivalents to 0.042 mg/kg equivalents. Total extracted radioactivity ranged from 83% to 92% TRR. Chlorantraniliprole residues form a large proportion of the radioactivity, ranging from approximately 64% to 85% of the TRR in lettuce sown from 0 days to 365 days after application. Metabolites that were identified include IN-F6L99, IN-F9N04 and IN-GAZ70, individually ≤ 5.2% TRR.

Table 24 Nature of ^{14}C residues in lettuce from crops sown at various intervals following a single 300 g ai/ha application of [^{14}C]chlorantraniliprole to soil and harvested at maturity

Lettuce	Days after soil treatment [a]			
	0	30	120	365
TRR (mequiv/kg)	0.023	0.042	0.029	0.031
%TRR				
Total extracted	91	88	83	92
chlorantraniliprole	85	70	72	64
IN-F6L99	ND	1.4	ND	ND
IN-F9N04	ND	1.6	3.5	5.2
IN-GAZ70	1.3	1.6	0.8	1.9
Unextracted	2.5	3.8	3.2	NQ

[a] Time of sowing of lettuce; sample for residues collected at time of crop maturity.

ND = Not detected.

For samples of both wheat grain and beet roots, TRRs at all sowing time points were not characterized further, due to the low levels of radioactivity present following application at 300 g ai/ha. The data for lettuce above show that there is likely to be some carry-over or transfer of residues into follow crops that may be sown up to 1 year after direct application of chlorantraniliprole to soil.

Table 25 Nature of ^{14}C residues in beet tops from crops sown at various intervals following a single 300 g ai/ha application of ^{14}C-chlorantraniliprole to soil and harvested at maturity

Beet foliage	Days after soil treatment [a]			
	0	30	120	365
TRR	0.063			
%TRR				
Total extracted	83	97		
Chlorantraniliprole	4.7	4.8		
IN-F6L99				
IN-F9N04	4.8			
IN-EVK64	2			

Beet foliage	Days after soil treatment [a]			
	0	30	120	365
TRR	0.063			
%TRR				
IN-HXH44	3.6			
IN-H2H20	2.8			
Unextracted	6			

[a] Time of sowing of beets; sample for residues collected at time of crop maturity

The characterisation and identification of the radioactivity in soil and wheat grain sown after application at an exaggerated rate of 900 g ai/ha is shown in Table 26. Soil analyses were conducted on samples taken at depths of 0–15 cm and also 15–30 cm. The components identified at both depths were identical as were the compositions of the extracted radioactivity and therefore data for the samples taken at a depth of 0 –15 cm only are shown.

The composition of the extracted radioactivity in wheat grain and in the soil at time of harvest is similar. The only components that were not detected in soil that were present in wheat grain were IN-HXH44 and IN-H2H20. Apart from chlorantraniliprole, all identified metabolites were present at < 0.002 mg/kg or < 2% TRR.

Table 26 Nature of ^{14}C residues in soil and in wheat from crops sown after application of [^{14}C]chlorantraniliprole to soil and harvested at maturity

Component	Time after application to soil		
	0 DAT Soil	108 DAT Soil	0 DAT Wheat grain [a]
TRR (mequiv/kg)	0.81	1.007	0.044
%TRR			
Extracted	92	96	61
Chlorantraniliprole	90	88	42
IN-F6L99	0.6	0.6	1.3
IN-F9N04	–	3	0.4
IN-GAZ70	–	0.4	0.2
IN-KAA24	–	0.2	–
IN-EQW78	–	2.7	1.6
IN-HXH44	–	–	1.3
IN-H2H20	–	–	0.7
Unextracted	NQ	NQ	21

[a] Time of sowing of wheat; sample taken at time of crop harvest which was 108 days after sowing.

NQ = Not quantifiable.

The data confirm the findings above, that is, detectable residues may be present in wheat grain sown in soil treated with chlorantraniliprole, with harvest of the wheat at 108 days after application.

The characterisation and identification of the radioactivity in wheat forage harvested from soil treated at 300 g ai/ha is shown in Table 27. The data reported are for fresh forage; sowing on days 0, 30, 120 and 365 after application to soil correspond to 28, 99, 159 and 409 days after the soil application. The 120 DAT soil sample had to be prepared separately and may explain the difference in samples from wheat grown in this soil compared to the day 0, 30 and 365 day samples.

Chlorantraniliprole is the major component of the extracted radioactivity in the wheat forage, ranging from 54 to 85% TRR over the four sampling points. Unextracted radioactivity ranged from 4.5% to 8.8% TRR. Several metabolites were identified, individually present at < 6% TRR. These metabolites included IN-F9N04, IN-GAZ70, IN-HXH44, IN-KAA24, IN-K7H29 and IN-EQW78. In relation to detectable concentrations, only chlorantraniliprole is present at levels that would require further consideration.

The results above confirm that chlorantraniliprole residues are likely to be present at detectable levels in forage grown up to 1 year after direct application to soil. This result is similar to that found in lettuce and beet foliage.

Table 27 Nature of ^{14}C residues in wheat forage from crops sown after application of ^{14}C-chlorantraniliprole to soil and harvested at maturity

Wheat forage	Days after soil treatment [a]			
	0	30	120	365
TRR (mequiv/kg)	0.082	0.109	0.25	0.062
%TRR				
Extracted	92	92	91	93
chlorantraniliprole	84	54	76	77
IN-HXH40/IN-HXH44	–	5.7	–	–
IN-HXH40	–	–	0.5	–
IN-F6L99	–	–	0.5	–
IN-F9N04	–	1.4	1.4	3.2
IN-GAZ70	–	0.7	0.3	0.9
IN-HXH44	–	–	0.2	0.4
IN-KAA24	–	2.1	0.5	0.1
IN-K7H29	–	2.2	–	0.2
IN-EQW78	–	1.4	0.8	1.2
Unextracted	8.8	7	4.5	8.6

[a] Time of sowing of wheat; samples collected at growth stages ranging 4 leaves, 5 tillers to 5 leaves, 5 tillers.

The data for wheat hay and straw are shown in Tables 28 and 29. Samples of wheat hay were dried after collection prior to extraction, whereas straw samples were collected at maturity and were dry. The reported TRRs for hay and straw are higher than in forage, explained by the dry nature of the feed commodities and therefore the values represent residues on a dry weight basis. Sowing on days 0, 30, 120 and 365 after application correspond to 56, 127, 181 and 431 days after application to soil for hay and 108, 165, 248 and 573 days after application to soil for straw.

Extracted radioactivity ranged from 86-100% TRR and 84–95% TRR for hay and straw, respectively. Chlorantraniliprole comprises the majority of the extracted radioactivity in hay and straw, ranging 50–73% TRR and 36–69% TRR, respectively. The metabolite composition of the radioactivity in hay and straw is similar with metabolites IN-F6L99, IN-F9N04, IN-GAZ70, IN-HXH44, and IN-EQW78 individually present at ≤ 3% TRR.

Table 28 Nature of ^{14}C residues in wheat hay from crops sown after application of ^{14}C-chlorantraniliprole to soil and harvested at maturity

Wheat hay	Days after soil treatment [a]			
	0	30	120	365
TRR (mequiv/kg)	0.393	0.54	1.721	0.329
%TRR				
Extracted	86	87	101	92

Wheat hay	Days after soil treatment [a]			
	0	30	120	365
TRR (mequiv/kg)	0.393	0.54	1.721	0.329
%TRR				
Chlorantraniliprole	61	73	51	67
IN-HXH40	2.2	–	–	–
IN-F6L99	–	0.2	2.4	2.1
IN-F9N04	0.8	1.8	2.1	2.1
IN-GAZ70	0.4	0.8	2.5	–
IN-HXH44	3.1	1.2	0.5	–
IN-KAA24	–	–	0.4	–
IN-K7H29	0.7	–	–	–
IN-EQW78	1.1	2.9	1.4	–
IN-H2H20	–	1.5	–	–
Unextracted	4.5	7.1	8.2	7.1

[a] Time of sowing of wheat; samples collected at growth stages ranging onset of flowering to early dough.

Table 29 Nature of ^{14}C residues in wheat straw from crops sown after application of ^{14}C-chlorantraniliprole to soil and harvested at maturity

Wheat straw	Days after soil treatment [a]			
	0	30	120	365
TRR (mequiv/kg)	0.371	0.339	1.939	0.467
%TRR				
Extracted	95	89	84	88
Chlorantraniliprole	69	63	64	37
IN-HXH40/IN-HXH44	–	1.9	–	–
IN-HXH40	–	1.1	–	–
IN-F6L99	–	0.5	0.6	1.7
IN-F9N04	–	1.6	1.3	2
IN-GAZ70	0.9	0.9	0.9	–
IN-HXH44	1.2	0.7	2.6	2.5
IN-KAA24	2.6	–	2.3	–
IN-K7H29	1.8	–	0.6	0.7
IN-EQW78	2.4	2.3	2.2	1
IN-H2H20	2.5	2	–	–
Unextracted	8.2	6.7	9.4	8.3

[a] Time of sowing of wheat; samples collected at normal crop harvest.

Table 30 Comparison of metabolites found in follow crop wheat hay and straw

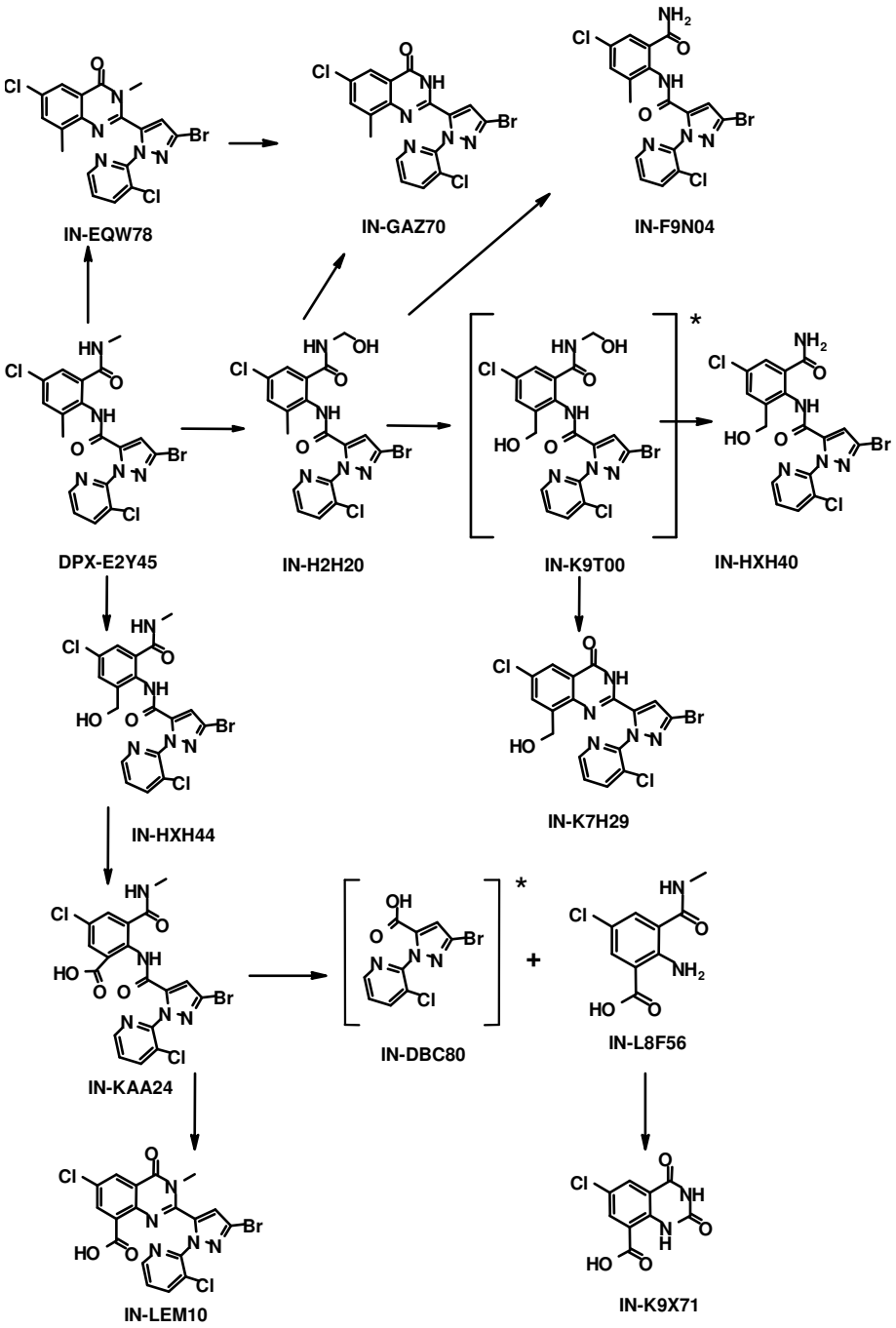
	Wheat hay		Wheat straw	
	0 DAT [a]	365 DAT [a]	0 DAT [a]	365 DAT [a]
TRR (mequiv/kg)	3.623	1.191	2.409	1.916
%TRR				
Extracted	88	102	95	85

	Wheat hay		Wheat straw	
	0 DAT [a]	365 DAT [a]	0 DAT [a]	365 DAT [a]
TRR (mequiv/kg)	3.623	1.191	2.409	1.916
%TRR				
Chlorantraniliprole	71	75	69	48
IN-HXH40	0.4	–	0.8	0.3
IN-HXH44	2.8	–	1.4	2.2
IN-F6L99	0.1	–	0.4	1.1
IN-F9N04	0.8	2.3	1.1	2.1
IN-GAZ70	0.4	–	0.8	–
IN-KAA24	0.9	–	3	0.8
IN-K7H29	0.6	–	3	0.4
IN-EQW78	1	1.4	2.4	2.3
IN-H2H20	0.6	–	1.6	–
IN-EVK64	–	–	0.4	–
Unextracted	5.2	8.7	6.3	12

[a] Time of sowing of wheat where DAT = days after soil treatment; samples collected at normal crop harvest.

The metabolites found in wheat straw are also found in wheat grain.

In summary, the results of the confined crop rotation study show that following application of [pyrazole carbonyl-^{14}C]-chlorantraniliprole to soil at 300 g ai/ha, detectable radioactivity was found in lettuce, wheat forage, hay and straw sown up to 365 days after application to soil. A large proportion of the radioactivity was extracted and it mostly comprised parent chlorantraniliprole. Although several metabolites were also identified from the extracted radioactivity in each of the commodities, their concentrations were < 5% TRR.



*** IN-DBC80 and IN-K9T00 shown in the brackets were not detected**

Figure 6 The proposed metabolic pathway for confined rotational crops

In an additional study Brown *et al*. (2005 12700) investigated the uptake of [^{14}C]chlorantraniliprole residues into rotational crops and the nature of the residues. [^{14}C]chlorantraniliprole (either [benzamide carbonyl-^{14}C] or [pyrazole carbonyl-^{14}C]chlorantraniliprole) was applied to 22 pots containing sandy loam soil; eleven pots per radiolabel at a rate equivalent to 150 g ai/ha. Water containing 0.6% Agridex surfactant was added to the spray solutions immediately before application, and then the spray applied to the soil surface of each pot. After application, the pots were kept in a greenhouse for a week and then outdoors. At 30 days after application, the pots were moved into the greenhouse and sown with wheat (cv Katepawa), soya beans (cv Williams 82) and radish (cv Cherry belle).

Table 31 Distribution of ^{14}C in various follow crops planted after application of [^{14}C]chlorantraniliprole to soil

Crop		DAT	DAP	TRR chlorantraniliprole (mg/kg equivalents)	
				benzamide carbonyl -[^{14}C]-	pyrazole carbonyl - [^{14}C]-
Radish [a]	Foliage	51	21	(0.089)	0.06
	Foliage	77	47	0.803	0.288
	Roots	51	21	(0.252)	0.039
	Roots	77	47	0.517	0.07
	Soil (harvest)	77	–	0.039	0.044
Wheat [b]	Foliage	51	21	0.613	0.218
	Foliage	86	56	0.512	0.35
	Seed/heads	86	56	0.088	0.061
	Straw	113	83	NA	0.682
	Chaff	113	83	NA	0.411
	Grain	113	83	NA	0.014
	Soil	113	NA	NA	(0.034)
	Straw	135	105	1.084	NA
	Chaff	135	105	0.447	NA
	Grain	135	105	0.017	NA
	Soil	135	NA	(0.049)	NA
Soya bean [c]	Foliage	77	47	(0.113)	(0.041)
	Foliage	87	57	NA	0.093
	Foliage	98	68	(0.135)	NA
	Foliage	176	146	0.126	0.147
	Pods	176	146	0.022	ND
	Beans	176	146	0.007	ND
	Soil	176	NA	(0.042)	(0.045)

DAT = days after soil treatment and is the planting date

DAP = samples for residue analysis collected at *x* days after planting

Values in parentheses are based on combustion values; all other values are based on extraction data.

NA = Not applicable.

[a] Immature radish samples (foliage and roots collected 21 days after planting; final harvest (foliage and roots) at 47 days after planting.

[b] Immature wheat samples (foliage and/or seed heads) were collected at 21 and 56 days after planting; final harvest (foliage, chaff and grain) at 83 days after planting for pyrazole carbonyl label and 105 days after planting for benzamide carbonyl label.

[c] Immature soya bean samples (foliage) collected at 47, 57 and 68 days after planting; final harvest (foliage, pods and beans) at 146 days after planting.

TRRs in the commodities grown in soil treated with [^{14}C]chlorantraniliprole ranged between 0.01and 0.02 mg/kg equivalents for wheat grain, 0.07–0.52 mg/kg equivalents for radish roots, and ≤ 0.01 mg/kg equivalents for soya beans. Residue levels in radish from the [benzamide-carbonyl ^{14}C] treated plot were high and explained as being due to fungal disease leading to the formation of small roots. TRRs in wheat commodities taken from soil treated with [^{14}C]chlorantraniliprole were 0.21–0.61 mg/kg equivalents for forage, 0.35–0.51 mg/kg equivalents for samples that would correspond to green hay, and 0.68–1.08 mg/kg equivalents for straw. The TRRs in soya bean commodities were 0.04–0.11 mg/kg equivalents for forage, 0.09–0.14 mg/kg equivalents for samples that would

correspond to fodder and 0.13–0.15 mg/kg equivalents for straw. TRRs in radish foliage were 0.29–0.81 mg/kg equivalents.

The nature of the radioactivity in soil samples is shown in Table 32.

Table 32 Nature of ^{14}C in soil samples collected at time of harvest of various follow crops planted after application of [^{14}C]chlorantraniliprole to soil

Soil Sample	DAT	TRR (mg/kg)	Extracted % TRR	Chlorantraniliprole % TRR
[benzamide-carbonyl -^{14}C]- chlorantraniliprole				
Radish harvest	77	0.039	85	60
Wheat harvest	135	0.049	NC	NA
Soybean harvest	176	0.042	NC	NA
[pyrazole carbonyl -^{14}C]- chlorantraniliprole				
Radish harvest	77	0.045	86	68
Wheat harvest	113	0.037	NC	NA
Soybean harvest	176	0.045	NC	NA

DAT = days after soil treatment

NC = not conducted; only soils collected at radish harvest were extracted

NA = not available since soils were not extracted

The majority (~85% TRR) of the soil residues were readily extracted from the soil samples collected at 77 days after treatment for the radish plot. The major component of the radioactivity extracted from the soil was chlorantraniliprole at 60–68% TRR. Levels of other soil components, tentatively identified as IN-F9N04 and IN-EQW78, were insignificant (individually less than 7% TRR; < 0.01 mg/kg).

The distribution and characterisation of the radioactivity in various plant matrices following application of either labelled solution are shown in Tables below.

Table 33 Distribution and nature of ^{14}C in various follow crops planted after application of [benzamide-carbonyl -^{14}C]chlorantraniliprole to soil.

Sample	DAT	TRR (mg/kg)	Extracted % TRR	Chlorantraniliprole % TRR
Radish				
Foliage	51	(0.089)	NC	NC
Foliage	77	0.803	95	76
Wheat				
Foliage [a]	51	0.613	93	86
Foliage[b]	86	0.512	90	56
Straw	135	1.084	87	73
Soya beans				
Foliage[a]	77	(0.113)	NC	NC
Foliage[b]	98	(0.135)	NC	NC

Sample	DAT	TRR (mg/kg)	Extracted % TRR	Chlorantraniliprole % TRR
Straw	176	0.126	80	64
Pods	176	0.022	77	NA[c]

DAT = days after soil treatment

NC = sample analysis was not conducted. Analysis of samples collected at a later time point indicated chlorantraniliprole was the principal extractable component and that no significant metabolites were present. Therefore, analyses of the earlier samples were not conducted/completed.

NA = not analysed.

Data in () are combustion results

[a] Forage sample

[b] Green-hay sample

[c] [benzamide-carbonyl -^{14}C] soya bean pods were extracted, but not analysed further due to extremely low levels of radioactivity and the oily nature of the extracts. No detectable TRR were found in the [pyrazole carbonyl -^{14}C] soya bean pods.

Table 34 Distribution and nature of ^{14}C in various follow crops planted after application of [pyrazole carbonyl -^{14}C]chlorantraniliprole to soil

Sample	DAT	TRR (mg/kg)	Extracted % TRR	Chlorantraniliprole % TRR
Radish				
Foliage	51	0.060	96	NC
Foliage	77	0.288	91	54
Wheat				
Foliage[a]	51	0.218	88	75
Foliage[b]	86	0.350	95	80
Straw	135	0.682	88	69
SoyA beans				
Foliage[a]	77	(0.041)[c]	NC	NC
Foliage[b]	98	0.093	94	68
Straw	176	0.147	71	45

DAT = days after soil treatment

NC = sample analysis was not conducted. Analysis of samples collected at a later time point indicated chlorantraniliprole was the principal extractable component and that no significant metabolites were present. Therefore, analyses of the earlier samples were not conducted/completed.

[a] Forage sample

[b] Green-hay sample

[c] Data in () are combustion results

The majority of the radioactivity in plant commodities was readily extracted (71–95% TRR) with aqueous acetonitrile. Chlorantraniliprole was the principal radioactive component in radish (54–76% TRR), wheat (56–86%), and soya bean (45–80%) livestock feed items. Minor metabolites (individually present at ≤ 3.0% TRR) detected in radish, wheat, and soya bean fodder was identified as IN-EQW78, IN-GAZ70, and IN-F9N04.

Table 35 Nature of ^{14}C in various follow crops planted after application of ^{14}C-chlorantraniliprole to soil and comparison of results for [pyrazole carbonyl -^{14}C]- and [benzamide-carbonyl -^{14}C]-labelled chlorantraniliprole.

Crop commodity	Radish foliage	Radish root	Wheat straw	Wheat chaff	Wheat grain	Soya bean fodder	Soya bean fodder
Label	pyrazole carbonyl	pyrazole carbonyl	benzamide-carbonyl	benzamide-carbonyl	benzamide-carbonyl	benzamide-carbonyl	pyrazole carbonyl
DAT	77	77	135	135	135	176	176
TRR (mg equiv/kg)	0.288	0.07	1.084	0.447	0.017	0.126	0.147
%TRR							
Total Extracted	91	98	87	93	92	80	71
Chlorantraniliprole	54	68	73	87	86	64	45
IN-F9N04	1.1	2.9	1.2	ND	ND	1.8	2
IN-GAZ70			0.4	ND	ND	6.4	3
IN-EQW78	1.5	1.4	1.4	0.8	ND	1	0.6
Unextracted	9	2.3	10	7.3	7.5	15	21

DAT = days after soil treatment

Minor components of the extracted radioactivity in soybean fodder comprise less than 10% TRR.

In summary, the transfer of chlorantraniliprole residues from soil that was treated with ^{14}C compound at 150 g ai/ha at 30 days prior to sowing radish, wheat and soybeans was low. The majority of the extracted radioactivity was composed of parent chlorantraniliprole at 45–86% TRR in all commodities. Minor metabolites that were detected in various commodities included IN-F9N04, IN-GAZ70 and IN-EQW78, all present at less than 10% TRR for any individual commodity. Parent chlorantraniliprole is the major component of the residue in follow or succeeding crops.

Field crop rotational studies

Residues of chlorantraniliprole (parent compound) in follow crops were studied at five sites in Canada and the USA. Leafy vegetables (lettuce, spinach and Swiss chard), pulses (soya beans), cereals (oats, wheat) and root and tuber vegetables (beetroot, radish and turnip) were planted 13–279 days after applications of chlorantraniliprole to bare soil or a crop at rates totalling 200, 225 and 600 g ai/ha. At harvest, residues in leafy vegetables were < 0.003 to 0.01 mg/kg. In leaves/tops of radish, beetroot and turnip residues ranged from < 0.003 to 0.16 mg/kg. Residues in roots of vegetables grown as follow-crops were < 0.003 to 0.010 mg/kg. In soya bean follow-crops, residues in grain were < 0.003 and 0.004 mg/kg while residues in forage and hay were 0.027 to 0.055 mg/kg. Residues in oat and wheat grain were < 0.003–0.006 mg/kg for plant back intervals of 15 to 238 days. In forage, straw and hay residues ranged from 0.003 to 0.015 mg/kg.

Table 36 Residues of chlorantraniliprole in various follow crops planted after application of chlorantraniliprole to bare soil or cropped plots (12775, 12776, 12777, 14818, 17045)

Location	Application rate (g ai/ha)	Crop (variety)	Matrix	DAP (days)	PBI (days)	Residues (mg/kg)	Average (mg/kg)
Corvallis[a], OR, USA 2004	200	NA	Soil	NA	30	0.142 0.128 0.121	0.130
		Oats (Cayuse)	Forage	49	30	0.012 0.015	0.013
			Grain	99	30	0.004 0.004	0.004
			Straw	99	30	0.032 0.028	0.030
			Hay	65	30	0.054 0.04	0.051
		Turnip	Roots	72	30	< 0.003 < 0.003	< 0.003

Location	Application rate (g ai/ha)	Crop (variety)	Matrix	DAP (days)	PBI (days)	Residues (mg/kg)	Average (mg/kg)
		(Shogoin)	Tops	72	30	0.003 0.003	0.003
		Beets (Red Ace)	Roots	72	30	< 0.003 < 0.003	< 0.003
			Tops	72	30	< 0.003 < 0.003	< 0.003
Donna[b],	50	NA	Soil	NA	40	0.042 0.046 0.042	0.043
TX, USA	50	NA	Soil	NA	61	0.031 0.033 0.038	0.034
2003	100	Swiss Chard (Silverado)	Leaves	59	61	< 0.003 < 0.003	< 0.003
		Radish	Root	44	40	0.005 0.004	0.005
		(Champion)	Tops	44	40	0.066 0.070	0.068
	63	NA	Soil	NA	130	0.028 0.022 0.030	0.026
	37	NA	Soil	NA	151	0.025 0.030 0.032	0.029
	50 50	Swiss Chard (Silverado)	Leaves	59	151	< 0.003 < 0.003	< 0.003
		Radish	Root	44	130	< 0.003 0.003	0.003
		(Champion)	Tops	44	130	0.030 0.030	0.030
	150	NA	Soil	NA	40	0.103 0.108 0.129	0.11
	151	NA	Soil	NA	61	0.113 0.085 0.091	0.096
	299	Swiss Chard (Silverado)	Leaves	59	61	0.010 0.007	0.009
		Radish	Root	44	40	0.009 0.010	0.010
		(Champion)	Tops	44	40	0.17 0.16	0.16
	189	NA	Soil	NA	130	0.085 0.083 0.073	0.080
	114	NA	Soil	NA	151	0.067 0.066 0.065	0.066
	149 150	Swiss Chard (Silverado)	Leaves	59	151	0.004 0.005	0.005
		Radish	Root	44	130	0.005 0.004	0.005
		(Champion)	Tops	44	130	0.078 0.061	0.070
Madera[c],	50	NA	Soil	NA	122	0.17 0.13 0.12	0.14
CA, USA	48	Wheat (Yecorro	Forage	95	31	0.032 0.030	0.031
2003	50	Rogo)	Grain	192	31	0.004 0.004	0.004
	50		Straw	192	31	0.045 0.033	0.039
			Hay	140	31	0.043 0.042	0.043
		Lettuce (Great Lakes, Head)	Leaves	141	31	0.004 0.003	0.003
		Beets (Detroit	Roots	142	31	0.006 0.006	0.006
		Dark Red)	Tops	142	31	0.016 0.013	0.015
	149	NA	Soil	NA	122	0.041 0.047 0.044	0.044
	150	Wheat (Yecorro	Forage	95	122	0.032 0.012	0.022
	152	Rogo)	Grain	192	122	0.003 0.003	0.003
	151		Straw	192	122	0.017 0.020	0.018
			Hay	140	122	0.022 0.008	0.015
		Lettuce (Great Lakes, Head)	Leaves	141	122	0.003 0.010	0.005
	49	NA	Soil	NA	31	0.33 0.30 0.30	0.31
	51	Wheat	Forage	95	31	0.086 0.080	0.083

Location	Application rate (g ai/ha)	Crop (variety)	Matrix	DAP (days)	PBI (days)	Residues (mg/kg)	Average (mg/kg)
	49	(Yecorro Rogo)	Grain	192	31	0.005 0.006	0.006
	50		Straw	192	31	0.14 0.10	0.12
			Hay	140	31	0.15 0.16	0.15
		Lettuce (Great Lakes, Head)	Leaves	141	31	0.014 0.006	0.010
		Beets (Detroit	Roots	142	31	0.007 0.009	0.008
		Dark Red)	Tops	142	31	0.028 0.041	0.034
	146	NA	Soil	NA	122	0.17 0.13 0.12	0.23
	148	Wheat (Yecorro	Forage	95	122	0.049 0.055	0.052
	147	Rogo)	Grain	192	122	0.004 0.007	0.006
	148		Straw	192	122	0.086 0.078	0.082
			Hay	140	122	0.098 0.10	0.10
		Lettuce (Great Lakes, Head)	Leaves	141	122	0.007 0.003	0.005
Rochelle[d],	50	NA	Soil	NA	30	0.16 0.13 0.16	0.15
IL, USA	52	Oats (Moraine)	Forage	39	30	0.014 0.017	0.016
2003	51		Grain	85	30	< 0.003 < 0.003	< 0.003
	50		Straw	85	30	< 0.003 0.004	0.003
			Hay	58	30	0.005 0.005	0.005
		Lettuce (Green Salad Bowl)	Leaves	71	30	< 0.003 < 0.003	< 0.003
		Beets (Detroit	Roots	73	30	< 0.003 < 0.003	< 0.003
		Medium Top)	Tops	73	30	< 0.003 < 0.003	< 0.003
	50	NA	Soil	NA	123	0.075 0.081 0.071	0.076
	50	Radish	Root	51	123	< 0.003 < 0.003	< 0.003
	51	(Champion)	Tops	51	123	< 0.003 < 0.003	< 0.003
	51	Spinach (Melody)	Leaves	69	123	0.006 0.005	0.005
		Winter Wheat	Grain	302	123	< 0.003 < 0.003	< 0.003
		(Pioneer 25R47)	Straw	302	123	0.016 0.011	0.014
			Hay	265	123	0.015 0.019	0.017
			Forage	223	123	0.007 0.009	0.008
	150	NA	Soil	NA	30	0.47 0.39 0.34	0.40
	151	Oats (Moraine)	Forage	39	30	0.040 0.038	0.039
	154		Grain	85	30	< 0.003 < 0.003	< 0.003
	152		Straw	85	30	0.011 0.011	0.011
			Hay	58	30	0.027 0.036	0.031
		Lettuce (Green Salad Bowl)	Leaves	71	30	< 0.003 < 0.003	< 0.003
		Beets (Detroit	Roots	73	30	< 0.003 < 0.003	< 0.003
		Medium Top)	Tops	73	30	< 0.003 < 0.003	< 0.003
	151	NA	Soil	NA	123	0.25 0.27 0.18	0.23
	153	Radish	Root	51	123	< 0.003 < 0.003	< 0.003
	154	(Champion)	Tops	51	123	0.009 0.011	0.010
	153	Spinach (Melody)	Leaves	69	123	0.011 0.008	0.010
		Winter Wheat	Grain	302	123	0.009 0.004	0.006

Location	Application rate (g ai/ha)	Crop (variety)	Matrix	DAP (days)	PBI (days)	Residues (mg/kg)	Average (mg/kg)
		(Pioneer 25R47)	Straw	302	123	0.028 0.035	0.032
			Hay	265	123	0.059 0.057	0.058
			Forage	223	123	0.020 0.020	0.020
Berwick[e], NS, Canada 2005	220 (~3 days before harvest of the broccoli cover crop)	NA	Soil	NA	15	0.076 0.089 0.087	0.084
		NA			238	0.046 0.041 0.044	0.044
		NA	Soil	N/A	279	0.0083 0.035 0.033	0.026
		Turnip (Purple Top White Globe)	Roots	110	279	ND ND	ND
			Tops	110	279	0.004 0.003	0.004
		Soya bean (Vista RR)	Forage	32	279	0.037 0.045	0.041
			Hay	73	279	0.064 0.045	0.055
			Seed	114	279	ND ND	ND
		Winter Wheat (AC Sampson) .	Forage	245	15	0.021 0.022	0.022
			Hay	258	15	0.049 0.040	0.045
			Grain	301	15	0.004 0.004	0.004
			Straw	301	15	0.062 0.059	0.061
		Spring Wheat (AC Helena)	Forage	60	238	0.042 0.043	0.043
			Hay	71	238	0.14d 0.15d	0.14
			Grain	71	238	0.003 0.003	0.003
			Straw	114	238	0.076 0.080	0.078
	225 (~14 days before planting of the rotational crops)	NA	Soil	N/A	131	0.073 0.097 0.067	0.079
		Turnip (Purple Top White Globe)	Roots	77	13	ND ND	ND
			Tops	77	13	0.005 0.004	0.005
		Spinach (Longstanding Bloomsdale)	Leaves	77	13	0.005 0.004	0.005
		Soya bean (Vista RR)	Forage	62	13	0.035 0.019	0.027
			Hay	109	13	0.036 0.038	0.037
			Seed	112	13	0.004 ND	0.004

PBI = Plant back interval which is the number of days between last application to treated crop and sowing of succeeding crops.

DAP = days after planting and is the number of days between sowing and sampling of mature succeeding crop commodities.

[a] Corvallis, OR, USA 2004 Silty Clay Loam; %OM 2.2; pH 6.0; CEC meq/100 g 14.2; rainfall/irrigation 5.6 – 13.9 cm

[b] Donna, TX, USA 2003 Clay %OM 1.1; pH 8.2; CEC 19.47 meq/100 g; rainfall/irrigation 0 - 16.8 cm

[c] Madera, CA, USA 2003 Loam %OM 1.4; pH 7.1; CEC 25.2meq/100 g; rainfall/irrigation 0.00 – 6.1 cm

[d] Rochelle, IL, USA 2003 Silty Clay Loam %OM 4.0; pH 5.4: CEC 23.1 meq/100 g; rainfall/irrigation 0.89 – 19.2 cm

[e] Berwick, NS, Canada 2005 Loamy Sand %OM 3.0; pH 6.2 CEC 9.9 meq/100 g; rainfall/irrigation 2.18-23.3 cm

Low levels of chlorantraniliprole could be detected in follow-crops.

METHODS OF RESIDUE ANALYSIS

Analytical methods

A number of different analytical methods have been reported for the analysis of chlorantraniliprole in plant and animal matrices. The basic approach employs extraction by homogenisation with acetonitrile:water, and column clean-up using hydrophilic-lipophilic balanced polymeric (HLB) and strong anion exchange (SAX) SPE columns in sequence). Residues are determined by gas chromatography (GC) with an electron capture detector (ECD) or liquid chromatography with mass spectra detection (MS/MS). In addition, the German DFG S19 multi-residue method with LC/MS/MS detection has shown to be applicable to the analysis of chlorantraniliprole residues in crops and animal tissues. The limit of quantitation was usually 0.01 mg/kg while the LOD was approximately 0.003 mg/kg. The table below provides a summary of some methods for chlorantraniliprole analysis of crops and animal tissues and milk. Details of recoveries reported as part of method validation are reported in tables that follow for the major methods used for determination of chlorantraniliprole in the residue trials.

Table 37 Summary of major analytical methods used for the determination of chlorantraniliprole, including some metabolites, in various matrices

Method/reference	Matrix	Extraction	Clean-up	Detection, LOQ
11374 (also 13294, 13295, 13292)	Plant commodities, including fruits, leafy vegetables, oilseeds, legumes, cereal grains, forage and fodder, dried fruit, and cereal grains	CH_3CN:H_2O	SPE (SAX, HLB) Elute with acetonitrile:ethyl acetate (4:1)	LC/MS/MS Chlorantraniliprole: LOQ 0.01 mg/kg
13291	Plant commodities, including cereal grains, tree nuts, oilseeds, legumes, forage, high acid and high water content fruit and vegetables	CH_3CN:H_2O	SPE (SAX, HLB) Elute with hexane/ethyl acetate (1:1)	Base hydrolysis for 2 hours to convert chlorantraniliprole to thermally stable IN-EQW78. GC/ECD. LOQ 0.01 mg/kg
13261 (also 18611) German official multi-residue method (DFG-S19)	Tomato, orange, wheat grain, almond	Tomato and wheat grain: extraction with water and acetone Orange: aqueous sodium bicarbonate and acetone. Almond: acetone/ CH_3CN in the presence of calcium silicate and diatomaceous earth, filtration, evaporation and reconstitution in ethyl acetate/cyclohexane.	Tomato and wheat: partition with ethyl acetate/cyclohexane/NaCl Orange: partition with ethyl acetate/cyclohexane/NaCl All: gel permeation chromatography	LC/MS/MS Chlorantraniliprole: LOQ 0.01 mg/kg
14314	Processed plant commodities, including raisins, tomato ketchup, apple juice, grape juice, orange peel,	Oil samples: dilute with hexane Solid samples: CH_3CN/H_2O Juice samples:	Oil samples: methanol partition, or by SPE Solid and juice: SPE	LC/MS/MS Chlorantraniliprole: LOQ 0.01 mg/kg

Method/reference	Matrix	Extraction	Clean-up	Detection, LOQ
	cooked spinach, grape pomace, and cotton seed oil.	dilute with CH_3CN		
11376 (also 18100, 17123)	Animal commodities (milk, cream, muscle, fat, liver, kidney and egg)	CH_3CN/H_2O	Partition with hexane, aqueous extracts cleaned up using SPE (SAX + HLB)	LC/MS/MS Chlorantraniliprole: LOQ 0.01 mg/kg
19533	Animal commodities (bovine muscle, liver, kidney, fat, milk, and chicken egg)	CH_3CN/H_2O	Partition with hexane, aqueous extracts cleaned up using SPE (SAX + HLB), convert chlorantraniliprole to the thermally stable metabolite IN-EQW78 by heating with aqueous base	GC-ECD Chlorantraniliprole: LOQ 0.01 mg/kg
15025, 18610, German official multi-residue method (DFG-S19)	Animal commodities (muscle, fat, liver, milk and egg)	Milk, muscle, egg and liver: acetonitrile and water, followed by partition with ethyl acetate and cyclohexane. Fat: dissolve in ethyl acetate/cyclohexane.	Extracts cleaned up by gel permeation chromatography	LC/MS/MS Chlorantraniliprole: LOQ 0.01 mg/kg

Method 11374 (Hill and Stry 2004): Samples were soaked in water for 20 minutes, then acetonitrile was added and the sample homogenised. The supernatant was decanted and the samples extracted with additional acetonitrile. An aliquot of the combined extracts was diluted with water and cleaned-up on a strong anion exchange and an HLB SPE cartridge connected in series. The cartridges were washed with water/acetonitrile, air dried, and chlorantraniliprole eluted from the HLB cartridge using acetonitrile and ethyl acetate. The eluant was evaporated to dryness and the residue was reconstituted in acetonitrile and diluted with water for analysis of chlorantraniliprole by LC/MS/MS operating in the positive ionization mode. The following ion transitions were monitored (484 → 453, 484 → 286, or 284 → 112, 284 → 177).

Studies 13294, 13295, 13292 describe minor modifications to the method that included changes in extraction and elution volumes and additional validation results.

Table 38 Analytical recoveries for various matrices fortified with chlorantraniliprole and analysed using method 11374

Matrix	Fortification (mg/kg)	Individual recoveries (%)	Mean recovery (%)	% RSD	Reference
Potatoes	0.010 0.10 10.0	83 104 95 96 103 109 92 108	94 103 100	11 6 11	13294
Potatoes	0.01 0.10	79 72 104 96 106 99 105 93 92 93	91 96	16.6 5.9	13295
Sugar beets (tops)	0.010 0.10 10.0	106 105 107 105 106 104 93 98	106 105 95	1 1 4	13294
Lettuce	0.010 0.10	97 106 97			11374

Matrix	Fortification (mg/kg)	Individual recoveries (%)	Mean recovery (%)	% RSD	Reference
Lettuce	0.010 0.10 10.0	110 110 100 93 94 90 84 84	107 92 84	5 3 1	13294
Lettuce	0.01 0.10	108 105 99 112 106 97 85 110 105 88	106 97	4.6 11.2	13295
Broccoli	0.010 0.10 10.0	107 85 103 103 104 111 101 107	98 106 104	12 4 4	13294
Soya beans	0.01 0.10	84 95 89			11374
Soya beans	0.010 0.10 10.0	95 108 95 94 92 93 122 114	99 93 118	7 1 5	13294
Soya bean forage	0.010 0.10 10.0	108 103 113 110 102 90 113 110	108 101 112	5 10 2	13294
Cotton seed	0.01 0.10	93 99 90			11374
Cotton gin trash	0.01 0.10	87 99 86			11374
Peppers	0.01 0.10	87 96 97			11374
Tomatoes	0.01 0.10	91 106 101			11374
Tomatoes	0.010 0.10 10.0	94 96 97 115 102 105 104 111	95 107 108	2 6 5	13294
Tomatoes	0.01 0.10	112 97 89 82 121 105 107 107 115 107	100 108	16.3 3.6	13295
Cucumbers	0.010 0.10 10.0	97 94 84 99 99 94 98 93	92 97 96	7 3 4	13294
Oranges	0.010 0.10 10.0	87 95 93 97 97 122 89 91	92 105 90	5 14 1	13294
Lemons	0.01 0.10	103 123 99 102 91 110 97 100 115 104	104 105	11.3 7.0	13295
Apples	0.010 0.10 10.0	100 94 101 115 103 113 99 94	98 110 97	3 6 4	13294
Apples	0.01 0.10	91 78 88 104 71 90 101 92 78 88	86 90	14.7 9.2	13295
Pears	0.010 0.10 10.0	106 95 112 97 97 97 99 92	105 97 96	8 0.27 6	13294
Peaches	0.010 0.10 10.0	105 103 100 101 101 108 94 93	103 103 93	2 4 0.39	13294
Peaches	0.01 0.10	75 87 97 118 87 79 62 97 101 98	93 87	17.6 18.8	13295

Matrix	Fortification (mg/kg)	Individual recoveries (%)	Mean recovery (%)	% RSD	Reference
Almonds (nut meat)	0.010 0.10 10.0	91 99 95 91 93 93 111 127	95 92 119	4 1 10	13294
Almonds (nut meat)	0.01 0.10	123 108 105 109 107 105 116 118 122 105	110 113	6.5 6.9	13295
Almonds (nut meat)	0.01 0.10	93 78 72 73 94 72 92 73 77 90	82 81	13 12	13292
Apples	0.01 0.10	92 78 108 104 107 86 89 85 104 107	98 94	13 10	13292
Rice grain	0.010 0.10 10.0	93 92 91 97 100 106 90 94	92 101 92	1 5 4	13294
Rice (white)	0.01 0.10	90 93 86			11374
Rice (brown)	0.01 0.10	85 99 92			11374
Wheat grain	0.010 0.10 10.0	85 107 111 115 117 113 108 111	101 115 109	14 2 2	13294
Wheat grain	0.01 0.10	108 115 97 94 93 95 91 110 108 85	101 98	9.6 11.3	13295
Wheat grain	0.01 0.10	89 106 94 105 102 111 110 105 105 108	99 108	7.5 2.6	13292
Wheat straw	0.01 0.10	81 89 73 78 78 70 69 77 71 71	80 72	7.5 4.2	13295
Wheat hay	0.010 0.10 10.0	96 84 96 111 100 98 75 85	92 103 80	7 7 9	13294
Corn fodder	0.01 0.10	81 88 85 88			11374
Corn stover	0.010 0.10 10.0	99 95 97 104 101 103 88 85	97 102 87	2 2 2	13294
Alfalfa forage	0.010 0.10 10.0	93 93 97 99 107 104 104 92	94 103 108	3 4 8	13294
Cotton seed	0.010 0.10 10.0	108 83 96 90 85 93 87 78	95 89 82	13 4 8	13294
Grapes	0.010 0.10	96 87			11374
Grapes	0.010 0.10 10.0	91 90 92 103 119 114 105 114	91 112 110	1 8 5	13294
Grapes	0.01 0.10	115 105 90 104 110 105 97 100 104 104	105 102	9.0 3.4	13295
Raisins	0.010 0.10	95 95 97			11374

Matrix	Fortification (mg/kg)	Individual recoveries (%)	Mean recovery (%)	% RSD	Reference
Corn grain	0.010 0.10 10.0	88 94 97 90 94 91 94 114	93 92 104	5 2 14	13294
Rape seed	0.01 0.10	110 93 103 107 106 108 104 108 105 111	104 107	6.3 2.6	13295
Tea	0.01 0.10	84 88			11374

Method 13291 (Gagnon *et al.* 2005): Chlorantraniliprole residues were extracted from homogenised sample material with acetonitrile:water. The supernatant was decanted and the samples extracted with additional acetonitrile. The extracts were combined and an aliquot diluted with water. The aqueous acetonitrile extract was sequentially filtered through a SAX and an HLB SPE cartridge. After washing the cartridges with water/acetonitrile, the cartridges were air dried, and the HLB cartridge eluted with hexane/ethyl acetate (1:1). The eluant was evaporated to dryness and then heated with 0.15% ammonia solution at 70 °C for two hours to convert chlorantraniliprole to IN-EQW78, which was then extracted into ethyl acetate. The extract was evaporated to dryness and reconstituted in acetonitrile, then made up to volume with ethyl acetate/acetonitrile (1:1) before analysis by GC-ECD.

Table 39 Analytical recoveries for various matrices fortified with chlorantraniliprole and analysed using method 13291

Matrix	Fortification (mg/kg)	Range recoveries (%) (n=5)	Mean recovery (%)	% RSD	Reference
Rice grain	0.010 0.10	85-93 77-81	89 79	3.3 2.0	13291
Maize grain	0.01 0.10	89-99 71-82	95 80	4.1 3.2	13291
Tomatoes	0.010 0.10	93-113 73-87	103 81	8.3 8.9	13291
Almonds	0.010 0.10	91-94 72-77	92 75	1.2 3.5	13291
Apples	0.010 0.10	93-120 77-89	107 83	9.9 6.2	13291
Alfalfa (lucerne) forage	0.010 0.10	75-106 74-80	94 77	12.2 7.3	13291
Grapes	0.010 0.10	85-112 80-88	101 83	10.1 4.0	13291
Cottonseed	0.010 0.10	83-106 70-84	96 77	9.5 7.3	13291
Cucumber	0.010 0.10	100-118 76-83	110 79	7.5 4.5	13291
Soya bean	0.010 0.10	85-100 70-84	91 78	6.6 8.6	13291

Method 13261: Rzepka (2005) described incorporation of chlorantraniliprole in the German official modular multi residue method for analysis of chlorantraniliprole residues in four representative crops (tomato, orange, wheat grain and almonds). For the multi-residue method, plant samples were extracted using module E1 for tomato, E2 for wheat grain, E3 for orange and E7 for almond nut meat.

The E1 module involved extracting chlorantraniliprole with water/acetone, partitioning the extract with ethyl acetate/cyclohexane in the presence of NaCl. The E2 module involved soaking the grain sample in water before homogenising with acetone, partitioning the extract with ethyl acetate/cyclohexane in the presence of NaCl. The E3 module involved extracting the orange sample with sodium bicarbonate aqueous solution mixed with acetone. The aqueous extract was partitioned with ethyl acetate/cyclohexane in the presence of NaCl.

For E1, E2 and E3: An aliquot of the organic phase was removed and filtered through cotton wool covered with Na_2SO_4. The filtrate was concentrated to yield an aqueous phase, and ethyl acetate, Na_2SO_4 and NaCl were added. Cyclohexane was added to the mixture, and the salts were allowed to settle before gel permeation chromatography of an aliquot of the solvent.

The E7 module involved blending almond nut meat with acetone, acetonitrile, Calflo E (calcium silicate) and celite (diatomaceous earth). The mixture was filtered under vacuum and the filtrate was filtered again through Calflo E. Iso-octane was added and the extract concentrated at 40 °C and evaporated to dryness under a stream of nitrogen. The residue was reconstituted in ethyl acetate/cyclohexane before gel permeation chromatography.

For E1, E2, E3 and E7, gel permeation chromatography an aliquot of the final extract was injected onto a column of Bio Beads S-X3 polystyrene gel. Chlorantraniliprole was eluted off the column with ethyl acetate/cyclohexane. Cleaned-up samples were analysed by LC/MS/MS operating in the positive ionization mode. Two ion transitions were monitored at 484 → 453 and 484 → 286 amu for determination. Quantification was achieved by external standard linear regression calibration. As significant matrix effects were observed for orange, matrix-matched external standard were used for oranges. The limit of quantification (LOQ) was 0.01 ppm. Chlorantraniliprole was not detectable (< 0.003 ppm) in control samples.

van Scheik (2006, 18611) provided additional validation data for method 13261.

Table 40 Recovery validation of the LC-MS/MS multi-residue method 13261 for chlorantraniliprole

Matrix	Fortification (mg/kg)	Range recoveries (%) (n=5)	Mean recovery (%)	% RSD	Reference
Tomatoes	0.01	102-116	108	5.1	13261
	0.10	102-117	110	5.5	
Tomatoes	0.01	84-95	90	5.2	18611
	0.10	80-91	86	4.5	
Oranges	0.01	93-99	95	2.8	13261
	0.10	82-106	96	9.9	
Oranges	0.01	85-106	93	9.9	18611
	0.10	71-95	84	12.1	
Almond (nut meat)	0.01	78-84	81	2.7	13261
	0.10	75-91	83	7.3	
Almond (nut meat)	0.01	92-109	99	7.0	18611
	0.10	63-95	77	18.2	
Wheat grain	0.01	88-96	92	3.6	13261
	0.10	93-109	99	6.2	
Wheat grain	0.01	97-102	100	2.3	18611
	0.10	93-106	100	5.4	

Method 14314 (Hill and Stry 2004 14314a): A method was developed for determination of chlorantraniliprole and IN-EQW78, IN-ECD73 and IN-F6L99 in processed crop fractions, including raisins, tomato ketchup, orange peel, grape pomace, cooked spinach, apple juice, grape juice and cottonseed oil and meal. Residues of chlorantraniliprole, IN-ECD73 and IN-EQW78 were extracted from solid processed crop fractions following pre-soaking in water. The homogenised samples were extracted twice with acetonitrile (ketchup samples were not homogenized). An aliquot of the

combined extracts was diluted with water and cleaned-up on an HLB (hydrophilic-lipophilic balanced polymeric) solid phase extraction (SPE) cartridge. After washing and drying the cartridge, chlorantraniliprole, IN-ECD73 and IN-EQW78 were eluted off the HLB cartridge with acetonitrile and ethyl acetate. The eluant was dried and the sample dissolved in acetonitrile/0.01 M aqueous formic acid for analysis by LC/MS/MS.

Residues of IN-F6L99 were extracted from solid processed crop fractions as above until the extracts were combined and diluted with water. At that point an aliquot was removed and taken to near dryness. The sample was reconstituted in water before clean-up on an HLB SPE cartridge. After washing and drying the cartridge, IN-F6L99 was eluted off with acetonitrile. After drying the eluant, the samples were prepared for LC-MS/MS analysis by dissolving in methanol/0.01 M aqueous formic acid.

Residues of chlorantraniliprole, IN-ECD73 and IN-EQW78 were extracted from liquid processed crop fractions with acetonitrile. An aliquot of the extract was then cleaned-up using on an HLB SPE cartridge. After washing and drying the cartridge, chlorantraniliprole, IN-ECD73 and IN-EQW78 were eluted off the HLB cartridge with acetonitrile/ethyl acetate. An aliquot was taken to dryness and taken up in acetonitrile/0.01 M aqueous formic acid for LC-MS/MS analysis.

Residues of IN-F6L99 were extracted from liquid processed crop fractions as above. An aliquot of the extract was purified using an HLB SPE cartridge. After washing and drying the cartridge, IN-F6L99 was eluted off with acetonitrile. An aliquot of the eluant was taken to dryness and the sample was prepared for LC-MS/MS analysis in methanol/0.01 M aqueous formic acid.

In a modification by Bilas and Stry (2005 14314b supplement1), the extraction of chlorantraniliprole and the degradation products IN-EQW78, IN-ECD73 and IN-F6L99 in vegetable oil samples was described.

Chlorantraniliprole, IN-EQW78 and IN-ECD73. Samples of oil were diluted with hexane, cleaned-up on a silica Solid Phase Extraction (SPE) cartridge pre-conditioned with hexane. The cartridges were washed with hexane and eluted with acetonitrile. Formic acid was added to the acetonitrile phase and sample was loaded onto a strong anion exchange (SAX) SPE cartridge pre-conditioned with 98:2 acetonitrile/formic acid. The combined eluates were made up to volume and an aliquot reduced in volume before adding 0.01% formic acid aqueous solution and analysis by LC/MS/MS.

Due to its higher polarity, IN-F6L99 was extracted by a different procedure. Oil samples were diluted with hexane and partitioned several times against methanol/formic acid. An aliquot of the combined methanol extracts was reduced in volume by evaporation, partitioned several times with hexane, and the methanol layer made to volume with 0.01 mol/L aqueous formic acid. Analysis was by LC/MS/MS.

Quantification was by LC/MS/MS. Transitions monitored were: chlorantraniliprole 484 → 453, 484 → 286 (alternative), IN-EQW78 466 → 188, 466 → 76, 466 → 186, IN-ECD73 279 → 244, 279 → 209 (quantification transition), 244 → 209, IN-F6L99 204 → 172 (used for validation), 204 → 66 (used for most samples).

Table 41 Recovery validation of the LC-MS/MS multi-residue method 14314 for chlorantraniliprole and degradation products in processed plant commodities

Matrix	Fortification level (mg/kg)	Recovery range (%) (n = 5)	Mean recovery (%)	% RSD	
Chlorantraniliprole					
Ketchup	0.010 0.10	103-113 109-125	109 116	3.5 6.1	14314
Raisins	0.010 0.10	91-108 94-101 (n=4)	103 98	6.5 3.0	14314
Orange Peel	0.010 0.10	92-113 89-93	103 92	8.5 2.0	14314

Matrix	Fortification level (mg/kg)	Recovery range (%) (n = 5)	Mean recovery (%)	% RSD	
Grape Pomace	0.010 0.10	103-109 98-104	106 101	2.7 2.7	14314
Cooked Spinach	0.010 0.10	97-106 92-101	103 96	3.7 3.5	14314
Grape Juice	0.010 0.10	96-104 85-109	101 95	3.3 9.2	14314
Apple Juice	0.010 0.10	83-108 91-102	97 98	10.0 5.5	14314
Cotton seed oil	0.010 0.10	105-121 107-116	111 113	5.6 3.6	Bilas and Stry 2005
Vegetable oil	0.010 0.10	103-116 96-110	110 106	4.6 5.5	Bilas and Stry 2005
IN-EQW78					
Ketchup	0.010 0.10	80-121 84-101	106 87	8.8 7.1	14314
Raisins	0.010 0.10	87-108 95-103	99 98	7.7 3.2	14314
Orange Peel	0.010 0.10	81-102 94-101	91 97	9.5 3.0	14314
Grape Pomace	0.010 0.10	85-97 94-100	91 98	5.5 2.4	14314
Cooked Spinach	0.010 0.10	90-127 96-104	105 100	15.0 3.0	14314
Grape Juice	0.010 0.10	95-114 74-102	106 88	7.6 12.1	14314
Apple Juice	0.010 0.10	89-110 78-101	99 88	7.2 10.2	14314
Cotton seed oil	0.010 0.10	80-95 86-90	88 87	8.0 2.0	Bilas and Stry 2005
Vegetable oil	0.010 0.10	89-108 80-84	97 82	7.3 2.2	Bilas and Stry 2005
IN-ECD73					
Ketchup	0.010 0.10	95-101 80-89	97 86	4.9 6.5	14314
Raisins	0.010 0.10	92-99 89-98	95 92	3.0 4.0	14314
Orange Peel	0.010 0.10	94-107 88-93	101 90	5.8 1.8	14314
Grape Pomace	0.010 0.10	86-100 86-94	93 91	6.1 3.5	14314
Cooked Spinach	0.010 0.10	82-99 83-94	89 88	8.6 5.8	14314
Grape Juice	0.010 0.10	97-108 80-96	102 86	4.9 6.9	14314
Apple Juice	0.010 0.10	86-97 81-97	93 87	4.2 7.0	14314
Cotton seed oil	0.010 0.10	106-116 93-106	111 98	3.6 5.4	Bilas and Stry 2005

Matrix	Fortification level (mg/kg)	Recovery range (%) (n = 5)	Mean recovery (%)	% RSD	
Vegetable oil	0.010 0.10	87-126 91-100	112 95	14.5 4.6	Bilas and Stry 2005
IN-F6L99					
Ketchup	0.010 0.10	99-112 94-109	107 100	3.9 4.5	14314
Raisins	0.010 0.10	85-117 103-110	107 106	11.9 2.8	14314
Orange Peel	0.010 0.10	91-118 91-98	102 95	11.6 2.8	14314
Grape Pomace	0.010 0.10	97-112 98-102	102 100	5.8 1.7	14314
Cooked Spinach	0.010 0.10	90-104 87-100	97 93	5.4 6.5	14314
Grape Juice	0.010 0.10	94-123 83-104	103 92	11.5 10.7	14314
Apple Juice	0.010 0.10	98-112 82-99	107 91	5.3 8.2	14314
Cotton seed oil	0.010 0.10	82-109 83-87	92 85	14.3 1.9	Bilas and Stry 2005
Vegetable oil	0.010 0.10	90-95 90-97	92 94	2.3 3.5	Bilas and Stry 2005

Method 11376: A method used for the determination of residues of chlorantraniliprole in bovine tissues and milk in residue trials was reported by Bilas *et al.* (2005 11376). Acetonitrile/water was added to milk and cream and the samples partitioned several times against hexane. The hexane was partitioned against further volumes of acetonitrile/water and the combined acetonitrile/water diluted with acetonitrile. An aliquot was removed and diluted with acetonitrile.

Muscle or liver were allowed to stand with water for 20 minutes, before blending with acetonitrile and hexane. The homogenised sample was centrifuged and the supernatant decanted. The sample pellet was blended with further acetonitrile, centrifuged and the solvent decanted. The combined extracts were allowed to separate and the lower (water/acetonitrile) layer removed, diluted with additional acetonitrile and an aliquot diluted with water.

Fat, kidney or egg samples were allowed to stand with water for 20 minutes, homogenised with acetonitrile/hexane, before centrifuging and decanting the lower (water/acetonitrile) layer through filter paper. The hexane fraction was extracted with further acetonitrile and the acetonitrile/water extracts combined and made to volume with additional acetonitrile. An aliquot was removed and diluted with water.

The sample extracts were all cleaned up using a strong anion exchange (SAX) cartridge in series with an HLB cartridge. The cartridges were then dried and the analytes eluted from the HLB cartridge with acetonitrile, followed by 0.5% formic acid in ethyl acetate. The combined eluates were collected and if a lower (aqueous) layer formed, it was discarded. The ethyl acetate/acetonitrile eluate was evaporated to dryness then reconstituted in acetonitrile/water. Quantitation was by LC/MS/MS. Chlorantraniliprole: 484 → 453 and 484 → 286 (total ion count), IN-K9T00: 469 → 415, IN-HXH44: 482 → 386, IN-GAZ70: 451 → 414, IN-EQW78: 466 → 188 and 466 → 76 (total ion count).

Additional validation data were reported by Fraser *et al.* (2006 18100) and Rzepka (2006a 17123).

Table 42 Recovery validation of the LC-MS/MS multi-residue method 11376 for chlorantraniliprole and degradation products in animal commodities

Matrix	Fortification Level (mg/kg)	Range (n = 5)	Mean	%RSD	Reference
Chlorantraniliprole					
Whole milk	0.01	103-108	107	2.2	11376
	0.10	105-116	110	4.5	
Milk	0.01	73-106	89	17	18100
	0.10	90 -111	97	9.7	
Milk	0.01	90-140	125	17	17123
	0.10	104-112	109	3.1	
Skim milk	0.01	92-109	103	6.6	11376
	0.10	106-112	109	2.2	
Cream	0.01	102-118	109	5.8	11376
	0.10	97-115	108	6.3	
Cream	0.01	65-96	84	15	18100
	0.10	80-99	90	8.7	
Fat	0.01	96-110	103	6.6	11376
	0.10	100-118	107	7.5	
Fat	0.01	75-102	92	11.	18100
	0.10	92-107	98	7.6	
Fat	0.01	96-140	109	17	17123
	0.10	95-108	108	5.8	
Kidney	0.01	97-121	102	11.4	11376
	0.10	90-120	103	14.9	
Kidney	0.01	76-86	82	5.1	18100
	0.10	76-81	79	2.2	
Muscle	0.01	100-108	104	2.9	11376
	0.10	97-103	101	2.1	
Meat	0.01	75-92	79	9.5	18100
	0.10	105-115	110	3.2	
Meat	0.01	87-102	96	6.4	17123
	0.10	85-103	96	7.5	
Liver	0.01	90-109	100	7.4	11376
	0.10	98-102	101	1.6	
Liver	0.01	72-94	82	11.	18100
	0.10	86-103	97	6.7	
Liver	0.01	76-108	97	13	17123
	0.10	100-109	103	3.5	
Egg	0.01	97-106	100	3.7	11376
	0.10	95-102	99	2.9	
Egg	0.01	88-105	93	7.6	17123
	0.10	93-99	95	2.5	
IN-EQW78					
Milk	0.01	94-111	103	6.1	11376
	0.10	86-110	94	10.2	

Matrix	Fortification Level (mg/kg)	Range (n = 5)	Mean	%RSD	Reference
Milk	0.01	67-96	82	16	18100
	0.10	81-102	88	9.5	
Milk	0.01	57-103	91	21	17123
	0.10	93-110	102	6.7	
Skim milk	0.01	99-116	103	7.5	11376
	0.10	78-93	83	7.1	
Cream	0.01	98-107	104	3.6	11376
	0.10	89-95	91	2.4	
Cream	0.01	72-109	88	18	18100
	0.10	77-92	88	6.8	
Fat	0.01	87-105	99	6.8	11376
	0.10	84-91	87	3.5	
Fat	0.01	66-80	74	8.0	18100
	0.10	81-90	85	4.1	
Fat	0.01	80-99	90	8.4	17123
	0.10	69-96	78	14	
Kidney	0.01	85-114	98	12.4	11376
	0.10	84-101	91	8.5	
Kidney	0.01	64-90	77	13	18100
	0.10	70-79	74	5.1	
Muscle	0.01	97-110	103	6.0	11376
	0.10	83-95	89	5.4	
Meat	0.01	73-94	84	8.7	18100
	0.10	89-99	95	4.3	
Meat	0.01	88-105	93	7.3	17123
	0.10	80-90	94	5.1	
Liver	0.01	87-100	94	5.5	11376
	0.10	88-94	91	2.4	
Liver	0.01	69-83	76	6.8	18100
	0.10	77-88	83	5.4	
Liver	0.01	63-102	91	18	17123
	0.10	94-101	96	3.0	
Egg	0.01	73-84	78	5.3	11376
	0.10	80-91	86	5.7	
Egg	0.01	78-99	85	9.6	17123
	0.10	81-88	84	3.2	
IN-GAZ70					
Whole milk	0.01	90-112	101	8.1	11376
	0.10	105-118	112	4.4	
Milk	0.01	72-96	83	12.	18100
	0.10	87-110	94	10.	
Milk	0.01	92-234	130	21	17123
	0.10	100-140	119	12	
Skim milk	0.01	75-103	89	11.9	11376
	0.10	88-104	94	6.4	

Matrix	Fortification Level (mg/kg)	Range (n = 5)	Mean	%RSD	Reference
Cream	0.01	94-113	101	7.9	11376
	0.10	92-113	100	7.9	
Cream	0.01	69-110	92	20	18100
	0.10	82-105	98	9.7	
Fat	0.01	81-100	90	8	11376
	0.10	80-91	85	4.8	
Fat	0.01	82-114	98	12	18100
	0.10	77-92	87	7.4	
Fat	0.01	101-357	160	69 [a]	17123
	0.10	85-122	105	15	
Kidney	0.01	84-106	97	10.9	11376
	0.10	86-116	97	12.3	
Kidney	0.01	74-91	83	9.8	18100
	0.10	73-81	78	3.8	
Muscle	0.01	79-88	85	5.7	11376
	0.10	81-85	83	1.8	
Meat	0.01	76-80	77	2.2	18100
	0.10	92-105	99	4.7	
Meat	0.01	99-131	116	11	17123
	0.10	104-119	110	6.2	
Liver	0.01	70-86	82	8.3	11376
	0.10	85-96	89	5.5	
Liver	0.01	69-85	76	8.0	18100
	0.10	74-90	84	7.9	
Liver	0.01	83-118	103	12	17123
	0.10	115-123	118	2.8	
Egg	0.01	82-101	93	7.1	11376
	0.10	84-99	94	6.7	
Egg	0.01	72-91	81	11	17123
	0.10	75-85	80	5.5	
IN-HXH44					
Whole milk	0.01	99-110	105	4.4	11376
	0.10	100-124	112	8.1	
Milk	0.01	82-106	94	12	18100
	0.10	96-114	102	7.1	
Milk	0.01	78-226	140	63 [a]	17123
	0.10	72-91	83	9.4	
Skim milk	0.01	96-114	104	7.3	11376
	0.10	84-106	94	8.9	
Cream	0.01	100-123	108	8.9	11376
	0.10	97-109	105	4.3	
Cream	0.01	80-119	101	17.	18100
	0.10	85-110	102	10	
Fat	0.01	108-120	115	4.7	11376
	0.10	107-117	112	3.7	

Matrix	Fortification Level (mg/kg)	Range (n = 5)	Mean	%RSD	Reference
Fat	0.01	78-110	95	14	18100
	0.10	92-106	102	5.6	
Fat	0.01	57-106	77	23	17123
	0.10	68-86	79	11	
Kidney	0.01	92-107	99	5.6	11376
	0.10	88-105	96	6.8	
Kidney	0.01	95-108	101	6.2	18100
	0.10	90-101	95	4.8	
Muscle	0.01	105-119	114	5.1	11376
	0.10	101-123	109	8.8	
Meat	0.01	73-89	80	8.1	18100
	0.10	88-106	96	6.9	
Meat	0.01	71-85	77	6.9	17123
	0.10	60-80	72	13	
Liver	0.01	94-107	101	5.0	11376
	0.10	105-123	111	6.7	
Liver	0.01	75-99	89	10.	18100
	0.10	95-104	98	3.8	
Liver	0.01	62-80	72	10	17123
	0.10	74-93	81	10	
Egg	0.01	87-105	97	7.3	11376
	0.10	93-102	98	4.1	
Egg	0.01	61-87	73	14	17123
	0.10	72-81	76	4.7	
IN-K9T00					
Whole milk	0.01	85-118	98	13.5	11376
	0.10	105-131	119	8.4	
Milk	0.01	27-94	73	38 [a]	18100
	0.10	88-108	97	8.4	
Milk	0.01	59-87	70	16	17123
	0.10	71-86	82	7.8	
Skim milk	0.01	81-118	104	14.5	11376
	0.10	94-112	103	6.9	
Cream	0.01	85-109	94	9.9	11376
	0.10	74-111	90	16.2	
Cream	0.01	76-110	95	17	18100
	0.10	86-106	96	9.4	
Fat	0.01	95-103	98	3.7	11376
	0.10	104-118	110	4.9	
Fat	0.01	72-140	83	34 [a]	18100
	0.10	84-99	92	7.2	
Fat	0.01	60-105	82	21	17123
	0.10	61-95	76	19	
Kidney	0.01	89-109	100	10.7	11376
	0.10	88-103	94	6	

Matrix	Fortification Level (mg/kg)	Range (n = 5)	Mean	%RSD	Reference
Kidney	0.01	88-117	103	10	18100
	0.10	91-100	96	3.7	
Muscle	0.01	88-116	104	11.3	11376
	0.10	88-109	97	9.2	
Meat	0.01	69-81	74	6.1	18100
	0.10	90-104	95	5.5	
Meat	0.01	63-84	74	10	17123
	0.10	59-84	70	13	
Liver	0.01	90-108	102	7.9	11376
	0.10	93-109	103	8.2	
Liver	0.01	74-115	89	18	18100
	0.10	88-103	97	6.6	
Liver	0.01	63-85	76	11	17123
	0.10	64-78	70	7.9	
Egg	0.01	86-100	93	7.3	11376
	0.10	93-99	95	2.5	
Egg	0.01	63-84	70	12	17123
	0.10	66-74	71	4.1	

[a] Outside acceptable RSD, 20%. If extreme values are treated as outliers, the range of recovery values and the %RSD return to acceptable levels.

Method 19533: A routine method for the determination of residues of chlorantraniliprole in bovine tissues and milk has been reported (Stry 2006 19533). The extraction is as for method 11376. After the SPE cleanup, a derivatisation step was undertaken to convert the thermally labile chlorantraniliprole to IN-EQW78 through heating in aqueous base. The acetonitrile eluates from the HLB cartridge were reduced to dryness, reconstituted in a smaller volume of acetonitrile, reduced to dryness again, reconstituted in a still-smaller volume of acetonitrile, and diluted with an aqueous ammonia solution. These solutions were heated at 75 °C for 2 h, cooled, and partitioned between aqueous formic acid solution and ethyl acetate/hexane. The layers were allowed to separate, and the upper (ethyl acetate/hexane) layer transferred to a centrifuge tube, while the aqueous layer was extracted twice more with ethyl acetate/hexane and the extracts combined in the centrifuge tube. The extracts were reduced to dryness and reconstituted in acetonitrile for analysis by GC/ECD with confirmation by LC/MS/MS.

Table 43 Recovery validation of GC-ECD method 19533 for chlorantraniliprole in animal commodities

Sample	Fortification level (mg/kg)	Range (n = 5)	Mean recovery (%)	%RSD	Reference
Bovine muscle	0.01	76-83	79	3.9	19533
	0.1	63-81	75	10.1	19533
Bovine kidney	0.01	84-93	87	3.8	19533
	0.1	70-84	79	7.3	19533
Bovine fat	0.01	73-82	78	5.7	19533
	0.1	71-80	74	5.7	19533
Whole milk	0.01	81-91	85	5.1	19533
	0.1	77-84	80	3.2	19533

Sample	Fortification level (mg/kg)	Range (n = 5)	Mean recovery (%)	%RSD	Reference
Eggs	0.01	83-97	90	6.8	19533
	0.1	75-86	80	6.3	19533

Table 44 Recovery validation of the LC/MS/MS confirmatory method 19533 for chlorantraniliprole in animal commodities

Sample	Fortification level (mg/kg)	Range (n = 5)	Mean recovery	%RSD	Reference
Bovine muscle	0.01	76-83	79	3.9	19533
	0.1	63-81	75	10	19533
Bovine kidney	0.01	84-93	87	3.8	19533
	0.1	70-84	79	7.3	19533
Bovine fat	0.01	73-82	78	5.7	19533
	0.1	71-80	74	5.7	19533
Whole milk	0.01	81-91	85	5.1	19533
	0.1	70-84	79	7.3	19533
Eggs	0.01	83-97	90	6.8	19533
	0.1	75-86	80	6.3	19533

German official multi-residue method (DFG-S19) Rzepka (2006b 15025) described the validation of the German official multi-residue method (DFG-S19, L 00.00-34) for analysis of chlorantraniliprole and the metabolites IN-K9T00, IN-HXH44, IN-GAZ70, and IN-EQW78 in animal tissues (meat, fat, and liver), milk and eggs.

Milk, muscle, egg, and liver samples were extracted using module E1. Acetone was added to samples of milk, meat, egg and liver. Sufficient water was added to maintain a constant 2:1 ratio of acetone to water and the samples homogenised before partitioning against ethyl acetate/cyclohexane (1:1 v/v mixture, NaCl added to promote phase separation). An aliquot of the organic phase was filtered using cotton covered with and the filtrate evaporated to an aqueous residue. Ethyl acetate was added to dissolve the residue and a 1:1 (w/w) mixture of NaCl:Na_2SO_4 added together with cyclohexane. The salt mixture was allowed to settle.

Fat samples were extracted using module E6. Samples of fat were dissolved in 1:1 (v/v) ethyl acetate/cyclohexane.

All samples were then cleaned up by gel permeation chromatography (GPC). Aliquots of the extracts of milk, meat, liver or egg were cleaned up on Bio Beads S-X3 polystyrene gel using 1:1 (v/v) ethyl acetate/cyclohexane as the eluant. The eluate was concentrated and made up to volume with ethyl acetate.

An aliquot of cleaned up extracts were evaporated to dryness, reconstituted in methanol and 0.1% acetic acid added to each sample. Samples were analysed by LC/MS/MS.

Van Schaik (2006 18610) made minor modifications during a validation study.

Table 45 Validation data for the determination of chlorantraniliprole and metabolites IN-K9T00, IN-HXH44, IN-GAZ70, and IN-EQW78 in animal tissues, milk, and eggs via DFG S 19 with LC/MS-MS

Matrix	Fortification level (mg/kg)	Range (n = 5)	Mean	%RSD	Reference
Chlorantraniliprole					
Milk	0.01	99-103	101	1.8	15025
	0.10	94-114	103	7.5	
Milk	0.01	96-99	97	1.5	18610
	0.10	86-99	91	5.8	
Egg	0.01	93-116	105	8.8	15025
	0.10	96-108	100	4.7	
Egg	0.01	94-101	98	3.0	18610
	0.10	90-95	93	2.3	
Meat	0.01	103-115	107	4.2	15025
	0.10	91-110	103	7.8	
Meat	0.01	104-106	105	0.9	18610
	0.10	92-102	97	4.1	
Liver	0.01	97-118	110	8.5	15025
	0.10	84-113	95	12	
Liver	0.01	89-104	98	5.9	18610
	0.10	92-98	94	2.5	
Animal fat	0.01	101-107	104	2.5	15025
	0.10	95-106	101	4.2	
IN-EQW78					
Milk	0.01	92-108	100	5.9	15025
	0.10	89-112	102	8.3	
Milk	0.01	55-81	66 [a]	17.	18610
	0.10	55-65	61 [a]	7.2	
Egg	0.01	88-126	104	17	15025
	0.10	86-116	99	13	
Egg	0.01	56-87	71	16.	18610
	0.10	78-90	83	5.9	
Meat	0.01	97-122	105	10	15025
	0.10	89-108	101	8.1	
Meat	0.01	98-106	103	3.2	18610
	0.10	95-103	98	3.2	
Liver	0.01	90-114	104	8.9	15025
	0.10	76-102	87	14 (n = 4)	
Liver	0.01	66-102	90	16.	18610
	0.10	82-87	85	2.0	
Animal fat	0.01	69-110	91	19	15025
	0.10	68-129	96	26	
IN-GAZ70					
Milk	0.01	98-111	103	5.0	15025
	0.10	95-117	105	7.5	

Matrix	Fortification level (mg/kg)	Range (n = 5)	Mean	%RSD	Reference
Milk	0.01	73-78	76	3.2	18610
	0.10	64-77	70	8.7	
Egg	0.01	89-135	107	20	15025
	0.10	85-128	104	19	
Egg	0.01	65-94	77	16.	18610
	0.10	74-80	76	2.9	
Meat	0.01	100-121	108	7.8	15025
	0.10	92-110	103	8.2	
Meat	0.01	97-103	100	2.3	18610
	0.10	87-95	91	3.8	
Liver	0.01	87-129	111	16	15025
	0.10	61-110	86	24	
Liver	0.01	78-92	85	8.1	18610
	0.10	81-84	83	1.5	
Animal fat	0.01	79-117	97	16	15025
	0.10	77-122	98	20	
IN-HXH44					
Milk	0.01	96-104	99	3.4	15025
	0.10	93-114	103	7.4	
Milk	0.01	89-108	98	8.0	18610
	0.10	88-98	93	4.5	
Egg	0.01	90-110	100	9.5	15025
	0.10	90-113	98	9.5	
Egg	0.01	86-100	94	6.4	18610
	0.10	80-90	87	4.6	
Meat	0.01	99-108	103	3.8	15025
	0.10	86-108	100	8.7	
Meat	0.01	101-112	104	4.4	18610
	0.10	91-101	96	4.6	
Liver	0.01	96-110	104	6.1	15025
	0.10	87-114	97	11	
Liver	0.01	94-101	98	3.1	18610
	0.10	88-96	92	3.2	
Animal fat	0.01	98-106	102	3.5	15025
	0.10	94-105	101	4.2	
IN-K9T00					
Milk	0.01	94-100	97	2.3	15025
	0.10	91-111	101	7.0	
Milk	0.01	87-102	96	5.8	18610
	0.10	88-96	92	3.6	
Egg	0.01	58- 81	68 [a]	13	15025
	0.10	60-70	66 [a]	5.8	
Egg	0.01	79-92	86	6.1	18610
	0.10	74-85	79	5.7	

Matrix	Fortification level (mg/kg)	Range (n = 5)	Mean	%RSD	Reference
Meat	0.01	68-90	79	12	15025
	0.10	61-93	79	17	
Meat	0.01	122-130	130 [a]	2.7	18610
	0.10	87-96	93	3.8	
Liver	0.01	52-71	59 [a]	14	15025
	0.10	54-78	64 [a]	14	
Liver	0.01	96-103	99	2.8	18610
	0.10	86-93	89	3.6	
Animal fat	0.01	93-105	97	4.7	15025
	0.10	98-110	105	4.9	

[a] Outside acceptable recovery range 70–120%.

Extraction efficiency

Extraction efficiency for chlorantraniliprole was reported by Kidd and Davidson (2005 13260). Samples with incurred residues from metabolism studies were subject to analysis using method 0673 (same extraction is used for 13294 and 13295). Greater than 90% of the total radioactive residues were extracted using the proposed method.

Table 46 Extraction efficiency for method 0673

Sample	Profiling method	Residue method		
	Chlorantraniliprole (%TRR)	Extraction efficiency (%TRR)	Mean extraction efficiency (%TRR)	Chlorantraniliprole (%TRR)
Lettuce	86.5	95.9 91.0	93.5	95.3 83.3
Apple	100	101 112	107	100 109

Stability of residues in stored analytical samples

Chlorantraniliprole was stable in homogenized samples stored frozen for at least 24 months for apple, grape, tomato, lettuce, cauliflower, potato, wheat grain, wheat straw, alfalfa hay and cotton seed. Chlorantraniliprole and metabolites (IN-EQW78, INECDW73 and IN-F6L99) were stable for at least 12 months, the period of frozen storage studied for the processed commodities tomato ketchup, raisin, cotton seed meal, cotton seed oil, and apple juice. Residues of chlorantraniliprole and the metabolites IN-K9T00, IN-HXH44, IN-GAZ70 and IN-EQW78 were stable in bovine liver, kidney, muscle, fat and milk stored frozen for at least 12 months.

Storage stability of frozen fortified samples of plant matrices was studied by Grant (2006, 12985). Samples of apple, grape, tomato, leaf lettuce, cauliflower, potato, wheat grain, wheat straw, alfalfa hay, and cottonseed were ground and homogenized with dry ice. For chlorantraniliprole stability testing, 10 g of the test matrix were weighed into polypropylene bottles to provide 53 samples for each matrix. For each stability time point, one aliquot was retained as a control (no fortification), two aliquots were fortified at 0.10 mg/kg with the appropriate standard solution of chlorantraniliprole in acetonitrile or acetonitrile containing 0.01 M formic acid, and two aliquots were stored until analysis and then fortified at 0.10 mg/kg with the appropriate standard solution of chlorantraniliprole in acetonitrile or acetonitrile containing 0.01 M formic acid. After samples were fortified for storage, all the bottles were stored at about -20 °C until required for analysis. Samples were removed from storage and analysed after 0, 3, 6, 12, 18, and 24 months (± one week). At each interval, one control, two stored fortified samples, and two stored control samples fortified upon removal from storage were analysed.

Stored samples were analysed for chlorantraniliprole using procedures described in the analytical method based on DuPont-11374 with minor modifications.

Results are summarized in the following table:

Table 47 Stability of chlorantraniliprole in various raw agricultural commodities fortified at 0.10 mg/kg and stored at –20 °C

Storage interval (months)	Matrix	Residue remaining (mg/kg)	Concurrent recovery	Matrix	Residue remaining (mg/kg)	Concurrent recovery
0	Apple	-	106	Potato	-	109
3		0.080 0.081	80		0.090 0.090	83
6		0.101 0.098	71		0.105 0.100	77 [a]
12		0.090 0.089	88		0.090 0.082	84
18		0.094 0.094	96		0.095 0.091	92
24		0.088 0.092	93		0.109 0.101	106
0	Grape	-	104	Wheat grain	-	99
3		0.070 0.069	71		0.072 0.074	74
6		0.107 0.100	101		0.096 0.103	110
12		0.088 0.087	87		0.077 0.080	83
18		0.099 0.099	92		0.082 0.088	97
24		0.089 0.093	80		0.072 0.079	82
0	Tomato	-	100	Wheat straw	-	106
3		0.076 0.077	75		0.072 0.074	76
6		0.097 0.101	70		0.078 0.077	81
12		0.088 0.097	85		0.063 0.067	73
18		0.096 0.098	95		0.076 0.072	79
24		0.097 0.095	80		0.078 0.075	77
0	Leaf lettuce	-	107	Alfalfa hay	-	102
3		0.082 0.081	78		0.071 0.079	85
6		0.103 0.106	77 [a]		0.090 0.092	95
12		0.083 0.087	82		0.081 0.086	91
18		0.093 0.095	93		0.085 0.079	92
24		0.094 0.099	84		0.081 0.073	77
0	Cauliflower	-	106	Cottonseed	-	97
3		0.070 0.069	73		0.073 0.073	71
6		0.093 0.097	104		0.102 0.095	96
12		0.073 0.078	83		0.076 0.069	73
18		0.082 0.083	89		0.086 0.090	90
24		0.098 0.094	97		0.086 0.082	79

[a] Concurrent samples fortified at 0.01 mg/kg, rather than the target 0.10 mg/kg

The data indicate that the residues of chlorantraniliprole are stable at approximately -20 °C for at least 24 months in 10 representative crops, including water-, oil-, protein-, and starch-containing commodities as represented by fruits, a fruiting vegetable, a root crop, a non-oily grain, and an oilseed.

Additional information on the storage stability of processed crop fractions was reported by Cairns and Hunter (2006 13255). Samples of all commodities except cottonseed meal and oil were obtained at a local grocer in the UK. Cottonseed oil was obtained from a commercial source, and

cottonseed meal was supplied by DuPont. No sample pre-processing was required for apple juice, tomato ketchup or cottonseed oil. Cottonseed meal was received pre-processed and no further processing occurred. Raisins were ground and homogenized with dry ice using a food chopper. The control samples displayed no significant background levels of chlorantraniliprole, IN-ECD73, IN-EQW78, and IN-F6L99.

Sixteen 10 g aliquots of each solid test matrix were fortified for frozen storage stability, and a further 24 × 10-g aliquots were used for fresh recovery samples (concurrent) and controls. Thirty-two aliquots of each liquid or oil test matrix (5 g liquids; 16 × 5 g and 16 × 10 g oils) were fortified for frozen storage stability. A further forty-eight aliquots (5 g liquids; 24 × 5 g and 24 × 10g oils) were also weighed for fresh recovery samples and controls. All storage stability samples were fortified with a standard acetonitrile solution of chlorantraniliprole, IN-ECD73, IN-EQW78 and IN-F6L99. The nominal fortification level was 0.10 mg/kg for each analyte. All aliquots, including unfortified controls, were then stored at *ca* –20 °C (no range provided) until required for analysis. Samples were removed from storage and analysed after approximately 0, 3, 6, 9 and 12 months. Stability samples were analysed for chlorantraniliprole, IN-ECD73, IN-EQW78 and IN-F6L99 using procedures described in the LC-MS/MS analytical method based on DuPont-14314.

The following tables summarize the results of the storage stability:

Table 48 Stability of chlorantraniliprole and metabolites in various process commodities fortified at 0.10 mg/kg and stored at –20 °C

Matrix	Storage Interval (months)	Chlorantraniliprole		IN-EQW78		IN-ECDW73		IN-F6L99	
		Residue (mg/kg)	Concurrent recovery (%)	Residue (mg/kg)	Concurrent recovery (%)	Residue (mg/kg)	Concurrent recovery (%)	Residue (mg/kg)	Concurrent recovery (%)
Tomato ketchup	0	-	106	-	101	-	98	-	99
	3	0.108 0.107	108	0.095 0.095	99	0.075 0.081	85	0.093 0.090	88
	6	0.109 0.103	95	0.094 0.102	93	0.091 0.102	105	0.081 0.080	75
	9	0.105 0.096	94	0.104 0.099	98	0.093 0.081	94	0.112 0.110	106
	12	0.107 0.109	79	0.105 0.102	73	0.090 0.086	72	0.099 0.107	105
Raisin	0	-	91	-	101	-	97	-	98
	3	0.109 0.101	100	0.090 0.086	92	0.080 0.078	82	0.094 0.100	87
	6	0.096 0.094	95	0.095 0.092	96	0.101 0.100	100	0.080 0.084	75
	9	0.106 0.093	74	0.101 0.095	71	0.086 0.075	64	0.097 0.094	95
	12	0.107 0.095	79	0.093 0.093	78	0.089 0.087	74	0.124 0.114	109
Cotton seed meal	0	-	87	-	95	-	91	-	81
	3	0.109 0.086	96	0.099 0.076	84	0.081 0.073	81	0.094 0.085	82
	6	0.081 0.081	80	0.079 0.082	81	0.093 0.091	92	0.082 0.081	73
	9	0.098 0.099	93	0.099 0.098	95	0.087 0.080	94	0.094 0.087	82
	12	0.079 0.105	77	0.073 0.096	74	0.073 0.098	76	0.076 0.081	107

Matrix	Storage Interval (months)	Chlorantraniliprole		IN-EQW78		IN-ECDW73		IN-F6L99	
		Residue (mg/kg)	Concurrent recovery (%)	Residue (mg/kg)	Concurrent recovery (%)	Residue (mg/kg)	Concurrent recovery (%)	Residue (mg/kg)	Concurrent recovery (%)
Cotton seed oil	0	-	96	-	102	-	105	-	105
	3	0.094 0.093	101	0.091 0.081	106	0.084 0.079	90	0.104 0.105	102
	6	0.110 0.112	106	0.091 0.089	99	0.089 0.077	90	0.105 0.100	97
	9	0.108 0.107	103	0.098 0.105	102	0.078 0.086	92	0.099 0.097	98
	12	0.120 0.120	110	0.110 0.110	111	0.090 0.086	87	0.100 0.100	102
Apple juice	0	-	103	-	108	-	99	-	105
	3	0.098 0.097	95	0.104 0.108	104	0.095 0.099	98	0.100 0.099	93
	6	0.113 0.112	108	0.103 0.106	107	0.089 0.098	100	0.101 0.101	97
	9	0.107 0.113	110	0.099 0.102	103	0.069 0.088	81	0.106 0.103	95
	12	0.110 0.110	111	0.100 0.110	109	0.100 0.100	107	0.110 0.120	111

The storage stability study demonstrates that residues of chlorantraniliprole, IN-ECD73, IN-EQW78 and IN-F6L99 are stable for at least 12 months when stored in processed crop fractions including predominantly water-, oil-, protein-containing and dry processed fractions (raisins, ketchup, apple juice, cottonseed meal and cottonseed oil) at approximately –20 °C.

The freezer storage stability of cattle tissues and milk was reported by Fraser and Kinney (2007 17004). Control bovine liver, kidney, muscle and fat were homogenized with dry ice. The milk obtained was fresh and required no sample preparation. The control samples displayed no significant background levels of chlorantraniliprole or its metabolites. Seventy-seven aliquots of milk, liver, kidney, muscle and fat, each 2.5 g, were weighed into plastic centrifuge tubes. Thirty-three aliquots of each matrix were fortified with a standard acetonitrile solution containing chlorantraniliprole and its metabolites IN-HXH44, IN-K9T00, IN-GAZ70 and IN-EQW78. The fortification level was a nominal 0.10 mg/kg for each analyte. The remaining samples were not fortified and were stored with the fortified storage samples to be used as control samples and freshly fortified samples (concurrent) extracted with the storage stability samples. Milk and tissue samples were removed from storage and analysed after approximately 0, 1, 3, 6, 9 and 12 months. The samples were analysed by an LC-MS/MS method, DuPont-11376, validated as DuPont–18100.

Table 49 Stability of chlorantraniliprole residues in representative bovine tissues and milk samples fortified at 0.10 mg/kg following storage at -20 ± 10 °C

Commodity	Storage Interval (months)	Residue (mg/kg)			Concurrent recovery (%)
Milk	0	0.0896	0.0981	0.0876	94.9
	0.25 (7 days)	0.103	0.101	0.0995	105
	1	0.0879	0.0917	0.0731	84.6
	3	0.0966	0.0699	0.0802	86.4
	6	0.0998	0.104	0.0998	91.9
	9	0.0685	0.0893	0.0823	88.4

Commodity	Storage Interval (months)	Residue (mg/kg)			Concurrent recovery (%)
	12	0.0968	0.104	0.0975	101
Liver	0	0.0902	0.0813	0.0837	92.3
	1	0.0936	0.0974	0.0967	93.5
	3	0.0853	0.0840	0.0772	89.6
	6	0.0873	0.0870	0.0892	89.4
	9	0.107	0.0963	0.105	105
	12	0.103	0.0926	0.0968	110
Kidney	0	0.0866	0.0750	0.0747	79.2
	1	0.0902	0.102	0.106	97.0
	3	0.0922	0.0880	0.0849	93.2
	6	0.0762	0.0822	0.0773	86.1
	9	0.105	0.104	0.106	108
	12	0.0945	0.0907	0.0893	96.5
Muscle	0	0.0848	0.0859	0.0822	81.2
	1	0.0971	0.0921	0.0968	107
	3	0.0925	0.0935	0.0848	78.6
	6	0.0993	0.104	0.101	101
	9	0.0831	0.0801	0.0806	86.7
	12	0.0955	0.104	0.106	87.6
Fat	0	0.107	0.107	0.109	97
	1	0.0904	0.0883	0.0988	94.9
	3	0.0773	0.0842	0.0779	82.4
	6	0.0928	0.103	0.103	110
	9	0.0841	0.0769	0.0790	80.9
	12	0.116	0.108	0.104	98.9

Table 50 Stability of IN-K9T00 residues in representative bovine tissues and milk samples fortified at 0.10 mg/kg following storage at -20 ± 10 °C

Commodity	Storage Interval (months)	Residue (mg/kg)			Concurrent recovery (%)
Milk	0	0.0890	0.0968	0.0819	92.6
	0.25 (7 days)	0.0903	0.0842	0.0805	91.8
	1	0.0931	0.0963	0.0910	103
	3	0.0874	0.0447	0.0709	91.2
	6	0.0952	0.0969	0.0967	96.8
	9	0.0698	0.0838	0.0834	95.1
	12	0.0883	0.0866	0.0839	116
Liver	0	0.101	0.0753	0.0796	92.0
	1	0.0942	0.0900	0.0877	106
	3	0.0846	0.0773	0.0765	98.2
	6	0.0949	0.0872	0.0953	112
	9	0.0774	0.0845	0.0974	92.1
	12	0.0839	0.0334	0.0728	112

Commodity	Storage Interval (months)	Residue (mg/kg)			Concurrent recovery (%)
Kidney	0	0.109	0.0954	0.0833	102
	1	0.0733	0.0739	0.0799	88.1
	3	0.0901	0.0873	0.0815	112
	6	0.0909	0.0707	0.0656	77.1
	9	0.0828	0.0700	0.0809	101
	12	0.0579	0.0617	0.0702	87.3
Muscle	0	0.0743	0.0849	0.0859	78.4
	1	0.0652	0.0716	0.0715	84.3
	3	0.0941	0.0970	0.0977	91.5
	6	0.0656	0.0804	0.0713	75.8
	9	0.101	0.0974	0.102	96.5
	12	0.0750	0.0781	0.0758	85.5
Fat	0	0.0857	0.980	0.0859	93.1
	1	0.0668	0.0606	0.0724	75.6
	3	0.0755	0.0732	0.0811	91.8
	6	0.0773	0.0788	0.0814	95.2
	9	0.0823	0.0746	0.0760	88.7
	12	0.0907	0.0918	0.0920	97.8

Table 51 Stability of IN-HXH44 residues in representative bovine tissues and milk samples fortified at 0.10 mg/kg following storage at -20±10 °C

Commodity	Storage Interval (months)	Residue (mg/kg)			Concurrent recovery (%)
Milk	0	0.0916	0.0966	0.0956	99.4
	1 week	0.112	0.109	0.106	115
	1	0.104	0.0948	0.102	102
	3	0.0957	0.0721	0.0796	94.8
	6	0.109	0.102	0.101	93.9
	9	0.0693	0.0950	0.0833	92.2
	12	0.0978	0.0999	0.0948	99.5
Liver	0	0.107	0.101	0.0969	109
	1	0.0971	0.112	0.105	105
	3	0.0993	0.0951	0.0984	104
	6	0.0957	0.0769	0.0843	84.5
	9	0.107	0.102	0.113	107
	12	0.0872	0.0823	0.0851	108
Kidney	0	0.0919	0.0868	0.0843	90.6
	1	0.108	0.106	0.117	104
	3	0.109	0.106	0.103	115
	6	0.0769	0.0758	0.0805	79.2
	9	0.0913	0.0916	0.0934	98.1
	12	0.0723	0.0775	0.0788	102

Commodity	Storage Interval (months)	Residue (mg/kg)			Concurrent recovery (%)
Muscle	0	0.0870	0.0848	0.0856	83.2
	1	0.0836	0.0820	0.0917	93.3
	3	0.0995	0.105	0.0997	89.9
	6	0.0982	0.109	0.102	102
	9	0.119	0.0977	0.124	105
	12	0.0849	0.0879	0.0901	89.8
Fat	0	0.102	0.101	0.109	94.2
	1	0.0861	0.0874	0.090	83.6
	3	0.0973	0.0988	0.0907	96.8
	6	0.101	0.109	0.0871	104
	9	0.101	0.0841	0.0955	97.5
	12	0.101	0.0982	0.101	107

Table 52 Stability of IN-GAZ70 residues in representative bovine tissues and milk samples fortified at 0.10 mg/kg following storage at -20 ± 10 °C

Commodity	Storage Interval (months)	Residue (mg/kg)			Concurrent recovery (%)
Milk	0	0.0586	0.0433	0.0611	61.8 [a]
	1 week	0.0916	0.0891	0.0875	95.7
	1	0.0878	0.0858	0.0903	92.0
	3	0.0919	0.0683	0.0806	79.3
	6	0.0842	0.0889	0.0852	78.4
	9	0.0414	0.0877	0.0710	83.0
	12	0.0928	0.0940	0.0901	103
Liver	0	0.0773	0.0756	0.0701	81.2
	1	0.0827	0.0843	0.0775	85.8
	3	0.0689	0.0626	0.0615	73.7
	6	0.0924	0.0822	0.0874	98.3
	9	0.0820	0.0763	0.0754	89.4
	12	0.0829	0.0755	0.0746	95.9
Kidney	0	0.0922	0.0802	0.0773	86.2
	1	0.0749	0.0819	0.0874	81.4
	3	0.0824	0.0791	0.0709	86.3
	6	0.135	0.0639	0.0781	82.3
	9	0.0889	0.0904	0.0905	110
	12	0.0932	0.0932	0.0993	92.3
Muscle	0	0.0694	0.0690	0.0791	80.2
	1	0.0763	0.0776	0.0725	82.5
	3	0.0985	0.0953	0.0878	91.8
	6	0.0802	0.0730	0.0757	83.1
	9	0.104	0.0808	0.108	107
	12	0.0770	0.0752	0.0771	77.7

Commodity	Storage Interval (months)	Residue (mg/kg)			Concurrent recovery (%)
Fat	0	0.0900	0.0838	0.0974	86.8
	1	0.0741	0.0707	0.0746	75.0
	3	0.0904	0.0959	0.0907	93.9
	6	0.0936	0.0864	0.0916	97.3
	9	0.0836	0.0764	0.0786	92.6
	12	0.0753	0.0671	0.0660	75.4

[a] Method not performing. See 1 week results.

Table 53 Stability of IN-EQW78 residues in representative bovine tissues and milk samples fortified at 0.10 mg/kg following storage at -20 ± 10 °C

Commodity	Storage Interval (months)	Residue (mg/kg)			Concurrent recovery (%)
Milk	0	0.0604	0.0491	0.0613	60.6
	0.25 (7 days)	0.0981	0.0960	0.0934	99.9
	1	0.0937	0.0874	0.0915	97.4
	3	0.0945	0.0656	0.0783	82.5
	6	0.0895	0.0915	0.0844	74.1
	9	0.0442	0.0922	0.0883	82.4
	12	0.0859	0.0899	0.0865	89.9
Liver	0	0.0861	0.0798	0.0756	84.0
	1	0.0899	0.0945	0.0914	94.9
	3	0.0681	0.0675	0.0550	68.1
	6	0.0858	0.0874	0.0879	92.4
	9	0.0891	0.0850	0.0858	88.2
	12	0.0899	0.0859	0.0802	96.6
Kidney	0	0.0822	0.0704	0.0716	71.3
	1	0.0715	0.0868	0.0935	89.0
	3	0.0797	0.0709	0.0672	72.9
	6	0.167	0.0762	0.0750	78.5
	9	0.0864	0.0860	0.0890	93.5
	12	0.0937	0.0861	0.0953	101
Muscle	0	0.0753	0.0740	0.0701	71.7
	1	0.0632	0.0713	0.0687	76.5
	3	0.0916	0.0907	0.0846	76.4
	6	0.0918	0.0796	0.0831	82.7
	9	0.106	0.0829	0.104	92.7
	12	0.0863	0.0897	0.0906	85.0
Fat	0	0.0983	0.0857	0.0992	84.3
	1	0.0810	0.0771	0.0803	83.1
	3	0.0860	0.0898	0.0814	79.9
	6	0.0924	0.0925	0.0902	91.9
	9	0.0830	0.0771	0.0788	82.1
	12	0.0924	0.0847	0.0846	94.4

When stored at approximately–20 °C residues of chlorantraniliprole, IN-K9T00, IN-HXH44, IN-GAZ70 and IN-EQW78 are stable in milk, liver, kidney, muscle and fat for at least 12 months.

USE PATTERN

Chlorantraniliprole is an insecticide belonging to the anthranilic diamide class of chemistry. It is highly active against a broad range of lepidopteran larvae and certain other insects and is currently being developed for agricultural use on fruits, vegetables, and cotton.

Formulations containing chlorantraniliprole are registered for use on a wide variety of crops in Argentina, Australia, Canada, China, Indonesia, Pakistan, Ukraine and the USA. Registered uses include foliar spray on vegetables, pome and stone fruit, tree nuts and cotton.

Table 54 Registered uses of chlorantraniliprole

Crop	Country		Spray, L/ha	Spray conc, g ai/hL	Rate, g ai/ha	No.	Interval	PHI Days
Apple	Argentina	SC		4		1-2	7-14	14
Peach	Argentina	SC		5		1-2	7-14	7
Potato	Canada	SC	> 100 > 50✈		50-75/season max 225	1-4	5	14
Fruiting vegetable except cucurbits [a]	Canada	SC	> 100		50-75/season max 225	1-4	5	1
Brassica vegetables [b]	Canada	SC	> 100		50/season max 200 [c]	1-4	3	3
Leafy vegetables [d]	Canada	SC	> 100		50/season max 200	1-4	3	1
Pome fruit	Canada	WG	> 450		51-100/season max 226	1-3	10	14
Grapes	Canada	WG	> 450		51-100/season max 226	1-3	7	14
Stone fruit	Canada	WG	> 450		51-100/season max 226	1-3	7	10
Rice	China	SC			15-30	1-3	14	7
Apple	China	WG	3000		14-50	1-2	14	14
Cabbage	China	SC			22-40	1-3	7	1
Cabbage	Indonesia	SC	600		1.9-2.8	1-4	5-7	1
Eggplant	Indonesia	SC	800		2.5-3.75	1-4	5-7	1
Rice	Indonesia	SC	300		1.87-3.75	1-2	14	3
Bean, string	Indonesia	SC	800		2.5-3.75	1-4	5-7	1
Cotton	Pakistan	SC			25-40			28
Grape [g]	USA	WG	935-1870		49-111/season max 224	1-4	7	14
Pome fruit [e]	USA	WG	935-1870		62-111/season max 224	1-4	10	14
Stone fruit [f]	USA	WG	935-1870		74-111/season max 224	1-4	7	10
Cotton [g]	USA	WG	-		49-110/season max 224	-	5	21
Potato	USA	WG	-		49-74/season max 224	-	5	14
Brassica (cole) leafy vegetables [b, h]	USA	SC	-		50-73/season max 224	-	3	3

Crop	Country		Spray, L/ha	Spray conc, g ai/hL	Rate, g ai/ha	No.	Interval	PHI Days
Cucurbit vegetables [i]	USA	SC	-		29-100/season max 224	-	5 (10 drip)	1
Fruiting vegetables [a, j]	USA	SC	-		29-110/season max 224	-	5 (10 drip)	1
Leafy vegetables [d, ♠]	USA	SC	-		50-110/season max 224	-	3 (10 drip)	1

[a] Eggplant, ground cherry, pepino, pepper, tomatillo, tomato

[b] Broccoli, Chinese broccoli, broccoli raab, Brussels sprouts, cabbage, Chinese cabbage, Chinese mustard, cauliflower, cavalo broccoli, collards, kale, kohlrabi, mizuna, mustard greens, mustard spinach, rape greens

[c] For optimal control apply with a modified seed oil adjuvant

[d] Amaranth, arugula, cardoon, celery, Chinese celery, celtuce, chevril, chrysanthemum, corn salad, cress, dandelion, dock, endive, Florence fennel, lettuce, orach, parsley, purslane, radicchio, rhubarb, spinach, spinach vine, New Zealand spinach, Swiss chard

[e] Do not use an adjuvant with applications less than 60 days before harvest

[f] Cherries (sweet and tart), do not use with an adjuvant

[g] Do not use with an adjuvant

[h] For best performance use an effective adjuvant

[i] chayote, Chinese wax gourd, citron melon, cucumber, gherkin, edible gourd, (includes hyotan, cucuzza, hechima, Chinese okra), Momordica spp., muskmelon, pumpkin, summer squash, winter squash, watermelon.

Do not use adjuvant with cucurbits except cucumber, Chinese waxgourd, gherkin and Momordica spp.

[j] Do not use adjuvant with chili pepper or pimento

♠ Do not use adjuvant with leafy vegetables (non-brassica) except cardoon, celery, Chinese celery, celtuce, Florence fennel, lettuce, radicchio, rhubarb and Swiss chard

✈ Aerial application

RESIDUES RESULTING FROM SUPERVISED TRIALS

The Meeting received information on supervised field trials for chlorantraniliprole on the following crops:

Commodity	Group	Table No.
Apples	Pome fruit	Tables 55–59
Pears		
Apricots	Stone fruit	Tables 60–62
Cherry		
Peach		
Plum		
Grapes		Tables 63–66
Brussels sprouts	Brassica vegetables	Tables 67–68
Broccoli		
Cabbage		
Cauliflower		
Cucumber	Fruiting vegetables, cucurbits	Tables 69–71
Melons		
Summer squash		
Zucchini		
Tomato	Fruiting vegetables, other than cucurbits	Tables 72–75
Peppers		Tables 76–81
Lettuce	Leafy vegetables	Tables 82–86
Spinach		
Mustard greens		
Potatoes	Root and tuber vegetables	Tables 87–88
Celery	Stalk and stem vegetables	Table 89

Commodity	Group	Table No.
Cotton	Oilseeds	Tables 90–91
Almonds	Tree nuts	Table 92
Pecans		
Cotton gin-trash	Animal feed	Tables 93–94
Almond hulls		Table 95

When residues were not detected they are shown as below the LOQ (e.g., < 0.01 mg/kg). Application rates and spray concentrations have generally been rounded to two significant figures. Residues greater than the LOD but the less than the LOQ have generally been rounded to one significant figure. Limited rounding has been used to facilitate the best use of the data in exploring statistical methods of estimation of maximum residue levels. Residue values from the trials conducted according to maximum GAP have been used for the estimation of maximum residue levels. Those results included in the evaluation are double underlined.

Conditions of the supervised residue trials were generally well reported in detailed field reports. Most trial designs used non-replicated plots. Most field reports provided data on the sprayers used, plot size, field sample size and sampling date.

Pome fruit

Australian trials on apples and pears 2004/5 and 2005/2006 (pressurised backpack or mounted misters/sprayers; 4 trees per treatment). All samples were analysed within four months of harvest. Recovery values for fresh control fortifications run concurrently with treated samples from the 2004–5 and 2005–6 season Australia trials were within 83.7–111.9% (n = 12) for pome fruit. New Zealand trials employed knapsack sprayers or hand-gun sprayers, 2–4 trees/plot. At each of the three sites, small plots (2–4 trees) of mature (10–25 years) apple trees were treated with a 350 WG formulation of chlorantraniliprole using knapsack sprayers or a handgun. At Havelock North, triplicate plots were treated, while single plots were treated at Mangateretere and Riwaka. All samples were received at the laboratory within 5 months of harvest. Analyses were completed within 8 months of the first samples being collected. Argentinean trials employed 3 trees per plot with application by hand-gun sprayers. Samples were analysed within 5 months of harvest and were kept at –20 °C between harvest and analysis. Fruit samples were analysed using a GC method with electron capture detection (13291).

Table 55 Residues of chlorantraniliprole in apples and pears from Australian, Argentina and New Zealand trials

Country	FL	Application					Portion analysed	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS [a]				
Apple										
Mooroopna, Victoria, Australia, 2005, Golden Delicious	SC	3 (14,13)	46.8 46.8 45.0	3 3 3	1559 1559 1500	Fruit 4-6 cm Fruit 4-7 cm Some small fruit	fruit	14* 0 7 14 21 28	0.14 0.34 0.26 0.18 0.15 0.07	DPX-E2Y45-Apples
	SC	3 (14, 13)	101 108 93.5	6 6 6	1676 1794 1559	Fruit 4-6 cm Fruit 4-7 cm Some small fruit	fruit	14* 0 7 14 21 28	0.30 0.64 0.48 0.48 0.29 0.23	

Country	FL	Application					Portion analysed	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS [a]				
Spreyton, Tasmania, Australia, 2005, Fuji	SC	3 (14, 14)	58.0 51.8 56.9	3 3 3	1933 1727 1896	Fruit 6-7 cm Fruit 7 cm Fruit 8 cm	fruit	14* 0 7 14 21 28	0.13 0.33 0.25 0.16 0.16 0.06	DPX-E2Y45-Apples
	SC	3 (14, 14)	108 114 114	6 6 6	1796 1896 1896	Fruit 6-7 cm Fruit 7 cm Fruit 8 cm	fruit	14* 0 7 14 21 28	0.37 0.86 0.58 0.38 0.36 0.17	
Pozieres, Queensland, Australia, 2005, Red Delicious	WG	3 (14, 14)	36.0 34.6 30.4	3 3 3	1201 1152 1014	Fruit development	fruit	14	0.09	19725 Agral 0.025%
	WG	3 (14, 14)	72.1 69.1 60.8	6 6 6	1201 1152 1014	Fruit development	fruit	14	0.25	Agral 0.025%
	SC	3 (14, 14)	36.0 34.6 30.4	3 3 3	1201 1152 1014	Fruit development	fruit	14	0.14	none
Spreyton, Tasmania, Australia, 2005, Fuji	WG	3 (14, 14)	69.3 61.4 58.1	3 3 3	2310 2045 1935	Fruit 8-9 cm Fruit 8-10 cm Fruit mature	fruit	0 7 14 21 28	0.29 0.25 0.10 0.10 0.06	19725 Agral 0.025%
	WG	3 (14, 14)	160 109 131	6 6 6	2661 1821 2187	Fruit 8-9 cm Fruit 8-10 cm Fruit mature	fruit	0 7 14 21 28	0.56 0.40 0.17 0.15 0.11	Agral 0.025%
	SC	3 (14, 14)	76.5 60.9 58.8	3 3 3	2549 2031 1959	Fruit 8-9 cm Fruit 8-10 cm Fruit mature	fruit	0 7 14 21 28	0.32 0.26 0.12 0.11 0.07	none
Batlow, New South Wales, Australia, 2005, Braeburn	WG	3 (14, 14)	79.1 81.2 79.1	3 3 3	2638 2708 2638	Fruit colour Fruit colouring Near maturity	fruit	14* 0 7 14 21 28	0.07 0.22 0.18 0.11 0.07 0.06	19725 Agral 0.025%
	WG	3 (14, 14)	158 162 158	6 6 6	2638 2708 2638	Fruit colour Fruit colouring Near maturity	fruit	14* 0 7 14 21 28	0.18 0.49 0.33 0.26 0.21 0.13	Agral 0.025%

Country	FL	Application					Portion analysed	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS [a]				
Havelock, Hawkes Bay, NZ, 2006, Gala	WG	2 (25)	46.6 46.6	3.15 3.15	1480 1480	Fruit 0.3-1.0 cm Fruit 2.0-3.0 cm	Fruit 12 g Fruit 146 g Fruit 170 g Fruit 210 g	0 56 70 84	0.12, 0.13 < 0.01 < 0.01 < 0.01 (3)	19904 Contact 0.025%
	WG	3 (21, 21)	48.4 48.0 48.1	3.15 3.15 3.15	1537 1523 1528	Fruit 4.0-5.0 cm Fruit 4.5-5.5 cm Fruit 5.0-6.0 cm	Fruit 170 g Fruit 170 g Fruit 181 g Fruit 210 g Fruit 185 g Fruit 210 g	21[b] 0 7 14 21 28	0.04 0.19 0.15 0.08, 0.10, 0.09 0.11 0.07	Contact 0.025%
Havelock, Hawkes Bay, NZ, 2006, Gala	WG	2 (25)	93.2 93.2	6.3 6.3	1480 1480	Fruit 0.3-1.0 cm Fruit 2.0-3.0 cm	Fruit 12 g Fruit 146 g Fruit 170 g Fruit 210 g	0 56 70 84	0.27, 0.24 0.01 0.02 < 0.01 (3)	19904 Contact 0.025%
	WG	3 (21, 21)	96.8 95.9 96.3	6.3 6.3 6.3	1537 1523 1528	Fruit 4.0-5.0 cm Fruit 4.5-5.5 cm Fruit 5.0-6.0 cm	Fruit 170 g Fruit 170 g Fruit 181 g Fruit 210 g Fruit 185 g Fruit 210 g	21[b] 0 7 14 21 28	0.09 0.12 0.08, 0.08 0.14, 0.11, 0.07 0.12 0.10	Contact 0.025%
Mangateretere, Hawkes Bay, NZ, 2006, Fuji	WG	2 (25)	47.9 48.0	3.15 3.15	1520 1525	Fruit 0.3-1.0 cm Fruit 2.0-3.0 cm	Fruit 15 g Fruit 99 g Fruit 130 g Fruit 139 g Fruit 182 g	0 56 70 84 112	0.17 0.01 < 0.01 < 0.01 < 0.01	19904 Contact 0.025%
	WG	3 (21, 21)	48.8 49.3 49.0	3.15 3.15 3.15	1550 1565 1556	Fruit 4.0-5.5 cm Fruit 4.0-7.0 cm Fruit 6.0-7.5 cm	Fruit 164 g Fruit 164 g Fruit 177 g Fruit 182 g Fruit 190 g Fruit 207 g	21[b] 0 7 14 21 28	0.03 0.08 0.05, 0.05 0.06 0.05 0.03	Contact 0.025%
Mangateretere, Hawkes Bay, NZ, 2006, Fuji	WG	2 (25)	95.8 96.1	6.3 6.3	1520 1525	Fruit 0.3-1.0 cm Fruit 2.0-3.0 cm	Fruit 15 g Fruit 99 g Fruit 130 g Fruit 139 g Fruit 182 g	0 56 70 84 112	0.25 0.02 0.01 < 0.01 < 0.01	19904 Contact 0.025%
	WG	3 (21, 21)	97.7 98.9 98.3	6.3 6.3 6.3	1550 1565 1556	Fruit 4.0-5.5 cm Fruit 4.0-7.0 cm Fruit 6.0-7.5 cm	Fruit 164 g Fruit 164 g Fruit 177 g Fruit 182 g Fruit 190 g Fruit 207 g	21[b] 0 7 14 21 28	0.10 0.19, 0.15 0.19 0.13 0.13 0.10	Contact 0.025%

Country	FL	Application					Portion analysed	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS [a]				
Riwaka, Nelson, NZ, 2006, Royal Gala	WG	2 (25)	35.3 37.8	3.15 3.15	1120 1200	Fruit 1.5-2.0 cm Fruit 2.0-3.0 cm	Fruit 15 g Fruit 141 g Fruit 160 g	0 70 84	0.07 < 0.01 < 0.01	19904 Contact 0.025%
	WG	3 (22, 19)	37.2 38.3 34.0	3.15 3.15 3.15	1180 1215 1080	Fruit 4.0-5.0 cm Fruit 6.0-7.5 cm	Fruit 131 g Fruit 130 g Fruit 135 g Fruit 153 g Fruit 171 g Fruit 171 g	19[b] 0 7 14 21 28	0.03 0.07 0.02 0.02 0.04 0.03	Contact 0.025%
Riwaka, Nelson, NZ, 2006, Royal Gala	WG	2 (25)	70.6 75.6	6.3 6.3	1120 1200	Fruit 1.5-2.0 cm Fruit 2.0-3.0 cm	Fruit 15 g Fruit 141 g Fruit 160 g	0 70 84	0.12 < 0.01 0.01	19904 Contact 0.025%
	WG	3 (22, 19)	74.3 76.5 68.6	6.3 6.3 6.3	1180 1215 1080	Fruit 4.0-5.0 cm Fruit 5.5-6.0 cm Fruit 6.0-7.5 cm	Fruit 131 g Fruit 130 g Fruit 135 g Fruit 153 g Fruit 171 g Fruit 171 g	19[b] 0 7 14 21 28	0.05 0.12 0.07, 0.08 0.06 0.04 0.06	Contact 0.025%
Tunuyán, Mendoza, Argentina, 2006, Royal Gala	SC	2 (14)	120 120	4 4	3000 3000	Fruit development	Mature	14	< 0.06	20737
	SC	2 (14)	240 240	8 8	3000 3000	Fruit development	Mature	14	< 0.06	
General Roca, Río Negro, Argentina, 2006, Red Delicious	SC	2 (14)	120 120	4 4	3000 3000	Fruit development	Mature	14	0.120	20737
	SC	2 (14)	240 240	8 8	3000 3000	Fruit development	Mature	14	0.240	
Guerrico, Río Negro, Argentina, 2006, Red Delicious	SC	2 (14)	120 120	4 4	3000 3000	Fruit development	Mature	14	0.193	20737
	SC	2 (14)	240 240	8 8	3000 3000	Fruit development	Mature	14	0.591	
Pear										
Mooroopna, Victoria, Australia, 2005, Packham	WG	3 (14, 14)	41.7 41.7 41.7	3 3 3	1389 1389 1389	Fruit ¾ size Almost full Almost ripe	fruit	14* 0 7 14 21 28	0.11 0.25 0.18 0.14 0.12 0.07	19725 Agral 0.025%
	WG	3 (14, 14)	83.3 83.3 83.3	6 6 6	1389 1389 1389	Fruit ¾ size Almost full Almost ripe	fruit	14* 0 7 14 21 28	0.18 0.60 0.31 0.32 0.15 0.14	Agral 0.025%

Country	FL	Application					Portion analysed	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS [a]				
Paracombe, South Australia, Australia, 2005,	WG	3 (15, 14)	50.1 50.1 50.1	3 3 3	1670 1670 1670	Fruit sizing Fruit sized Mature fruit	fruit	14	0.11	19725 Agral 0.025%
Packham	WG	3 (15, 14)	100 100 100	6 6 6	1670 1670 1670	Fruit sizing Fruit sized Mature fruit	fruit	14	0.20	Agral 0.025%
	SC	3 (15, 14)	50.1 50.1 50.1	3 3 3	1670 1670 1670	Fruit sizing Fruit sized Mature fruit	fruit	14	0.17	None
Karagullen, West Australia, Australia, 2005,	WG	3 (14, 14)	71.4 79.7 71.4	3 3 3	2380 2658 2380	Fruit 5.0×5.5 cm Fruit 5.0×6.0 cm Fruit 7.5×7.5 cm	fruit	14	0.18	19725 Agral 0.025%
Packham	WG	3 (14, 14)	143 159 143	6 6 6	2380 2658 2380	Fruit 5.0×5.5 cm Fruit 5.0×6.0 cm Fruit 7.5×7.5 cm	fruit	14	0.32	Agral 0.025%
	SC	3 (14, 14)	71.4 79.7 71.4	3 3 3	2380 2658 2380	Fruit 5.0×5.5 cm Fruit 5.0×6.0 cm Fruit 7.5×7.5 cm	fruit	14	0.19	none

*Sampled before the 3rd application, 14 days after the 2nd spray.

[a] GS = Growth stage

[b] Sampled before the 3rd application, 19 or 21 days after the 2nd spray

NOTE: NZ trials recovery at 0.01 mg/kg was 102% and at 0.1 mg/kg 90%. Results were corrected for mean recovery of the 0.1 mg/kg spike samples.

NOTE: all NZ trials + 25mL/hL Contact® surfactant containing 600 g/L non-ionic polysaccharide ethoxylates

Trials on pome fruit were conducted in Europe over several seasons. In 2004 decline trials were conducted in Spain and France. At each trial site, two treated plots were established and chlorantraniliprole 20SC or 35WG formulations were applied twice by foliar application at targeted application rates of 35–47.5 g ai/ha (3.5–7 g ai/hL) for the 1st application and 42–52.5 g ai/ha (3.5–7 g ai/hL) for the 2nd. No surfactants or adjuvants were added to the applications.

Additional trials were conducted in 2005 and 2006. Twelve residue trials were conducted; one each in Germany, Belgium, Hungary, Poland, and Italy; two each in Greece and north France, and three in south France. In addition, six reverse decline trials were conducted, two in The Netherlands, one in north France, two in Spain, and one in south France. Ten trials were conducted in apples and eight trials were conducted in pears. At fourteen of eighteen trial locations, 20SC formulation was applied twice as a foliar broadcast spray at a target application rate of 4.0 g ai/hL (40–60 g ai/ha). At the remaining four trial locations (2 each in 2005 and 2006), low spray volumes (400 L/ha) were employed at application rates of 40.0 g ai/ha (10.0 g ai/hL) in 2005 and 50.0 g ai/ha (12.5 g ai/hL) in 2006 for each of the two applications. The two applications of 20SC were made at 14-day (±1) intervals with the last application occurring approximately 0–90 days before the predicted commercial

harvest. No surfactants or adjuvants were added to the applications. All samples were analysed within 6 months of sampling. The mean percent recovery for chlorantraniliprole from control apple fruit specimens fortified at 0.01–0.10 mg/kg was 96 ± 9 (2004). For European trials on apples and pears (2005), concurrent recoveries from control samples fortified at 0.010–0.30 mg/kg with chlorantraniliprole ranged from 74–110% (mean = 92 ± 11%). For 2006 trials on apples and pears, concurrent recoveries from control apple specimens fortified at 0.010–0.20 mg/kg of chlorantraniliprole ranged from 75–114% with a mean recovery of 93 ± 12% (n = 10), concurrent recoveries from control pear specimens fortified at 0.010–0.20 mg/kg of chlorantraniliprole ranged from 86–98% with a mean recovery of 94 ± 4% (n = 6).

Table 56 Residues for chlorantraniliprole in apples from European trials

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS				
El Palau, d'Anglesola, Catalunya, Spain, 2004, Golden Delicious	SC	2 (14)	36.8	3.67	1002	77	81	(-1 h)	0.049	14141
			44.4	3.67	1207		81	(+3h)	0.077	
							85	7	0.097	
							85	14	0.077	
							87	21	0.048	
							87	28	0.062	
							89	35	0.061	
	WG	2 (14)	35.8	3.57	1002	77	81	(-1 h)	0.024	
			43.0	3.56	1204		81	(+3h)	0.059	
							85	7	0.043	
							85	14	0.055	
							87	21	0.035	
							87	28	0.026	
							89	35	0.025	
St Sylvestre Cappel, Norde-Pas-de-Calais, France, 2004, Jonagold	SC	2 (15)	49.4	7.35	672	81	77	(-1 h)	0.033	14141
			54.6	7.34	745		77	(+2h)	0.086	
							78-81	7	0.11	
							83-85	14	0.091	
							87	21	0.081	
							89	28	0.11	
							89-90	35	0.072	
	WG	2 (15)	49.1	7.18	683	81	77	(-1 h)	0.068	
			52.7	7.15	737		77	(+2h)	0.14	
							78-81	7	0.068	
							83-85	14	0.071	
							87	21	0.048	
							89	28	0.076	
							89-90	35	0.070	
Kalkar, North Rhine-Westphalia, Germany, 2005, Elstar	SC	2 (14)	41.8	4.01	1043	81	85	(+2h)	0.036	16577
			40.1	4.01	1000	85	87	14	0.054	
Groesbeek, Limburg, Netherlands, 2005, Elstar	SC	2 (14)	41.7	4.01	1040	71	87	90	< 0.01	16577
			40.4	4.01	1009	72				
		2 (15)	41.8	4.01	1044	73-74	87	56	0.016	
			39.8	4.01	993	75				
		2 (14)	39.9	4.01	996	77	87	28	0.052	
			41.0	4.01	1022	81				

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS				
		2 (15)	40.4 40.4	4.01 4.01	1007 1007	81 83	87	14	0.069	
		2 (14)	40.6 42.0	4.01 4.01	1013 1049	83-85 83-85	87	7	0.066	
		2 (14)	40.8 39.9	4.01 4.01	1018 996	85 87	87	(-1 h) (+2 h)	0.034 0.036	
Herlies, Norde-Pas-de-Calais, France, 2005, Jonagold	SC	2 (13)	41.5 40.3	10.0 10.0	415 402	72 72	87	84	< 0.01	16577
		2 (14)	36.6 39.2	10.0 10.0	396 392	74 75	87	56	0.020	
		2 (15)	40.5 40.5	10.0 10.0	405 404	76 77	87	27	0.027	
		2 (13)	40.7 41.1	10.0 10.0	407 410	77 81-85	87	14	0.068	
		2 (13)	40.9 39.4	10.0 10.0	409 393	79-81 85	87	7	0.090	
		2 (15)	40.5 39.6	10.0 10.0	404 395	81-85 87	87	(-1 h) (+2 h)	0.051 0.081	
Almenar, Cataluña, , Spain 2005, Golden Delicious	SC	2 (14)	60.3 60.1	4.0 4.0	1504 1497	72 74	88	90	0.028	16577
		2 (14)	60.3 60.7	4.0 4.0	1506 1512	76 76	88	56	0.054	
		2 (14)	60.3 60.3	4.0 4.0	1505 1506	76 80	88	28	0.066	
		2 (14)	59.7 60.5	4.0 4.0	1489 1509	80 83-84	88	14	0.051	
		2 (14)	60.3 60.7	4.0 4.0	1504 1515	81-82 82	88	7	0.13	
		2 (14)	60.7 60.3	4.0 4.0	1514 1501	83-84 88	88	(-1 h) (+2 h)	0.062 0.12	
Platani, Pella, Central Macedonia, Greece, 2005, Granny Smith	SC	2 (14)	60.5 61.3	4.0 4.0	1509 1528	78 84	84 89	(+2 h) 14	0.073 0.024	16577
Châteamneuf sur Isíre, Rhône-Alpes, France, 2005, Pink Lady	SC	2 (14)	40.7 41.3	10 10	408 413	81 85	85 87	(+2 h) 14	0.088 0.039	16577
Molembaix, Hainant, Belgium, 2006, Golden	SC	2 (14)	40.5 40.3	4.0 4.0	1012 1007	79-81 85	85 87	(+1 h) 14	0.036 0.13	18752
Casalnoceto, Piemonte, Italy, 2006, Golden	SC	2 (14)	60.1 60.3	4.0 4.0	1500 1505	79-81 81	81 89	(+1 h) 14	0.059 0.048	18752

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS				
Osieczek, Pniewy, Mazovian Region, Poland, 2006, Idared	SC	2 (14)	43.8 44.2	4.0 4.0	1097 1104	78 78	78 89	(+2 h) 14	< 0.01 0.010	18752
Platani, Pella, Central Macedonia, Greece, 2006, Granny Smith	SC	2 (14)	62.1 60.4	4.0 4.0	1550 1539	81-85 87-88	87-88 89	(+2 h) 15	0.048 0.022	18752

Table 57 Residues for chlorantraniliprole in pears from European trials

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS				
Groesbeek, Limburg, Netherlands, 2005, Doyenné du Cornice	SC	2 (14)	40.9 39.2	4.01 4.01	1021 979	71 72	87	90	0.014	16577
		2 (15)	39.5 39.6	4.01 4.01	985 988	73-74 75	87	56	0.021	
		2 (14)	40.7 38.7	4.01 4.01	1017 967	77 81	87	28	0.064	
		2 (15)	39.4 38.6	4.01 4.01	983 965	81 83	87	14	0.082	
		2 (14)	39.1 40.2	4.01 4.01	975 1004	83-85 83-85	87	7	0.085	
		2 (14)	39.7 38.7	4.01 4.01	992 967	85 87	87	(-1 h) (+2 h)	0.036 0.087	
Godewaersvelde, Norde-Pas-de-Calais, France, 2005, Conference	SC	2 (14)	39.2 39.4	4.01 4.00	979 985	77 79-81	79-81 87-89	(+2 h) 14	0.073 0.046	16577
El Palau d'Aglesola, Cataluña, Spain, 2005, Blanca de Aranjuez	SC	2 (14)	60.5 60.1	4.0 4.0	1510 1499	71-72 72	86	90	0.016	16577
		2 (13)	60.5 60.1	4.0 4.0	1511 1496	74-75 75	86	57	0.024	
		2 (14)	60.5 60.7	4.0 4.0	1509 1514	77-78 78-79	86	28	< 0.01	
		2 (14)	60.5 60.5	4.0 4.0	1509 1507	78-79 80	86	14	0.034	
		2 (15)	60.3 60.5	4.0 4.0	1503 1507	80 82-83	86	7	0.064	
		2 (14)	60.3 59.7	4.0 4.0	1501 1488	80 86	86	(-1 h) (+2 h)	0.022 0.033	
La Motte-Servolex, Rhône-Alpes, France, 2005,	SC	2 (14)	39.2 40.9	4.0 4.0	980 1024	72 73	87	90	< 0.01	16577
		2 (15)	40.7 40.5	4.0 4.0	1018 1013	74 75	87	55	0.014	

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS				
Duchesse de bered		2 (15)	40.1 40.5	4.0 4.0	1003 1013	77 81	87	27	0.030	
		2 (13)	41.1 36.6	4.0 4.0	1027 991	81 85	87	14	0.053	
		2 (14)	40.7 39.7	4.0 4.0	1015 993	85 85	87	7	0.047	
		2 (14)	40.7 40.3	4.0 4.0	1018 1008	85 87	87	(-1 h) (+2 h)	0.022 0.028	
Herlies , Nord Pas de Calais, France, 2006, Doyenne du Cornice	SC	2 (13)	50.9 49.7	12.51 12.48	407 398	81-85 87	87 89	(+1 h) 13	0.18 0.090	18752
Loire/Rhône, Rhône-Alpes, France, 2006, Beurré Hardy	SC	2 (14)	39.9 40.1	4.0 4.0	994 1002	77 77	77 87	(+1 h) 14	0.19 0.096	18752
Dommartin, Rhône-Alpes, France, 2006, Louise Bonne	SC	2 (14)	50.3 49.5	12.48 12.49	403 396	77 81	81 85-87	(+3 h) 14	0.084 0.024	18752
Mor, Fejér County, Hungary, 2006, Bosc Kobak	SC	2 (14)	52.0 50.1	4.0 3.9	1297 1278	83 89	89 90	(+2 h) 14	0.085 < 0.01	18752

Trials on apples and pears were conducted in 2005 at 28 locations in Canada and the United States (16576). Chlorantraniliprole (35WG formulation) was applied twice as foliar spray at the rate of 112 g ai/ha/application to apples and pears when the crop was at growth stage BBCH 75 to 89. No surfactants or adjuvants were added to the applications. The applications were made at 10-day intervals using airblast or foliar broadcast sprayers. All samples were analysed within 151 days of sampling using an LC/MS/MS method (13294). Concurrent recoveries from control apple fruit fortified at 0.010–0.50 mg/kg of chlorantraniliprole ranged from 81–113% (mean = 93 ± 11%, $n = 10$). Concurrent recoveries from control pear fruit fortified at 0.010–0.10 mg/kg of chlorantraniliprole ranged from 73–98% (mean = 90 ± 8.8%, $n = 6$).

Table 58 Residues for chlorantraniliprole in apples from Canadian and USA trials

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS				
North Rose, NY, USA, 2005 Idared	WG	2 (11)	112 112	12 12	935 934	85 85	89	14	0.022	16576
Orefield, PA, USA 2005 Jonamac	WG	2 (10)	114 113	12 12	935 935	79 81	87	14	0.056	16576
Hereford, PA, USA 2005 Starkrimson Red Delicious	WG	2 (10)	114 112	12 12	935 935	78 79	87	14	0.11	16576

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS				
Berwick, NS, Canada 2005 McIntosh	WG	2 (10)	111 111	12 12	908 944		88	10	0.074	16576
Lula, GA, USA 2005 Arkansas Black	WG	2 (10)	113 109	12 11	962 976	83-84 83-84	87	15	0.073	16576
Hart, MI, USA 2005 Empire	WG	2 (10)	111 111	12 12	898 898	81 85	87-89	14	0.038	16576
Paynesville, MN, USA 2005 Haralson	WG	2 (10)	113 111	12 12	959 959	81 85	mature	14	0.010	16576
St. George, ON, Canada 2005 Red Delicious	WG	2 (10)	110 113	12 12	920 935	76 77-78	85-87	15	0.072	16576
St. Paul-D'Abbotsford, QC, Canada 2005 Paula Red	WG	2 (10)	106 111	11 11	942 974	75 77	88	14	0.012	16576
St. Paul-D'Abbotsford, QC, Canada 2005 Spartan	WG	2 (11)	114 112	12 12	953 953	78 83	89	15	0.030	16576
Perry, UT, USA 2005 Gala	WG	2 (11)	112 112	11 11	997 1002	81 85	87	14	0.088	16576
Sanger, CA, USA 2005 Lady	WG	2 (10)	111 112	12 12	946 969	85 87	89	14	0.045	16576
Ephrata, WA, USA 2005 Braeburn	WG	2 (10)	112 112	12 12	934 930	81 83	89	14	0.093	16576
Parkdale, OR, USA 2005 Jonagold	WG	2 (11)	111 112	13 12	873 906	81 85	mature	14	0.061	16576
Wamic, OR, USA 2005 Gala	WG	2 (10)	114 118	12 13	935 935	79 81	89	14	0.23	16576
Payette, ID, USA 2005 Law Rome	WG	2 (11)	111 113	12 12	935 935	79 85	89	14	0.078	16576
Ephrata, WA, USA 2005 Red Delicious	WG	2 (10)	112 112	12 12	937 937	76 79	79 79 80 85 89 89	-0 0 7 14 21 28	0.068 0.13 0.10 0.088 0.066 0.067	16576

Table 59 Residues for chlorantraniliprole in pears from Canadian and USA trials

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS				
North Rose, NY, USA 2005 Bartlett	WG	2 (10)	112 112	12 12	935 934	81 85	87	14	0.026	16576
Berwick, NS, Canada 2005 Clapps	WG	2 (10)	110 111	12 11	942 967		88	10	0.070	16576
St. George, ON, Canada 2005 Bartlett	WG	2 (10)	118 113	13 12	884 935	76 77-78	87-89	13	0.059	16576
St. George, ON, Canada 2005 Bosc	WG	2 (10)	118 113	13 12	890 918	76 77-78	87-89	13	0.085	16576
Elginfield, ON, Canada 2005 Bosch	WG	2 (10)	115 112	12 12	947 947	77 79	87	14	0.10	16576
Parlier, CA, USA 2005 Honsui Asian	WG	2 (11)	112 113	12 12	930 936	87 89	89	14	0.016	16576
Madera, CA, USA 2005 Asian	WG	2 (10)	113 115	12 12	935 935	81 85	87	10	0.054	16576
Ephrata, WA, USA 2005 Concord	WG	2 (10)	112 112	12 12	940 946	76 78	89	14	0.12	16576
Parkdale, OR, USA 2005 Cascade	WG	2 (11)	112 112	12 12	946 964	81 85	mature	14	0.13	16576
Hood River, OR, USA 2005 Starcrimsen	WG	2 (10)	112 112	12 12	945 958	78 79	87	13	0.033	16576
Fruitland, ID, USA 2005 Bartlett	WG	2 (10)	112 112	12 12	935 935	77 78	87	14	0.070	16576

Stone Fruit

Residue trials on plums and cherries were conducted in 2005 at 19 locations in Canada and the United States. A 35WG formulation was applied twice as a foliar broadcast spray at the rate of 112 g ai/ha/application to plum, sweet cherry, and sour cherry when the crop was at growth stage BBCH 75 to 87. No adjuvant was added to the spray mixtures for 14 of the trials. At three plum trials and two cherry trials, three treatments were tested - one treatment without adjuvant, one treatment with Hasten™ modified vegetable oil at a target rate of 0.25% v/v, and one treatment with Induce® non-ionic surfactant at a rate of 0.125% v/v. The applications of chlorantraniliprole were made at 7-day intervals with the last application occurring approximately 10 days before normal harvest.

For peaches, field trials were conducted in 2005 at 17 locations in Canada and the United States. At each trial, two foliar applications of 35WG formulation were made at 112 g ai/ha at 7 day intervals with the last application approximately 10 days before commercial harvest. No adjuvant was added to the spray mixtures for 14 of the trials. At three trials, three treatments were tested - one treatment without adjuvant, one treatment with Hasten™ (modified vegetable oil) at a rate of 0.25% v/v, and one treatment with Induce® non-ionic surfactant at a rate of 0.125% v/v. All samples were

analysed within 129 days of sampling (plum/cherry). The samples were analysed within 146 days from harvest (peach). Concurrent recoveries from control cherry samples fortified at 0.010–1.5 mg/kg of chlorantraniliprole were 78–109% with an average of 92 ± 9.8% (n = 9). Concurrent recoveries from control plum samples fortified at 0.010–0.10 mg/kg of chlorantraniliprole were 77–103% with an average of 90 ± 9.1% (n = 13). Recoveries of peach samples fortified with chlorantraniliprole at 0.01 mg/kg ranged from 74 to 108%, with an average recovery of 91 ± 12% (n = 8). The average recovery of samples fortified at 0.01 to 5.0 mg/kg was 86 ± 9% (n = 33).

Table 60 Residues for chlorantraniliprole in stone fruit from Canadian and USA trials

Country	Application						Sample GS	PHI (days)	Residue (mg/kg)	Reference
	FL	No (interval)	g ai/ha	g ai/hL	L/ha	Application GS				
Plum										
Grand Pre', NS, Canada 2005 Italian Prune	WG	2 (7)	112 112	14.8 14.2	758 786	81 85	87	10	0.026	16569
Beamsville, ON, Canada 2005, Vanette	WG	2 (7)	105 112	12.2 14.2	861 786	75 85	89	10	0.017	16569
	+	2 (7)	108 111	12.3 14.3	879 776			10	0.049	
	++	2 (7)	118 112	12.3 13.8	963 814			10	0.076	
St. Catharines, ON, Canada 2005 Italian prune	WG	2 (7)	110 112	13.7 13.8	804 814	75-76 77	89	10	0.067	16569
Conklin, MI, USA 2005 Stanley	WG	2 (7)	112 112	16.9 16.2	664 692	81 85	87	10	0.066	16569
Marengo, IL, USA 2005 Stanley	WG	2 (7)	112 108	13.4 14.8	833 730	81 85	87	10	< 0.01	16569
Porterville, CA, USA 2005 Angelino's	WG	2 (7)	112 112	17.1 17.1	655 655	84 85	89	10	0.015	16569
Dinuba, CA, USA 2005 Black Amber	WG	2 (7)	112 112	15.8 15.8	711 711	85 87	89	10	< 0.01	16569
Orange Cove, CA, USA 2005 Angelino's	WG	2 (7)	112 112	15.0 14.8	748 758	84 85	89	10	< 0.01	16569
Reedley, CA, USA 2005 King Midas Yellow	WG	2 (7)	112 112	20.6 19.0	543 589	81 83	88	10	< 0.01	16569
Royal City, WA, USA 2005 Pluot	WG	2 (7)	112 112	12.1 12.1	917 926	81 85	85 85 85 89 89 89	-0 0 5 10 14 21	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	16569

Country	Application						Sample GS	PHI (days)	Residue (mg/kg)	Reference
	FL	No (interval)	g ai/ha	g ai/hL	L/ha	Application GS				
	+[a]	2 (7)	112 111	12.2 12.1	917 917	81 85	89	10	0.011	
	++	2 (7)	112 111	12.2 12.1	917 917	81 85	89	10	0.011	
Dallas, OR, USA 2005	WG	2 (6)	112 112	11.6 11.6	963 963	81-85 85	89	10	< 0.01	16569
Moyer	+	2 (6)	112 112	11.6 11.6	963 963	81-85 85	89	10	0.022	
	++	2 (6)	112 112	11.6 11.6	963 963	81-85 85	89	10	0.029	
Cherry										
Conklin, MI, USA 2005 Napoleon	WG	2 (6)	111 112	13.6 14.4	814 776	78 85	87	10	0.26	16569
Plainview, CA, USA 2005 Tulare	WG	2 (7)	112 112	16.2 16.4	692 683	83 84	87	10	0.11	16569
Ephrata, WA, USA 2005 Bing	WG	2 (7)	112 112	15.8 15.8	711 711	81 85	89	10	0.10	16569
	+[b]	2 (7)	112 112	15.8 15.8	711 711			10	0.15	
	++	2 (7)	112 112	15.8 15.8	711 711			10	0.19	
Caldwell, ID, USA 2005 Skenna	WG	2 (7)	110 112	12.0 12.0	917 935	81 85	89	10	0.056	16569
Lyons, NY, USA 2005	WG	2 (7)	112 112	14.8 14.8	758 758	81 85	87	10	0.36	16569
Montmorency	+	2 (7)	112 112	14.8 15.0	758 748			10	0.48	
	++	2 (7)	112 112	14.8 15.0	758 748			10	0.57	
Conklin, MI, USA 2005 Montmorency	WG	2 (7)	112 112	16.9 16.9	664 664	78 85	87	10	0.21	16569
Marengo, IL, USA 2005 North Star	WG	2 (6)	112 112	14.4 12.6	776 889	81 85	87-89	9	0.18	16569
Perry, UT, USA 2005 Montmorency	WG	2 (6)	112 112	14.4 15.8	776 711	83 85	87	10	0.45	16569
Peach										
Sodus NY, USA 2005 Harcrest	WG	2 (7)	116 114	12.0 12.0	964 949			11	0.090	09389
Bridgeton NJ USA 2005 Suncrest	WG	2 (6)	117 113	12.3 12.6	953 897			10	0.247	09389

Country	Application						Sample GS	PHI (days)	Residue (mg/kg)	Reference
	FL	No (interval)	g ai/ha	g ai/hL	L/ha	Application GS				
Bridgeton NJ USA 2005 Dixie Red	WG	2 (7)	110 111	11.9 11.9	924 936			11	0.105	09389
Fennville MI USA 2005 Elberta	WG	2 (7)	113 111	12.0 11.8	940 945			10	0.144	09389
Jackson Springs NC USA 2005 Contender	WG	2 (6)	112 112	10.8 10.8	1041 1034			1 3 8 11 15	0.318 0.246 0.289 0.255 0.172	09389
Jackson Springs NC USE 2005 Emery	WG	2 (6)	114 115	11.0 11.0	1035 1048			11	0.309	09389
Troy TN USA 2005 Red Skin	WG	2 (6)	112 111	20.1 20.1	559 551			9	0.072	09389
Fredricksburg TX USA 2005 Gold Prince	WG	2 (6)	112 112	21.8 21.8	514 515			9	0.125	09389
Parlier CA USA 2005 O'Henry	WG	2 (6)	113 114	6.3 6.3	1792 1790			10	0.132	09389
Davis CA USA 2005 Dr Davis Cling	WG	2 (7)	112 113	13.2 13.1	852 859			11	0.183	09389
Parlier CA USA 2005 Flavour Crest	WG	2 (7)	116 116	17.8 17.5	652 663			1 3 8 10 14	0.158 0.101 0.074 0.118 0.114	09389
Madera CA USA 2005 Angelos	WG	2 (7)	113 113	9.6 9.6	1176 1174			10	0.165	09389
Summerland BC 2005 Harbrite	WG	2 (6)	111 112	9.8 9.8	1135 1140	81 85		10	0.064	09389
	+	2 (6)	113 112	9.8 9.8	1154 1151	81 85		10	0.106	
	++	2 (6)	112 110	9.8 9.8	1144 1128	81 85		10	0.114	
Jordan Station ON 2005 Baby Gold 5	WG	2 (7)	111 114	11.1 11.1	1000 1018			10	0.092	09389
Jordan Station ON 2005 Harrow Diamond	WG	2 (7)	115 116	11.2 11.2	1023 1038			10	0.101	09389
Jordan Station ON 2005	WG	2 (7)	114 116	11.2 11.2	1023 1040			10	0.083	09389
Harrow Beauty	+	2 (7)	114 115	11.2 11.2	1026 1034			10	0.089	

Country	Application						Sample GS	PHI (days)	Residue (mg/kg)	Reference
	FL	No (interval)	g ai/ha	g ai/hL	L/ha	Application GS				
	++	2 (7)	115 115	11.2 11.2	1029 1033			10	0.132	
Jordan Station ON 2005 Loring	WG	2 (7)	116 116	11.2 11.2	1033 1037			10	0.122	09389
	+	2 (7)	115 115	11.2 11.2	1026 1029			10	0.142	
	++	2 (7)	116 116	11.2 11.2	1037 1035			10	0.101	

+ = Hasten ® modified vegetable oil at 0.25%-1% v/v

++ = Induce ® Non-ionic Low Foam Wetter/Spreader @ 0.125% v/v

+[a] = + Hasten ® modified vegetable oil at 1% v/v for the 1st spray and 0.25% v/v for the 2nd

+[b] = + Hasten ® modified vegetable oil at 1% v/v

*pips removed, data on flesh

Trials on stone fruit were conducted in the EU in 2004, 2005 and 2006. In 2004 decline trials were conducted at two locations in Italy. An SC formulation was applied twice by foliar application to peaches at target application rates of 47.5 g ai/ha and 52.5 g ai/ha. The last application occurred 13–14 days before the commercial harvest date. No surfactants or adjuvants were added to the applications. In 2005, two magnitude of residue studies on peaches were conducted; one in Spain and one in Greece. One reverse decline peach trial was conducted in Spain. Two reverse decline apricot trials were conducted, one in Italy and one in France (south). At the trial locations an SC formulation was applied as a foliar broadcast spray. Trials in 2006 were conducted at one location in Spain, one location in Greece, two locations in southern France and one location in Italy. Two normal and three reverse decline trials were conducted in peaches and apricots. At all trial locations, SC formulation was applied as a foliar broadcast spray with a target application rate of 60 g ai/ha (4.0 g ai/hL). All samples were analysed within 7 months of sampling.

For the 2004 trials, concurrent recoveries from control specimens fortified at 0.010, 0.10 and 0.50 mg/kg of chlorantraniliprole ranged from 72–98% (mean = 84 ± 9.9%). For the 2005 trials, the mean percent recovery for chlorantraniliprole from 7 control specimens fortified at 0.010 mg/kg was 87 ± 6.2%. The mean percent recovery for chlorantraniliprole from 7 control specimens fortified at 0.10 mg/kg was 92 ± 9.0%. The mean percent recovery for chlorantraniliprole from 2 control specimens fortified at 0.50 mg/kg was 92%. In 2006 concurrent recoveries from control peach specimens fortified at 0.010 mg/kg and 0.10 mg/kg of chlorantraniliprole ranged from 67–109% (mean = 86 ± 13%). Concurrent recoveries from control apricot specimens fortified at 0.010, 0.10 and 0.50 mg/kg of chlorantraniliprole ranged from 88–117% (mean = 102 ± 10%).

Table 61 Residues for chlorantraniliprole in peaches and apricots from European trials

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS				
Peach										
Emilia-Romagna, Crespellano, Italy 2004, Gugcielmina	SC	2 (15)	50.0 55.1	3.68 3.68	1361 1498	78-79 81	81 81 81-85 85-87 87 89	(-1 h) 0 (+2 h) 7 14 21 28	0.040 0.16 0.059 0.044 0.040 0.034	14144

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS				
Emilia-Romagna, Monticelli d'Ongina, Italy 2004, Royal Glory	SC	2 (15)	49.6 55.1	3.66 3.67	1354 1500	73-75 75	75 75 85 88 89 89	(-1 h) 0 (+2 h) 6 13 20 27	< 0.01 0.033 0.014 0.015 0.023 < 0.01	14144
Tocina, Andalucia, Spain 2005 Spring Crest	SC	2 (9)	60.1 61.1	4.0 4.0	1498 1525	75 81-85	81-85 89	(+2 h) 14	0.045 0.029	16568
Imathia, Central Macedonia, Greece 2005 Andros	SC	2 (11)	58.5 60.7	4.0 4.0	1460 1514	79 84	84 89	(+2 h) 14	0.040 0.045	16568
Almenar, Catalunya, Spain 2005 Ryansun	SC	2 (9)	60.3 60.7	4.0 4.0	1507 1517	73-74 74-75	88	72	0.031	16568
		2 (10)	60.3 60.3	4.0 4.0	1506 1503	75 75	88	57	0.014	
		2 (10)	59.9 60.5	4.0 4.0	1497 1512	77 78	88	29	0.014	
		2 (11)	59.9 51.1	4.0 4.0	1496 1502	78 82	88	14	0.019	
		2 (10)	57.0 60.3	4.0 4.0	1421 1507	81 84	88	7	0.029	
		2 (10)	60.3 60.3	4.0 4.0	1506 1505	82-83 84	88	(-1 h) (+3 h)	0.028 0.075	
Alcarras, Lleida, Spain 2006 Andros	SC	2 (10)	60.1 60.1	4.0 4.0	1500 1502	73 75	86-88	55	0.028	18749
		2 (10	58.8 59.5	4.0 4.0	1467 1482	75 75	86-88	27	0.027	
		2 (10)	60.5 60.3	4.0 4.0	1511 1505	76 80	86-88	13	0.036	
		2 (10)	60.1 60.1	4.0 4.0	1499 1500	78 83	86-88	6	0.093	
		2 (9)	60.1 62.8	4.0 4.0	1501 1567	82 86-88	86-88	(-1 h) (+2 h)	0.040 0.065	
Volpedo, Piemonte, Italy 2006 Roberta	SC	2 (11)	50.1 60.3	3.3 4.0	1500 1505	73-75 77	85-87	14	0.021	18749
Thurins, Rhône-Alpes, France 2006 Sanguine Magnard	SC	2 (9)	60.3 60.1	12 12	502 501	75 75	87	57	0.011	18749
		2 (10)	61.4 60.5	12 12	512 504	75 75	87	28	0.032	
		2 (9)	60.3 60.3	12 12	502 503	77 77-81	87	14	0.030	

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS				
		2 (9)	60.5 61.2	12 10	504 611	77 85	87	7	0.033	
		2 (10)	60.5 60.7	10 10	606 607	81 87	87	(-1 h) (+2 h)	0.032 0.049	
Apricot										
Volpedo, Piemonte, Italy 2005 Bergeron	SC	2 (10)	39.6 40.5	4.0 4.0	998 1012	72 75	89	70	0.024	16568
		2 (10)	40.1 40.5	4.0 4.0	1000 1008	75 75	89	56	0.032	
		2 (11)	39.6 40.9	4.0 4.0	992 1022	76 78	89	27	0.037	
		2 (10)	40.3 37.1	4.0 4.0	1006 926	78-79 81	89	14	0.052	
		2 (10)	39.4 40.3	4.0 4.0	982 1006	79 85	89	7	0.091	
		2 (10)	40.3 40.1	4.0 4.0	1005 1002	85 89	89	(-1 h) (+1 h)	0.034 0.081	
Saint Marcel les Valence, Rhône-Alpes, France 2005 Tardive de Vain	SC	2 (10)	39.9 41.3	8.0 8.0	498 516	71 73	87	70	0.028	16568
		2 (10)	39.6 40.7	8.0 8.0	496 508	73 73-75	87	56	0.041	
		2 (9)	40.1 39.9	8.0 8.0	502 499	75 75	87	28	0.050	
		2 (10)	40.7 39.9	8.0 8.0	509 499	77 81	87	14	0.12	
		2 (10)	40.5 40.9	8.0 8.0	506 512	81 85	87	7	0.14	
		2 (10)	39.9 41.1	8.0 8.0	497 514	81 87	87	(-1 h) (+1 h)	0.063 0.18	
St Marcel Les Valence, Rhône-Alpes, France 2006 Tardive di Tain	SC	2 (12)	59.5 60.7	6.0 6.0	993 1014	72 73	87	54	0.064	18749
		2 (11)	60.7 59.5	6.0 6.0	1011 992	73-75 75	87	28	0.095	
		2 (11)	59.9 60.3	6.0 6.0	1000 1004	75 81	87	14	0.11	
		2 (10)	60.3 60.9	6.0 6.0	1005 1015	81 85	87	7	0.14	
		2 (10)	60.7 60.5	6.0 6.0	1012 1008	85 87	87	(-1 h) (+2 h)	0.12 0.20	
Pella, Central Macedonia, Greece 2006 Bebecou	SC	2 (11)	59.4 60.2	4.0 4.0	1482 1502	77-79 78-79	89	14	0.12	18749

Two Argentinean trials on peaches conducted in the 2005–6 season were reported. At each site, two trials were conducted, one at the proposed GAP rate and a second at 2× the proposed GAP rate. An SC formulation was applied two times by foliar application at application rates of 5 and 10 g ai/hL (all applications in 1800 L/ha for ground rates of 90 and 180 g ai/ha). No surfactants or adjuvants were added to the applications. The applications were made at 14-day (± 1) intervals with the last application occurring approximately 14 days before the predicted commercial harvest.

Trials on peaches conducted in the 2005–6 season were also available from Australia. A WG formulation was applied four times as a foliar broadcast spray at application concentrations of 3.15 and 6.3 g ai/hL and with retreatment intervals of 14–16 days, the last application occurring approximately 14 days before normal commercial harvest. Three treated plots were established, two at a 1× rate but with differing adjuvants (modified seed oil or non-ionic surfactant), and a third at a 2× rate with non-ionic surfactant. Recovery values for untreated control samples fortified with 0.010 and 1.0 mg/kg chlorantraniliprole run concurrently with treated samples in all the trials were within 82.4–89% (n = 4). Analysis was within 6 months of sample collection.

Table 62 Residues for chlorantraniliprole in peaches from Argentinean and Australian trials

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS				
Tupungato, Mendoza, Argentina 2006 Red Haven	SC	2 (14)	90 90	5.0 5.0	1800 1800	Fruit developing	Mature	14	< 0.05	20738
		2 (14)	180 180	10 10	1800 1800	Fruit developing	Mature	14	0.183	
Tupungato, Mendoza, Argentina 2006 O'Henry	SC	2 (14)	90 90	5.0 5.0	1800 1800	Fruit developing	Mature	14	< 0.05	20738
		2 (14)	180 180	10 10	1800 1800	Fruit developing	Mature	14	0.129	
Undera, Victoria, Australia 2006 Tatura 204	WG +	4 (14 14 16)	41.5 41.5 33.6 28.4	3.15 3.15 3.15 3.15	1317 1317 1066 902	Small fruit Fruit 4 cm Fruit 5 cm Fruit green/yellow	Mature	14	0.18	20921
	++	4 (14 14 16)	41.5 41.5 33.6 28.4	3.15 3.15 3.15 3.15	1317 1317 1066 902	Small fruit Fruit 4 cm Fruit 5 cm Fruit green/yellow	Mature	14	0.25	
	++	4 (14 14 16)	83 83 67.2 56.8	6.3 6.3 6.3 6.3	1317 1317 1066 902	Small fruit Fruit 4 cm Fruit 5 cm Fruit green/yellow	Mature	14	0.67	

+ = Hasten ®, a modified seed oil used at ca. 2 l/ha

++ = Agral ®, a non-ionic surfactant used at 0.025% v/v

Berries and small fruit

North American trials on grapes were conducted in 2005 at 17 locations in Canada and the United States. At each trial, two foliar applications of WG formulation were made at approximately 112 g ai/ha. No adjuvant was added to the spray mixtures for 14 of the trials. At three trials, three treatments were tested - one treatment without adjuvant, one treatment with Hasten™ modified vegetable oil at a rate of 0.25% v/v, and one treatment with Induce® non-ionic surfactant at a rate of

0.125% v/v. Applications were made at 7-day intervals with the last application occurring approximately 14 days before normal harvest. The samples were analysed within 306 days from harvest. Analytical recoveries for samples fortified at 0.01 to 0.51 mg/kg ranged from 71 to 106%.

Table 63 Residues for chlorantraniliprole in grapes from Canadian and USA trials

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS				
Dundee, NY 2005 Concord	WG	2 (7)	113 112	12.0 12.0	941 935	85 85		14	0.083	09388
Bridgeton, NJ 2005 Concord	WG	2 (7)	119 116	22.7 22.7	524 509			1 2 7 13 23	0.040 0.036 0.039 0.013 0.015	09388
Parlier, CA 2005 Thompson seedless	WG	2 (7)	113 113	9.7 9.7	1160 1167			14	0.042	09388
Parlier, CA 2005 Thompson seedless	WG	2 (7)	113 114	9.8 10.5	1150 1090			14	0.093	09388
Fresno, CA 2005 Thompson seedless	WG	2 (7)	113 115	9.6 9.6	1175 1204			14	0.175	09388
Madera, CA 2005 Merlot	WG	2 (7)	115 113	12.0 12.0	959 940			14	0.335	09388
Ukiah, CA 2005 Chardonnay	WG	2 (7)	113 114	16.1 16.0	704 711			14	0.257	09388
Ukiah, CA 2005 Zinfandel	WG	2 (7)	115 110	16.1 16.1	715 681			14	0.522	09388
Davis, CA 2005 French Columbard	WG	2 (6)	112 112	11.4 11.4	985 983			1 4 7 15 20	0.429 0.296 0.335 0.248 0.320	09388
Davis, CA 2005 Thompson seedless	WG	2 (6)	110 111	13.7 13.7	806 814			15	0.477	09388
Prosser, WA 2005 Lemberger	WG	2 (6)	112 112	9.6 9.9	1167 1123			15	0.119	09388
Monroe, OR 2005 Chardonnay	WG	2 (7)	114 114	12.0 12.0	952 948			13	0.199	09388
Summerland, BC 2005	WG	2 (7)	111 109	10.9 10.8	1022 1004	83-85 83-85		15	0.189	09388
Chancellor	+	2 (7)	109 108	10.9 10.8	1004 995	83-85 83-85		15	0.371	

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS				
	++	2 (7)	109 108	10.9 10.8	1001 995	83-85 83-85		15	0.461	
Jordan Station, ON 2005 Vidal	WG	2 (6)	114 114	11.2 11.2	1019 1018			14	0.108	09388
Jordan Station, ON 2005 Cabernet Sauvignon	WG	2 (6)	112 112	14.0 14.0	798 801			14	0.044	09388
Jordan Station, ON 2005 Concord	WG	2 (6)	115 113	11.2 11.2	1026 1014			14	0.043	09388
	+	2 (6)	114 114	11.2 11.2	1018 1022			14	0.044	
	++	2 (6)	115 115	11.2 11.2	1028 1027			14	0.091	
Jordan Station, ON 2005 Riesling	WG	2 (7)	112 113	11.2 11.2	1001 1008			14	0.036	09388
	+	2 (7)	112 112	11.2 11.2	1001 1004			14	0.044	
	++	2 (7)	112 113	11.2 11.2	1004 1010			14	0.041	

+ = + Hasten ®

++ = + Induce ®

Trials on grapes were conducted in the EU. In 2005 and 2006 residues in grapes were studied at three locations in Spain, three locations in Greece, two locations in southern France and two locations in Italy. Four normal and six reverse decline table grape trials were conducted. For the two low-volume trials conducted in southern France, a SC formulation was applied twice by foliar application at the target rate of 35–40 g ai/ha. For the remaining eight trials, SC formulation was applied twice by foliar application at the target rate of 3.5 g ai/hL (42 g ai/ha). For all five trials conducted in 2006, a WG formulation was applied with the same application rates and intervals as for the SC-treated plots sampled with a 3-day PHI. The applications of SC or WG were made at 10–12-day intervals with the last application occurring approximately 0–90 days before normal commercial harvest. No surfactants or adjuvants were added to the applications. All samples were analysed within 8 months of sampling. Recoveries from table grapes fortified at 0.010–0.30 mg/kg of chlorantraniliprole ranged from 81–102% (mean = 90 ± 7.3% (RSD = 8.1%, n = 10) for study 16566 and for samples fortified at 0.010–0.20 mg/kg ranged from 79–108% (mean = 93 ± 10% (RSD = 11%, n = 10) for 18751.

Table 64 Residues for chlorantraniliprole in table grapes from European trials

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS				
Los Palacios, Andalucia, Spain 2005 Shelva	SC	2 (12)	42.4 42.2	3.5 3.5	1206 1205	77 87	87	3	0.020	16566 Berries, entire sample homogenised

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS				
Anchialos, Thessaloniki, Central Macedonia, Greece 2005 Roditis	SC	2 (11)	43.7 42.6	3.5 3.5	1247 1216	84 88	89	3	0.035	16566 Berries, entire sample homogenised
Serres, Provence-Alpes-	SC	2 (10)	35.3 33.2	17.5 17.5	202 190	71 73-75	89	90	0.016	16566 Berries, entire sample
Côte d'Azur, France 2005		2 (9)	35.5 35.3	17.6 17.5	202 201	77 77-79	89	56	0.082	homogenised
Italia		2 (8)	36.3 35.7	17.5 17.5	207 204	81 83	89	29	0.088	
		2 (9)	36.3 34.2	17.5 17.5	207 195	85 85	89	14	0.12	
		2 (11)	35.1 36.1	17.5 17.5	200 206	85 89	89	3	0.23	
		2 (10)	35.7 35.1	17.6 17.5	203 200	89 89	89	(-1 h) (+2 h)	0.053 0.18	
Eleftheres, Kavala, East	SC	2 (11)	43.7 40.0	3.5 3.5	1255 1149	78 79	89	56	0.12	16566 Berries, entire sample
Macedonia, Greece 2005		2 (11)	40.1 41.4	3.5 3.5	1153 1189	83 83	89	28	0.11	homogenised
Sultanina		2(10)	43.5 41.9	3.5 3.5	1252 1205	84 85	89	14	0.051	
		2 (11)	40.4 41.6	3.5 3.5	1161 1195	85 88	89	3	0.11	
		2 (10)	42.1 41.8	3.5 3.5	1211 1203	86 89	89	(-1 h) (+2 h)	0.036 0.068	
Diolo (Piacenza),	SC	2 (12)	40.9 42.0	3.5 3.5	1166 1195	57 63	89	89	0.006	16566
Emilia Romagna, Italy		2 (11)	41.5 42.2	3.5 3.5	1185 1204	75 75	89	55	0.038	Berries, entire sample
2005 Moscato d'Adda		2 (10)	41.5 42.4	3.5 3.5	1185 1206	79 81	89	28	0.063	homogenised
		2 (10)	41.7 41.5	3.5 3.5	1188 1184	81 85	89	14	0.13	
		2 (10)	42.2 42.6	3.5 3.5	1203 1214	85 89	89	3	0.12	
		2 (10)	42.4 42.2	3.5 3.5	1208 1204	85 89	89	(-1 h) (+1 h)	0.037 0.10	
Los Palacios y Villafranca,	SC	2 (10)	42.0 42.0	3.5 3.5	1199 1198	80 83	88	28	0.043	18751
Andalucia, Spain 2006		2 (10)	42.2 42.2	3.5 3.5	1202 1205	84 85	88	14	0.082	
Varlade		2 (11)	42.2 42.4	3.5 3.5	1202 1210	85 86	88	3	0.10	

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS				
		2 (10)	42.0 42.0	3.5 3.5	1203 1205	85 88	88	(-1 h) (+2 h)	0.045 0.079	
	WG	2 (11)	42.2 42.2	3.5 3.5	1201 1200	85 86	88	3	0.036	
Los Palacios y Villafranca, Andalucia,	SC	2 (11)	42.2 42.2	3.5 3.5	1204 1203	85 86	87	3	0.065	18751
Spain 2006 Matilde	WG	2 (11)	42.7 42.0	3.5 3.5	1213 1195	85 86	87	3	0.069	
Carpentras–Serres,	SC	2 (12)	41.1 38.2	10 10	412 382	81 81	89	27	0.048	18751
Provence-Alpes Cote d'Azur,		2 (11)	39.6 40.1	10 10	397 402	83 89	89	13	0.068	
France 2006 Alphouse		2 (10)	39.9 41.5	10 10	398 415	89 89	89	3	0.069	
Lavallié		2 (10)	40.3 41.1	10 10	404 412	89 89	89	(-1 h) (+1 h)	0.044 0.068	
	WG	2 (10)	40.2 39.9	10 10	403 398	89 89	89	3	0.066	
Kato Milia, Pieria, Central	SC	2 (10)	42.7 42.5	3.5 3.5	1218 1213	83 88	89	3	0.12	18751
Macedonia, Greece 2006 Muchat	WG	2 (10)	43.3 42.2	3.5 3.5	1234 1202	83 88	89	3	0.083	
Contrada, Mazzarronella	SC	2 (10)	41.7 42.5	3.5 3.5	1189 1211	79 81	89	28	0.087	18751
Sicily, Italy 2006 Italia		2 (10)	42.8 42.6	3.5 3.5	1222 1215	83 85	89	14	0.050	
		2 (11)	42.7 42.3	3.5 3.5	1217 1207	85 88	89	3	0.044	
		2 (10)	42.8 43.2	3.5 3.5	1222 1233	87 89	89	(-1 h) (+2 h)	0.13 0.017	
	WG	2 (11)	42.4 41.8	3.5 3.5	1207 1189	85 88	89	3	0.061	

Trials in the EU on wine grapes were conducted in the 2004, 2005 and 2006 seasons. In 2004 decline trials were conducted at one location in Southern France and at one location in Northern France. A SC formulation was applied once by foliar application at growth stage BBCH 73–75 to wine grapes (berries and small fruit) at a target application rate of 45.0 g ai/ha. In 2005 and 2006, twelve reverse decline grapes trials were conducted, two in Spain, two in Germany, two in southern France, four in northern France and two in Italy. Seven other grape trials were conducted, three in Spain, one in Greece, one in northern France, and two in Germany. In 14 of 19 trials, a single foliar broadcast application of chlorantraniliprole (SC formulation) was applied at a target application rate of 3.5 g ai/hL (52.5 g ai/ha). In 1 of 19 trials, a single foliar broadcast application of chlorantraniliprole (SC formulation) was applied at a target application rate of 52.5 g ai/ha (7.0 g ai/hL) to represent reduced spray volumes consistent with the crop canopy and local practice. In the remaining 4 of 19 trials, conducted in France, a single foliar broadcast application of chlorantraniliprole (SC formulation) was applied at target application rates of 35.0 g ai/ha

(17.5 g ai/hL) or 40 g ai/ha (10 g ai/hL) to represent low-volume applications consistent with the crop canopy and local practice. No surfactants or adjuvants were added to the applications. Recoveries for samples fortified at 0.01–0.1 mg/kg were

92 ± 12% (RSD = 13%, *n* = 4) for study 14139, 89 ± 7.9% (RSD = 8.8%, *n* = 12) for study 16567 and for samples fortified at 0.01–0.3 mg/kg 83 ± 11% (RSD = 13%, *n* = 22) for study 19306. Samples were stored frozen for up to 8 months prior to analysis.

Table 65 Residues for chlorantraniliprole in wine grapes from European trials

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS				
Les Charmes, Bourgogne, 2004, Pinot Noir	SC	1	47.1	3.2	1490	73	75 81 89 89	30 44 59 75	0.022 0.012 0.016 0.014	14139 Berries, entire sample homogenised
Villié Morgon,Rhône-Alpes, 2004 Gamay	SC	1	47.9	3.1	1523	75	81 85 89 89	30 45 60 75	0.058 0.016 0.010 < 0.01	14139 Berries, entire sample homogenised
Los Palacios, Andalucia,Spain 2005 Merlot	SC	1	52.6	3.5	1500	73	89	90	0.031	16567
		1	53.0	3.5	1514	77	89	56	0.016	
		1	52.0	3.5	1483	77	89	45	0.013	
		1	52.6	3.5	1502	79	89	28	0.018	
		1	53.0	3.5	1509	83	89	14	0.011	
		1	52.8	3.5	1504	89	89	(-1 h) (+2 h)	< 0.01 0.039	
Veldenz Rhineland-Palatinate, Germany 2005 Müller-Thurgau	SC	1	54.3	8.8	619	73	89	90	0.017	16567
		1	50.4	7.0	719	77	89	56	0.055	
		1	55.1	7.0	786	79-81	89	45	0.051	
		1	54.1	7.0	771	83	89	28	0.12	
		1	51.8	7.0	738	85	89	14	0.097	
		1	51.8	7.0	738	89	89	(-1 h) (+1 h)	< 0.01 0.094	
Lamié, Rhône-Alpes, France 2005 Gameny	SC	1	34.0	17.5	195	68-71	89	84	< 0.01	16567
		1	35.7	17.6	203	79	89	55	< 0.01	
		1	35.9	17.5	205	79	89	41	0.019	
		1	36.3	17.5	207	81	89	29	0.010	
		1	35.9	17.5	205	85	89	15	0.013	
		1	34.6	17.5	198	89	89	(-1 h) (+1 h)	< 0.01 < 0.01	
Marfaux, Champagne-Ardennes, France 2005 Chardonnay	SC	1	51.1	3.5	1457	65	89	84	< 0.01	16567
		1	53.8	3.5	1538	77	89	56	0.021	
		1	52.0	3.5	1482	77	89	46	0.012	
		1	50.9	3.5	1452	81	89	28	0.018	
		1	51.6	3.5	1470	83-85	89	14	0.031	
		1	51.8	3.5	1478	89	89	(-1 h) (+1 h)	< 0.01 0.034	

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS				
Fleury-La-Riviere, Champagne, France, 2005 Meunier	SC	1	34.8	17.5	199	65	89	90	< 0.01	16567
		1	34.4	17.5	197	77	89	56	< 0.01	
		1	35.9	17.5	205	77	89	46	< 0.01	
		1	35.3	17.5	201	81	89	28	0.019	
		1	34.2	17.5	195	83-85	89	14	0.014	
		1	34.4	17.5	197	89	89	(-1 h) (+1 h)	< 0.01 0.029	
Miradolo Terme, Lombardia, Italy, 2005 Trebbiano	SC	1	50.9	3.5	1455	65	89	88	0.011	16567
		1	52.0	3.5	1485	77	89	53	0.046	
		1	52.6	3.5	1502	77	89	45	0.052	
		1	52.8	3.5	1507	79	89	29	0.15	
		1	52.4	3.5	1496	83	89	15	0.074	
		1	52.2	3.5	1488	85-89	89	(-1 h) (+1 h)	< 0.01 0.25	
Los Palacios y Villafranca, Andalucia Spain 2006 Merlot	SC	1	52.2	3.5	1490	73	86	90	0.022	19306
		1	52.4	3.5	1496	77	86	56	0.039	
		1	52.8	3.5	1511	81	86	31	0.026	
		1	53.2	3.5	1520	83	86	14	0.057	
		1	52.6	3.5	1506	86	86	(-1 h) (+1 h)	< 0.01 0.011	
Villalba del Alcor, Andalucia, Spain 2006 Zalema	SC	1	52.2	3.5	1495	77	88	90	< 0.01	19306
		1	52.8	3.5	1510	80	88	56	< 0.01	
		1	52.2	3.5	1492	82	88	28	0.033	
Marfaux, Champagne-Ardenne France 2006 Chardonnay	SC	1	40.5	10	405	60-69	89	87	< 0.01	19306
		1	39.9	10	397	75	89	53	< 0.01	
		1	39.9	10	398	81	89	30	0.014	
		1	39.9	10	398	83-85	89	14	0.035	
		1	39.6	10	395	89	89	(-1 h) (+1 h)	< 0.01 < 0.01	
Veldnez, Rhineland-Palatinate, Germany 2006 Müller-Thurgau	SC	1	52.9	3.5	1514	75	89	83	0.025	19306
		1	52.6	3.5	1505	77	89	56	0.041	
		1	51.8	3.5	1481	81	89	30	0.074	
		1	52.8	3.5	1510	85	89	14	0.075	
		1	51.9	3.5	1486	89	89	(-1 h) (+1 h)	< 0.01 < 0.01	
Miradolo, Lombardia, Italy, 2006 Trebbiano	SC	1	52.8	3.5	1509	71	87-89	90	0.013	19306
		1	52.2	3.5	1493	75	87-89	54	0.029	
		1	53.4	3.5	1526	83	87-89	32	0.13	
		1	52.6	3.5	1507	83	87-89	14	0.028	
		1	50.2	3.5	1490	87-89	87-89	(-1 h) (+1 h)	< 0.01 0.21	

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS				
Lancié, Rhône-Alpes, France 2006 Gaunay	SC	1	39.9	10	398	57	89	90	< 0.01	19306
		1	39.2	10	391	77	89	54	< 0.01	
		1	40.1	10	399	81	89	30	< 0.01	
		1	39.6	10	396	85	89	14	< 0.01	
		1	41.3	10	413	89	89	(-1 h) (+1 h)	< 0.01 0.045	
Les Charmes, Bourgogne France 2006 Pinot	SC	1	53.0	3.5	1518	73	89	81	0.015	19306
		1	52.0	3.5	1487	77-79	89	49	0.024	
		1	52.8	3.5	1510	83	89	24	0.030	
		1	52.4	3.5	1496	89	89	11	0.058	
		1	52.8	3.5	1513	89	89	(-1 h) (+1 h)	< 0.01 0.055	
La Roche Vineuse Bourgogne, France 2006 Chardonnay	SC	1	53.2	3.5	1522	71	89	90	0.012	19306
		1	53.4	3.5	1531	75	89	55	0.013	
		1	530	3.5	1516	85	89	31	0.036	
Verdu, Lleida, Spain, 2006 Macaben	SC	1	53.0	3.5	1519	75-77	89	90	0.016	19306
		1	52.6	3.5	1502	81	89	56	0.017	
		1	52.4	3.5	1497	81	89	31	0.061	
Sant Marti de Malda, Lleida, Spain 2006 Syrah	SC	1	52.6	3.5	1503	75-77	89	92	0.016	19306
		1	52.8	3.5	1510	81	89	56	0.041	
		1	52.6	3.5	1507	81	89	31	0.080	
Kesten, Rhineland-Palatinate, Germany 2006 Domfelder	SC	1	53.0	3.5	1515	75	89	83	0.017	19306
		1	52.5	3.5	1501	77	89	56	0.026	
		1	52.7	3.5	1508	81	89	30	0.044	
Anchialos, Thessaloniki, Central Macedonia, Greece 2006 Muschat	SC	1	52.6	3.5	1506	71	89	90	0.011	19306
		1	53.1	3.5	1518	73-75	89	56	0.010	
		1	52.6	3.5	1516	80-82	89	30	0.036	
Radebeul, Saxony, Germany 2006 Kerner	SC	1	55.2	3.5	1574	77	89	90	0.013	19306
		1	52.4	3.5	1495	79-81	89	57	0.068	
		1	54.8	3.5	1563	83	89	30	0.061	

Trials on grapes were also conducted in Australia in the 2005–6 season. Two residue trials were conducted at the same vineyard with two grape varieties (Riesling and Merlot). For each variety, two rates were studied, one at the proposed GAP rate and a second at 2× the proposed GAP rate. A WG formulation was applied twice as a foliar broadcast spray at application concentrations of 3.15 and 6.3 g ai/hL (500–650 l/ha). The non-ionic surfactant Agral was added to each application at 0.025%. The applications were made at an interval of 29 days with the last application occurring

approximately 56 days before normal commercial harvest. Samples were stored frozen for up to 4 months prior to analysis. Recoveries for samples fortified at 0.010–1.0 mg/kg were 81–87% (n = 4).

Table 66 Residues for Chlorantraniliprole in grapes from Australian trials

Country	FL	Application					Residues			Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS	Sample GS	PHI (days)	(mg/kg)	
Orange, New South Wales, Australia 2006 Riesling	WG ++	1	31.5	6.3	500	80% capfall		85	< 0.01	20921 Berries
		2 (29)	15.8 20.5	3.15 3.15	500 650	80% cap fall Pre-bunch closure		56	0.02	
		2 (29)	31.5 41.0	6.3 6.3	500 650	80% cap fall Pre-bunch closure		56	0.03	
Orange, New South Wales, Australia 2006 Merlot	WG ++	1	31.5	6.3	500	80% capfall		85	< 0.01	20921
		2 (29)	15.8 20.5	3.15 3.15	500 650	80% cap fall Pre-bunch closure		56	< 0.01	
		2 (29)	31.5 41.0	6.3 6.3	500 650	80% cap fall Pre-bunch closure		56	0.02	
Coldstream, Victoria, Australia 2006 Chardonnay	WG ++	1	18.9	3.15	600	80% capfall		90	< 0.01	20921
	WG ++	2 (23)	9.5 16.2	1.58 1.58	600 1025	80% cap fall Pre-bunch closure		67	< 0.01	
	WG ++	2 (23)	18.9 32.3	3.15 3.15	600 1025	80% cap fall Pre-bunch closure		67	< 0.01	
	WG ++	2 (23)	37.8 64.6	6.3 6.3	600 1025	80% cap fall Pre-bunch closure		67	0.02	
	SC ++	2 (23)	18.0 30.8	3 3	600 1025	80% cap fall Pre-bunch closure		67	< 0.01	

++ = Agral®, a non-ionic surfactant used at 0.25% v/v

Brassica and Cole vegetables

Trials were conducted in Australia on brassica vegetables (broccoli, cabbage, and Brussels sprouts) in the 2004–5 season and on broccoli, cauliflower, cabbage, and Brussels sprouts in the 2005–6 season. An SC formulation was applied three times by foliar application when heads or buttons were developing at application rates of 20 and 40 g ai/ha for seasonal application rates of 60 and 120 g ai/ha. At all sites, applications were made with the addition of a non-ionic surfactant at 0.0125% v:v. The applications were made at 7-day (±1) intervals with the last application occurring approximately 7 days before the predicted commercial harvest. Decline samples were collected immediately before the last application, after the last application and 3, 7, and 10 days after the last application. Samples were stored frozen for 3–35 days prior to being transported (frozen) to the laboratory. At the laboratory, homogenised samples were stored frozen from 2 weeks to 3 months prior to analysis, meaning that all samples had been stored for 3–4 months before analysis. Recoveries for samples fortified at 0.01–0.5 mg/kg were 93±3.1% (n = 8) for study 19726 and for samples fortified at 0.01–1.0 mg/kg 95.7 ± 4.7% (n = 6) for study DPX-E2Y45.

New Zealand trials on brassica vegetables (broccoli and cabbage) were conducted in the 2005–6 season. A SC formulation was applied three times by foliar application when the heads were 50 to 250 mm in size, at application rates of 20 and 40 g ai/ha. At all sites, applications were made with the addition of a non-ionic surfactant at 0.007% v:v. The applications were made at 7-day (±1) intervals with decline samples collected immediately before the last application, after the last application and 3, 7, and 10 days after the last application. Samples were stored frozen for up to 5 months prior to being transported (frozen) to the laboratory. At the laboratory, homogenised samples were stored frozen for 1 month prior to analysis. In total, samples had been stored frozen for up to 6 months between harvest and analysis. Recoveries for cabbage samples fortified at 0.01–0.1 mg/kg ranged from 90–108% and for broccoli 78–91%.

Table 67 Residues for chlorantraniliprole in brassica and cole from Australian and NZ trials

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS				
Broccoli										
Werribee, Victoria, Broccoli Atomic	SC ++	3 (7 6)	20 20 20	4.2 4.4 4.0	476 455 500	Head 2.0-5.0 cm Heads 6.0 cm Harvest size	Mature	6** 0 3 7 10	0.096 0.207 0.181 0.156 0.079	19726 Agral 0.0125%
	SC ++	3 (7 6)	40 40 40	8.4 8.8 8.0	476 455 500	Head 2.0-5.0 cm Heads 6.0 cm Harvest size	Mature	6** 0 3 7 10	0.178 0.494 0.423 0.271 0.192	
Shepparton, Victoria Broccoli Mascot	SC ++	3 (7 7)	20 20 20	4.0 4.0 4.0	500 500 500	Early head Early head Mature	Mature	7** 0 3 7 10	0.07 0.18 0.16 0.12 0.06	DPX-E2Y45 Brassica AU Agral 0.0125%
	SC ++	3 (7 7)	40 40 40	8.0 8.0 8.0	500 500 500	Vegetative Early head Early head	Mature	7** 0 3 7 10	0.16 0.39 0.33 0.22 0.15	
Pukekohe, NZ 2006 broccoli Viper	SC +++	3 (7 7)	20 20 20	5.0 5.0 5.0	400 400 400	Head ≤ 5 cm Head ≤ 12.5 cm Head ≤ 20 cm	Mature	7** 0 3 7 10	0.07 0.15 0.13 0.07 0.04	19727 Actiwet 0.007%
		3 (7 7)	40 40 40	10 10 10	400 400 400	Head ≤ 5 cm Head ≤ 12.5 cm Head ≤ 20 cm	Mature	7** 0 3 7 10	0.17 0.28 0.21 0.12 0.04	
Pukekohe, NZ 2006 broccoli Marathon	SC +++	3 (7 7)	20 20 20	4.1 4.1 4.1	490 490 492	Head ≤ 5 cm Head ≤ 10 cm Head ≤ 15 cm	Mature	** 0 3 7 10	0.01 0.06 0.03 0.03 0.01	19727 Actiwet 0.007%

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS				
		3 (7 7)	40	8.2	490	Head ≤ 5 cm	Mature	**	0.04	
			40	8.2	490	Head ≤ 10 cm		0	0.11	
			40	8.1	492	Head ≤ 15 cm		3	0.05	
								7	0.07	
								10	0.05	
Cauliflower										
Yanchep, Western Australia, cauliflower, Avron	SC ++	3 (7 7)	20	3.2	626	Pre-heading	Mature	7	0.109	19726 Agral 0.0125%
			20	3.2	626	Head 5 cm				
			20	3.2	626	Head 15 cm				
	SC ++	3 (7 7)	40	6.4	626	Pre-heading	Mature	7	0.233	
			40	6.4	626	Head 5 cm				
			40	6.4	626	Head 15 cm				
Cabbage										
Pozieres, Queensland, Cabbage	SC ++	3 (7 7)	20	2.7	737	Developing head	Mature	7	0.081	19726 Agral 0.0125%
			20	2.7	737	Developing head				
			20	2.7	745	Developing head				
Camborne	SC ++	3 (7 7)	40	5.4	737	Developing head	Mature	7	0.171	
			40	5.4	737	Developing head				
			40	5.4	745	Developing head				
Cranbourne, Victoria, cabbage Green cornet	SC ++	3 (7 7)	20	3.6	550	Head enlarging	Mature	7	0.086	19726 Agral 0.0125%
			20	3.6	550	Head enlarging				
			20	3.6	550	Mature				
	SC ++	3 (7 7)	40	7.3	550	Head enlarging	Mature	7	0.198	
			40	7.3	550	Head enlarging				
			40	7.3	550	Mature				
The Summit, Queensland, cabbage Drum Head	SC ++	3 (7 9)	20	3.3	614	Head developing	Mature	7**	0.04	DPX-E2Y45 Brassica AU Agral 0.0125%
			20	3.2	626	Head developing		0	0.12	
			20	3.3	614	Head developing		3	0.10	
								7	0.05	
								10	0.03	
	SC ++	3 (7 9)	40	6.5	614	Head developing	Mature	7**	0.08	
			40	6.4	626	Head developing		0	0.19	
			40	6.5	614	Head developing		3	0.15	
								7	0.13	
								10	0.07	
Pukekohe NZ 2006 cabbage Cabaret	SC +++	3 (8 7)	20	5.0	400	Head 10 cm	Head 20-25 cm	7**	0.02	19727 Actiwet 0.007%
			20	5.0	400	Head 15 cm		0	0.05	
			20	5.0	400	Head 20 cm		3	0.08	
								7	0.03	
								10	0.07	

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS				
		3 (8 7)	40 40 40	10 10 10	400 400 400	Head 10 cm Head 15 cm Head 20 cm	Head 20-25 cm	7** 0 3 7 10	0.03 0.09 0.14 0.08 0.06	
Pukekohe NZ 2006 cabbage Cabaret	SC +++	3 (7 7)	20 20 20	4.0 4.1 4.1	497 492 493	Head 10 cm Head 15 cm Head 25 cm	Mature	** 0 3 7 10	< 0.01 0.06 < 0.01 < 0.01 < 0.01	19727 Actiwet 0.007%
		3 (7 7)	40 40 40	8.0 8.1 8.1	497 492 493	Head 10 cm Head 15 cm Head 25 cm	Mature	** 0 3 7 10	0.02 0.06 0.01 < 0.01 < 0.01	
Brussels sprouts										
Moriarty, Tasmania, Brussels sprouts	SC ++	3 (7 7)	20 20 20	4.9 4.8 4.9	410 416 410	0.4-0.5 cm 1.0-4.0 cm 1.0-4.0 cm	Mature/ semi-mature	7	0.185	19726 Agral 0.0125%
Maximus	SC ++	3 (7 7)	40 40 40	9.8 9.6 9.8	410 416 410	0.4-0.5 cm 1.0-4.0 cm 1.0-4.0 cm	Mature/ semi-mature	7	0.275	
Nairne, South Australia, Brussels sprouts Abacus	SC ++	3 (6 8)	20 20 20	3.3 3.3 3.3	600 600 600	Forming buttons Forming buttons Maturing buttons		7** 0 3 7 10	0.04 0.14 0.12 0.08 0.06	DPX-E2Y45 Brassica AU Agral 0.0125%
	SC ++	3 (6 8)	40 40 40	6.7 6.7 6.7	600 600 600	Forming buttons Forming buttons Maturing buttons		7** 0 3 7 10	0.11 0.27 0.28 0.20 0.12	

** sample 6-7 days after 2nd application

++ = Agral ®, a non-ionic surfactant used at 0.125% v/v

+++ = Actiwet ® at 0.7% v/v

North American trials on brassica vegetables were conducted in 2005 at 27 locations in Canada and the United States. An SC formulation was applied twice as a foliar broadcast spray at the rate of 112 g ai/ha/application to brassica vegetables when the crop was at growth stage BBCH 15 to 87. Adjuvants were applied according to typical agricultural practices. The applications of were made at 3-day intervals with the last application occurring approximately 3 days before normal harvest. All samples were analysed within 258 days of sampling using an LC/MS/MS method (13294). Untreated control samples fortified with 0.010 to 12.5 mg/kg chlorantraniliprole were analysed concurrently with the treated samples to verify method performance. Concurrent recoveries were 99 ± 11% (n = 21). For broccoli/cauliflower, recoveries ranged from 79 to 118% with an average of 98 ± 12% (n = 7). For cabbage, recoveries ranged from 85 to 103% with an average of 93 ± 7% (n = 8). For mustard greens, recoveries ranged from 93 to 119% with an average of 107 ± 9% (n = 6).

Table 68 Residues for chlorantraniliprole in brassica and cabbage from USA trials (16570)

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Spray Additive
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS				
Broccoli										
Germansville, PA, USA 2005 triathlon	SC	2 (2)	114 115	31 31	374 375	Early/mid head formation	mature	3	0.32	16570 Dyne-Amic (0.5%)
Delavan, WI, USA 2005 Premium Crop	SC	2(3)	114 114	54 56	212 204	48 48	49	3	0.30	X-77 (0.25%)
Branchton, ON, Canada 2005	SC	2 (3)	109 118	45 49	242 239	47 47	47	3	0.40	Agral 90 (0.03%)
St-Marc-sur-Richelieu, QC, Canada 2005 Packman	SC	2 (3)	110 109	44 44	244 247	47 48	49	3	0.38	Citowett Plus (0.25%)
Lakeport, CA, USA 2005 Arcadia	SC	2 (4)	110 113	29 30	374 374	49 49	49	3	0.32	Siluet L77 (0.03%)
Madera, CA, USA 2005 Heritage	SC	2 (3)	116 116	41 41	286 286	49 49	49	3	0.41	Penetrator Plus (0.75%)
San Ardo, CA, USA 2005 Patron	SC	2 (3)	114 112	36 36	315 313	78 78-79	79	3	0.35	
Corvallis, OR, USA 2005 Emerald Pride	SC	2 (3)	115 116	27 27	420 422	Head developing Head developing	1st harvest	3	0.12	R-11 (0.25%)
Paynesville, MN, USA 2005 Gypsy	SC	2 (3)	113 114	59 60	191 191	77-79 79	77-79 77-79 77-79 77-79 79 79	-0 +0 1 3 7 10	0.56 0.46 0.67 0.56 0.10 0.042	NIS (0.25%)
Cabbage										
Germansville, PA, USA 2005 Blue Lagoon	SC	2 (3)	115 115	35 35	328 328	Head 15 cm Head 15 cm	mature	3	0.64	Dyne-Amic (0.5%)
Norman Park, GA, USA 2006 Rio Verde	SC	2 (3)	116 118	56 53	209 222	87 87	untrimmed trimmed 88	3	0.28 0.037	
Needmore, FL, USA 2005 Bravo	SC	2 (4)	116 115	32 32	359 359	48 48	51	3	0.033	Agri-Dex (0.5%)
Rochelle, IL, USA 2005 Blue Gem	SC	2 (3)	112 112	40 40	278 279	47 49	49	3	0.51	

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Spray Additive
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS				
Gardner, ND, USA 2005 Stonehead	SC	2 (3)	114 115	61 60	186 187	46 48	48	3	0.48	
St-Marc-sur-Richelieu, QC, Canada 2005 Stonehead	SC	2 (3)	111 113	45 45	248 253	48 48	49	3	0.066	Citowett Plus (0.25%)
Rougemont, QC, Canada 2005 Bantley	SC	2 (3)	112 104	34 34	333 306	49 49	49	3	0.29	Agral 90 (0.03%)
East Bernard, TX, USA 2005 Early Jersey Wakefield	SC	2 (3)	113 115	48 49	233 235	48 49	Untrimmed Trimmed 49	3	1.1 0.078	Dyne-Amic (0.5%)
Fresno, CA, USA 2005 Golden Acre	SC	2 (3)	110 113	30 30	369 377	47 48	Untrimmed Trimmed mature	3	0.75 0.077	
Abbotsford, BC, Canada 2005 Bartolo	SC	2 (3)	112 116	50 50	223 232	70-80 70-80	80-90	4	0.10	

Cabbage untrimmed = cabbage with wrapper leaves intact;

Cabbage, trimmed = cabbage with wrapper leaves removed.

Fruiting vegetables, Cucurbits

In 2006 trials on cucumbers and zucchini were conducted at two locations in each Spain, Italy, southern France, and the Netherlands, and one location in Greece. A WG formulation was applied twice at the target rate of 60 g ai/ha (4.0 g ai/hL) at both applications to cucumbers and zucchini grown under protected cover. The applications were made at 6–7-day intervals with the last application occurring at 0–1 days before the first commercial harvest for (continuously ripening) protected cucumbers and zucchinis. No surfactants or adjuvants were added to the applications.

All samples were analysed within 9 months of sampling. Concurrent recoveries from control cucumber specimens fortified at 0.010–0.20 mg/kg of chlorantraniliprole ranged from 75–104% (mean = 91% ± 10%. Concurrent recoveries from control zucchini specimens fortified at 0.010–0.20 mg/kg of chlorantraniliprole ranged from 84–104% (mean = 95% ± 7%).

Table 69 Residues for chlorantraniliprole in cucumbers and zucchini grown under protected cover from European trials

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS				
Cucumber										
Los Palacios Andalucia, Spain 2006 Sol Verde	WG	2 (7)	60.2 60.5	4.0 4.0	1498 1506	75 88	88 88 89 89 89 89	(-1 h) (+2 h) 1 7 14 21	< 0.01 0.022 < 0.01 < 0.01 < 0.01 < 0.01	18760

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS				
Los Palacios, Andalucia, Spain 2006 Suso	WG	2 (7)	60.2 60.5	4.0 4.0	1501 1506	71 76	76 76 76 77 77 79	(-1 h) (+2 h) 1 7 14 21	0.030 0.026 0.10 0.031 0.032 0.013	18760
Lucenay Rhône- Alpes, France 2006 Loustik	WG	2 (7)	60.2 59.5	4.0 4.0	1510 1490	89 89	89	1	0.058	18760
Siebengewalde, The Netherlands 2006 Fitness	WG	2 (7)	60.4 60.7	4.0 4.0	1513 1519	83-85 89	89	1	0.039	18760
Profitis. Thessaloniki Central Macedonia, Greece 2006 Luberon	WG	2 (7)	59.7 60.7	4.0 4.0	1489 1513	85 89	89	(-1 h) (+1 h) 1 7 14 21	0.015 0.066 0.083 0.013 < 0.01 < 0.01	18760
Zucchini										
Triginto di Mediglia, Lombardia, Italy 006 President	WG	2 (7)	58.4 61.9	4.0 4.0	1459 1542	72 81/82	81/82 81/82 81/82 83 85 87	(-1 h) (+2 h) 1 7 14 21	0.011 0.17 0.13 0.027 < 0.01 < 0.01	18760
Murello, Piemonte, Italy 2006 Isotta	WG	2 (7)	59.1 59.4	4.0 4.0	1483 1487	70 71	71	1	0.064	18760
Pernes-les-Fontaines Provence-Alpes-Côte d'Azur, France 2006 Radiant	WG	2 (6)	59.1 60.2	4.0 4.0	1475 1505	75 76	76 76 76 78 78 > 79	(-1 h) (+1 h) 1 7 14 21	< 0.01 0.029 0.021 < 0.01 < 0.01 < 0.01	18760
Siebengewalde, The Netherlands 2006 Cora	WG	2 (7)	60.6 59.3	4.0 4.0	1520 1488	75-77 89	89	1	0.016	18760

The field program was conducted in 2006. Four magnitude of residue and five normal decline melon trials were conducted, two in Spain, three in Italy, two in southern France and two in Greece. Chlorantraniliprole (35WG formulation) was applied twice at the target rates 60.0 g ai/ha (4.0 g ai/hL) to protected melons. The applications were made at 7-day intervals with the last application occurring at normal harvest. No surfactants or adjuvants were added to the applications.

All samples were analysed within 6 months of sampling. Concurrent recoveries from control melon peel specimens fortified at 0.010, 0.10 and 0.30 mg/kg of chlorantraniliprole ranged from 74–126%, mean = 96 ± 19% (RSD = 20%, n = 16). Concurrent recoveries from control melon pulp specimens fortified at 0.010 and 0.10 mg/kg of chlorantraniliprole ranged from 85–119%, mean = 97 ± 9% (RSD = 9%, n = 15).

Table 70 Residues for chlorantraniliprole in melons grown under protected cover from European trials

Country	FL	Application					Sample GS	DALT (days)	Residue (mg/kg)			Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	GS			Peel	Pulp	Whole	
Los Palacios y Villafranca, Andalucia, Spain 2006 Nicolas	WG	2 (7)	59.4 60.2	4.0 4.0	1480 1501	85 85	85 85 85 85 89 89	(-1 h) (+1 h) 1 7 14 21	0.029 0.074 0.014 0.048 0.022 0.020	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.013 0.030 0.007 0.019 0.010 0.009	18761
Casteldidon, Lombardia, Italy 2006 Macigno	WG	2 (7)	59.4 60.5	4.0 4.0	1483 1510	78 83	83 83 83 85 87 88-89	(-1 h) (+1 h) 1 7 14 21	0.016 0.081 0.037 0.091 0.069 0.038	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.011 0.049 0.021 0.038 0.032 0.009	18761
Lograto, Lombardia, Italy 2006 Macigno	WG	2 (7)	59.4 60.5	4.0 4.0	1481 1509	79 83	83 83 83 84-85 86-87 88-89	(-1 h) (+1 h) 1 7 14 21	0.016 0.027 0.058 0.025 0.048 0.015	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.009 0.015 0.030 0.013 0.021 0.009	18761
Sanlucar de Barrameda, Andalucia, Spain 2006 Siglo	WG	2 (7)	60.5 60.2	4.0 4.0	1519 1508	80 89	85	1	0.091	< 0.01	0.032	18761
Pernes les Fontaines, Provence-Alpes-Cote d'Azur, France	WG	2 (7)	60.5 60.2	4.0 4.0	1512 1496	73 73-85	73-85 73-85 73-85 85 87 89	(-1 h) (+1 h) 1 8 14 21	0.018 0.016 0.017 0.021 0.018 0.013	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.011 0.010 0.009 0.010 0.010 0.008	18761
Profitis, Central Macedonia, Greece 2006 Gallia F1	WG	2 (7)	60.4 58.6	4.0 4.0	1506 1462	73-79 79-82	89 89 89 89 89 89	(-1 h) (+1 h) 1 7 14 21	0.028 0.040 0.080 0.026 0.043 0.049	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.014 0.025 0.032 0.016 0.016 0.028	18761
Pernes les Fontaines, Provence-Alpes-Cote d'Azur France 2006 Cesar	WG	2 (7)	60.2 60.5	4.0 4.0	1510 1522	67-73 73-85	73-85	1	0.15	< 0.01	0.068	18761
Svoronos, Central Macedonia, Greece 2006 Gallia F1	WG	2 (7)	60.4 59.0	4.0 4.0	1504 1469	75-79 80-84	89	1	0.054	< 0.01	0.023	18761

Country	FL	Application					Sample GS	DALT (days)	Residue (mg/kg)			Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	GS			Peel	Pulp	Whole	
Contrada Pozzo Bollente, Sicily, Italy 2006 Calbero	WG	2 (7)	58.0 60.4	4.0 4.0	1455 1515	81 87	88	1	0.039	< 0.01	0.019	18761

Residue trials in cucurbits were conducted in 2005 at 20 locations in the United States. An SC formulation was applied twice as a foliar broadcast spray at the rate of 112 g ai/ha/application to cucumber, summer squash, and cantaloupe/muskmelon when the crop was at growth stage BBCH 71-89. The applications were made at 5-day intervals with the last application occurring approximately 1 day before normal harvest. No surfactants or adjuvants were added to the applications. All samples were analysed within 128 days of sampling using an LC/MS/MS method (13294). Concurrent recoveries from control samples fortified at 0.010 to 0.10 mg/kg of chlorantraniliprole ranged from 71 to 110% with an overall average of 92 ± 11% (n = 17).

Table 71 Residues for chlorantraniliprole in cucurbits from USA trials

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS				
Cucumber										
Sycamore, GA, USA 2005 Straight Eight	SC	2 (6)	115 115	56 59	206 195	88 89	89	1	0.076	16572
Athens, GA, USA 2005 Long Green Improved	SC	2 (5)	111 114	44 45	255 252	72-73 87-88	88-89	1	0.011	16572
Zellwood, FL, USA 2005 FM5020 F1	SC	2 (3)	111 113	40 40	281 281	Near maturity Pickle size	Pickle size	1	0.015	16572
Delavan, WI, USA 2005 Marketmore 86	SC	2 (5)	114 114	52 52	219 218	71 73	Mature	1	< 0.01	16572
Gardner, ND, USA 2005 Straight Eight	SC	2 (5)	124 109	66 58	187 187	Fruit 15-20 cm 85	85	1	0.012	16572
Leonard, MO, USA 2005 Sweet Slice	SC	2 (5)	116 113	61 59	189 190	86 88	89	1	0.076	16572
East Bernard, TX, USA 2005 Straight Eight	SC	2 (5)	119 118	88 51	235 233	73 75	75 75 75 76 76 77	(-1 h) (+1 h) 1 3 7 9	< 0.01 0.022 0.017 0.013 < 0.01 < 0.01	16572

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS				
Cantaloupe										
Athens, GA, USA 2005 Hales Best Jumbo	SC	2 (5)	113 113	40 40	283 281	82-84 89	89	1	0.090	16572
Delavan, WI, USA 2005 Fast Break	SC	2 (5)	115 113	55 53	210 214	81 89	Mature	1	0.027	16572
East Bernard, TX, USA 2005 Hales Best Jumbo	SC	2 (4)	121 120	52 51	233 235	82 84	84	1	0.065	16572
Porterville, CA, USA 2005 Hales Best Jumbo	SC	2 (5)	112 110	30 29	373 379	73 78	79	1	0.100	16572
Madera, CA, USA 2005 Top Mark G Strain	SC	2 (5)	114 113	35 35	327 327	82 83	84	1	0.081	16572
Hughson, CA, USA 2005 Hales Best Jumbo	SC	2 (6)	112 114	40 41	280 280	85 87	89	1	0.052	16572
Muskmelon										
Hamilton City, CA, USA 2005 Canary Yellow	SC	2 (5)	114 113	41 40	281 281	Fruit 13 cm Fruit 13-18 cm	80% ripe	1	0.010	16572
Summer squash										
North Rose, NY, USA 2005 Clarita	SC	2 (6)	108 113	44 47	243 243	71 86	89	1	0.017	16572
Bumpass, VA, USA 2005 Summer Gold	SC	2 (6)	110 110	59 59	186 186		Mature	1	0.081	16572
Sycamore, GA, USA 2005 Crooked Neck	SC	2 (4)	116 116	56 56	206 206	88 89	Mature	1	0.023	16572
Bradenton, FL, USA 2005 Zucchini Non-classic	SC	2 (5)	121 114	31 30	388 374	Fruiting Fruiting	Mature	1	0.054	16572
Leonard, MO, USA 2005 Black Beauty	SC	2 (5)	112 112	59 60	189 188	85 86	86	1	0.076	16572

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS				
Porterville, CA, USA 2005 Early Summer Yellow Crooked Neck	SC	2 (4)	110 112	30 30	369 372	73 78	81	1	0.040	16572

Fruiting vegetables other than cucurbits

Tomatoes

A trial on tomatoes was conducted in the 2004–5 season at one site in Australia. Plots were treated 1× rate and 2× the proposed Australian rate. A SC formulation was applied four times as a foliar broadcast spray at application rates of 20 and 40 g ai/ha. No surfactants or adjuvants were added to the application. The applications were made at intervals of 9, 8, and 42 days with the last application occurring approximately 7 days before normal commercial harvest. All samples were analysed within 4 months of sampling. Recovery values for untreated control samples fortified with 0.010 and 1.0 mg/kg chlorantraniliprole run concurrently with treated samples in all the trials were within 82.0–93.0% ($n = 4$).

Table 72 Residues for chlorantraniliprole in tomatoes from Australian trials

Country	Application					Residues			Report No
	FL	No (interval)	g ai/ha	L/ha	GS	Sample GS	PHI (days)	(mg/kg)	
Mooroopna, Victoria Australia 2005 U941	SC	3 (9, 8)	20	500 500 500	Fruit mostly flowers Flowering 3rd truss Some ripe fruit	mature	42	< 0.01	20921
		4 (9, 8, 42)	20	500 500 500 500	Fruit mostly flowers Flowering 3rd truss Some ripe fruit Majority ripe fruit	mature	0 7	0.02 0.01	
		3 (9, 8)	40	500 500 500	Fruit mostly flowers Flowering 3rd truss Some ripe fruit	mature	42	< 0.01	
		4 (9, 8, 42)	40	500 500 500 500	Fruit mostly flowers Flowering 3rd truss Some ripe fruit Majority ripe fruit	mature	0 7	0.03 0.02	

Field grown tomatoes

Trials in 2004 were conducted in Spain and Italy using a WG formulation applied twice by foliar application at a target application rate 30 g ai/ha. The second application occurred 7 days before predicted commercial harvest. No surfactants or adjuvants were added to the applications. In 2005 trials were conducted at two locations in Spain, one location in Greece, one location in southern France and one location in Italy. Chlorantraniliprole (WG formulation) was applied twice by foliar application at the target rates of 3.5 g ai/hL (35 g ai/ha) to field tomatoes. The applications were made at target 7-day intervals with the last application occurring 0-35 days before the predicted commercial harvest. In 2006 trials were conducted at one location in Spain, one location in Greece, one location in

southern France and one location in Italy. Chlorantraniliprole (WG formulation) was applied twice at the target rate of 40 g ai/ha to field. In addition, chlorantraniliprole SC formulation was applied twice at the same targeted rate to separate plots at each location. The applications of WG and SC were made at 6–7-day intervals with the last application occurring approximately 0–35 days before normal commercial harvest. No surfactants or adjuvants were added to the applications during any field program. All samples were analysed within 7 months of sampling. Recovery values for samples fortified with chlorantraniliprole at 0.01–0.1 mg/kg were 87-119% (n = 23).

Trial 73 Residues for chlorantraniliprole in tomatoes from European trials (field crops)

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS				
Turano Lodiciano, Lombardia, Italy 2004, 9661	WG	2 (10)	30.4 30.8	3.1 3.1	988 1000	81 87	87 87 87 87 87 88 88	(–1 h) 0 (+2 h) 1 3 5 7 10	< 0.01 0.030 0.015 0.023 0.015 0.014 < 0.01	14153
Palafolls, Catalonia Spain 2004 Bond	WG	2 (10)	30.8 30.8	3.1 3.1	998 1000	71-73 83-85	83-85 83-85 84-85 85-86 87-88 87-88 88	(–1 h) 0 (+2 h) 1 3 5 7 11	0.022 0.038 0.055 0.015 0.014 0.018 0.012	14153
Los Palacios, Andalucia, Spain 2005 Juncal	WG	2 (10)	35.1 35.8	3.5 3.5	1001 1015	72 73	89	32	0.013	16581
		2 (7)	34.4 35.5	3.5 3.5	975 1004	75 76	89	21	0.033	
		2 (7)	35.1 35.8	3.5 3.5	998 1014	76 79	89	10	0.023	
		2 (7)	35.8 35.8	3.5 3.5	1021 1012	83 86	89	1	0.033	
		2 (7)	34.5 36.5	3.5 3.5	1008 1036	83 89	89	(-1 h) (+2 h)	< 0.01 0.023	
San Donato Milanese, Lombardia, Italy 2005 Pavia	WG	2 (6)	34.8 34.8	3.5 3.5	985 983	63 63	86	36	< 0.01	16581
		2 (6)	34.0 35.1	3.5 3.5	965 997	71 71-81	86	22	< 0.01	
		2 (6)	35.5 34.8	3.5 3.5	1009 991	83 84	86	10	0.011	
		2 (7)	35.5 34.8	3.5 3.5	1006 986	84 86	86	1	0.029	
		2 (7)	35.8 34.8	3.5 3.5	1014 987	84 86	86	(-1 h) (+2 h)	< 0.01 0.038	
Tauste, Navarra, Spain 2005 Talen	WG	2 (7)	35.1 36.2	3.5 3.5	995 1023	85 88-89	88-89	1	0.030	16581

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS				
Pusignan, Rhône-Alpes, France 2005 Perfect Peel	WG	2 (7)	34.0 35.0	3.5 3.5	970 999	85 87	87-89	1	0.025 c0.012	16581
Thessaloniki, Central Macedonia, Greece 2005 Titano M	WG	2 (8)	35.1 34.7	3.5 3.5	996 986	73 77	89	34	< 0.01	16581
		2 (7)	34.8 36.9	3.5 3.5	987 1047	80 82	89	21	< 0.01	
		2 (7)	36.9 35.0	3.5 3.5	1048 992	84 86	89	10	< 0.01	
		2 (7)	35.0 35.0	3.5 3.5	992 995	87 89	89	1	< 0.01	
		2 (7)	34.8 35.3	3.5 3.5	988 1001	87 89	89	(-1 h) (+2 h)	0.012 0.013	
Monticelli d'Ongina, Emilia Romagna, Italy 2006 Cilento	WG	2 (7)	39.9 38.8	4.0 4.0	1001 979	64 64	87	36	< 0.01	18756
		2 (7)	38.8 38.8	4.0 4.0	972 977	71 81	87	22	< 0.01	
		2 (7)	38.8 39.2	4.0 4.0	974 981	81 83	87	10	< 0.01	
		2 (6)	39.9 40.2	4.0 4.0	1007 1012	85 87	87	1	0.033	
		2 (7)	39.2 40.2	4.0 4.0	985 1009	85 87	87	(-1 h) (+2 h)	0.021 0.031	
	SC	2 (6)	39.2 40.5	4.0 4.0	983 1013	85 87	87	1	0.036	
Vaunaveys, Rhône-Alpes, France 2006 Leader	WG	2 (7)	40.6 40.9	4.0 4.0	1018 1025	87 87-89	87-89	1	0.032	18756
	SC	2 (7)	41.1 40.9	4.0 4.0	1029 1021	87 87-89	87-89	1	0.062	
Bellius, Lleida, Spain 2006 Raff	WG	2 (7)	39.9 39.9	4.0 4.0	1003 1007	61-65 71-72	84-85	35	< 0.01	18756
		2 (7)	39.9 39.9	4.0 4.0	1006 1004	72 73-74	84-85	21	0.017	
		2 (7)	39.9 40.6	4.0 4.0	1001 1017	76 78	84-85	10	0.017	
		2 (7)	40.2 40.2	4.0 4.0	1014 1007	76 82	84-85	1	0.030	
		2 (7)	40.6 40.2	4.0 4.0	1018 1018	78 82	84-85	(-1 h) (+2 h)	0.024 0.024	
	SC	2 (7)	40.5 40.3	4.0 4.0	1013 1007	76 82	84-85	1	0.041	
Profitis, Central Macedonia, Greece 2006 Santim	WG	2 (7)	40.2 40.5	4.0 4.0	1011 1018	85 89	89	1	0.011	18756
	SC	2 (7)	40.3 41.6	4.0 4.0	1007 1039	85 89	89	1	0.018	

EU trials on tomatoes grown under protected cover

In 2004 trials were conducted at one location each in Spain and Italy. A WG formulation was applied twice by foliar application to protected tomatoes at a target application rate 45 g ai/ha. The second application occurred 5 days before predicted commercial harvest. In 2005–6 trials were conducted; one in France, two in Spain, three in the Netherlands and one in Greece. Chlorantraniliprole (WG formulation) was applied twice by foliar application at the target rates of 3.5 g ai/hL (52.5 g ai/ha) to protected tomatoes, including cherry tomatoes. The applications were made at 7-day (± 1) intervals with the last application occurring on the day of the first commercial harvest for (continuously ripening) protected tomatoes.

For 2006 trials were conducted at two locations in Spain, one location in Greece, two locations in France and two locations in Italy. Chlorantraniliprole (WG formulation) was applied twice at the target rates of 60 g ai/ha (4.0 g ai/hL) to protected tomatoes, including cherry tomatoes. The applications were made at 6–7-day intervals with the last application occurring approximately 0–1 days before the first commercial harvest. No surfactants or adjuvants were added to the applications in any growing season. All samples were analysed within 10 months of sampling. Recovery values for samples fortified at 0.01–0.2 mg/kg were 76-132% (n = 41) for tomatoes, including cherry tomatoes, grown under protected cover.

Table 74 Residues for chlorantraniliprole in tomatoes from European trials (protected cover crops)

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS				
Mediglia, Lombardia, Italy 2004 Albenga	WG	2 (10)	45.9 46.2	3.1 3.1	1498 1507	86 88	88 88 88 88-89 88-89 89 89	(-1h) 0(+1h) 1 3 5 7 10	< 0.01 0.021 0.012 0.018 0.011 0.010 0.017	14154
Los Palacios, Andalucia, Spain 2004 Bond	WG	2 (10)	46.2 45.8	3.0 3.0	1518 1505	66 71	71 71 71 7 71 89 89	(-1h) 0(+2h) 1 3 5 7 10	< 0.01 0.012 0.010 < 0.01 0.011 0.015 0.011	14154
Los Palacios y Villafranca, Andalucia, Spain 2005 Bond	WG	2 (7)	52.3 52.7	3.5 3.5	1500 1505	83 84	84 84 84 84 86 89	(-1 h) (+2 h) 1 10 21 35	< 0.01 0.015 < 0.01 0.012 < 0.01 < 0.01	16582
Siebengewald, Limburg, The Netherlands 2005 Pannory	WG	2 (7)	52.4 53.2	3.5 3.5	1500 1522	85 87	87 87 88-89 89 89 89	(-1 h) (+2 h) 1 10 21 35	< 0.01 0.034 0.029 0.027 0.034 0.021	16582
Wellerlooi, Limburg, The Netherlands 2005 Relaxx	WG	2 (7)	54.6 54.8	3.5 3.5	1563 1531	85 87	88-89	1	0.095	16582

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS				
Profitis, Thessaloniki, Central Macedonia, Greece 2005 Alma	WG	2 (7)	51.1 50.6	3.5 3.5	1463 1450	78 81	89	1	< 0.01	16582
Moissieu, Pact, Rhônes-Alpes, France 2005 Félicia	WG	2 (7)	51.9 53.4	3.5 3.5	1486 1522	73 73	73 73 73 74-85 85 89	(-1 h) (+1 h) 1 10 21 35	< 0.01 < 0.01 < 0.01 < 0.01 0.015 < 0.01	16582
Triginto di Mediglia, Lombardia, Italy 2006 Oskar	WG	2 (7)	59.4 58.7	4.0 4.0	1488 1471	83 85	85 85 85 87 89 89	(-1 h) (+1 h) 1 10 21 35	0.013 0.043 0.028 0.012 0.037 0.027	18755
Pact, Rhône-Alpes, France 2006 Félicia	WG	2 (7)	62.3 60.2	4.0 4.0	1554 1506	81 81	82-83	1	0.079	18755
Lleida, Spain 2006 Caramba	WG	2 (7)	60.2 60.2	4.0 4.0	1500 1502	84-85 87	87 87 87 87 88 88	(-1 h) (+1 h) 1 11 21 35	0.023 0.072 0.061 0.039 0.028 0.022	18755
Profitis, Central Macedonia, Greece 2006 Alma	WG	2 (7)	59.3 60.8	4.0 4.0	1481 1519	74-80 80-82	89	1	< 0.01	18755
Los Palacios y Villafranca, Andalucia Spain 2006 Lupita	WG	2 (7)	52.3 52.7	3.5 3.5	1497 1504	73 83	83 83 83 84 84 89	(-1 h) (+2 h) 1 10 21 35	< 0.01 0.016 0.015 < 0.01 0.022 0.028	16584 Cherry tomato
Siebengewald, Limburg, The Netherlands 2005 Efwee 100	WG	2 (7)	52.9 52.4	3.5 3.5	1514 1500	85 87	87 87 88-89 89 89 89	(-1 h) (+2 h) 1 10 21 35	0.035 0.095 0.079 0.067 0.066 0.051	16584 Cherry tomato
Los Palacios y Villafranca, Andalucia, pain 2006 Lupita	WG	2 (7)	59.8 60.2	4.0 4.0	1502 1510	81 85	85 85 85 85 88 89	(-1 h) (+1 h) 1 10 21 45	0.037 0.12 0.11 0.10 0.071 0.044	18769 Cherry tomato

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS				
Roncoferraro, Lombardia, Italy 2006 Fiolino	WG	2 (6)	59.8 60.5	4.0 4.0	1503 1521	85 87	87 87 87 87 89 89	(-1 h) (+2 h) 1 10 22 36	0.061 0.066 0.090 0.011 c0.011 0.031 0.010	18769 Cherry tomato
Caphan, Provence-Alpes-Cote d'Azur, France 2006 Severino	WG	2 (7)	58.7 61.2	4.0 4.0	1472 1540	75-76 76	76 76 76 79 79 87	(-1 h) (+1 h) 1 10 21 35	0.080 0.11 0.088 0.15 0.15 0.099	18769 Cherry tomato

North American trials on tomatoes and peppers were conducted in 2005–6 at 40 locations in Canada and the United States. A SC formulation was applied twice as a foliar broadcast spray at the rate of 112 g ai/ha/application to fruiting vegetables (tomatoes, bell peppers, and non-bell peppers) when the crop was at a growth stage ranging from BBCH 49 to 90. The applications of were made at 5-day intervals with the last application occurring approximately 1 day before normal harvest. No surfactants or adjuvants were added to the applications.

All samples were analysed within 5 months of sampling. For tomato, pepper, and non-bell peppers, concurrent recoveries from control samples fortified at 0.010 to 1.00 mg/kg of chlorantraniliprole ranged from 88 to 113% with an overall average of 96 ± 6% (n = 24).

Table 75 Residues for chlorantraniliprole in tomatoes from USA trials

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS				
North Rose, NY, USA 2005 Floradade	SC	2 (5)	112 112	48 48	234 234	81 83	84	1	0.071	16575
Bumpass, VA, USA 2005 589	SC	2 (6)	111 112	59 59	187 189		81	1	0.040	16575
Needmore, FL, USA 2005 Florida 47	SC	2 (5)	118 118	29 29	401 402	51 51	51	1	0.018	16575
Jennings, FL, USA 2005 FLA 47	SC	2 (6)	120 115	35 35	343 329	50 51	Mature	1	0.032	16575
Rochelle, IL, USA 2005 Celebrity	SC	2 (3)	112 112	40 40	277 281	87 88	89	1	0.040	16575
Clarence, MO, USA 2005 Better Boy	SC	2 (5)	109 114	54 58	202 195	81 83	93	1	0.032	16575

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS				
Cambridge, ON, Canada 2005 CC 1069	SC	2 (5)	119 106	55 47	216 227	89 89	89	1	0.18	16575
Port Burwell, ON, Canada 2005 Plum	SC	2 (6)	112 118	45 47	248 252	88-89 89	89	1	0.14	16575
Thorndale, ON, Canada 2005 Roma Sunoma	SC	2 (5)	112 112	56 56	200 200	79 82-83	82-83	1	0.092	16575
Thamesford, ON, Canada 2005 Roma Sunoma	SC	2 (4)	112 115	56 58	200 200	79 82-83	82-83	1	0.14	16575
London, ON, Canada 2005 Sebring	SC	2 (5)	113 111	57 56	199 197	82-83 85	85	1	0.14	16575
St-Marc-sur-Richelieu, QC, Canada 2005 Celebrity	SC	2 (4)	112 109	37 37	299 296	75-81 84	84	1	0.044	16575
Madera, CA, USA 2005 ACE 55 VF	SC	2 (5)	114 115	41 41	280 284	86 86	86	1	0.059	16575
Madera, CA, USA 2005 Rio Grande	SC	2 (5)	114 114	41 40	281 284	86 86	86	1	0.051	16575
Fresno, CA, USA 2005 Roma	SC	2 (5)	112 114	41 41	275 281	87 88	89	1	0.061	16575
Glenn, CA, USA 2005 CXD 207	SC	2 (5)	114 114	49 49	234 234	50% red fruit 90% red fruit	Mature	1	0.11	16575
Terra Bella, CA, USA 2005 9557 processing	SC	2 (5)	113 112	31 30	369 374	75 77	77	1	0.095	16575
Porterville, CA, USA 2005 9557 processing	SC	2 (6)	115 113	31 30	371 374	75 77	77	1	0.10	16575
San Luis Obispo, CA, USA 2005 AB2	SC	2 (6)	118 118	28 28	419 420	83 86	86-87	1	0.082	16575
Langton, ON, Canada 2005 CC 1069	SC	2 (5)	113 113	47 47	241 240	88 89	89 89 89 89 89 89	-0 +0 1 3 7 10	0.046 0.14 0.049 0.058 0.052 0.070	16575

Peppers

Peppers (including chili) grown under protected cover

In the EU, trials on peppers grown under protected cover were conducted in 2005 and 2006 at four locations in Spain, three locations in Greece, three locations in the Netherlands, three locations in France, and one location in Italy. In the first season, chlorantraniliprole (WG formulation) was applied twice by foliar application at the target rates of 3.5 g ai/hL (35 g ai/ha) to protected peppers, including hot peppers. In the second season chlorantraniliprole (WG formulation) was applied twice at the target rates 43.75 g ai/ha (3.5 g ai/hL) to protected peppers and chili peppers. The applications were made at 5–8-day intervals. Specimens were harvested from the residue decline treated plots immediately before the last application (0 DBLA) and then +0, 1, 10, 21, and 35 days (BBCH 62–89) after the last application (DALA) to the treated plots. No surfactants or adjuvants were added to the applications. All samples were analysed within 6 months of sampling. Recoveries for samples fortified a

Table 76 Residues for chlorantraniliprole in peppers from European trials (protected cover crops)

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	GS				
Los Palacios, Spain 2005 Italico	WG	2 (7)	35.5 35.5	3.5 3.5	1007 1004	71 76	79	1	0.048	16580
Limburg, The Netherlands, 2005 Derby	WG	2 (7)	35.6 34.9	3.5 3.5	1010 990	87 89	89	1	0.049	16580
Meterik, The Netherlands, 2005 Corsica	WG	2 (7)	35.8 34.2	3.5 3.5	1015 970	71 73-75	89	35	0.019	16580
		2 (8)	36.3 35.2	3.5 3.5	1030 1000	77 79	89	21	0.022	
		2 (7)	36.3 35.8	3.5 3.5	1030 1015	81 83	89	10	0.023	
		2 (7)	36.3 35.8	3.5 3.5	1030 1015	85 89	89	1	0.058	
		2 (8)	34.7 34.7	3.5 3.5	985 985	85 89	89	(-1 h) (+2 h)	0.022 0.051	
Pact, France 2005 Hannibal	WG	2 (7)	34.8 35.5	3.5 3.5	994 1014	> 79 > 79	87-89	35	0.054	16580
		2 (7)	35.1 35.1	3.5 3.5	1005 1001	> 79 > 79	87-89	21	0.044	
		2 (7)	35.1 33.7	3.5 3.5	1006 965	> 79 > 79	87-89	10	0.062	
		2 (7)	35.8 35.5	3.5 3.5	1026 1017	> 79 87-89	87-89	1	0.055	
		2 (7)	35.5 35.8	3.5 3.5	1010 1024	87-89 87-89	87-89	(-1 h) (+1 h)	0.018 0.033	
Profitis, Greece 2005 Astor	WG	2 (7)	33.8 35.3	3.5 3.5	961 1002	72-79 79-81	89	35	0.029	16580
		2 (7)	34.8 35.5	3.5 3.5	988 1005	82 84	89	21	0.052	
		2 (7)	34.4 36.2	3.5 3.5	976 1029	85 85	89	10	0.035	

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	GS				
		2 (7)	33.6 36.7	3.5 3.5	954 1041	86 89	89	1	0.023	
		2 (7)	35.6 36.4	3.5 3.5	1010 1034	87 89	89	(-1 h) (+2 h)	< 0.01 0.014	
Los Palacios y Villafranca, Andalucia, Spain 2006 Palermo	WG	2 (7)	44.9 44.9	3.5 3.5	1282 1277	55 55	83	35	< 0.01	18754
		2 (5)	44.5 44.9	3.5 3.5	1272 1286	61 61	83	22	< 0.01	
		2 (7)	44.9 44.5	3.5 3.5	1277 1268	63 63	83	10	0.072	
		2 (7)	44.5 44.5	3.5 3.5	1270 1273	73 78-81	83	1	0.062	
		2 (7)	44.9 44.1	3.5 3.5	1284 1261	74-76 83	83	(-1 h) (+2 h)	0.022 0.14	
Murello, Piemonte, Italy 2006 Quadrato di Cuneo	WG	2 (7)	43.8 43.8	3.5 3.5	1248 1250	51 61-62	85	35	0.026	18754
		2 (6)	43.8 45.6	3.5 3.5	1252 1301	65 70	85	22	0.024	
		2 (6)	43.8 45.2	3.5 3.5	1249 1287	71 71	85	11	0.029	
		2 (6)	44.1 44.1	3.5 3.5	1259 1262	81-82 85	85	1	0.025	
		2 (7)	44.9 44.5	3.5 3.5	1280 1267	81-82 85	85	(-1 h) (+2 h)	0.013 0.059	
Pact, Rhône-Alpes, France 2006 Hannibal	WG	2 (7)	43.8 43.4	3.5 3.5	1246 1240	88 89	89	1	0.11	18754
Profitis, Thessaloniki, Greece 2006 Raiko	WG	2 (7)	45.9 45.6	3.6 3.6	1289 1280	85 87	89	1	0.036	18754

Peppers (including chili) grown in field

Trials were conducted in 2005 in field grown peppers at three locations in Spain, two locations in Greece and at two locations in Italy. Chlorantraniliprole (WG formulation) was applied twice by foliar application at the target rates of 35 g ai/ha (3.5 g ai/hL) to field peppers, including hot peppers. In 2006 residue trials were conducted at three locations in Spain, two locations in Greece, and two locations in Italy. Chlorantraniliprole (WG formulation) was applied twice at the target rates 40.0 g ai/ha to field peppers, including hot peppers. The applications were made at 7-day (±1) intervals. Specimens were harvested from the residue decline treated plots immediately before the last application (0 DBLA) and then +0, 1, 10, 21, and 35 days (BBCH 62–89) after the last application (DALA) to the treated plots. No surfactants or adjuvants were added to the applications. All samples were analysed within 7 months of sampling. For samples fortified at 0.01–0.3 mg/kg, recoveries were 72–11% (n = 36) for field peppers, including hot peppers.

Table 77 Residues for chlorantraniliprole in peppers from European trials (field crops)

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS				
Huelva, Spain 2005 Palermo	WG	2 (6)	35.1 35.5	3.5 3.5	1004 1006	73 74	74	1	0.022	16579
Profitis, Greece 2005 Astor	WG	2 (6)	33.6 34.6	3.5 3.5	957 986	83 86-87	89	1	0.037	16579
Murello, Italy 2005 Quadrato di Cuneo	WG	2 (6)	35.8 35.1	3.5 3.5	1016 995	62 62	85	36	< 0.01	16579
		2 (8)	35.8 35.5	3.5 3.5	1019 1013	62-71 62-71	85	21	< 0.01	
		2 (6)	35.1 36.2	3.5 3.5	999 1034	62-81 81	85	10	< 0.01	
		2 (6)	35.8 35.5	3.5 3.5	1018 1007	81-82 84-85	85	1	0.019	
		2 (7)	35.1 35.1	3.5 3.5	999 1002	81-82 85	85	(-1 h) (+1 h)	< 0.01 0.014	
Poirino, Italy 2005 Lungo	WG	2 (7)	34.8 34.8	3.5 3.5	993 990	63 72	86	36	< 0.01	16579
		2 (7)	35.8 36.2	3.5 3.5	1023 1031	81 81	86	21	< 0.01	
		2 (7)	35.8 35.5	3.5 3.5	1023 1015	82 83	86	10	< 0.01	
		2 (7)	35.8 34.0	3.5 3.5	1025 970	83 86	86	1	0.025	
		2 (7)	35.8 35.5	3.5 3.5	1019 1007	83 86	86	(-1 h) (+2 h)	< 0.01 0.028	
Navarra, Spain 2005 California	WG	2 (7)	34.8 35.8	3.5 3.5	993 1024	63-71 69-73	89	36	0.026	16579
		2 (7)	35.1 35.5	3.5 3.5	996 1011	83 85	89	21	0.020	
		2 (7)	36.2 35.5	3.5 3.5	1031 1008	85 86	89	11	0.060	
		2 (7)	35.5 35.5	3.5 3.5	1010 1012	86 88	89	1	0.066	
		2 (7)	35.5 35.8	3.5 3.5	1012 1021	87 89	89	(-1 h) (+2 h)	0.014 0.059	
Villalba del Alcor, Andalucia, Spain 2006 Infante	WG	2 (7)	39.5 39.5	4.0 4.0	998 994	55 55	72	33	< 0.01	18753
		2 (7)	38.4 39.5	4.0 4.0	968 998	61 63	72	21	< 0.01	
		2 (7)	39.9 39.5	4.0 4.0	1006 997	64 65	72	10	0.018	
		2 (6)	39.9 39.9	4.0 4.0	1006 1007	71 71-72	72	1	< 0.01	
		2 (7)	39.9 39.9	4.0 4.0	999 1004	71 72	72	(-1 h) (+2 h)	< 0.01 < 0.01	

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS				
Poirino, Piemonte, Italy 2006 Corno di Bue	WG	2 (7)	39.5 40.2	4.0 4.0	996 1013	80 80-81	87	36	< 0.01	18753
		2 (7)	40.6 39.5	4.0 4.0	1023 997	81 81	87	22	< 0.01	
		2 (7)	39.9 39.9	4.0 4.0	1005 1005	81 83	87	10	0.010	
		2 (7)	40.9 40.2	4.0 4.0	1028 1012	83-84 87	87	1	0.049	
		2 (8)	39.5 40.6	4.0 4.0	998 1018	83-84 87	87	(-1 h) (+1 h)	0.023 0.044	
Villalba del Alcor, Andalucia, Spain 2005 Italico	WG	2 (7)	40.2 39.9	4.0 4.0	1008 1003	89 89	89	1	0.020	18753
Profitis, Central Macedonia, Greece 2005 Staboli	WG	2 (7)	39.3 39.0	4.0 4.0	989 981	87 89	89	1	0.15	18753

Table 78 Residues for chlorantraniliprole in Bell peppers from USA trials (16575)

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS				
Bumpass, VA, USA 2005 Enza	SC	2 (5)	106 114	59 60	180 192		89	1	0.11	16575
Jennings, FL, USA 2005 Aristotle Bell	SC	2 (6)	112 110	40 47	277 234	89 89	89	1	0.069	16575
Rochelle, IL, USA 2005 Sweet California Wonder	SC	2 (5)	112 113	40 40	283 284	73 74	74	1	0.024	16575
Marysville, OH, USA 2005 Alliance	SC	2 (5)	116 111	60 56	195 197	89 90	89-90	1	0.090	16575
Cambridge, ON, Canada 2005 Aristotle	SC	2 (6)	105 116	43 46	245 255	87 89	89	1	0.013	16575
Cambridge, ON, Canada 2005 Aristotle	SC	2 (6)	119 112	48 44	245 255	85 89	89	1	0.022	16575
Cambridge, ON, Canada 2005 Aristotle	SC	2 (5)	118 118	53 54	220 217	87 87	89	1	0.019	16575
St-Marc-sur-Richelieu, QC, Canada 2005 Bell Boy	SC	2 (5)	112 114	37 38	299 303	81 84	84	1	0.11	16575

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS				
East Bernard, TX, USA 2006 California Wonder	SC	2 (5)	113 113	47 48	241 235	84 89	89	1	0.13	16575
Madera, CA, USA 2005 Maccabi	SC	2 (5)	114 114	41 41	281 282	87 87	87	1	0.18	16575
Porterville, CA, USA 2005 Ingra	SC	2 (4)	118 112	37 35	315 317	49 49	49	2	0.14	16575

Table 79 Residues for chlorantraniliprole in hot peppers (chili) from European trials (field)

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS				
Profitis Central Macedonia	WG	2 (7)	35.5 34.8	3.5 3.5	1007 989	77 79	89	35	0.028	16585
Greece 2005 Local		2 (7)	34.8 35.2	3.5 3.5	989 999	80 82	89	21	0.036	
Magnisias		2 (7)	35.1 34.8	3.5 3.5	996 989	84 85	89	10	0.032	
		2 (7)	35.1 34.0	3.5 3.5	996 965	85 88	89	1	0.13	
		2 (7)	35.5 34.4	3.5 3.5	1009 978	85-86 89	89	(-1 h) (+2 h)	0.027 0.15	
Alpicat, Lleida	WG	2 (7)	35.5 34.8	3.5 3.5	1008 983	62-70 62-70	85-86	35	0.019	16585
Spain 2005 Guindilla roja		2 (7)	35.5 35.8	3.5 3.5	1011 1014	71-72 71-73	85-86	22	0.059	
riojana		2 (7)	35.5 34.8	3.5 3.5	1005 987	71-73 73	85-86	11	0.070	
		2 (7)	35.5 35.8	3.5 3.5	1010 1022	78-79 83	85-86	1	0.20	
		2 (7)	35.8 35.8	3.5 3.5	1013 1019	79 85-86	85-86	(-1 h) (+2 h)	0.048 0.16	
Los Palacios y Villafranca,	WG	2 (7)	40.2 40.2	4.0 4.0	1008 1009	51 63	87	35	0.018	18765
Andalucia, Spain 2006		2 (7)	40.2 39.2	4.0 4.0	1008 983	81 81	87	21	0.040	
Flame Flare		2 (7)	40.6 40.2	4.0 4.0	1018 1012	81 85	87	10	0.056	
		2 (7)	40.2 40.2	4.0 4.0	1008 1015	85 87	87	1	0.11	
		2 (7)	39.9 39.9	4.0 4.0	1004 999	86 87	87	(-1 h) (+2 h)	0.014 0.16	

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS				
Nea Magnisia, Central Macedonia, Greece 2006 Local Magnisia	WG	2 (6)	40.4 40.1	4.0 4.0	1017 1008	67-70 70-72	89	36	< 0.01	18765
		2 (8)	40.2 40.0	4.0 4.0	1012 1007	72-74 74-78	89	21	0.031	
		2 (7)	40.0 40.2	4.0 4.0	1005 1010	79-81 81-83	89	10	0.079	
		2 (7)	38.5 39.6	4.0 4.0	968 997	82-86 86-88	89	1	0.089	
		2 (7)	40.2 40.2	4.0 4.0	1012 1010	82-86 87-88	89	(-1 h) (+2 h)	0.088 0.14	
Contrada Gelso Bianco, Catania, Italy 2006 Piros	WG	2 (7)	38.3 39.2	4.0 4.0	964 987	63 64	81	35	< 0.01	18765
		2 (7)	39.8 39.1	4.0 4.0	1000 982	66 67	81	21	0.060	
		2 (7)	38.6 39.1	4.0 4.0	971 982	71 72	81	10	0.10	
		2 (7)	39.4 39.1	4.0 4.0	991 982	72 81	81	1	0.18	
		2 (7)	40.1 41.3	4.0 4.0	1009 1040	72 81	81	(-1 h) (+2 h)	0.058 0.11	

Table 80 Residues for chlorantraniliprole in hot peppers (chili) from European trials (protected cover)

Country	FL						Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS				
Los Palacios y Villafranca, Andalucia, Spain 2006 Flame	WG	2 (6)	35.5 35.5	3.5 3.5	1021 1013	71 71-73	86	35	0.17	16586
		2 (7)	35.5 34.8	3.5 3.5	1014 1033	73 74	86	21	0.12	
		2 (7)	35.9 35.2	3.5 3.5	1026 1010	75-77 76	86	10	0.11	
		2 (7)	35.5 35.2	3.5 3.5	1013 1002	78 86	86	1	0.15	
		2 (7)	35.9 34.8	3.5 3.5	1024 999	81 86	86	(-1 h) (+2 h)	0.050 0.23	
Limburg, Netherlands, 2005 Piedno	WG	2 (7)	36.3 35.2	3.5 3.5	1031 1000	71 73-75	89	35	0.054	16586
		2 (7)	35.2 36.3	3.5 3.5	1000 1031	77 79	89	21	0.037	
		2 (7)	36.3 36.3	3.5 3.5	1031 1031	81 83	89	10	0.064	
		2 (7)	34.1 35.2	3.5 3.5	969 1000	87 89	89	1	0.039	
		2 (8)	35.2 36.3	3.5 3.5	1000 1031	87 89	89	(-1 h) (+2 h)	0.016 0.053	

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS				
Los Palacios y Villa Franca,	WG	2 (6)	43.4 43.4	3.5 3.5	1245 1244	61 62	89	36	0.11	18757
Andalucia, Spain 2006		2 (7)	43.8 44.1	3.5 3.5	1248 1256	62 68	89	21	0.57	
Flame Flare		2 (7)	43.8 44.1	3.5 3.5	1252 1260	68 74	89	10	0.32	
		2 (7)	43.8 43.8	3.5 3.5	1255 1255	74 86	89	1	0.14	
		2 (7)	43.8 43.4	3.5 3.5	1254 1242	78 89	89	(-1 h) (+2 h)	0.068 0.23	
Pact, Rhône-Alpes, France	WG	2 (7)	43.8 43.8	3.5 3.5	1250 1247	81 81	89	35	0.063	18757
2006 Nour		2 (7)	44.1 44.5	3.5 3.5	1261 1269	81 85	89	21	0.10	
		2 (7)	45.2 44.9	3.5 3.5	1293 1284	85 87-89	89	10	0.16	
		2 (7)	45.2 44.1	3.5 3.5	1295 1260	87-89 89	89	1	0.15	
		2 (7)	44.5 44.5	3.5 3.5	1267 1276	87-89 89	89	(-1 h) (+2 h)	0.077 0.11	
Profitis, Central	WG	2 (7)	45.2 42.9	3.5 3.5	1292 1226	60-61 67	89	34	0.011	18757
Macedonia, Greece 2006		2 (7)	44.4 42.8	3.5 3.5	1268 1221	68-75 68-75	89	20	0.11	
Raiko		2 (7)	44.8 45.0	3.5 3.5	1280 1284	75 83-84	89	10	0.39	
		2 (7)	45.1 43.6	3.5 3.5	1288 1246	85 87-89	89	1	0.37	
		2 (7)	43.6 45.0	3.5 3.5	1245 1284	84-86 87-89	89	(-1 h) (+2 h)	0.22 0.34	

Table 81 Residues for chlorantraniliprole in non-Bell peppers from USA trials

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS				
Marysville, OH, USA 2005 Jalapeno	SC	2 (5)	116 118	60 60	195 197	81-89 81-89	87-89	1	0.21	16575
Cambridge, ON, Canada 2005 Super Hot Hungarian	SC	2 (5)	114 114	54 55	220 217	87 89	89	1	0.019	16575
Cambridge, ON, Canada 2005 Inferno	SC	2 (6)	109 116	44 46	245 255	87 89	89	1	0.035	16575

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS				
Thorndale, ON, Canada 2005 Keywest	SC	2 (5)	113 113	57 57	198 200	72-74 74-82	74-82	1	0.066	16575
Thamesford, ON, Canada 2005 Hungarian Wax Pepper	SC	2 (4)	112 112	56 59	198 190	79 81	81	1	0.059	16575
St-Marc-sor-Richelieu, QC, Canada 2005 Cayenne	SC	2 (5)	114 119	37 37	307 318	79-90% final size 85	85	1	0.41	16575
Levelland, TX, USA 2005 Jalapeno M	SC	2 (5)	118 115	42 42	282 277	71 89	89	1	0.063	16575
Claude, TX, USA 2005 NuMex Chili	SC	2 (5)	116 116	42 41	280 282	86 89	89	1	0.13	16575
King City, CA, USA 2005 Jalapeno	SC	2 (6)	113 112	35 35	320 316	88 89	89	1	0.069	16575

Leafy vegetables

Lettuce

The field program was conducted in 2005 and 2006 at two locations in Spain, two locations in Greece, two locations in southern France, three locations in Italy, and two locations in northern France. Four magnitude of residue field lettuce trials and seven reverse decline field lettuce trials were conducted. Chlorantraniliprole 35WG was applied twice as a foliar broadcast spray at a target application rate of 40 g ai/ha. The applications of Chlorantraniliprole 35WG were made at 6–9-day intervals with the last application occurring approximately 0–42 days before normal commercial harvest. No surfactants or adjuvants were added to the applications. All samples were analysed within 9 months of sampling.

Europe 2005: Field. The mean percent recovery for chlorantraniliprole from five control field lettuce specimens, freshly fortified at 0.010 mg/kg was 85 ± 14% (RSD = 17%). The mean percent recovery for chlorantraniliprole from 5 control field lettuce specimens freshly fortified at 0.10 mg/kg was 86 ± 9.4% (RSD = 11%). The mean percent recovery for chlorantraniliprole from 2 control field lettuce specimens freshly fortified at 5.0 mg/kg was 86%.

Europe 2006 Field: The mean percent recovery for chlorantraniliprole from three control field lettuce specimens, freshly fortified at 0.010 mg/kg was 87 ± 8.6% (RSD = 10%). The mean percent recovery for chlorantraniliprole from 3 control field lettuce specimens freshly fortified at 0.10 mg/kg was 89 ± 3.0% (RSD = 3%). The mean percent recovery for chlorantraniliprole from 2 control field lettuce specimens freshly fortified at 0.15 mg/kg was 91 ± 1.7% (RSD = 2%).

Table 82 Residues for chlorantraniliprole in lettuce from European trials (field)

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS				
Olivares, Andalucia, Spain 2005 Filippu	WG	2 (9)	40.5 40.5	4.0 4.0	1011 1013	14 18	49	40	< 0.01	16573
		2 (7)	40.8 40.8	4.0 4.0	1019 1019	19 19	49	28	< 0.01	
		2 (8)	40.8 40.5	4.0 4.0	1019 1016	30 37	49	14	0.039	
		2 (7)	39.8 40.1	4.0 4.0	998 1001	37 47	49	7	0.31	
		2 (6)	39.4 39.4	4.0 4.0	984 989	47 49	49	1	0.14	
		2 (7)	39.4 39.8	4.0 4.0	987 999	47 49	49	(-1 h) (+2 h)	0.064 0.33	
Profitis, Thessaloniki, Central Macedonia, Greece 2005 Atraxion	WG	2 (7)	40.0 40.2	4.0 4.0	998 1003	46 49	49	1	1.7	16573
Lucenay, Rhône-Alpes, France 2005 Esstelle	WG	2 (7)	39.1 41.2	4.0 4.0	973 1033	47-49 47-49	47-49	1	0.88	16573
Milagro, Navarro, Spain 2005 Iceberg	WG	2 (6)	40.8 41.2	4.0 4.0	1014 1028	14-15 16	49	43	< 0.01	16573
		2 (7)	39.8 38.3	4.0 4.0	994 956	17 41-42	49	28	< 0.01	
		2 (7)	40.1 41.9	4.0 4.0	996 1048	45-47 47	49	14	< 0.01	
		2 (7)	42.6 41.6	4.0 4.0	1062 1033	47 48-49	49	7	< 0.01	
		2 (7)	40.5 39.8	4.0 4.0	1006 995	48 48-49	49	1	< 0.01	
		2 (7)	40.1 40.1	4.0 4.0	1003 1004	48-49 49	49	(-1 h) (+1 h)	< 0.01 0.13	
Triginio di Mediglia, Lombardia, Italy 2005 Gentilina	WG	2 (7)	40.1 41.2	4.0 4.0	999 1024	13 16	49	42	< 0.004	16573
		2 (7)	39.0 41.6	4.0 4.0	971 1040	18 20	49	28	< 0.01	
		2 (8)	40.8 39.4	4.0 4.0	1014 985	22 37	49	14	0.055	
		2 (7)	40.1 38.7	4.0 4.0	1002 964	37 38	49	7	0.18	
		2 (6)	39.4 39.0	4.0 4.0	982 972	38 49	49	1	0.37	
		2 (7)	39.0 39.0	4.0 4.0	973 970	38 49	49	(-1 h) (+2 h)	0.094 0.63	

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS				
Lucenay, Rhône-Alpes, France 2006 Feuille de Chêne	WG	2 (7)	41.6 40.6	4.0 4.0	1041 1014	15 19	49	27	< 0.01	18750
		2 (7)	38.8 39.2	4.0 4.0	968 980	43 47	49	14	0.032	
		2 (6)	38.4 39.5	4.0 4.0	964 991	47 47-49	49	7	0.075	
		2 (6)	38.4 39.2	4.0 4.0	958 982	47-49 49	49	1	0.45	
		2 (8)	39.5 39.9	4.0 4.0	990 999	47-49 49	49	(-1 h) (+1 h)	0.012 0.44	
Triginto di Mediglia, Lombardia, Italy 2006 Lollo	WG	2 (6)	38.8 39.2	4.0 4.0	979 987	14 16	49	29	< 0.01	18750
		2 (8)	38.4 39.9	4.0 4.0	968 1002	19 45	49	14	0.017	
		2 (7)	39.2 39.9	4.0 4.0	986 1003	45 47	49	7	0.31	
		2 (6)	38.8 39.2	4.0 4.0	973 984	47 49	49	1	0.86	
		2 (7)	39.2 40.9	4.0 4.0	983 1026	47 49	49	(-1 h) (+1 h)	0.22 1.0	
Castellazzo Bormida, Piemonte, Italy 2006 Diabless	WG	2 (7)	39.5 40.9	4.0 4.0	993 1031	47 49	49	1	0.46	18750
Thessaloniki, Central Macedonia, Greece 2006 Aberam	WG	2 (7)	40.6 40.6	4.0 4.0	1022 1020	42-43 47-48	49	1	< 0.01	18750
Milly-le-Foret, Ile-de-France, France 2006 Freestyle	WG	2 (7)	41.0 40.8	4.0 4.0	1025 1019	14 14	49	29	< 0.01	18750
		2 (7)	40.8 39.9	4.0 4.0	1019 998	18 19	49	15	0.15	
		2 (7)	40.4 40.6	4.0 4.0	1011 1016	19 47-49	49	6	0.52	
		2 (6)	40.8 40.4	4.0 4.0	1021 1010	47-48 49	49	1	0.83	
		2 (6)	39.4 39.7	4.0 4.0	984 992	47-48 49	49	(-1 h) (+2 h)	0.32 0.96	
St Lambert des Levees, France 2005 Anibrai	WG	2 (7)	40.4 40.9	4.0 4.0	1011 1023	14 17	47	28	0.014	18750
		2 (7)	39.9 40.5	4.0 4.0	997 1012	41 42-43	47	14	0.48	
		2 (7)	41.0 40.4	4.0 4.0	1025 1011	42-43 44	47	7	0.55	
		2 (7)	39.5 41.1	4.0 4.0	987 1027	43-44 47	47	1	1.0	

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS				
		2 (7)	39.5 40.8	4.0 4.0	988 1020	44 47	47	(-1 h) (+2 h)	0.28 1.3	

The field program was conducted in 2006 at two locations in the Netherlands, one location in Greece, three locations in France, four locations in Italy and at four locations in Spain. Four magnitude of residue (MOR) trials and ten reverse decline trials were conducted in protected lettuce, including lambs lettuce. Chlorantraniliprole (35WG formulation) was applied twice as a foliar broadcast spray at a target application rate of 40.0 g ai/ha. The applications were made at 6–8-day intervals with the last application occurring approximately 0–28 days before normal harvest. No surfactants or adjuvants were added to the applications. All samples were analysed within 10 months of sampling.

Europe 2006 - Protected cover: Concurrent recoveries from control specimens fortified at 0.01, 0.10 and 9.0 mg/kg of chlorantraniliprole ranged from 74–109%, with an overall mean = 90 ± 11% (RSD = 12%, *n* = 16).

Table 83 Residues for chlorantraniliprole in lettuce from European trials (protected)

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS				
Villafranca, Loa Palacios y Andalucia, Spain 2006 Iceberg	WG	2 (7)	40.6 39.5	4.0 3.9	1020 1003	16 17	49	28	< 0.01	18764
		2 (7)	39.9 39.9	4.0 4.0	1006 1008	19 35	49	14	< 0.01	
		2 (7)	39.9 39.5	4.0 3.9	1005 1004	35 45	49	7	0.072	
		2 (7)	39.9 39.5	4.0 3.9	1006 1004	45 49	49	1	0.093	
		2 (7)	39.5 39.5	3.9 3.9	1004 1004	45 49	49	(-1 h) (+1 h)	0.032 0.41	
Los Palacios, Andalucia, Spain 2006 Tunice	WG	2 (7)	39.9 39.9	4.0 4.0	1008 1009	16 17	49	28	0.046	18764
		2 (7)	39.2 39.9	4.0 4.0	989 1005	35 45	49	14	0.099	
		2 (7)	39.5 40.2	3.9 4.0	1004 1015	45 47	49	7	0.093	
		2 (6)	40.6 39.5	4.0 3.9	1023 1003	47 49	49	1	0.15	
		2 (7)	40.6 40.2	4.0 4.0	1023 1015	47 49	49	(-1 h) (+1 h)	0.11 0.35	
Barbate, Cadiz, Andalucia, Spain 2006 Iceberg	WG	2 (7)	33.2 33.2	3.3 3.3	1008 1003	45 49	49	1	0.38	18764

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS				
Azzano S.Paolo, Lombardia, Italy 2005 Tropi	WG	2 (6)	39.2 39.2	4.0 4.0	978 993	12 14	48	29	0.28	18764 Lambs lettuce
		2 (6)	38.8 39.5	3.9 3.9	985 1003	14 16	48	14	4.5	
		2 (7)	39.9 40.2	4.0 4.0	1006 1016	16 18	48	7	5.6	
		2 (6)	39.9 39.5	3.9 3.9	1013 1003	18 19-48	48	1	7.8	
		2 (7)	38.5 39.2	4.0 4.0	971 990	18 48	48	(-1 h) (+2 h)	3.4 7.1	
Azzano S.Paolo, Lombardia, Italy 2006 Justine	WG	2 (7)	39.5 39.2	4.0 4.0	988 978	15 19	49	28	< 0.01	18764
		2 (7)	39.9 39.5	4.0 4.0	998 987	19 42	49	14	0.25	
		2 (7)	39.2 40.6	4.0 4.0	986 1021	42 46	49	7	1.0	
		2 (6)	39.2 39.2	4.0 4.0	983 984	46 49	49	1	1.6	
		2 (7)	39.2 40.9	4.0 4.0	984 1024	46 49	49	(-1 h) (+2 h)	0.57 1.4	
Triginto di Mediglia, Lombardia, Italy 2006 Fantastic	WG	2 (7)	39.5 40.2	4.0 4.0	995 1012	47 49	49	1	1.3	18764 open
Vivy, Pays-dela-Loirre, France 2006 Trophy	WG	2 (7)	40.6 39.4	4.0 4.0	1029 997	14 18-20	49	28	0.32	18764
		2 (7)	41.4 41.2	4.0 4.0	1048 1042	19-42 19-44	49	14	1.4	
		2 (7)	39.9 40.8	4.0 4.0	1010 1032	19-44 19-46	49	7	4.1	
		2 (7)	38.7 38.6	4.0 4.0	980 976	19-46 49	49	1	2.8	
		2 (7)	40.5 39.4	4.0 4.0	1026 998	19-46 49	49	(-1 h) (+4 h)	1.7 3.3	
Lucenay, Rhone-Alpes, France 2006 Dedale	WG	2 (7)	38.8 38.5	4.0 4.0	979 975	> 19 > 19	47-49	28	0.77	18764 Open
		2 (7)	38.5 38.8	4.0 3.9	970 986	33 33	47-49	14	1.1	
		2 (7)	39.9 40.2	3.9 4.0	1012 1019	33 35-37	47-49	7	0.91	
		2 (6)	38.8 38.5	4.0 3.9	979 976	35-37 47-49	47-49	1	1.4	
		2 (7)	39.9 40.2	3.9 4.0	1012 1018	35-37 47-49	47-49	(-1 h) (+1 h)	0.56 1.7	

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS				
Pernes les Fontaines, Provence-Alpes-Cote d'Azur, France 2006 Kigalie	WG	2 (7)	41.0 40.6	4.0 4.0	1035 1028	47 49	49	1	2.0 c< 0.01	18764 open
Limburg, The Netherlands 2006 Pulsar	WG	2 (7)	38.5 41.2	8.0 8.0	483 517	12 19	49	28	0.12	18764
		2 (7)	39.9 41.2	8.0 8.0	500 517	41 45	49	14	2.2	
		2 (7)	41.2 38.5	8.0 8.0	517 483	45 47	49	7	3.2	
		2 (7)	39.9 39.9	8.0 8.0	500 500	47 49	49	1	3.1	
		2 (7)	39.9 39.9	8.0 8.0	500 500	47 49	49	(-1 h) (+1 h)	2.1 3.9	
Murchante, Navarra, Spain 2006 Hoja de roble	WG	2 (7)	39.9 40.2	4.0 4.0	1001 1012	18 19	49	30	1.1	18764
		2 (7)	39.5 39.9	4.0 4.0	996 1006	33 38	49	15	2.3	
		2 (7)	39.5 39.5	3.9 4.0	1004 1000	38 46-49	49	8	1.7	
		2 (6)	40.2 40.2	4.0 3.9	1015 1021	46-49 49	49	1	1.8	
		2 (7)	39.5 39.2	4.0 3.9	1015 1021	46-49 49	49	(-1 h) (+3 h)	1.5 2.2	
Boekend, Limburg, The Netherlands 2006 Ciriller	WG	2 (7)	39.9 41.2	8.0 8.0	500 517	12 19	49	28	0.098	18764
		2 (7)	41.2 39.9	8.0 8.0	517 500	41 45	49	14	2.3	
		2 (7)	41.2 41.2	8.0 8.0	517 517	45 47	49	7	3.0	
		2 (7)	41.2 39.9	8.0 8.0	517 500	47 49	49	1	4.1	
		2 (7)	41.2 38.5	8.0 8.0	517 483	47 49	49	(-1 h) (+1 h)	0.98 2.4	
Profitis, Thessaloniki, Greece 2006 Simson	WG	2 (7)	39.4 39.3	4.0 4.0	997 994	44 49	49	1	1.8	18764
Treviolo, Lombardia, Italy 2006 Vilmorin	WG	2 (7)	40.6 39.9	4.0 4.0	1020 997	12 14	49	28	0.065	18764
		2 (8)	38.8 39.2	4.0 4.0	969 986	16 19	49	14	2.0	
		2 (7)	38.8 39.9	4.0 4.0	973 997	19 49	49	7	3.3	

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS				
		2 (7)	40.6 40.2	4.0 4.0	1017 1013	49 49	49	1	8.0	
		2 (8)	39.5 39.5	4.0 4.0	988 984	49 49	49	(-1 h) (+2 h)	2.5 5.4	

A field trial program was conducted in 2005 at 28 locations in the United States. A 20 SC formulation was applied twice as a foliar broadcast spray at the rate of 112 g ai/ha/application to leafy vegetables when the crop was at growth stages BBCH 19 to 89. The applications were made at 3-day intervals with the last application occurring approximately 1 day before normal harvest. No surfactants or adjuvants were added to the applications. All samples were analysed within 237 days (7.8 months) of sampling.

USA 2005: An LC/MS/MS method (report number DuPont-13294) was used. For head lettuce, recoveries ranged from 89 to 104% with an average of 94 ± 5% (n = 6). For leaf lettuce, recoveries ranged from 88 to 114% with an average of 99 ± 10% (n = 6). For celery, recoveries ranged from 84 to 108% with an average of 91 ± 10% (n = 6). For spinach, recoveries ranged from 84 to 124% with an average of 98 ± 15% (n = 6).

Table 84 Residues for chlorantraniliprole in lettuce and spinach from USA trials

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS				
Lettuce										
North Rose, NY, USA 2005 Ithaca	SC	2 (3)	111 113	47 48	234 234	48 49	untrim trim 49	1	2.4 0.47	16571
Bradenton, FL, USA 2005 Ithaca	SC	2 (3)	111 109	24 24	459 452	Enlarging heads Heads	untrim	1	1.3	16571
Porterville, CA, USA 2005 Cannery Row	SC	2 (4)	115 112	37 35	314 316	47 48	untrim trim 49	1	0.43 0.043	16571
Madera, CA, USA 2005 Crown Fortune	SC	2 (3)	114 114	41 41	281 281	49 49	untrim trim 49	1	2.2 0.39	16571
Chico, CA, USA 2005 Cannery Row	SC	2 (4)	112 114	40 41	279 278	13 cm head 13-15 cm head	Untrim mature	1	0.012	16571
Glenn, CA, USA 2005 Cannery Row	SC	2 (4)	114 114	41 41	279 279	13 cm head 13-15 cm head	Untrim mature	1	< 0.01	16571
San Ardo, CA, USA 2005 Vandenberg	SC	2 (4)	111 113	35 36	317 312	45 45	45 45 45 46 47 49	-0 0 1 3 7 10	0.63 0.56 0.55 0.46 0.18 0.048	16571 untrim

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS				
Germansville, PA, USA 2005 Paris Island Tall	SC	2 (3)	118 116	36 36	328 326	Excellent vigour Excellent vegetative	Normal harvest Untrim	1	6.2	16571 Leaf lettuce
Bradenton, FL, USA 2005 New Red Fire	SC	2 (3)	110 111	24 24	453 456	19 Marketable leaf	Marketable Untrim	1	3.2	16571
Madera, CA, USA 2005 Waldmann's Green	SC	2 (4)	116 113	42 40	279 281	49 49	49 Untrim	1	3.9	16571
Porterville, CA, USA 2005 Red Fire	SC	2 (3)	113 114	37 37	303 305	87 89	49 Untrim	1	4.5	16571
Lakeport, CA, USA 2005 Waldmann's Dark Green	SC	2 (2)	110 111	29 30	374 374	49 49	mature Untrim	1	5.3	16571
San Luis Obispo, CA, USA 2005 Ocean Green	SC	2 (3)	116 116	28 28	413 417	48 49	49 Untrim	1	4.0	16571
Sanger, CA, USA 2005 Elisa	SC	2 (3)	113 115	45 47	250 246	49 49	49 Untrim	1	3.9	16571
Spinach										
Germansville, PA, USA 2005 Tyee	SC	2 (2)	116 118	36 36	327 331	Excellent vigour Excellent vigour	mature	1	6.8	16571
Bumpass, VA, USA 2005 Unipack 151	SC	2 (4)	114 112	59 59	193 189		49	1	8.6	16571
E. Bernard, TX, USA 2005 Melody	SC	2 (2)	110 111	46 46	237 239	49 49	49	1	7.4	16571
Jerome, ID, USA 2005 Russet Burbank	SC	2 (3)	112 111	46 45	246 246	47 48	48	1	5.6	16571
Madera, CA, USA 2005 Shasta	SC	2 (4)	114 115	41 41	281 281	49 49	49	1	8.9	16571
Hickman, CA, USA 2005 Shasta	SC	2 (2)	113 114	40 41	280 280	48 49	49	1	7.3	16571

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS				
San Ardo, CA, USA 2005 Amelia	SC	2 (4)	110 113	35 36	317 312	45 45	45 45 45 46 47 47	-0 0 1 3 7 10	0.77 3.7 3.4 3.1 2.4 2.3	16571

Table 85 Residues for chlorantraniliprole in mustard greens from USA trials (16570)

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Spray Additive
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS				
Sycamore, GA, USA 2005 Broad Leaf	SC	2 (2)	116 114	30 31	385 365	47 48	48	3	1.7	
Cheneyville, LA, USA 2005 Florida Broadleaf	SC	2 (4)	112 112	53 53	212 210	Mature Mature	Mature	3	4.6	Prime Oil (0.10%)
Delavan, WI, USA 2005 Giant Southern Curled	SC	2 (4)	116 113	52 49	222 229	48 49	49	3	1.2	X-77 (0.25%)
Arkansaw, WI, USA 2005 Florida Broadleaf India Mustard	SC	2 (3)	116 118	42 42	279 280	18 19	19	3	5.6	Induce (0.25%)
Thorndale, ON, Canada 2005 Florida Broadleaf	SC	2 (3)	113 116	56 58	201 200	17 19	19	3	2.9	Agral 90 (0.20%)
East Bernard, TX, USA 2005 Florida Broadleaf	SC	2 (3)	113 112	48 48	235 235	49 49	51	3	3.7 c< 0.01	Dyne-Amic (0.5%)
Hickman, CA, USA 2005 Florida Broadleaf	SC	2 (4)	113 114	40 41	280 280	48 49	51	3	4.8	Adjuvant (0.75%)
Abbotsford, BC, Canada 2005 Savanna	SC	2 (3)	113 111	41 41	276 273	15-16 15-16	15-17	3	2.2	

Trials on lettuce were conducted in Australia in 2006. At each site, two treated plots were established, one treated at the proposed GAP rate and a second treated at 2× the proposed GAP rate. A SC formulation was applied three times as a foliar broadcast spray at a target application rate of 30 and 60 g ai/. At both sites, applications were made with the addition of a non-ionic surfactant at 0.025%. The applications were made at targeted retreatment intervals of 7 days with the last application occurring approximately 3 days before normal commercial harvest. Samples were

analysed within 1 month of sample collection. Recovery values for untreated control samples fortified with 0.010 and 1.0 mg/kg chlorantraniliprole were 88.7-108% (n = 4).

Table 86 Residues for chlorantraniliprole in lettuce from Australian trials

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS				
Grantham, Queensland 2006 Titanic	SC+	3 (10 5)	30	6	500	¼ - ½ head	Mature	3	0.24	20921
			30	6	500	¾ head				
			30	6	500	Nearly fully formed				
			60	12	500	¼ - ½ head	Mature	3	0.33	
			60	12	500	¾ head				
			60	12	500	Nearly fully formed				
Lower Tenthill Creek, Queensland 2006 Titanic	SC+	3 (10 5)	30	6	500	¼ - ½ head	Mature	3	0.07	20921
			30	6	500	¾ head				
			30	6	500	Nearly fully formed				
			60	12	500	¼ - ½ head	Mature	3	0.19	
			60	12	500	¾ head				
			60	12	500	Nearly fully formed				

+ = Agral®, a non-ionic surfactant used at 0.025% v/v

Root and Tuber Vegetables

Potatoes

In the EU two trials were conducted in 2004 at two locations in Poland. An SC formulation was applied twice by foliar application at a target application rate of 10 g ai/ha. The second application occurred at growth stage BBCH 47, 21–22 days before the commercial harvest date. Trials were also conducted in 2005 and 2006 at one location in Spain, two in Greece, one in Germany, one location in Italy, one in Poland, and two in northern France. An SC formulation was applied twice by foliar application at the target rate of 12.5 g ai/ha. In 2005, the applications were made at 14 ± 1 day intervals. In 2006, the applications were made at 9–11-day intervals. In both growing seasons, the last application occurred 14–15 days before normal harvest. No surfactants or adjuvants were added to the applications. All samples were analysed within 6 months of sampling. Recovery values for samples fortified at 0.01–0.1 mg/kg were 73–105% (n = 16) for potato tubers.

Table 87 Residues for chlorantraniliprole in potatoes from European trials

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	GS				
Rozbity, Kamien, Poland 2004 Irga	WG	2 (14)	10.5	2.1	503	46	47	(-1 h)	< 0.01	14143
			10.5	2.1	503	47	47	(+1 h)	< 0.01	
							47	7	< 0.01	
							48	14	< 0.01	
							49	21	< 0.01	
							49	28	< 0.01	

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	GS				
Zawady, Poland 2004 Bila	WG	2 (13)	10.7 10.7	2.1 2.1	506 506	44 47	47 47 47 47-48 49 49	(-1 h) (+3 h) 7 14 22 28	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	14143
Olivares, Andalucia Spain 2005 Liseta	SC	2 (14)	12.3 12.7	2.1 2.1	594 606	43-45 45-47	49	14	< 0.01	16565
Chalkidona, Central Macedonia Greece 2005 Dailfa	SC	2 (14)	13.1 12.9	2.1 2.1	628 617	69 foliage 85 foliage	49 (tuber)	14	< 0.01	16565
Goch-Nierswalde, North Rhine-Westphalia, Germany 2005 Bintjie	SC	2 (14)	12.9 12.8	2.1 2.1	617 613	69 foliage 45-47 tuber	49 Tuber	14	< 0.01	16565
Allouagne, Nord pas de Calais France 2005 Amyla	SC	2 (14)	12.7 12.9	2.1 2.1	610 619	75 foliage 91 foliage	95-97 foliage	14	< 0.01	16565
Chalkidona, Central Macedonia, Greece 2006 Fabula	SC	2 (10)	12.7 12.8	2.1 2.1	607 611	41-42 42	49	14	< 0.01	18748
Allouagne, Nord Pas de Calais, France 2006 Amila	SC	2 (11)	12.7 12.3	2.1 2.1	607 590	47-48 47-48	49	14	< 0.01	18748
Tortona, Piemonte, Italy 2006 Monna Lisa	SC	2 (9)	12.3 12.9	2.1 2.1	593 617	39 48	49	15	< 0.01	18748
Rozbity Kamień, Podlasie, Poland 2006 Irga	SC	2 (9)	14.6 14.4	2.4 2.4	598 595	43-45 47-48	49	15	< 0.01	18748

Trials on potatoes were also conducted in North America. Trials conducted in 2004 at two locations in the United States used a WG formulation applied three times by foliar broadcast application at a target application rate of 50 g ai/ha/application when the crop was at growth stage BBCH 42–92. In 2005 trials were conducted at 27 locations in Canada and the United States with a WG formulation applied 3 times as a foliar broadcast spray at the rate of 75 g ai/ha/application to potato when the crop was at growth stage BBCH 33 to 95. The applications were made at 4 to 6-day intervals with the last application occurring approximately 14 days before normal harvest. No surfactants or adjuvants were added to the applications. All samples were analysed within 203 days (6.7 months) of sampling. Recoveries ranged from 68–91% with an average of 78 ± 8.4% (n = 8) for the 2004 trials. For USA/Canada 2005, concurrent recoveries from control samples fortified at 0.010

to 0.10 mg/kg of chlorantraniliprole ranged from 77 to 109% with an overall average of 94 ± 7% (n = 22).

Table 88 Residues for chlorantraniliprole in potatoes from Canada and USA trials

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS				
Paynesville, MN 2004 Red Pontiac	WG	3 (5 5)	49 49 49	26.2 26.2 26.2	187 187 187	42 45 47	Mature	-0 0 7 14 21 28	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	14149
Payette, ID 2004 Russett Burbank	WG	3 (5 6)	50 52 50	26.7 27.8 26.7	187 187 187	91 91 92	Mature	-1 0 7 15 21 28	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	14149
North Rose, NY, USA 2005 Castile	WG	3 (5 5)	76 75 76	32 32 32	234 234 234	47 47 48	49	14	< 0.01	16578
Germansville, PA, USA 2005 Andover	WG	3 (6 5)	78 77 77	45 44 44	173 173 173	Tuber bulking Tuber bulking Mature tubers	Mature	14	< 0.01	16578
Port Elgin, PE, Canada 2005 Norland	WG	3 (4 4)	75 78 76	22 22 21	351 358 352	Tuber bulking Tuber bulking 95	Mature	15	0.01	16578
Berwick, NS, Canada 2005 Superior	WG	3 (5 5)	74 74 74	23 23 23	326 326 325		Mature	14	< 0.01	16578
Canning, NS, Canada 2005 Norvalley	WG	3 (5 5)	74 73 74	23 22 23	326 325 325		Mature	14	< 0.01	16578
New Glasgow, PE, Canada 2005 Norland	WG	3 (4 4)	74 74 76	21 21 22	347 348 351	Tuber bulking Tuber bulking 95	Mature	14	< 0.01	16578
New Glasgow, PE, Canada 2005 Shepody	WG	3 (6 4)	75 76 74	21 22 21	350 353 348	Tuber bulking Tuber bulking 91	Mature	14	< 0.01	16578
Chula, GA, USA 2005 Red Pontiac	WG	3 (6 4)	75 75 75	55 58 61	138 129 123		Mature	14	< 0.01	16578
Bradenton, FL, USA 2005 Red Lasota	WG	3 (5 5)	76 78 75	16 16 16	471 479 466	Tuber enlargement	48-49	14	< 0.01	16578
Gardner, ND, USA 2005 Dakota Pearl	WG	3 (4 5)	78 78 78	42 42 42	187 187 187	44 44 45	48	15	< 0.01	16578

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS				
Arkansaw, WI, USA 2005 Russet Burbank	WG	3 (4 5)	76 76 76	41 41 41	187 187 187	47 47 47	48	14	< 0.01	16578
Marysville, OH, USA 2005 Red Lasoda	WG	3 (5 5)	74 77 74	36 37 35	208 210 210	91 91 91	99	15	< 0.01	16578
Paynesville, MN, USA 2005 Red Pontiac	WG	3 (5 5)	75 75 75	39 39 39	191 191 191	Maturing tubers Maturing tubers Mature tubers	Mature	14	< 0.01	16578
St-Paul D-Abbotsford, QC, Canada 2005 Chieftain	WG	3 (5 5)	74 74 76	53 32 32	139 234 239	33-34 45-46 47-48	Mature	14	< 0.01	16578
Taber, AB, Canada 2005 Russet	WG	3 (10 4)	73 75 74	49 50 50	149 150 149	65 69 69	69	15	< 0.01	16578
Jerome, ID, USA 2005 Russet Burbank	WG	3 (4 5)	74 74 75	34 35 35	218 212 213	47 47 48	49	14	< 0.01	16578
Fresno, CA, USA 2005 Red Lasoda	WG	3 (5 5)	76 76 75	41 41 41	186 185 184	48 48 48	49	14	< 0.01	16578
Ephrata, WA, USA 2005 Russet Burbank	WG	3 (5 5)	75 75 75	54 54 54	139 138 139	80% final mass 90% final mass 90% final mass	49	14	< 0.01	16578
Ephrata, WA, USA 2005 Russet Ranger	WG	3 (5 5)	75 75 75	33 33 33	227 227 226	80% final mass 90% final mass 90-100% final mass	48-49	14	< 0.01	16578
Madras, OR, USA 2005 Russet	WG	3 (5 6)	75 74 74	32 32 32	233 231 234	89 91 92	95	14	< 0.01	16578
Hermiston, OR, USA 2005 Russet Ranger	WG	3 (6 6)	74 74 75	31 32 31	236 232 239	43 45 47	99	14	< 0.01	16578
Payette, ID, USA 2005 Russet Burbank	WG	3 (5 6)	76 75 77	27 27 28	281 281 281	47 48 48	49	14	< 0.01	16578
Abbotsford, BC, Canada 2005 Russet Burbank	WG	3 (4 4)	75 75 78	50 27 27	152 276 283	68 69 69	91	14	< 0.01	16578

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS				
Josephburg, AB, Canada 2005 Norcoda	WG	3(4 4)	76 78 75	32 33 32	234 234 234	40-59 41-61 41-61	79-91	14	< 0.01	16578
Wellwood, MB, Canada 2005 Russet Burbank	WG	3 (4 6)	76 74 75	27 27 27	281 281 281	47 47 47	85	14	< 0.01	16578
North Rose, NY, Canada 2005 Castile	WG	3 (5 5)	75 76 74	32 32 32	234 234 234	47 47 48	48 48 48 48 49 49	-0 +0 3 7 14 21	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	16578
Paynesville, MN, USA 2005	WG	3 (5 5)	76 76 76	40 40 40	191 191 191	89 Mature Mature	Mature Mature Mature Mature Mature Mature	-0 +0 3 7 14 21	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	16578
	WG	3 (5 5)	376 380 376	197 199 197	191 191 191	Mature Mature Mature	Mature	14	< 0.01	5× processing

Stalk and stem vegetables

Celery

The field program was conducted in 2005 at 28 locations in the United States. 20SC formulation was applied twice as a foliar broadcast spray at the rate of 112 g ai/ha/application to leafy vegetables when the crop was at growth stage BBCH 19 to 89. The applications were made at 3-day intervals with the last application occurring approximately 1 day before normal harvest. No surfactants or adjuvants were added to the applications. All samples were analysed within 237 days (7.8 months) of sampling.

USA 2005. An LC/MS/MS method (report number DuPont-13294) was used. For head lettuce, recoveries ranged from 89 to 104% with an average of 94 ± 5% (n = 6). For leaf lettuce, recoveries ranged from 88 to 114% with an average of 99 ± 10% (n = 6). For celery, recoveries ranged from 84 to 108% with an average of 91 ± 10% (n = 6). For spinach, recoveries ranged from 84 to 124% with an average of 98 ± 15% (n = 6).

Table 89 Residues for chlorantraniliprole in celery from USA trials

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS				
Belle Glade, FL, USA 2005 AD52	SC	2 (2)	113 115	41 41	277 282	Mature mature	Mature Untrim	1	0.99	16571
Delavan, WI, USA 2005 Giant Pascal	SC	2 (3)	114 114	47 50	241 228	48 49	Untrim Trim mature	1	2.6 2.5	16571

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	Application GS				
Porterville, CA, USA 2005 Conquistador	SC	2 (3)	112 113	35 35	317 319	48 48	Untrim Trim 48	1	2.1 0.25	16571
Madera, CA, USA 2005 Salyor Sonora	SC	2 (3)	114 114	41 41	281 281	49 49	Untrim 49	1	3.6	16571
Hickman, CA, USA 2005 Conquistador	SC	2 (3)	114 114	35 35	326 326	49 49	Untrim Trim 49	1	2.1 0.19	16571
San Luis Obispo, CA, USA 2005 Conquistador	SC	2 (3)	116 118	28 28	417 420	48 49	Untrim 49	1	1.4	16571
Sanger, CA, USA 2005 Sonora	SC	2 (3)	114 115	32 33	356 350	49 49	49	1	3.6	16571

Cotton

Trials on cotton were conducted in the 2005–6 season at two sites in Australia. A WG formulation was applied three times as a foliar broadcast spray at application rates of 52.5 and 105 g ai/ha. A non-ionic surfactant was added to each application. The applications were made at retreatment intervals of 7–14 days with the last application occurring approximately 28 days before harvest. All samples were analysed within 2 months of harvest. Recovery values for untreated control cottonseed, trash, and lint samples fortified with 0.010 and 1.0 mg/kg chlorantraniliprole run concurrently with treated samples in all the trials were within 81.8–91.0% ($n = 6$). Samples were frozen within 3 h of harvest, and stored at –18 °C. Seed and lint samples were ginned approximately 6 weeks after harvest, then returned to freezer storage prior to transport to the laboratory two days later.

Table 90 Residues for chlorantraniliprole in cotton from Australian trials (20921)

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Comment
		No (interval)	g ai/ha	g ai/hL	L/ha	GS				
Kupunn, Queensland 2006 Sicot 80	WG +	3 (14 7)	52.5 52.5 52.5	42 42 42	125 125 125	5% terminal flowers 20-30% open 30% open	Mature	27	< 0.01	HHS, Hand picked
			105 105 105	84 84 84	125 125 125	5% terminal flowers 20-30% open 30% open	Mature	27	< 0.01	
Brookstead, Queensland, 2006 Sicot F1	WG +	3 (13 10)	52.5 52.5 52.5	42 42 42	125 125 125	90% terminal flowers 30% open 50% open	Mature	28	< 0.01	HHS, Hand picked
			105 105 105	84 84 84	125 125 125	90% terminal flowers 30% open 50% open	Mature	28	< 0.01	

+ = +Chemwett non-ionic surfactant at 0.125% v/v

Additional cotton trials were conducted in 2005 at 14 locations in the United States. A WG formulation was applied as a foliar broadcast spray at the rate of 112 g ai/ha/application to cotton when the crop was at growth stage BBCH 81 to 89. The applications were made at 5-day intervals with the last application occurring approximately 21 days before normal harvest. No surfactants or adjuvants were added to the applications. All samples were analysed within 135 days of sampling using an LC/MS/MS method (report number DuPont-13294). Recoveries ranged from 69–98% with an average of 84 ± 7% (n = 16). For undelinted cottonseed, recoveries ranged from 74–98% with an average of 85 ± 6% (n = 11). For cotton gin by-products, recoveries ranged from 69–87% with an average of 80 ± 8% (n = 5).

Table 91 Residues for Chlorantraniliprole in cotton from USA trials (16574)

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	GS				
Chula, GA, USA, 2005 DP555	WG	2 (5)	111 111	122 123	91 90	85 87	89	21	0.047	TMS, Mechanical picker
Newport, AR, USA, 2005 PM1218 BGRR	WG	2 (6)	111 113	118 120	94 94	87 89	89	21	0.082	TMS, Mechanical picker
Levelland, TX, USA, 2005 FM989 BR	WG	2 (4)	112 114	78 83	143 138	10% bolls open 15% bolls open	Mature	21	0.049	BPS, Mechanical stripper
Wolfforth, TX, USA, 2005 DP444	WG	2 (5)	110 109	77 77	142 141	10-20% bolls open 40% bolls open	Mature	21	0.13	BPS, Mechanical stripper
Edmonson, TX, USA, 2005 Fiber Max 958	WG	2 (6)	112 113	119 120	94 94	50% bolls open 60% bolls open	98% open	21	0.083	BPS, Mechanical stripper
Groom, TX, USA, 2005 Paymaster 2326	WG	2 (5)	111 110	123 121	90 91	83 84	99	22	0.054	BPS, Mechanical stripper
Tulare, CA, USA, 2005 Phytogen 710R	WG	2 (5)	113 114	106 104	107 110	81 84	89	22	0.081	TMS, Mechanical stripper
Washington, LA, USA, 2005 DPL 555	WG	2 (5)	113 111	75 66	150 167		Mature	21	0.022	HHS, Hand picked
Alexandria, LA, USA, 2005 DP434	WG	2 (5)	112 112	122 123	92 91	5-10% open 15-20% open	Mature	20	0.016	TMS, Hand picked
Pleasant Hill, NM, USA, 2005 Paymaster 2326RR	WG	2 (4)	111 112	87 90	127 124	5% open 5% open	90% open	22	0.031	HHS, Hand picked

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	GS				
Fresno, CA, USA, 2005 Acala Maxxa	WG	2 (5)	114 113	61 60	187 187	82 85	96	21	0.029	BPS, Hand picked
Tipton, CA, USA, 2005 DPX419	WG	2 (5)	112 113	80 81	140 140	84 85	89	23	< 0.01	BPS, Hand picked
Cheneyville, LA, USA, 2005 DP444 Bt/RR	WG	2 (5)	118 110	130 120	91 92	5-10% open 5-10% open	5-10% 5-10% 10-20% 20-30% 60-70% 90%	-0 0 7 14 21 28	0.041 0.078 0.061 0.029 0.011 c< 0.01 0.014	TMS, Hand picked
Uvalde, TX, USA, 2005 Deltapine 458	WG	2 (5)	112 110	80 79	140 140	87 87	87 87 87 89 89 89	-0 0 6 14 20 25	0.12 0.23 0.34 0.25 0.18 c< 0.01 0.21	TMS, Mechanical Picker

Seed cotton samples were collected using stripper equipment at 5 trial sites; picker equipment at three and were hand-picked at all other trial sites.

%moisture for cotton seed: Chula, GA/2005 12%; Newport, 11%; Levelland, TX/2005 16%; Wolfforth, TX/2005 10%; Edmonson, TX/2005 14%; Groom, TX/2005 14%; Tulare, CA/2005 12%; Washington, LA/2005 12%; Alexandria, LA/2005 15%; Pleasant Hill, NM/2005 13%; Fresno, CA/2005 16%; Tipton, CA/2005 14%; Cheneyville, LA/2005 14%; Uvalde, TX/2005 16%.

Tree nuts

Trials on almonds and pecans were conducted in 2006 at 12 locations in the United States. A WG formulation was applied twice as a foliar broadcast spray at the rate of 112 g ai/ha/application to almond and pecan when the crops were ranged in growth stage from initial drying, hull/shuck split, and BBCH 85 to drying, 10-day PHI, shuck split, and BBCH 96. No surfactants or adjuvants were added to the applications. The applications were made at 7-day intervals with the last application occurring approximately 10 days before normal harvest. All samples were analysed within 141 days of sampling. Concurrent recoveries fell within the range of acceptability specified by the protocol (70 to 120%). For almond nutmeat, recoveries ranged from 87 to 93% with an average of 91 ± 3% (n = 4). For almond hulls, recoveries ranged from 86 to 107% with an average of 97 ± 9% (n = 4). For pecan nutmeat, recoveries ranged from 89 to 101% with an average of 97± 6% (n = 4).

Table 92 Residues for Chlorantraniliprole in tree nuts from USA trials

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	GS				
Almond										
Kerman, CA, USA 2006 Nonpareil	WG	2 (7)	112 111	12 12	935 926	Initial drying Drying	Maturity	10	< 0.01	18803

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	GS				
Madera, CA, USA 2006 Nonpareil	WG	2 (7)	114 113	12 12	935 935	Drying Drying	Maturity	10	< 0.01	18803
Glenn, CA, USA 2006 Nonpareil	WG	2 (7)	112 112	12 12	935 935	Hull split	Maturity	10	< 0.01	18803
Terra Bella, CA, USA 2006 Pareil	WG	2 (7)	111 112	12 12	935 945	88 88	89	11	< 0.01	18803
Sanger, CA, USA 2006 Neplus	WG	2 (7)	112 112	12 12	945 935	85 85	89	10	< 0.01	18803
Sultana, CA, USA 2006 Carmel	WG	2 (7)	112 112	12 12	945 945	85 87	89	10	< 0.01	18803
Pecans										
Chula, GA, USA 2006 Sumner	WG	2 (7)	113 113	12 12	935 945	87 87	89	10	< 0.01	18803
Sycamore, GA, USA 2006 Sumner	WG	2 (7)	113 114	12 12	935 945	87 87	89	10	< 0.01	18803
Albany, GA, USA 2006 Sumner	WG	2 (7)	113 114	12 12	935 945	87 87	89	10	< 0.01	18803
Marked Tree, AR,USA 2006 Stuart	WG	2 (7)	113 112	12 11	935 992	95 96	Harvest	10	0.014	18803
Anton, TX, USA 2006 Western Schuley	WG	2 (7)	113 112	12 12	945 935	Shucks splitting Shucks split	Harvest	9	0.015	18803
D'Hanis, TX, USA 2006 Wichita	WG	2 (7)	114 112	13 13	879 870	85 85	89	10	< 0.01	18803

Almond hulls and nuts with shells were separated in the field and placed in separate composite piles. Hull samples were collected prior to the nutmeat samples. Hull samples were taken from the composite hull pile and placed in residue bags. In general, almond nuts and shells were then separated by hand; using disposable gloves; pecan nuts and shells were separated using commercially available crackers.

Animal Feed commodities

Table 93 Residues for chlorantraniliprole in cotton from USA trials (gin by-products) (16574)

Country	FL	Application				Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	L/ha	GS				
Chula, GA, USA, 2005	WG	2 (5)	111 111	91 90	85 87	89	21	12 c< 0.01	Mechanical picker
Newport, AR, USA, 2005	WG	2 (6)	111 113	94 94	87 89	89	21	6.4 c< 0.01	Mechanical picker

Country	FL	Application				Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	L/ha	GS				
Levelland, TX, USA, 2005	WG	2 (4)	112 114	143 138	10% bolls open 15% bolls open	Mature	21	3.3 c< 0.01	Mechanical stripper
Wolfforth, TX, USA, 2005	WG	2 (5)	110 109	142 141	10-20% bolls open 40% bolls open	Mature	21	4.1	Mechanical stripper
Edmonson, TX, USA, 2005	WG	2 (6)	112 113	94 94	50% bolls open 60% bolls open	98% open	21	2.4 c< 0.01	Mechanical stripper
Groom, TX, USA, 2005	WG	2 (5)	111 110	90 91	83 84	99	22	1.1	Mechanical stripper
Tulare, CA, USA, 2005	WG	2 (5)	113 114	107 110	81 84	89	22	13 c0.025	Mechanical stripper

%moisture: Chula, GA/2005 18%; Newport, AR/2005 12%; Levelland, TX/2005 42%; Wolfforth, TX/2005 23%; Edmonson, TX/2005 28%; Groom, TX/2005 28%; Tulare, CA/2005 18%

c – Control or untreated samples

Table 94 Residues for chlorantraniliprole in cotton from Australian trials (gin by-products) (20921)

Country	FL	Application				Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	L/ha	GS				
Kupunn, Queensland 2006 Sicot 80	WG +	3 (14 7)	52.5 52.5 52.5	125 125 125	5% terminal flowers 20-30% open 30% open	Mature	27	0.02	HHS, Hand picked
			105 105 105	125 125 125	5% terminal flowers 20-30% open 30% open	Mature	27	0.05	
Brookstead, Queensland, 2006 Sicot F1	WG +	3 (13 10)	52.5 52.5 52.5	125 125 125	90% terminal flowers 30% open 50% open	Mature	28	0.04	HHS, Hand picked
			105 105 105	125 125 125	90% terminal flowers 30% open 50% open	Mature	28	0.06	

+ = +Chemwett non-ionic surfactant at 0.125% v/v

Table 95 Residues for chlorantraniliprole in almond hulls from USA trials

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	GS				
Kerman, CA, USA 2006 Nonpareil	WG	2 (7)	112 111	12 12	935 926	Initial drying Drying	Maturity	10	0.88	18803

Country	FL	Application					Sample GS	PHI (days)	Residue (mg/kg)	Reference
		No (interval)	g ai/ha	g ai/hL	L/ha	GS				
Madera, CA, USA 2006 Nonpareil	WG	2 (7)	114 113	12 12	935 935	Drying Drying	Maturity	10	0.38	18803
Glenn, CA, USA 2006 Nonpareil	WG	2 (7)	112 112	12 12	935 935	Hull split	Maturity	10	0.59	18803
Terra Bella, CA, USA 2006 Pareil	WG	2 (7)	111 112	12 12	935 945	88 88	89	11	0.52	18803
Sanger, CA, USA 2006 Neplus	WG	2 (7)	112 112	12 12	945 935	85 85	89	10	1.6	18803
Sultana, CA, USA 2006 Carmel	WG	2 (7)	112 112	12 12	945 945	85 87	89	10	1.1	18803

Kerman, CA 22% moisture; Madera, CA 24%; Glenn, CA 23%; Terra Bella, CA 82%; Sanger, CA 24%; Sultana, CA 22%.

Fate of residues in storage and processing

Residues after Processing

Processing studies are necessary according to the uses and the residues of chlorantraniliprole on raw agricultural commodities. The fate of chlorantraniliprole residues during processing of raw agricultural commodities was investigated in several major registered crops (apples, plums, grapes and cottonseed) using important processing procedures.

As a measure for the transfer of residues into processed products, a transfer factor was used, which is defined as

$$TF = \frac{\text{Residue in processed product (mg/kg)}}{\text{Residue in raw agricultural commodity (mg/kg)}}$$

A concentration of residues takes place when TF > 1.

Chapleo (2004 12994) studied the effects of high temperature hydrolysis of residues of chlorantraniliprole under varying conditions. Solutions of two radiolabelled forms of chlorantraniliprole, [benzamide carbonyl-^{14}C]- and [pyrazole carbonyl-^{14}C]-chlorantraniliprole, were prepared in citrate buffer and subjected to hydrolysis at pH 4, 5 and 6 at high temperature. The conditions were selected to simulate hydrolysis under processing conditions and included:

The effect of pasteurisation (pH 4 and pasteurized at 90 °C for 20 minutes)

The effect of baking, brewing or boiling (pH 5 and baked at 100 °C for 60 minutes)

The effect of sterilisation (pH6 and autoclaving at 120 °C for 20 minutes)

Solutions of each radiolabel were prepared in 0.01M citrate buffer (pH 4, 5 and 6) at a nominal test concentration of 0.6 µg/mL. The pH ranged from 4.01–4.04 for the pH 4 samples, 5.03–5.05 for the pH 5 samples, and 5.97–6.02 for the pH 6 samples. The material balance of radioactivity throughout the study for the individual test systems was within the range of 98–107% of the added radioactivity (AR). The material balance of radioactivity following chromatographic analysis was within the range of 99–104% AR.

[^{14}C]chlorantraniliprole and its degradation products present in test solutions were identified by co-elution with authentic reference standards and quantified by reversed phase HPLC. The nature of the radioactivity and mass balance for each set of conditions are shown in Tables 96 and 97.

Table 96 Nature of residues in high temperature hydrolysis studies with [benzamide carbonyl-^{14}C]-chlorantraniliprole (% added radioactivity)

	pH 4; 90°C, 20 min.		pH 5.0; 100°C, 60 min.		pH 6.0; 120°C, 20 min.	
	Control*	Treated	Control*	Treated	Control*	Treated
Chlorantraniliprole	100	98	101	87	98	96
IN-EQW78	nd	0.58	nd	3.5	0.17	0.76
IN-ECD73	nd	1.2	0.31	11	0.68	1.6
Others [a]	nd	0.14	0.28	0.43	0.18	0.37
Equipment rinse [b]	0.84	0.35	0.51	0.69	0.21	0.44
Total recovery [c]	101	101	103	103	99	100

*control samples were not heated

nd = not detected

[a] Others = components not identified. Individual ^{14}C components detected by HPLC were less than 1% of the applied radioactivity.

[b] Equipment rinse = radioactivity not characterized.

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

Table 97 Nature of residues in high temperature hydrolysis studies with [pyrazole carbonyl-^{14}C]-chlorantraniliprole (% added radioactivity)

	pH 4; 90 °C, 20 min.		pH 5.0; 100 °C, 60 min.		pH 6.0; 120 °C, 20 min.	
	Control*	Treated	Control*	Treated	Control*	Treated
Chlorantraniliprole	100	99	103	86	102	96
IN-EQW78	nd	0.50	nd	2.8	nd	0.42
IN-ECD73	nd	1.3	0.44	14	0.69	2.9
Others [a]	0.15	0.22	nd	0.95	0.21	1.5
Equipment rinse [b]	0.37	0.44	0.65	0.41	0.36	0.47
Total recovery [c]	100	102	104	104	103	101

*control samples were not heated

nd = not detected

[a] Others = components not identified. Individual ^{14}C components detected by HPLC were less than 1% of the applied radioactivity.

[b] Equipment rinse = radioactivity not characterized.

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

Following heating of radiolabelled chlorantraniliprole at 90 °C for 20 minutes at pH 4 and baking at 120 °C for 20 minutes at pH 6, most of the applied radioactivity is recovered as parent compound, with 98% and 96% AR for the [benzamide carbonyl ^{14}C]-label and 99% and 96% AR for the [pyrazole carbonyl ^{14}C]-label.

With baking at 100 °C, for 60 minutes at pH 5, there is some degradation, with chlorantraniliprole accounting for 87% and 86% AR for the [benzamide carbonyl ^{14}C]- and [pyrazole carbonyl ^{14}C]-labelled compounds, respectively. The degradation products present include IN-F6L99 (13.6 % AR for the [pyrazole carbonyl ^{14}C]chlorantraniliprole), IN-ECD73 (11 % AR, [benzamide carbonyl ^{14}C]chlorantraniliprole) and IN-EQW78 (3.5 and 2.8 % AR [benzamide carbonyl ^{14}C]- and [pyrazole carbonyl ^{14}C]chlorantraniliprole, respectively). A proposed pathway for hydrolytic degradation is presented in Figure 7.

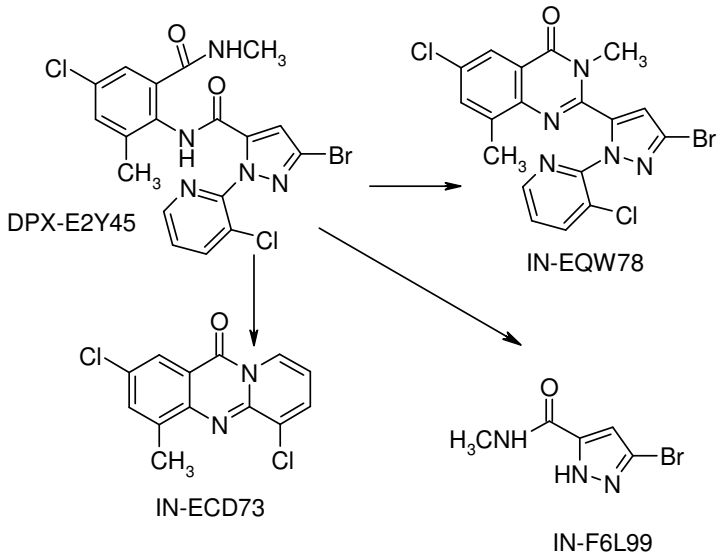


Figure 7 Proposed pathways for hydrolytic degradation of chlorantraniliprole

In summary, the data show that chlorantraniliprole is stable under processing conditions representative of pasteurization (90 °C for 20 minutes in pH 4 solution) and sterilization (120 °C for 20 minutes in pH 6 solution). During conditions representative of baking, brewing or boiling (100 °C for 60 minutes in pH 5 solution), a small amount of degradation of chlorantraniliprole led to the formation of IN-F6L99 (14 %AR), IN-ECD73 (11 %AR) and IN-EQW78 (2.8 -3.5%AR).

Apples

Foster *et al*. (2006 16587) studied the effect of processing (laboratory scale) on residues of chlorantraniliprole in apples. Apples with incurred residues were obtained from trials where trees were sprayed with two applications of chlorantraniliprole as an SC formulation at intervals of 13–15 days. Application rates were 4 g ai/hL with harvest 13–14 days after the last spray. Details of the trials are provided in Table 98.

Table 98 Details of field trials and residues in apples used for processing (Foster *et al*. 2006 16587)

Country	FL	No	g ai/ha	g ai/hL	GS Last appl'n	Sample	PHI (days)	Residue (mg/kg)
①Les Grand Chaux, Rhône-Alpes, France 2005 Pink Lady	SC	2 (14)	40 39	4 4	85	Fruit	14	0.026
②Kalkar, North Rhine, Westphalia, Germany 2005 Jonagold	SC	2 (13)	40 42	4 4	83-85	Fruit	14	0.027
③Chelmsford, Essex, UK 2005 Cox's Orange Pippin	SC	2 (15)	50 50	4 4	87	Fruit	13	0.081
④Lleida, Catalonya, Spain 2005 Golden Delicious	SC	2 (14)	60 61	4 4	83-89	Fruit	13	0.055

Preparation of washed fruit: The apples were with a constant spray of water at 0.5 L/kg apples.

Preparation of Apple Juice: The apples were crushed and the crushed fruit was pressed to separate raw juice and wet pomace. The wet pomace was dried at approximately 60 °C to produce dry pomace. Pectolytic enzymes (0.1 g/L) were added to the raw juice which was allowed to settle for at

least 12 h. The raw juice was racked to separate the juice from deposited solids and the clear juice filtered before pasteurisation at 85 °C for at least 60 seconds.

Preparation of Apple Sauce: The apples were blanched in boiling water (2 L/kg fruit) for 2 minutes and the blanched apples crushed and then sieved to separate the puree from the pips and peel (waste). Sugar was added to the puree and the volume reduced to obtain sauce with 24% Brix degree. The sauce was packaged into glass jars and sterilized by heating to 115–120 °C for 10 minutes.

Preparation of preserves/canned apples: The apples were peeled with a knife and the peeled apples blanched in boiling water for 2 minutes. The cores were removed and the fruit cut into pieces. A sugar syrup (200 g sugar: 800 g water, pH 3.5 adjusted with citric acid) was added in the ratio 2/3 apple to 1/3 syrup to prepare preserves and canned apples. Jars of preserves were pasteurized at 90 °C for at least 60 seconds while other jars were sterilized at 115–120 °C for 10 minutes to approximate canning.

Concurrent recoveries for samples of fruit, dry pomace and juice were within acceptable ranges for chlorantraniliprole and metabolites. Concurrent recoveries from apple fruit control specimens fortified at 0.010–0.10 mg/kg of chlorantraniliprole ranged from 75–97% (mean = 86 ± 8.5%, RSD = 10%, n = 6). Solid processed fractions (apple fruit, washed apple fruit, crushed apple, wet pomace, dried pomace, juice deposit, filter paper, blanched apple, puree, apple sauce, peeled apple, peels, peeled blanched apple, cores, peeled apple without cores, apple preserve and canned apple) and liquid processed fractions (washing water, raw juice, juice after filtration, apple juice and blanching water) were analysed by LC-MS/MS to determine residues of chlorantraniliprole, IN-EQW78, IN-ECD73, and IN-F6L99. Average recoveries using representative dry and liquid processed fraction matrices (dry pomace and apple juice) ranged from 80 ± 7.3%, RSD = 9.1 to 103 ± 13%, RSD = 13.

Table 99 Effect of processing on chlorantraniliprole (parent) residues in processed apple commodities

Matrix	①France		②Germany		③UK		④Spain	
	Residue (mg/kg)	PF	Residue (mg/kg)	PF	Residue (mg/kg)	PF	Residue (mg/kg)	PF
Fruit	0.026	-	0.027	-	0.081	-	0.055	-
Juicing								
Washed apple	0.049	1.9	0.032	1.2				
Wash water	0.007		0.009					
Crushed apple	0.098	3.8	0.034	1.2				
Raw juice	0.018	0.69	0.005	0.19				
Wet pomace	0.11	4.2	0.048	1.8	0.18	2.2	0.12	2.2
Dried pomace	0.32	12	0.35	13	0.75	9.3	0.60	11
Juice deposit	0.13	5.0	0.007	0.26				
Filter paper	0.003		0.00015					
Filtered juice	< 0.005	< 0.19	< 0.005	< 0.19				
Apple juice	< 0.005	< 0.19	< 0.005	< 0.19	< 0.005	< 0.06	< 0.005	< 0.09
Sauce								
Blanched apples	0.015	0.58	< 0.005	< 0.19				
Blanching water	0.016		0.009					
Crushed apples	0.013	0.50	0.011	0.41				
Purée	< 0.005	< 0.19	< 0.005	< 0.19	0.007	0.09	0.005	0.09
Waste	0.029		0.016					
Apple sauce	< 0.005	< 0.19	< 0.005	< 0.19	0.022	0.27	< 0.005	< 0.09
Preserves								
Peeled apples	0.014	0.54	0.009	0.33				

Matrix	①France		②Germany		③UK		④Spain	
	Residue (mg/kg)	PF	Residue (mg/kg)	PF	Residue (mg/kg)	PF	Residue (mg/kg)	PF
Peels	0.18	6.9	0.26	9.6				
Blanching water	< 0.004		< 0.005					
Peeled blanched apples	< 0.004	< 0.19	< 0.005	< 0.19				
Cores	0.010	0.38	< 0.005	< 0.19				
Peeled apples (no cores)	< 0.004	< 0.19	< 0.005	< 0.19				
Apple preserves	< 0.004	< 0.19	< 0.005	< 0.19	0.010	0.12	0.010	0.18
Canned apples	< 0.004	< 0.19	< 0.005	< 0.19	< 0.004	< 0.06	< 0.005	< 0.09

① Trial numbers correspond to site details in Table 98

Moisture in wet pomace ranged from 77–84%

Residues in washed apples were higher than in the RAC. As the samples are destroyed by analysis and the results are therefore for different batches of fruit the increases in residues are attributed to variability between batches of apples.

Plums

Carringer and Rodgers (2006 16591) studied the effect of processing plums on residues of chlorantraniliprole. Plum trees were treated with chlorantraniliprole (35WG formulation) as two foliar sprays at 112 g ai/ha (spray volume 954–963 L/ha) and at a 6 day interval. Harvest was 9 days after the last application.

The plums with incurred residues were processed (dried) to generate prunes. Prior to drying, the plum were sorted (leaves, stems, and other debris along with rotten or otherwise damaged fruit removed). The fresh fruit was washed with water for five minutes and the washed fruit placed on drying trays which were placed in a preheated Laboratory Tray Air Dryer at 74 °C. The trays were periodically removed from the dryer and weighed and the trays rotated to insure even drying. The fruit were dried for ~32 to 47 hours until the moisture content was 25.5–30.1%.

Samples were analysed by LC/MS/MS. For plum fruit, chlorantraniliprole recoveries were 75 and 77% with an overall average of 76 ± 1.5% (n = 2). For prunes, chlorantraniliprole recoveries ranged from 90 to 95% with an overall average of 92 ± 1.9% (n = 4); IN-EQW78 recoveries ranged from 88 to 92% with an overall average of 89 ± 2.5% (n = 4); IN-ECD73 recoveries ranged from 90 to 100% with an overall average of 95 ± 4.0% (n = 4); and IN-F6L99 recoveries ranged from 85 to 89% with an overall average of 87 ± 1.9% (n = 4). The LOQ and LOD were 0.010 and 0.003 mg/kg, respectively.

Mean residues of chlorantraniliprole found in replicate field samples of treated plums (stone/pit removed) and prunes (stone/pit removed) from the trial were 0.013 and 0.025 mg/kg, respectively. IN-EQW78, IN-ECD73, and IN-F6L99 were not detected (ND, < 0.003 mg/kg) in the prune samples. The processing/concentration factor (PF) for prunes was 1.9.

Table 100 Results of processing of plums on chlorantraniliprole (parent) residues (Carringer and Rodgers 2006 16591)

Country	FL	No	g ai/ha	L/ha	Last appl'n	Sample	PHI (days)	Residue (mg/kg)	PF
Dallas, OR,	WG	2	112	954	85	Fruit, stone removed	9	0.013	
USA 2005			112	963		Prune, with stone	-	0.022	-
Moyer						Prune, stone removed	-	0.025 [a]	1.9

[a] residue corrected for weight of stones estimated from similar fruit.

Grapes

Foster and Cairns (2005 14572) studied the effect of processing of grapes into wine on residues of chlorantraniliprole and metabolites. Red and white grape varieties were sprayed with a single application of chlorantraniliprole at 18 g ai/hL (90 g ai/ha) with harvest 43–44 days after application.

Red wine: Red grapes were crushed and stems removed. Samples of stems and must (crushed grapes) were collected. Potassium metabisulphite was added at 0.8 g/L to the must. Dry active yeast was added and the progress of the fermentation monitored by measuring the density, temperature and pH of the must. Wine was decanted from the solids, which were pressed to release the maximum amount of liquid. The solids (wet pomace) was dried at 60 °C to a constant weight to obtain dry pomace.

After inoculation with lactic bacteria (*Leuconostoc oenos*), monolactic fermentation of wine (anaerobic) was carried out in demijohns at room temperature. When complete, potassium metabisulphite was added at 0.8 g/L and solids allowed to settle. Samples of sediment (lees) were collected and the decanted wine clarified using dry gelatine (0.1 g/L and potassium metabisulphite (0.04 g/L). The wine was cooled to 5 °C, potassium metabisulphite added and filtered. Samples of the filtered wine were collected for analysis.

White wine: White wine grapes were crushed and pressed and a sample of the must collected. Pectolytic enzymes (0.02 g/L) and potassium metabisulphite (0.1 g/L) were added and the must decanted after 18 h of settling. Dry active yeast was added and the progress of the fermentation monitored by measuring the density, temperature and pH of the must. After racking, the wine was separated from the lees. After inoculation with lactic bacteria (*Leuconostoc oenos*), monolactic fermentation of wine (anaerobic) was carried out in demijohns at room temperature. When complete, potassium metabisulphite was added at 0.8 g/L and solids allowed to settle. Samples of sediment (lees) were collected and the decanted wine clarified using dry gelatine (0.1 g/L and potassium metabisulphite (0.04 g/L). The wine was cooled to 5 °C, potassium metabisulphite added and filtered. Samples of the filtered wine were collected for analysis.

Table 101 Recovery results for the method used for grape processing study

Matrix	analyte	nominal fortification range (mg/kg)	sample size (n)	mean recovery (%) ± std dev (%)	% relative standard deviation
Whole Grape Berries	chlorantraniliprole	0.010 - 0.50	6	96 ± 14	15
Dry Pomace	chlorantraniliprole	0.010 - 0.50	6	85 ± 16	19
	IN-EQW78	0.010 - 0.10	4	107 ± 20	19
	IN-ECD73	0.010 - 0.10	4	83 ± 6	8
	IN-F6L99	0.010 - 0.10	6	79 ± 13	17
Finished Wine	chlorantraniliprole	0.010 - 0.10	4	112 ± 14	12
	IN-EQW78	0.010 - 0.10	4	109 ± 11	10
	IN-ECD73	0.010 - 0.10	4	95 ± 10	11
	IN-F6L99	0.010 - 0.10	4	109 ± 10	9

Table 102 Results of processing of wine grapes on chlorantraniliprole (parent) residues (Foster and Cairns 2005 14572)

Country	FL	No	g ai/ha	g ai/hL	GS Last appl'n	Sample	PHI (days)	Residue (mg/kg)	PF
Languedoc-Roussillon, Gallician, France 2004 Grenache Noir	SC	1	90	18	81	Berries (red)	44	0.025	-
						Stems	-	0.17	6.8
						Must	-	0.038	1.5
						Wet pomace	-	0.089	3.6
						Dry pomace	-	0.30	12
						Alcoholic Fermentation Wine	-	0.022	0.88
						Malolactic Fermentation Wine	-	0.010	0.4
						Lees	-	0.036	1.4
						Finished red wine	-	0.019	0.76
Rhóne-Alpes, Tulette, France 2004 Ugni-Blanc	SC	1	89	18	81	Berries (white)	43	0.033	-
						Stems		0.14	4.2
						Must		0.014	0.42
						Wet pomace		0.059	1.8
						Dry pomace		0.20	6.1
						Must deposit		0.14	4.2
						Alcoholic Fermentation Wine		< 0.005	< 0.15
						Malolactic Fermentation Wine		< 0.005	< 0.15
						Finished white wine		< 0.005	< 0.15

Moisture contents for wet pomace were 68–72% (red) and 74% (white).

In a separate study Foster *et al.* (2006 16590) studied the concentration of chlorantraniliprole on processing of grapes to produce raisins, juice and wine. Table grapes were treated with 2 applications of an SC formulation of chlorantraniliprole at 8.7 or 3.5 g ai/hL (42 or 35 g ai/ha) and at 10–11 day intervals. Grape berries were harvested 3 days after the last application. Wine grapes (white and red) were treated with a single application of an SC formulation of chlorantraniliprole at 3.5 or 8.7 g ai/hL (35 or 52 g ai/ha) and harvested 43–45 days after application.

Table 103 Details of field trials and chlorantraniliprole (parent) residues in grapes used for processing (Foster *et al*. 2006 16590)

Country	FL	No	g ai/ha	g ai/hL	GS Last appl'n	Sample	PHI (days)	Residue (mg/kg)
①Serres, Provence-Alpes-Côte d'Azur, France 2005 Italia	SC	2 (11)	35 36	8.7 8.7	89	Fruit	3	0.055
②Lugagnano Val d'arda, Emilia Romagna, Italy 2005 Moscato d'Adda	SC	2 (10)	42 42	3.5 3.5	89	Fruit	3	0.083 (for juice) 0.096 (for raisins)
③Cormoyeux, Champagne-Ardenne, France 2005 Meunier	SC	1	35	8.7	77	Fruit	43	0.014 (for juice) 0.013 (for raisins) 0.017 (for wine)

Country	FL	No	g ai/ha	g ai/hL	GS Last appl'n	Sample	PHI (days)	Residue (mg/kg)
④Baldomar, Catalunya, Spain 2005 Cabenet (Franc)	SC	1	52	3.5	75	Fruit	45	0.036 (for juice) 0.062 (for raisins) 0.044 (for wine)

Samples from trials 3 and 4 were received in medium condition (presence of rot, trial 3, or due to length of shipment which was 2–3 days, trial 4).

Grape juice processing: Bunches were destemmed and manually crushed. After addition of pectolytic enzymes the destemmed crushed grapes were heated to 50 °C and maintained at 45–60 °C for 2 h. Raw juice was separated in a water press and a sample of wet pomace collected. The raw juice was clarified at 85 °C followed by cold storage (5–10 °C). After cold storage the juice was racked to obtain clear juice which was subsequently filtered (cellulose filter plate and/or 2.5 µm filter). The filtered juice was pasteurized for 1 minute at 85 °C.

Raisin preparation: Raisins were prepared by drying grapes in an oven at 60 °C for 3–4 days. The grapes were "worked over" each day and drying was continued until judged complete (visually). Raisins were manually destemmed.

White wine processing: Bunches were crushed and the stems discarded. The crushed grapes were pressed and the must recovered. After addition of pectolytic enzymes and potassium metabisulphite, the must was allowed to settle for 24 h and decanted. Alcoholic fermentation was initiated by the addition of yeast and the progress of fermentation monitored. As the alcohol content was insufficient for normal wine production, sugar was added to allow fermentation to a suitable content. After racking the wine was separated from the lees and bottled.

Monolactic fermentation was carried out in the absence of air in demijohns after in inoculation with lactic bacteria (*Leuconostoc oenos*). Due to difficulties with fermentation potassium bicarbonate was added to raise the pH and a second inoculation made. When the fermentation was complete 0.1 g/L potassium metabisulphite was added. After racking, the wine was separated from the lees and clarified by addition of gelatine and potassium metabisulphite and stored at 5 °C before decanting to remove sediment. Additional potassium metabisulphite was added to protect the wine from oxidation during filtration and the filtered wine bottled.

Red wine processing: Bunches of grapes were crushed and the stems discarded. The crushed grapes (must) were recovered in a large vat. After addition of potassium metabisulphite, alcoholic fermentation was initiated by the addition of yeast and the progress of fermentation monitored. When judged finished the wine was decanted from the solids and the solids pressed to recover as much wine as possible and the wine bottled.

Monolactic fermentation was carried out in the absence of air in demijohns after in inoculation of alcoholic fermentation wine with lactic bacteria (*Leuconostoc oenos*). When the fermentation was complete 0.1 g/L potassium metabisulphite was added. After racking, the wine was separated from the lees and clarified by addition of gelatine and potassium metabisulphite and stored at 5 °C before decanting to remove sediment. Additional potassium metabisulphite was added to protect the wine from oxidation during filtration and the filtered wine bottled.

Concurrent recoveries from whole berry control specimens fortified at 0.010–0.80 mg/kg of chlorantraniliprole ranged from 78–95% (mean = 86 ± 6.9%, RSD = 7.9%, n = 10). Solid processed fractions (must, must deposit, wet and dry pomace, filter papers, juice deposit, dried grapes and raisins) and liquid processed fractions (alcoholic fermentation wine, malolactic fermentation wine, lees, finished wine, raw juice, clear juice, juice after filtration and finished juice) were analysed by LC-MS/MS to determine residues of chlorantraniliprole, IN-EQW78, IN-ECD73, and IN-F6L99. Average recoveries using representative dry and liquid processed fraction matrices (dry pomace and finished wine) ranged from 88 ± 11%, RSD = 13 to 112 ± 18%, RSD = 16.

Table 104 Results of processing grapes on chlorantraniliprole (parent) residues for trials 1 and 2 (Foster *et al*. 2006 16590)

Matrix	①France		②Italy	
	Residue (mg/kg)	PF	Residue (mg/kg)	PF
Juicing				
Whole berries, prior to processing to juice	0.055	-	0.083	-
Stems/juice processing	0.49	8.9		
Raw juice	0.095	1.7		
Wet pomace/juice processing	0.18	3.3		
Clear juice	0.11	2.0		
Deposit/juice processing	0.15	2.7		
Filter paper	0.018	0.32		
Juice after filtration	0.11	2.0		
Juice	0.092	1.7	0.038	0.46
Dried Grape (inc. stems)/raisin processing				
Whole berries, prior to processing to raisins	0.070	-	0.096	-
Raisin	0.50	7.1	0.28	2.9

Table 105 Results of processing grapes on chlorantraniliprole (parent) residues for trials 3 and 4 (Foster *et al*. 2006 16590)

Matrix	③France		④Spain	
	Residue (mg/kg)	PF	Residue (mg/kg)	PF
Juicing				
Whole berries, prior to processing to juice	0.014	-	0.036	-
Stems/juice processing			0.64	18
Raw juice			0.050	1.4
Wet pomace/juice processing			0.027	0.75
Clear juice			0.037	1.0
Deposit juice			0.049	
Filter paper			0.0017 mg/total paper	
Filtered juice			0.033	0.92
Juice	0.006	0.43	0.035	1.0
Dried Grape (inc. stems)/raisin processing				
Whole berries, prior to processing to raisins	0.013	-	0.062	-
Dried stems			2.9	
Raisins	0.035	2.7	0.25	4.0
Wine making				
Whole berries, prior to processing to wine	0.017	-	0.044	-
Alcoholic fermentation wine	< 0.005	< 0.29	0.068	1.5
Malolactic fermentation wine	< 0.005	< 0.29	0.078	1.8
Finished white wine	< 0.005	< 0.29		
Finished red wine			0.072	1.6

Tomatoes

A processing study was conducted to determine if residues would concentrate in tomato processed products following treatment with chlorantraniliprole (Foster *et al*. 2006 16588). Tomatoes with incurred residues were obtained from trials where plants were sprayed with two applications of chlorantraniliprole as a WG formulation at 7 day intervals. Application rates were 3.5 g ai/hL (35 g ai/ha) with harvest 1day after the last spray. The tomatoes were harvested without their calyxes. Details of the trials are provided in Table 106.

Table 106 Details of field trials and residues in tomatoes used in further processing (Foster *et al*. 2006 16588)

Country	FL	No	g ai/ha	g ai/hL	GS Last appl'n	Sample	PHI (days)	Residue (mg/kg)
①Vaunaveys la Rochette, Rhône-Alpes France 2005 Leader	WG	2 (7)	36 34	3.5 3.5	89	Fruit	1	0.037
②San Donato Milanese, Lombardia, Italy 2005 Pavia	WG	2 (7)	34 35	3.5 3.5	87	Fruit	1	0.018
③Trobal, Sevilla, Andalucia Spain 2005 Juncal	WG	2 (7)	35 35	3.5 3.5	86	Fruit	1	0.018
④Tudela, Navarre, Spain 2005 Talen	WG	2 (7)	35 35	3.5 3.5	83-89	Fruit	1	0.035

Preparation of washed fruit: The tomatoes were washed with a constant spray of water at 0.5 L/kg tomatoes.

Preparation of tomato juice: Tomatoes were crushed and sieved to separate juice from seeds and peels (wet pomace). Salt was added to the juice at 7 g/kg and the pH adjusted with citric acid to 3.5 if required. The juice was transferred to glass jars, sealed and pasteurized at 82–85 °C for one minute.

Preparation of tomato puree: The tomatoes were crushed and the crushed tomatoes put in a double jacketed saucepan for reduction in the volume. The reduction was stopped when the Brix degree reached 12–14%. The reduced puree was sieved to remove peels and seeds (waste), salt added at 4 g/kg and the pH adjusted with citric acid to 3.5. The puree was packaged in glass jars and the sealed jars sterilized at 115 °C for at least 10 minutes.

Preparation canned tomatoes: The tomatoes were peeled by placing in boiling water (1 L/kg fruit) for 1 minute and plunged into cold water to split the skins. The peel was removed with the aid of a knife. Peeled tomatoes and juice in proportions 2/3 tomatoes to 1/3 juice were added to glass jars and sealed before sterilizing at 115 °C for at least 10 minutes.

Preparation of paste: Tomatoes were crushed and the crushed tomatoes concentrated using a double jacket saucepan to remove moisture. Reduction was stopped when the measured Brix degree reached 24–25%. The reduced tomatoes were sieved to separate juice from seeds and peels (waste). Salt was added to the puree at 4 g/kg and the pH adjusted to 3.5 with citric acid before transferring to glass jars, sealing and sterilizing at 115 °C for at least 10 minutes.

Table 107 Residues of chlorantraniliprole (parent) in tomatoes and processed commodities, trials 1 and 2

Matrix	①France				②Italy			
	chlorantraniliprole	IN-EQW78	IN-ECD73	IN-F6L99	chlorantraniliprole	IN-EQW78	IN-ECD73	IN-F6L99
Fruit	0.037				0.018			
washed tomatoes	0.014	nd	nd	nd	< 0.01	nd	nd	nd

Matrix	①France				②Italy			
	chlorantraniliprole	IN-EQW78	IN-ECD73	IN-F6L99	chlorantraniliprole	IN-EQW78	IN-ECD73	IN-F6L99
washing water	0.017	nd	nd	nd	0.010	nd	nd	nd
peeled tomatoes	nd	nd	nd	nd	Nd	nd	nd	nd
Peels	0.25	nd	nd	nd	0.50	nd	nd	nd
Purée								
crushed tomatoes	0.071	nd	nd	nd	0.023	nd	nd	nd
reduced tomatoes	0.20	0.013	0.015	0.010	0.044	< 0.01	< 0.01	nd
waste sieving	0.36	0.022	0.022	0.011	0.057	< 0.01	< 0.01	nd
raw purée	0.079	< 0.01	< 0.01	< 0.01	0.026	nd	nd	nd
Purée	0.055	< 0.01	0.012	0.012	0.030	nd	nd	nd
Canning								
peeled tomatoes`	< 0.01				< 0.01			
Peels	0.25				0.50			
blanched tomatoes	0.012	nd	nd	nd	< 0.01	nd	nd	nd
blanching water	0.015	nd	nd	nd	0.016	nd	nd	nd
cooling water	< 0.01	nd	nd	nd	< 0.01	nd	nd	nd
canned tomatoes	0.024	nd	nd	nd	< 0.01	nd	nd	nd
Juicing								
crushed tomatoes	0.037	nd	nd	nd	0.022	nd	nd	nd
wet pomace	0.050	nd	nd	nd	0.021	nd	nd	nd
raw juice	0.026	nd	nd	nd	0.019	nd	nd	nd
Juice	0.042	nd	nd	nd	0.016	nd	nd	nd
Paste								
crushed tomatoes	0.065	nd	nd	nd	0.023	nd	nd	nd
reduced tomatoes	0.22	0.015	0.017	0.012	0.068	0.013	0.013	< 0.01
waste (sieving)	0.36	0.028	0.029	0.012	0.057	< 0.01	0.011	< 0.01
raw paste	0.065	0.010	0.014	0.013	0.030	< 0.01	< 0.01	< 0.01
Paste	0.075	0.012	0.016	0.015	0.043	< 0.01	< 0.01	< 0.01
Ketchup								
crushed tomatoes	0.037	nd	nd	nd	0.025	nd	nd	nd
reduced tomatoes	0.10	0.013	0.017	0.011	0.055	< 0.01	< 0.01	nd
waste (sieving)	0.16	0.025	0.028	0.012	0.072	0.010	< 0.01	nd
raw purée	0.064	0.008	0.012	0.011	0.019	nd	nd	nd
Ketchup	0.043	0.010	0.012	0.011	0.028	nd	nd	nd

Table 108 Residues of chlorantraniliprole (parent) in tomatoes and processed commodities, trials 3 and 4

Matrix	③Spain				④Spain			
	chlorantraniliprole	IN-EQW78	IN-ECD73	IN-F6L99	chlorantraniliprole	IN-EQW78	IN-ECD73	IN-F6L99
Fruit	0.018				0.035			
Purée	0.022	nd	nd	nd	0.050	nd	< 0.01	< 0.01
canned tomatoes	0.006	nd	nd	nd	0.008	nd	nd	nd
Juice	0.014	nd	nd	nd	0.020	nd	nd	nd
Paste	0.011	nd	nd	< 0.01	0.037	< 0.01	0.010	0.014
ketchup	0.013	nd	nd	nd	0.026	nd	nd	< 0.01

Chlorantraniliprole was the major component of the residue in the processed commodities. Processing factors were calculated solely for residues of chlorantraniliprole.

Table 109 Summary of processing factors for tomatoes

Matrix	①France	②Italy	③Spain	④Spain
Washed tomatoes	0.38	0.39		
Wet pomace	1.4	1.2		
Purée	1.5	1.7	1.2	1.4
Canned tomatoes	0.65	< 0.28	0.33	0.23
Juice	1.1	0.89	0.78	0.57
Paste	2.0	2.4	0.61	1.1
Ketchup	1.2	1.6	0.72	0.74

Cotton

Rice and Rodgers (2006 16589) studied the effect of processing of cotton seed on residues of chlorantraniliprole residues in cotton seed processed commodities. Chlorantraniliprole was applied a 2 applications of a WG formulation to cotton plants at 5 day intervals, Uvalde, Texas USA. The application rate was 225 g ai/ha (140 L/ha). Seed was harvested 21 days after the last application using a mechanical picker and ginned to produce undelinted cotton seed which was processed to produce refined oil, meal and hulls.

Cotton seed (11–15% remaining lint) was delinted, approximately 3% lint remaining. The seed was mechanically cracked and screened to separate most of the hulls from kernels. If moisture of kernels is greater than 12% the kernels are dried at 54–71 °C. After heating at 79–91 °C for 15–30 minutes, the kernel material was flaked with a flaking roll (0.008–0.013”). The flaked material was passed through an expander/extruder and steam injected to produce collets which were dried at 66–82 °C for 30–40 minutes. The collets were extracted three times with hexane at 49–60 °C. The miscella (crude oil and hexane) were passed through a recovery unit to separate the crude oil and hexane. The crude oil was heated to 73–90 °C to remove hexane. The crude oil was alkali refined and after miscella refining, the refined oil separated from the soapstock.

Chlorantraniliprole recoveries in cottonseed were 83 and 84% with an average of 83 ± 1% (n = 2). For refined oil, recoveries ranged from 81–100% with an average of 91 ± 8% (n = 4). For meal, recoveries ranged from 84–108% with an average of 93 ± 11% (n = 4). For hulls, recoveries ranged from 84–97% with an average of 92 ± 6% (n = 4). IN-EQW78 recoveries in refined oil ranged from 74–96% with an average of 85 ± 9% (n = 4). For meal, recoveries ranged from 79–107% with an average of 92 ± 12% (n = 4). For hulls, recoveries ranged from 77–110% with an average of 91 ± 15% (n = 4). IN-ECD73 recoveries in refined oil ranged from 81–94% with an average of 87 ± 5%

(n = 4). For meal, recoveries ranged from 85–92% with an average of 89 ± 4% (n = 4). For hulls, recoveries ranged from 87–95% with an average of 92 ± 4% (n = 4). IN-F6L99 recoveries in refined oil ranged from 89-92% with an average of 91 ± 1% (n = 4). For meal, recoveries ranged from 75–92% with an average of 84 ± 8% (n = 4). For hulls, recoveries ranged from 67–75% with an average of 71 ± 4% (n = 4).

Table 110 Results of processing cotton seed on chlorantraniliprole (parent) residues (Rice and Rodgers 2006 16589)

Country	FL	No	g ai/ha	l/ha		Sample	PHI (days)	Residue (mg/kg)	PF
Uvalde, Texas, USA 2005 DPL 458	WG	2 (5)	219	140	85	seed	21	0.016	-
			227	140	87	Refined oil		< 0.01	< 0.25
						Meal		0.012	0.75
						Hulls		0.033	2.1

The results of processing studies are summarised in the table below.

Table 111 Summary of processing factors for chlorantraniliprole residues

Raw agricultural commodity (RAC)	Processed commodity	Calculated processing factors	PF (Mean, median or best estimate)	RAC-STMR (mg/kg)	STMR-P(mg/kg)
Apple	Pomace, wet	1.8 2.2 2.2 4.2	2.2	0.07	0.154
	Pomace, dry	9.3 11 12 13	11.5		0.805
	Juice	< 0.06 < 0.09 < 0.19 < 0.19	< 0.14		< 0.0098
	Purée	0.09 0.09 < 0.19 < 0.19	0.09		< 0.0063
	Sauce	< 0.09 < 0.19 < 0.19 0.27	0.27		0.0189
	Peeled	0.33 0.54	0.435		0.0304
	Peel	6.9 9.6	8.25		0.578
	Preserves, canned	< 0.06 < 0.09 < 0.19 < 0.19	< 0.14		< 0.0098
Plum	Prune	1.9	1.9	0.015	0.0285
Grape	Pomace wet	0.75 1.8 3.3 3.6	2.55	0.119	0.303
	Pomace dry	6.1 12	9		1.07
	Juice	0.43 0.46 1.0 1.7	0.73		0.0869
	Raisin	2.7 2.9 4.0 7.1	3.45		0.411
	Wine	< 0.15 < 0.29 0.76 1.6	0.525		0.0625
Tomato	Washed tomatoes	0.38 0.39	0.385	0.0705	0.0271
	Canned tomatoes	< 0.2 0.23 0.33 0.65	0.28		0.0197
	Juice	0.57 0.78 0.89 1.1	0.835		0.0589
	Ketchup	0.72 0.74 1.2 1.6	0.98		0.0691
	Purée	1.2 1.4 1.5 1.7	1.45		0.102
	Paste	0.61 1.1 2.0 2.4	1.55		0.109
	Pomace, wet	1.2 1.4	1.3		0.0916
Cotton	Hulls	2.1	2.1	0.049	0.103
	Meal	0.75	0.75		0.0368
	Oil, refined	0.25	0.25		0.0122

Residues in Animal Commodities

Fraser and McLellan (2006, 17817) dosed lactating Holstein/Friesian dairy cows (average daily weights for dose groups 550-750 kg, average 12–22 kg milk/day) with chlorantraniliprole at levels corresponding to the equivalent of 1, 3, 10 and 50 ppm in the diet based on actual feed consumption and doses. The animals were given hay and water *ad libitum* in addition to a protein concentrate ration, which was given twice a day at milking (8 kg ration/day).

Table 112 Description of dosing regime per treatment group for lactating cows

Target dose (ppm) [a]	Administered dose (mg/cow/day)	Feed consumption (kg/cow/day)	Equivalent residue level in feed (ppm) [b]
1	17.8–20.1	16.6–19.4	0.93–1.18
3	52–57.1	16.3–18.8	2.95– .40
10	175.2–188.5	16.5–18.2	9.61–11.2
50	843–945.1	16.6–18.2	47.9–53.0
50 (depuration)	840.6–935.1	16.6–18.2	48.0–53.1

[a] Dose stability was confirmed by analysis of 3 capsules at each dose level on the day of preparation. The remaining capsules were stored at −20 °C and analysed after 24 hours and 10 days. The 10 days storage and analysis was sufficient to cover the longest period from preparation to last dosing of a treatment group. Recoveries of chlorantraniliprole over the 10 day storage period ranged from 102 to 122%.

[b] Expressed on a dry weight basis.

Milk was collected twice daily (AM and PM), and samples of milk from the afternoon milking (PM) were combined with samples from the next morning milking (AM), to make a sample for a single day. Milk samples were taken on days 1, 3, 5, 7, 10, 14, 17, 21, 24, and 28 days; all samples except those from days 17 and 24 were analysed. Samples of skim milk and cream were prepared from milk collected on days 14 and 21, and were analysed separately. Cream and skim milk were prepared using a Lehmans' cream separator (centrifugation). Milk samples from the depuration group of animals were taken on the days indicated above, and also on days 1, 3, 5, 7, 10 and 14 days after cessation of dosing. Only milk samples from days 1, 3, 5 and 7 after cessation of dosing were analysed.

Cows were sacrificed 23–24 h after the last morning dose, except for the animals in the depuration group, which were sacrificed on days 9 and 23 after cessation of dosing. One of the control animals was not sacrificed.

All samples were stored frozen until analysis. The maximum frozen storage intervals were 20 days for milk, 57 days for skim milk, 34 days for cream, and 87 days for all tissues (liver, kidney, muscle, and fat were 83, 80, 87 and 76 days, respectively).

Residues of chlorantraniliprole and metabolites IN-HXH44, IN-K9T00, IN-EQW78, and IN-GAZ70 were determined in all samples, using method DuPont-11376 with modifications (the modified method was validated as DuPont-18100).

Table 113 Summary of concurrent recoveries of chlorantraniliprole, IN-K9T00, IN-HXH44, IN-GAZ70, and IN-EQW78 from animal matrices

					Recoveries (%)		
			chlorantraniliprole	IN-K9T00	IN-HXH44	IN-GAZ70	IN-EQW78
Matrix	Fort Level (mg/kg)	N	Mean ± SD	Mean ± SD	Mean ± SD	Mean ± SD	Mean ± SD
Milk	0.01	19-20	97.1 ± 10.6	95.3 ± 16.8	102 ± 10.5	90.8 ± 10.5	89.8 ± 11.0
	0.1	19–20	100 ± 13.2	99.6 ± 14.2	106 ± 6.9	90.0 ± 12.8	88.5 ± 11.9
	Overall	38–40	98.6 ± 11.9	97.4 ± 15.5	104 ± 9.0	90.4 ± 11.6	89.2 ± 11.3

					Recoveries (%)		
			chlorantraniliprole	IN-K9T00	IN-HXH44	IN-GAZ70	IN-EQW78
Matrix	Fort Level (mg/kg)	N	Mean ± SD	Mean ± SD	Mean ± SD	Mean ± SD	Mean ± SD
Skim Milk	0.01	4–6	84.4 ± 19.4	94.6 ± 8.2	100 ± 12.0	72.1 ± 9.7	93.0 ± 6.8
	0.1	4–6	91.1 ± 16.0	101 ± 10.4	91.0 ± 14.7	72.2 ± 11.0	87.5 ± 18.5
	Overall	8–12	87.8 ± 16.8	98.0 ± 9.6	95.5 ± 13.3	72.1 ± 9.6	90.3 ± 13.2
Cream	0.01	4–5	99.0 ± 11.1	93.6 ± 7.8	106 ± 9.4	92.5 ± 8.9	80.6 ± 7.7
	0.1	4–5	100 ± 5.9	97.5 ± 5.3	104 ± 3.9	88.5 ± 5.5	88.3 ± 10.0
	Overall	8–10	99.7 ± 8.4	95.6 ± 6.5	105 ± 6.7	90.5 ± 7.2	84.5 ± 9.3
Liver	0.01	2	81.6	89.6	81.2	73.9	92.4
	0.1	2	81.7	77.4	89.4	66.9	76.7
	Overall	4	81.7 ± 11.9	83.5 ± 7.5	85.3 ± 4.9	70.4 ± 7.8	84.5 ± 9.1
Kidney	0.01	2	89.5	77.2	85.8	75.7	82.1
	0.1	2	81.7	77.4	83.1	72.8	83.9
	Overall	4	85.6 ± 8.6	77.3 ± 12.6	84.4 ± 8.1	74.2 ± 9.9	83.0 ± 6.6
Muscle	0.01	2	76.7	69.6	86.2	82.1	104
	0.1	2	79.1	72.9	93.8	74.0	73.0
	Overall	4	77.9 ± 14.4	71.2 ± 9.3	90.0 ± 10.6	78.1 ± 6.0	88.3 ± 20.5**
Fat	0.01	4	88.3	79.1	93.5	87.7	84.7
	0.1	3	95.4	83.9	93.1	78.3	83.5
	Overall	7	91.3 ± 10.2	81.1 ± 10.3	93.3 ± 4.5	83.7 ± 10.5	84.2 ± 9.4

*SD not calculated for sample sizes < 3.

** = The relative standard deviation, CV, exceeds 20%, however this is not expected to have any effect on the results since all residues were ND (< 0.003 mg/kg) for IN-EQW78 in muscle.

Table 114 Residues of chlorantraniliprole and metabolites in milk from lactating cows dosed with chlorantraniliprole.

Feeding level (ppm)	Dosing days	Residue (mg/kg)		
		chlorantraniliprole	IN-K9T00	IN-HXH44
3	1	< 0.01	< 0.01	< 0.01
	3	< 0.01	< 0.01	< 0.01
	5	< 0.01	< 0.01	< 0.01
	7	< 0.01	< 0.01	< 0.01
	10	< 0.01	< 0.01	< 0.01
	14	< 0.01	< 0.01	< 0.01
	21	< 0.01	< 0.01	< 0.01
	28	< 0.01	< 0.01	< 0.01
10	1	< 0.01	< 0.01	< 0.01
	3	< 0.01	< 0.01	0.011
	5	< 0.01	< 0.01	0.010
	7	< 0.01	< 0.01	0.013
	10	< 0.01	< 0.01	0.013
	14	< 0.01	< 0.01	0.011
	21	< 0.01	< 0.01	0.011

Feeding level (ppm)	Dosing days	Residue (mg/kg)		
		chlorantraniliprole	IN-K9T00	IN-HXH44
	28	< 0.01	< 0.01	0.013
50	1	< 0.01	< 0.01	0.010
		< 0.01	< 0.01	0.015
	3	0.021	< 0.01	0.029
		0.020	0.011	0.035
	5	0.024	< 0.01	0.025
		0.020	< 0.01	0.031
	7	0.027	0.012	0.030
		0.027	0.013	0.043
	10	0.020	0.013	0.029
		0.024	0.014	0.039
	14	0.024	0.011	0.027
		0.028	0.011	0.039
	21	0.016	0.009	0.026
		0.018	0.012	0.038
	28	0.017	0.011	0.029
		0.021	0.013	0.045
	+1	0.012	< 0.01	0.028
	+3	< 0.01	< 0.01	< 0.01
	+5	< 0.01	< 0.01	< 0.01
	+7	< 0.01	< 0.01	< 0.01

Based upon the mean daily intake, the actual mean weekly dose levels were equivalent to 1.0–1.1 mg/kg feed, 3.0-3.2 mg/kg feed, 10–11 mg/kg feed, and 49–52 mg/kg feed.

The LOQ for each analyte in all matrices was 0.01 mg/kg; the LOD was 0.003 mg/kg.

Residues of chlorantraniliprole, IN-K9T00, and IN-HXH44 plateaued in whole milk within 7 to 10 days of dosing. After 10 days, residues of IN-HXH44 were highest in milk followed by (in decreasing order) chlorantraniliprole and IN-K9T00. Residues detected in whole milk were dose dependent. There were no detectable residues (< 0.003 mg/kg) of IN-GAZ70 or IN EQW78 in whole milk, cream, or skim milk in any dose group.

Table 115 Residue data for cream and skim milk from dairy cow feeding study with chlorantraniliprole.

Day	Feed level (ppm)	Average Residues (mg/kg)		
		chlorantraniliprole	IN-K9T00	IN-HXH44
Skim milk				
14	1	< 0.01	< 0.01	< 0.01
	3	< 0.01	< 0.01	< 0.01
	10	< 0.01	< 0.01	0.012
	50	0.018	0.012	0.030
21	1	< 0.01	< 0.01	< 0.01
	3	< 0.01	< 0.01	< 0.01
	10	< 0.01	< 0.01	0.012
	50	0.013	0.011	0.026

Day	Feed level (ppm)	Average Residues (mg/kg)		
		chlorantraniliprole	IN-K9T00	IN-HXH44
Cream				
14	1	< 0.01	< 0.01	< 0.01
	3	< 0.01	< 0.01	< 0.01
	10	0.027	< 0.01	0.015
	50	0.13	< 0.01	0.035
21	1	< 0.01	< 0.01	< 0.01
	3	0.015	< 0.01	< 0.01
	10	0.025	< 0.01	0.010
	50	0.086	< 0.01	0.020

Based upon the mean daily intake, the actual mean weekly dose levels were equivalent to 0.998–1.066 mg/kg feed, 3.034-3.164 mg/kg feed, 10.069–10.599 mg/kg feed, and 49.459–51.783 mg/kg feed.

The LOQ for each analyte in all matrices was 0.01 mg/kg; the LOD was 0.003 mg/kg.

A residue of 0.008 mg/kg (< LOQ) was detected for IN-GAZ70 in the Day 21 cream sample from Animal 8 (dose 3 mg/kg feed). This residue was excluded from the calculation of the mean since this result was considered to be unlikely. No residues of IN-GAZ70 were detected in any other milk, skim milk, cream, or tissue samples at any dose level.

As observed in whole milk, residues of IN-HXH44 were highest in skim milk followed by (in decreasing order) chlorantraniliprole and IN-K9T00. In cream, residues of chlorantraniliprole were highest followed by IN-HXH44 and IN-K9T00. Concentrations of chlorantraniliprole in cream were significantly higher than in skim milk (Table 114). The average chlorantraniliprole residues in cream and skim milk from the 50 mg/kg dosing group were 0.11 mg/kg and 0.016 mg/kg, respectively. There were no detectable residues (ND, < 0.003 mg/kg) of IN-GAZ70 or IN-EQW78 in skim milk or cream in any dose group.

Using the day 14 data for milk and cream, residues of chlorantraniliprole concentrate by a factor of 5.3× from whole milk to cream and residues of IN-HXH44 concentrate by a factor of 1.3×, from the 10 and 50 ppm feed groups. Residues of chlorantraniliprole are designated as slightly “fat-soluble” as confirmed by the milk and cream data.

Residues of chlorantraniliprole and metabolites in liver, kidney, muscle and fat are shown in Table 116. Data for individual animals are reported.

Table 116 Residue data for tissues from dairy cow feeding study with chlorantraniliprole.

Tissue	Animal number	Feed Level (ppm)	Residues (mg/kg)[a]			
			chlorantraniliprole	IN-EQW78	IN-HXH44	IN-K9T00
Liver	4	1	< 0.01	< 0.01	< 0.01	< 0.01
	5		< 0.01	< 0.01	< 0.01	< 0.01
	6		< 0.01	< 0.01	< 0.01	< 0.01
	7	3	< 0.01	< 0.01	< 0.01	< 0.01
	8		< 0.01	< 0.01	< 0.01	< 0.01
	9		0.014	< 0.01	< 0.01	< 0.01
	10	10	0.031	< 0.01	0.014	< 0.01
	11		0.021	< 0.01	0.019	< 0.01
	12		0.035	< 0.01	0.017	< 0.01
	13	50	0.133	< 0.01	0.05	< 0.01
	14		0.118	< 0.01	0.037	< 0.01
	15		0.129	< 0.01	0.047	< 0.01

Tissue	Animal number	Feed Level (ppm)	Residues (mg/kg)[a] chlorantraniliprole	IN-EQW78	IN-HXH44	IN-K9T00
	16	+9 days	< 0.01	< 0.01	< 0.01	< 0.01
	17	+23 days	< 0.01	< 0.01	< 0.01	< 0.01
kidney	4	1	< 0.01	< 0.01	< 0.01	< 0.01
	5		< 0.01	< 0.01	< 0.01	< 0.01
	6		< 0.01	< 0.01	< 0.01	< 0.01
	7	3	< 0.01	< 0.01	< 0.01	< 0.01
	8		< 0.01	< 0.01	< 0.01	< 0.01
	9		< 0.01	< 0.01	< 0.01	< 0.01
	10	10	0.021	< 0.01	0.011	< 0.01
	11		0.01	< 0.01	< 0.01	< 0.01
	12		0.035	< 0.01	0.011	< 0.01
	13	50	0.055	< 0.01	0.042	0.014
	14		0.081	< 0.01	0.042	0.012
	15		0.068	< 0.01	0.033	0.011
	16	+9 days	< 0.01	< 0.01	< 0.01	< 0.01
	17	+23 days	< 0.01	< 0.01	< 0.01	< 0.01
muscle	4	1	< 0.01	< 0.01	< 0.01	< 0.01
	5		< 0.01	< 0.01	< 0.01	< 0.01
	6		< 0.01	< 0.01	< 0.01	< 0.01
	7	3	< 0.01	< 0.01	< 0.01	< 0.01
	8		< 0.01	< 0.01	< 0.01	< 0.01
	9		< 0.01	< 0.01	< 0.01	< 0.01
	10	10	< 0.01	< 0.01	< 0.01	< 0.01
	11		< 0.01	< 0.01	< 0.01	< 0.01
	12		< 0.01	< 0.01	< 0.01	< 0.01
	13	50	0.029	< 0.01	0.012	< 0.01
	14		< 0.01	< 0.01	< 0.01	< 0.01
	15		0.024	< 0.01	0.011	< 0.01
	16	+9 days	< 0.01	< 0.01	< 0.01	< 0.01
	17	+23 days	< 0.01	< 0.01	< 0.01	< 0.01
Fat	4	1	< 0.01	< 0.01	< 0.01	< 0.01
	5		< 0.01	< 0.01	< 0.01	< 0.01
	6		< 0.01	< 0.01	< 0.01	< 0.01
	7	3	< 0.01	< 0.01	< 0.01	< 0.01
	8		< 0.01	< 0.01	< 0.01	< 0.01
	9		0.015	< 0.01	< 0.01	< 0.01
	10	10	0.036	< 0.01	< 0.01	< 0.01
	11		0.022	< 0.01	< 0.01	< 0.01
	12		0.028	< 0.01	< 0.01	< 0.01
	13	50	0.132	< 0.01	< 0.01	< 0.01
	14		0.156	< 0.01	0.02	< 0.01
	15		0.125	< 0.01	< 0.01	< 0.01
	16	+9 days	< 0.01	< 0.01	< 0.01	< 0.01
	17	+23 days	< 0.01	< 0.01	< 0.01	< 0.01

[a] LOQ for each analyte = 0.01 mg/kg; LOD = 0.003 mg/kg.

+*x* days = number of days after cessation of dosing

At sacrifice, residue levels were highest in liver followed by fat, kidney, and muscle. In fat, liver, and muscle, the major residue was chlorantraniliprole, followed by IN-HXH44. No residues of IN-K9T00, IN-GAZ70, or IN-EQW78 were detected (< 0.003 mg/kg) in fat, liver, or muscle with the exception of a residue of 0.003 mg/kg IN-EQW78 in fat and residues of 0.005 and 0.008 mg/kg in liver for the 50 mg/kg dose group. These residue levels were below the validated LOQ of 0.01 mg/kg. In kidney, the major residue was chlorantraniliprole, followed by IN-HXH44 then IN-K9T00. No residues of IN-GAZ70 or IN-EQW78 were detected in kidney. Residues in liver, fat, kidney, and muscle were dose dependent.

Using residues data for muscle and fat from the 10 and 50 ppm feed groups, chlorantraniliprole residues in fat are a factor of 4.7× higher than in muscle.

Following a 9-day depuration period for a cow dosed at 50 ppm, no residues of any analyte were detected in liver, kidney, muscle, or fat with the exception of a residue of 0.004 mg/kg chlorantraniliprole in liver. Following a 23-day depuration period for a cow dosed at 50 mg/kg, no residues of any analyte were detected in liver, kidney, muscle, or fat. Residues in milk collected from the depuration animals were < 0.003 mg/kg at 3 days after cessation of dosing.

A summary of the chlorantraniliprole tissue residues data is shown in Table 117.

Table 117 Summary of residue data for tissues from a dairy cow feeding study with chlorantraniliprole

Tissue	Feed Level (ppm)	Average Residues (mg/kg) chlorantraniliprole	IN-K9T00	IN-HXH44	IN-EQW78
Liver	1	< 0.01	< 0.01	< 0.01	< 0.01
	3	0.010	< 0.01	< 0.01	< 0.01
	10	0.029	< 0.01	0.016	< 0.01
	50	0.13	< 0.01	0.045	< 0.01
	50 (+9-day depuration)	< 0.01	< 0.01	< 0.01	< 0.01
	50 (+23-day depuration)	< 0.01	< 0.01	< 0.01	< 0.01
Kidney	1	< 0.01	< 0.01	< 0.01	< 0.01
	3	< 0.01	< 0.01	< 0.01	< 0.01
	10	0.022	< 0.01	0.010	< 0.01
	50	0.068	0.012	0.039	< 0.01
	50 (+9-day depuration)	< 0.01	< 0.01	< 0.01	< 0.01
	50 (+23-day depuration)	< 0.01	< 0.01	< 0.01	< 0.01
Muscle	1	< 0.01	< 0.01	< 0.01	< 0.01
	3	< 0.01	< 0.01	< 0.01	< 0.01
	10	< 0.01	< 0.01	< 0.01	< 0.01
	50	0.019	< 0.01	< 0.01	< 0.01
	50 (+9-day depuration)	< 0.01	< 0.01	< 0.01	< 0.01
	50 (+23-day depuration)	< 0.01	< 0.01	< 0.01	< 0.01
Fat	1	< 0.01	< 0.01	< 0.01	< 0.01
	3	< 0.01	< 0.01	< 0.01	< 0.01
	10	0.029	< 0.01	< 0.01	< 0.01
	50	0.14	< 0.01	0.012	< 0.01
	50 (+9-day depuration)	< 0.01	< 0.01	< 0.01	< 0.01
	50 (+23-day depuration)	< 0.01	< 0.01	< 0.01	< 0.01

Based upon the mean daily intake, the actual mean weekly dose levels were equivalent to 1.0–1.1 mg/kg feed,

3.0-3.2 mg/kg feed, 10–11 mg/kg feed, and 49–52 mg/kg feed.

The LOQ for each analyte in all matrices was 0.01 mg/kg; the LOD was 0.003 mg/kg.

In summary, there were no average residues greater than 0.01 mg/kg of any analyte in any sample at the 1 and 3 ppm feed levels, with the exception of chlorantraniliprole residues at 0.015 mg/kg in day 21 cream from the 3 ppm feed group.

Residues of chlorantraniliprole, IN-HXH44, and IN-K9T00 were not detected (< 0.003 mg/kg) in whole milk from the lowest dose group (1 ppm feed) but were dose dependent, increasing at higher doses. Residues in milk reached a plateau within 7 to 10 days of dosing. Chlorantraniliprole residues concentrate by a factor of 5.3× in cream compared to whole milk.

Residues of chlorantraniliprole, IN-HXH44, and IN-K9T00 were detected in fat, kidney, liver, and muscle. Residues were dose dependent, increasing with higher doses. Residues of IN-GAZ70 or IN-EQW78 were not detected (< 0.003 mg/kg) in any sample from any dose group with the exception of a residue of 0.003 mg/kg for IN-EQW78 in fat from the 50-mg/kg feed group. Chlorantraniliprole residues in fat are a factor of 4.7× higher than in muscle.

Following cessation of dosing, residues in milk and tissues rapidly declined to non-detectable levels (< 0.003 mg/kg) in the milk samples from 3 days post last dose and in tissue samples collected from the earliest sacrifice time at 9 days after cessation of dosing.

National Residue Definitions

In considering the need to include metabolites in the residue definition, regulators have taken different approaches. For example, Canada has utilised the following residue definitions:

risk assessment and enforcement in plant products – chlorantraniliprole;

enforcement in animal commodities and risk assessment in animal tissues–chlorantraniliprole;

risk assessment in milk - chlorantraniliprole and metabolites IN-HXH44 and IN-K9T00;

risk assessment in eggs - chlorantraniliprole and metabolites IN-H2H20, IN-GAZ70 and IN-K7H29;

Australia has proposed the following residue definitions:

plant products and commodities of animal origin other than milk: chlorantraniliprole

milk and milk products: sum of chlorantraniliprole IN-K9T00 and IN-HXH44, expressed as chlorantraniliprole; and

the USA and New Zealand have proposed the following simple definition:

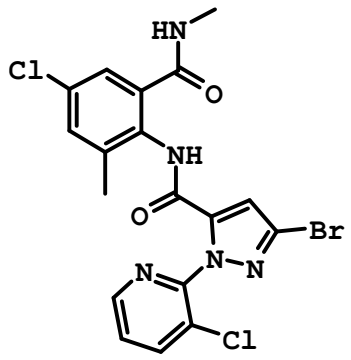
plant and animal products – chlorantraniliprole.

APPRAISAL

Chlorantraniliprole was considered for the first time by the present Meeting. The Meeting received information on chlorantraniliprole metabolism and environmental fate, methods of residue analysis, freezer storage stability, national registered use patterns, supervised residue trials, farm animal feeding studies and fate of residues in processing.

The 2008 JMPR established an ADI and ARfD for chlorantraniliprole of 0-2 mg/kg bw/day and not required respectively.

Chlorantraniliprole is 3-bromo-*N*-[4-chloro-2-methyl-6-[(methylamino)carbonyl]phenyl]-1-(3-chloro-2-pyridinyl)-1*H*-pyrazole-5-carboxamide.



The following abbreviations are used for the metabolites discussed below:

IN-DBC80	3-Bromo-1-(3-chloro-2-pyridinyl)-1H-pyrazole-5-carboxylic acid
IN-EQW78	2-[3-Bromo-1-(3-chloro-2-pyridinyl)-1H-pyrazol-5-yl]-6-chloro-3, 8-dimethyl-4(3H)-quinazolinone
IN-ECD73	2,6-dichloro-4-methyl-11H-pyrido[2,1-b]quinazolin-11-one
IN-F9N04	N-[2-(Aminocarbonyl)-4-chloro-6-methylphenyl]-3-bromo-1-(3-chloro-2-pyridinyl)1H-pyrazole-5-carboxamide
IN-F6L99	5-Bromo-N-methyl-1H-pyrazole-3-carboxamide
IN-GAZ70	2-[3-Bromo-1-(3-chloro-2-pyridinyl)-1H-pyrazol-5-yl]-6-chloro-8-methyl-4(3H)-quinazolinone
IN-H2H20	3-Bromo-N-[4-chloro-2-[[(hydroxymethyl)amino]carbonyl]-6-methylphenyl]-1-(3-chloro-2-pyridinyl)-1H-pyrazole-5-carboxamide
IN-HXH44	3-Bromo-N-[4-chloro-2-(hydroxymethyl)-6-[(methylamino)carbonyl]phenyl]-1-(3-chloro-2-pyridinyl)-1H-pyrazole-5-carboxamide
IN-L8F56	2-Amino-5-chloro-3-[(methylamino)carbonyl]benzoic acid
IN-LEM10	2-[5-Bromo-2-(3-chloro-pyridin-2-yl)-2H pyrazol-3-yl]-6-chloro-3,4-dihydro-3-methyl-4-oxo-8-quinazolinecarboxylic acid
IN-K9T00	3-Bromo-N-[4-chloro-2-(hydroxymethyl)-6-[[(hydroxymethyl)amino)carbonyl]phenyl]-1-(3-chloro-2-pyridinyl)-1H-pyrazole-5-carboxamide
IN-K3X21	2-[3-Bromo-1-(3-chloro-2-pyridinyl)-1H-pyrazol-5-yl]-6-chloro-8-(hydroxymethyl)-3-methyl-4(3H)-quinazolinone
IN-K7H29	2-[3-Bromo-1-(3-chloro-2-pyridinyl)-1H-pyrazol-5-yl]-6-chloro-8-(hydroxymethyl)-4(3H)-quinazolinone
IN-KAA24	2-[[[3-Bromo-1-(3-chloro-2-pyridinyl)-1H-pyrazol-5-yl]carbonyl]amino]-5-chloro-3-[(methylamino)carbonyl]benzoic acid

Animal metabolism

Radiolabelled chlorantraniliprole (separately [^{14}C]labelled at the benzamide-carbonyl and pyrazole-carbonyl positions) was used in the metabolism and environmental studies. The metabolism of laboratory animals was qualitatively the same as for farm animals though some species related differences were noted. The proposed major route of chlorantraniliprole metabolism in livestock is via (i) hydroxylation of the N-methyl group (to IN-H2H20) or hydroxylation of the tolyl methyl group (to

IN-HXH44); (ii) cyclization with loss of water to a quinazolinone derivative (IN-EQW78); and (iii) N-demethylation via IN-H2H20 to IN-F9N04.

Lactating goats were orally dosed with a 1:1 mixture of [benzamide carbonyl-^{14}C] and [pyrazole carbonyl-^{14}C]chlorantraniliprole at 0.36 mg/kg bw for 7 consecutive days equivalent to 10 ppm in the feed.

The majority of the administered dose was recovered in excreta (79% in faeces, 11% in urine) with an additional 3.9% recovered from the cage wash. Radioactivity retained in tissues, bile or secreted in milk accounted for approximately 1.3% of the administered dose. Overall 95% of administered radioactivity was accounted for.

Radiocarbon content in various tissues were highest in liver (0.64 mg/kg) followed by kidney (0.076 mg/kg), fat (0.07 mg/kg) and muscle (0.016 mg/kg) while in milk residues were 0.067 mg/kg for a composite sample from 1 through 7 days of the study. Chlorantraniliprole was the major component of the extracted radioactivity identified in kidney (19%), muscle (41%), and fat (35–75%) samples and was also present in liver (4%) where IN-L8F56 was the major component (7.5%). In milk chlorantraniliprole (24% TRR), IN-K9T00 (26% TRR) and IN-HXH44 (27% TRR) were the major components identified.

Laying hens were orally dosed with a 1:1 mixture of [benzamide carbonyl-^{14}C] and [pyrazole carbonyl-^{14}C]chlorantraniliprole at 0.8 mg/kg bw/day for 14 days. The majority of the administered radioactivity was excreted (98% over the 14 day dosing period), with 5% recovered from cage wash and approximately 3% in eggs (white and yolks). In tissues, the highest concentrations of radioactivity were in liver (0.52 mg/kg), followed by fat (0.052 mg/kg) and muscle (0.022 mg/kg). Chlorantraniliprole (25–30%) and IN-GAZ70 (29–37%) were the major components of the radioactivity in eggs with a large number of metabolites individually present at < 10% TRR, principally IN-K7H29, IN-H2H20, IN-EQW78 and IN-F9N04. In liver and muscle, no single component (unchanged parent compound or metabolite) was present at levels > 10% TRR with chlorantraniliprole present at only 2.2–3.7% TRR. Chlorantraniliprole formed the major component of the residue in skin with fat at 18% TRR. No other metabolite exceeded 9% TRR in skin and fat.

Plant metabolism

The Meeting received information on the fate of [^{14}C]chlorantraniliprole after foliar application to apple, tomato, lettuce and cotton and as a soil drench to rice.

Metabolism studies in apples, tomato, lettuce and cotton demonstrated that following foliar application, chlorantraniliprole was not metabolized to any great extent. With up to three consecutive foliar applications of chlorantraniliprole to apples (3×100 g ai/ha), tomatoes (3×100 g ai/ha) and lettuce (3×100 g ai/ha), and following a single application to cotton (1×150 g ai/ha), parent compound was the major component of the radioactive residues at 85%, 92%, 89% and 57% of the TRR respectively for apples, tomatoes, lettuce and cotton seed. When applied as a soil drench to rice crops (1×300 g ai/ha), the metabolism was complex due to uptake of degradates in water through the roots. Parent compound was the major component of the TRR in grain at harvest (51% TRR). For straw, numerous metabolites were identified in addition to parent compound. IN-GAZ70 (0.049 mg/kg) and IN-EQW78 (0.039 mg/kg) were two major metabolites in the rice straw but were present at less than 7% of the TRR. Minor metabolites (< 0.035 mg/kg) identified in rice straw included IN-KAA24, IN HXH40, IN H2H20, IN-HXH44, and IN-F6L99.

Environmental fate

Hydrolysis in water is pH dependent. Chlorantraniliprole is considered stable at pH 4 and 7 but is hydrolysed at pH 9 with a half-life of < 10 days. At pH 9, chlorantraniliprole undergoes cyclization followed by irreversible dehydration to form IN-EQW78. Abiotic hydrolysis is unlikely to contribute significantly to the degradation of chlorantraniliprole residues in aquatic systems unless the pH is high.

The aerobic degradation of chlorantraniliprole in soil is primarily by abiotic cyclization followed by dehydration to form IN-EQW78, with subsequent demethylation forming IN-GAZ70. Alternative pathways include abiotic rearrangement followed by cleavage to form IN-F6L99 and IN-ECD73. Ultimately mineralisation to $^{14}CO_2$ occurs. The half-life for degradation of chlorantraniliprole in soil is estimated to be > 100 days and sometimes > 1000 days. The degradation is sometimes limited by sequestration (or aging) of the compound in soil. The sequestration of chlorantraniliprole in soil makes the compound more difficult to extract and protects the compound from degradation, while limiting mobility. Chlorantraniliprole is considered to be persistent.

The log Kow of chlorantraniliprole (log Kow 2.86, pH 7) and the results of the rice metabolism study suggests chlorantraniliprole may be translocated in plants. In confined and field rotational crop studies, residues of chlorantraniliprole were found in leafy vegetables, root vegetables and cereal grain. Residues of chlorantraniliprole and metabolites were also detected in forage and fodder. It is concluded that rotational crops may contain significant residues of chlorantraniliprole.

Methods of Analysis

Several different analytical methods have been reported for the analysis of chlorantraniliprole and selected metabolites/degradates in plant material (IN-EQW78, IN-ECD73, IN-F6L99) and animal commodities (IN-K9T00, IN-HXH44, IN-GAZ70, IN-EQW78). The basic approach employs extraction by homogenisation with acetonitrile:water, and column clean-up using SPE (hydrophilic-lipophilic balanced polymer and strong anion exchange in sequence). Residues are determined by gas chromatography with an electron capture detector or liquid chromatography with mass spectra detection.

The analytical methods for chlorantraniliprole and selected metabolites have been extensively validated with numerous recoveries on a wide range of substrates with LOQs of 0.01 mg/kg for each analyte.

German official multi-residue method (DFG-S19) with LC-MS/MS detection was validated for chlorantraniliprole in plant and chlorantraniliprole, IN-K9T00, IN-HXH44, IN-GAZ70 and IN-EQW78 in animal commodities. LOQs were 0.01 mg/kg for each analyte.

Stability of pesticide residues in stored analytical samples

Freezer storage stability was tested for a range of representative substrates. Residues of chlorantraniliprole were stable in fortified sample crops and their processed products for the duration of the studies. Chlorantraniliprole was stable in homogenized samples stored frozen for at least 24 months for apple, grape, tomato, lettuce, cauliflower, potato, wheat grain, wheat straw, alfalfa hay and cotton seed. Chlorantraniliprole and metabolites (IN-EQW78, IN-ECDW73 and IN-F6L99) were stable for at least 12 months, the period of frozen storage studied for the processed commodities tomato ketchup, raisin, cotton seed meal, cotton seed oil, and apple juice. Residues of chlorantraniliprole and the metabolites IN-K9T00, IN-HXH44, IN-GAZ70 and IN-EQW78 were stable in bovine liver, kidney, muscle, fat and milk stored frozen for at least 12 months.

Residue definition

The residue following use of chlorantraniliprole on crops following foliar application is predominantly chlorantraniliprole. Similarly, chlorantraniliprole is the major component of the residue in rotational crops.

In the lactating goat metabolism study, chlorantraniliprole is the major component of the residue in edible tissues while in milk IN-HXH44 and IN-K9T00, and in eggs from the laying hen study IN-GAZ70, were present at slightly higher levels than chlorantraniliprole. Residues of chlorantraniliprole and metabolites decline rapidly on removal of exposure sources. None of the metabolites were identified by the 2008 JMPR as being of toxicological concern. Chlorantraniliprole and metabolites are considered to have low toxicity. At low doses the metabolites IN-HXH44 and IN-K9T00 are detected in milk in the absence of parent compound in the lactating dairy cow feeding study.

The Meeting recommended that the residue definition for plant and animal commodities, for compliance with MRLs and for estimation of dietary intake should be chlorantraniliprole.

The log Kow of chlorantraniliprole (log Kow 2.86, pH 7) suggests that chlorantraniliprole might be borderline fat soluble. The ratio of chlorantraniliprole residues in muscle and fat observed in the livestock metabolism and feeding studies (lactating goat: 1:3.7–1:7.8; lactating cow 1:4.7, laying hen 1:12) and ratio of residues in whole milk to cream (1:5.4) support the conclusion that chlorantraniliprole is fat soluble.

The Meeting recommended that chlorantraniliprole be described as fat-soluble

Proposed definition of the residue (for compliance with MRL and for estimation of dietary intake): *chlorantraniliprole*.

The residue is fat-soluble.

Results of supervised residue trials on crops

Supervised trials were available for the use of chlorantraniliprole on numerous crops: apples, pears, apricots, peaches, nectarines, plums, cherries, grapes, strawberries, Brassica vegetables (broccoli, Brussels sprouts, cabbage, cauliflower and Chinese cabbage), peppers, tomatoes, lettuce, spinach, mustard greens, celery, potatoes, cotton, almonds and pecans.

Residue trial data was made available from Argentina, Australia, New Zealand, Canada, member states of the European Union and the USA. As information on GAP of Australia, New Zealand and members states of the European Union were not supplied, trials from these countries were not considered in estimating maximum residue levels, however, the results are summarized in the 2008 JMPR Monograph.

Apples and pears

Data were available from supervised trials on apples in several countries including Argentina, Canada and the USA for which GAP information was available.

In Argentina chlorantraniliprole is permitted to be used on apples with a maximum of two foliar sprays at a spray concentration of 4 g ai/hL and a PHI of 14 days. Three trials complied with the GAP of Argentina with residues of < 0.06, 0.12 and 0.19 mg/kg.

The GAPs of Canada and the USA are similar and the GAP of the USA was used to evaluate trials on pome fruit from the two countries (USA GAP: 111 g ai/ha, PHI 14 days with a maximum seasonal application of 224 g ai/ha).

Residues of chlorantraniliprole in apples from 16 trials in Canada and the USA complying with GAP of the USA were: 0.010, 0.012, 0.022, 0.030, 0.038, 0.045, 0.056, 0.061, 0.072, 0.073, 0.078, 0.088, 0.088 and 0.093, 0.11 and 0.23 mg/kg.

Nine of eleven trials on pears from Canada and the USA complying with GAP of the USA had residues of chlorantraniliprole of: 0.016, 0.026, 0.033, 0.059, 0.070, 0.085, 0.10, 0.12 and 0.13 mg/kg.

The Meeting noted that the use patterns for apple and pears in the USA were the same and that the residues populations for each crop could be used to support the other. The Meeting decided to combine the data for apples and pears to increase the database for the purposes of estimating a maximum residue level, STMR and HR and to make a recommendation for pome fruit.

Residues in rank order (n = 25), median underlined, were: 0.010, 0.012, 0.016, 0.022, 0.026, 0.030, 0.033, 0.038, 0.045, 0.056, 0.059, 0.061, 0.070, 0.072, 0.073, 0.078, 0.085, 0.088, 0.088, 0.093, 0.10, 0.11, 0.12, 0.13 and 0.23 mg/kg.

The Meeting estimated maximum residue level and STMR values for chlorantraniliprole in pome fruit of 0.4 and 0.07 mg/kg respectively.

Stone fruit

Data were available from supervised trials on stone fruit in Argentina, Australia, member states of the European Union, Canada and the USA. GAP information was only available for Argentina, Canada and the USA.

In Argentina chlorantraniliprole is permitted to be used on peaches with a maximum of two foliar sprays at a spray concentration of 5 g ai/hL and a PHI of 7 days. No trials complied with GAP of Argentina.

The GAPs of Canada and the USA are similar and the GAP of the USA was used to evaluate trials on stone fruit from the two countries (USA GAP: 111 g ai/ha, PHI 10 days with a maximum seasonal application of 224 g ai/ha). The USA GAP advises against the use of adjuvants when spraying cherries. As GAP of Canada does not advise against the use of adjuvants for cherries, where trials were conducted at the same location with and without adjuvants, the value from the trial plot with the highest residue was selected for estimating maximum residue levels. As there were no restrictions for other stone fruit, data were also selected from the plot at a trial location with the highest residue that complied with GAP.

Residues of chlorantraniliprole in cherries from eight trials in Canada and the USA complying with GAP of the USA were: 0.056, 0.11, 0.18, 0.19, 0.21, 0.26, 0.45 and 0.57 mg/kg.

Residues of chlorantraniliprole in peaches from 17 trials in Canada and the USA complying with GAP of the USA were: 0.072, 0.090, 0.092, 0.10, 0.10, 0.11, 0.12, 0.12, 0.13, 0.13, 0.14, 0.14, 0.16, 0.18, 0.25, 0.26 and 0.31 mg/kg.

Eleven trials on plums from Canada and the USA complied with GAP of the USA with residues of: < 0.01 (4), 0.011, 0.015, 0.026, 0.029, 0.066, 0.067 and 0.076 mg/kg. The STMR for plums is 0.015 mg/kg.

The use pattern in the USA is for stone fruit and the residues populations for each crop could be used to support a crop group recommendation. The Meeting decided to use the data on the crop with the highest residues, cherries, in estimating a maximum residue level and STMR for stone fruit.

The Meeting estimated maximum residue level and, STMR values for chlorantraniliprole in stone fruit of 1 and 0.20 mg/kg respectively.

Grapes

Data were available from supervised trials on grapes in Australia, member states of the European Union, Canada and the USA. GAP information was only available for Canada and the USA.

The GAPs of Canada and the USA are similar. The GAP of Canada was used to evaluate trials on grapes from the two countries (Canada GAP: 111 g ai/ha, PHI 14 days with a maximum seasonal application of 224 g ai/ha) as GAP of Canada does not advise against the use of adjuvants for grapes. The Meeting noted that the residue populations corresponding to treatments with and without adjuvants were from similar populations and where they were from the same location should be treated as replicates with the value from the trial plot with the highest residue selected for estimating maximum residue levels.

Residues of chlorantraniliprole in grapes from 17 trials in Canada and the USA, complying with GAP of the USA, were (in rank order, median underlined): 0.015, 0.042, 0.044, 0.044, 0.083, 0.091, 0.093, 0.11, 0.119, 0.18, 0.20, 0.26, 0.32, 0.34, 0.46, 0.48 and 0.52 mg/kg.

The Meeting estimated maximum residue level and STMR values for chlorantraniliprole in grapes of 1 and 0.119 mg/kg respectively.

Brassica vegetables

Chlorantraniliprole is registered in the USA for use on Brassica vegetables at 73 g ai/ha, PHI of 3 days and a maximum application per season of 224 g ai/ha. Trials were available from Canada and the

USA in which crops were treated twice at three day intervals at 112 g ai/ha with harvest 3 days after the last spray. The trials did not comply with GAP of Canada and the USA and could not be used to estimate a maximum residue level.

Fruiting vegetables, Cucurbits

Trials on cucurbits were reported from Canada and the USA (USA GAP: 100 g ai/ha, PHI of 1 day and a maximum application per season of 224 g ai/ha).

Chlorantraniliprole residues on cucumbers in seven trials from the USA matching GAP in rank order were: < 0.01, 0.011, 0.012, 0.015, 0.017, 0.076 and 0.076 mg/kg.

Residues on melons (cantaloupe, muskmelon) in seven trials from the USA matching GAP in rank order were: 0.010, 0.027, 0.052, 0.065, 0.081, 0.090 and 0.10 mg/kg. Data on residues in the edible portion for melons in trials complying with USA GAP were not available.

Chlorantraniliprole residues on summer squash (including zucchini) in six trials from the USA matching GAP, in rank order were: 0.017, 0.023, 0.040, 0.054, 0.076 and 0.081 mg/kg.

The use-pattern in the USA is for fruiting vegetables, cucurbits and the Meeting decided to use the data on the crop with the highest residues (melons) to estimate a maximum residue level for the group.

The Meeting estimated a maximum residue level and an STMR value for chlorantraniliprole in fruiting vegetables, cucurbits of 0.3 and 0.065 mg/kg respectively.

Fruiting vegetables, other than Cucurbits

Trials on tomatoes were reported from Canada and the USA (USA GAP: 110 g ai/ha, PHI of 1 day and a maximum application per season of 224 g ai/ha).

Chlorantraniliprole residues in twenty trials from the USA matching GAP in rank order (median underlined) were: 0.018, 0.032, 0.032, 0.040, 0.040, 0.044, 0.051, 0.059, 0.061, 0.070, 0.071, 0.082, 0.092, 0.095, 0.10, 0.11, 0.14, 0.14, 0.14 and 0.18 mg/kg.

Trials on peppers were reported from the USA (GAP: 110 g ai/ha, PHI of 1 day and a maximum application per season of 224 g ai/ha).

Chlorantraniliprole residues in eleven trials on peppers (Bell) from the USA matching GAP in rank order (median underlined) were: 0.013, 0.019, 0.022, 0.024, 0.069, 0.090, 0.11, 0.11, 0.13, 0.14 and 0.18 mg/kg.

Chlorantraniliprole residues in chili peppers in nine trials from the USA matching GAP in rank order were (median underlined): 0.019, 0.035, 0.059, 0.063, 0.066, 0.069, 0.13, 0.21 and 0.41 mg/kg.

The Meeting decided that the trials in tomatoes, sweet and chili peppers could be used to support a crop group maximum residue level for fruiting vegetables, other than Cucurbits except mushrooms and sweet corn. The Meeting decided to use the data on the crop with the highest residues (chili peppers) to estimate a maximum residue level for the group.

The Meeting estimated a maximum residue level and STMR value for chlorantraniliprole in fruiting vegetables other than cucurbits (except mushrooms and sweet corn) of 0.6 and 0.066 mg/kg respectively.

Leafy vegetables

Trials on lettuce, spinach and mustard greens were reported from Canada and the USA (GAP: 110 g ai/ha, PHI of 1 day and a maximum application per season of 224 g ai/ha).

Chlorantraniliprole residues in fourteen trials on lettuce from Canada and the USA matching GAP in rank order were: < 0.01, 0.012, 0.43, 0.55, 1.3, 2.2, 2.4, 3.2, 3.9, 3.9, 4.0, 4.5, 5.3 and 6.2 mg/kg.

Chlorantraniliprole residues in seven trials on spinach from Canada and the USA matching GAP in rank order were: 3.4, 5.6, 6.8, 7.3, 7.4, 8.6 and 8.9 mg/kg.

Mustard greens are classified as a brassica vegetable in the US crop classification system and as a leafy vegetable according to the Codex classification. In considering trials on mustard greens and as explained for Brassica vegetables, the Meeting considered the trials did not comply with GAP of the USA.

The Meeting noted that the registered use of chlorantraniliprole in the USA is for leafy vegetables and decided to recommend a group MRL. The Meeting decided to use the data on the crop with the highest residues (spinach) to estimate a maximum residue level for the group. The Meeting estimated a maximum residue level and STMR value for chlorantraniliprole in leafy vegetables of 20 and 7.3 mg/kg respectively.

Celery

Chlorantraniliprole residues in seven trials on celery from Canada and the USA matching GAP (same as for leafy vegetables) in rank order were (median underlined): 0.99, 1.4, 2.1, 2.1, 2.6, 3.6 and 3.6 mg/kg. The Meeting estimated a maximum residue level and STMR value for chlorantraniliprole in celery of 7 and 2.1 mg/kg respectively.

Potatoes

Trials on potatoes were reported from Canada and the USA (US GAP: 49–74 g ai/ha, PHI of 14 days and a maximum application per season of 224 g ai/ha).

Chlorantraniliprole residues in twenty-seven trials from the USA matching GAP in rank order were (median underlined): < 0.01 (27) mg/kg.

Uptake of persistent residues from soil may also give rise to residues in potatoes tubers. Maximum residue levels and the potential for residues in succeeding and/or rotational crops are discussed under rotational crops below.

Tree nuts

Trials were available from the USA on residues of chlorantraniliprole in almonds and pecans but were unable to be evaluated as no relevant GAP existed at the time of evaluation.

Cotton seed

Trials on cotton were reported from the USA (GAP: 110 g ai/ha, PHI of 21 days and a maximum application per season of 224 g ai/ha).

Chlorantraniliprole residues in thirteen trials from the USA matching GAP in rank order were (median underlined): < 0.01, 0.016, 0.022, 0.029, 0.031, 0.047, 0.049, 0.054, 0.081, 0.082, 0.083, 0.13 and 0.25 mg/kg.

The Meeting estimated a maximum residue level and STMR value for chlorantraniliprole in cotton seed of 0.3 and 0.049 mg/kg respectively.

Animal feedstuffs

Cotton gin-trash

Chlorantraniliprole field trials on cotton were made available to the Meeting from the USA (GAP: 110 g ai/ha, PHI of 21 days and a maximum application per season of 224 g ai/ha).

Chlorantraniliprole residues on cotton gin-trash were 1.1, 2.4, 3.3, 4.1, 6.4, 12 and 13 mg/kg (fresh weight basis). The Meeting estimated an STMR value for chlorantraniliprole in cotton gin-trash of 4.1 mg/kg.

Almond hulls

The trial data could not be evaluated as no GAP was available.

Rotational crops

Residues of chlorantraniliprole are persistent in soil and may be taken up by following crops. In the USA the total seasonal application rate for crops is 220 g ai/ha. Studies of residues in rotational crops were made available to the meeting where in confined rotational crop studies soil was treated at 300–900 g ai/ha and in field studies bare soil and preceding crops were treated at 200–600 g ai/ha and 220 g ai/ha respectively.

Residues in leafy vegetables were < 0.01 (5) and 0.010 mg/kg in lettuce, < 0.01 (2) and 0.010 mg/kg in spinach and < 0.01 (4) mg/kg in Swiss chard. The levels in leafy vegetables from rotational crops are adequately covered by the recommendation for leafy vegetables of 20 mg/kg. Similarly residues of chlorantraniliprole in leaves/tops of turnips were < 0.01 (3) mg/kg, in beets < 0.01 (3), 0.015 and 0.034 mg/kg and in radish tops < 0.01, 0.010, 0.030, 0.068, 0.070 and 0.16 mg/kg and are also covered by the recommendation for leafy vegetables.

Residues in root and tuber vegetables grown as follow-crops were < 0.01 (3) mg/kg for turnip roots, < 0.01 (5) mg/kg for beet roots and < 0.01 (5) and 0.010 mg/kg for radish roots. Residues were observed at levels between the LOD and LOQ of the analytical method. Trials on root vegetables for foliar application according to GAP only supported a maximum residue level recommendation for potatoes of 0.01 mg/kg; no data on residues in potatoes grown as follow crops or on the combined effect of potatoes grown in soils containing residues (follow crops) and foliar application were made available to the Meeting. Residues in other root vegetables at harvest after planting as follow-crops were: < 0.01 (13) and 0.010 mg/kg.

Noting the residue data on follow-crops, the Meeting decided to recommend a maximum residue level for root and tuber vegetables of 0.02 mg/kg and an STMR of 0.01 mg/kg. The estimated maximum residue level for residues taken up from soil would accommodate residues arising from foliar application to potatoes.

Residues in follow-crop cereal grains were < 0.01 (3) mg/kg for oats and < 0.01 (8) mg/kg. As residues were observed in grain at levels above the LOD but below the LOQ of the analytical method, the Meeting decided to combine the data on follow-crop cereal grains and recommend maximum residue level and STMR values of 0.02 and 0.01 mg/kg respectively for cereal grain.

Corresponding residues in cereal forage (oat and wheat) were: < 0.01, 0.013, 0.016, 0.020, 0.022, 0.022, 0.031, 0.039, 0.043, 0.052 and 0.083 mg/kg. The Meeting decided to combine the data on forage of follow-crop cereals and recommend STMR and highest residue values of 0.022 and 0.083 mg/kg respectively for forage of cereals.

Residues in cereal hay (oat and wheat) were: < 0.01, 0.015, 0.017, 0.031, 0.043, 0.045, 0.051, 0.058, 0.10, 0.14 and 0.15 mg/kg. The estimated STMR and highest residue values for hay of cereals are 0.045 (or 0.051 mg/kg on a dry weight basis) and 0.15 mg/kg (or 0.17 mg/kg on a dry weight basis) respectively.

Residues in cereal straw (oat and wheat) were: < 0.01, 0.011, 0.014, 0.018, 0.030, 0.032, 0.039, 0.061, 0.078, 0.082 and 0.12 mg/kg. The estimated STMR and highest residue values for straw of cereals are 0.032 (or 0.036 mg/kg on a dry weight basis) and 0.12 mg/kg (or 0.136 mg/kg on a dry weight basis) respectively.

Residues in hay were higher than straw and the Meeting decided to use the hay data on follow-crop cereals and recommend a maximum residue level, STMR and highest residue for straw and hay of cereals of 0.3, 0.051 and 0.17 mg/kg respectively.

Two trials on residues in pulses (soya bean) with residues in seed of < 0.01 (2) mg/kg were available. Residues in forage were 0.027 and 0.041 mg/kg while residues in hay were 0.037 and

0.055 mg/kg. The Meeting considered two trials on pulses grown as rotational crops to be inadequate for the purposes of estimating maximum residue levels, STMRs and highest residues.

No trials on residues in follow-crops were available brassica vegetables, stalk and stem vegetables, legume vegetables, bulb vegetables, pulses, oilseeds, grass/pasture and legume animal feeds.

Fate of residues during processing

The fate of chlorantraniliprole residues has been examined in apples, grapes, plum and cotton processing studies. Processing of tomatoes into purée and paste showed a slight increase of chlorantraniliprole residues in the processed commodities when compared to the RAC. Whilst there was a decrease in residues found in the corresponding juice and ketchup. Apples and grapes showed a decrease in residues found in the juice, but an increase in pomace, raisins and apple peel. There was a concentration into the hulls of cottonseed. Estimated processing factors and STMR-Ps are summarised below.

Summary of processing factors for chlorantraniliprole residues.

Raw agricultural commodity (RAC)	Processed commodity	Calculated processing factors	PF (Mean, median or best estimate)	RAC-STMR (mg/kg)	STMR-P(mg/kg)
Apple	Pomace, dry	9.3 11 12 13	11.5	0.07	0.805
	Juice	< 0.06 < 0.09 < 0.19 < 0.19	< 0.14		< 0.0098
	Purée	0.09 0.09 < 0.19 < 0.19	0.09		< 0.0063
	Sauce	< 0.09 < 0.19 < 0.19 0.27	0.27		0.0189
	Preserves, canned	< 0.06 < 0.09 < 0.19 < 0.19	< 0.14		< 0.0098
Plum	Prune	1.9	1.9	0.015	0.0285
Grape	Pomace dry	6.1 12	9	0.119	1.07
	Juice	0.43 0.46 1.0 1.7	0.73		0.0869
	Raisin	2.7 2.9 4.0 7.1	3.45		0.411
	White wine	< 0.15 < 0.29	< 0.22		0.0262
	Red wine	0.76 1.6	1.18		0.140
Tomato	Canned tomatoes	< 0.2 0.23 0.33 0.65	0.28	0.066	0.0197
	Juice	0.57 0.78 0.89 1.1	0.835		0.0589
	Ketchup	0.72 0.74 1.2 1.6	0.98		0.0691
	Purée	1.2 1.4 1.5 1.7	1.45		0.102
	Paste	0.61 1.1 2.0 2.4	1.55		0.109
	Pomace, wet	1.2 1.4	1.3		0.0916
Cotton	Hulls	2.1	2.1	0.049	0.103
	Meal	0.75	0.75		0.0368
	Oil, refined	0.25	0.25		0.0122

Chlorantraniliprole concentrated in prunes, fruit pomace (apple, grape and tomato), raisins, cotton seed meal and hulls. As the estimated residues for the processed commodities raisins, cotton seed hulls and meal in the table above, are below the maximum residue levels proposed for the raw agricultural commodities the Meeting decided it was not necessary to make recommendations for maximum residue levels for these processed commodities.

The Meeting decided to estimate a maximum residue for chili pepper (dried) of 5 mg/kg following application of a default dehydration factor of 7 to the estimated maximum residue level of 0.6 mg/kg for chili pepper (7×0.6 = 4.2 mg/kg).

Farm animal dietary burden

The Meeting estimated the dietary burden of chlorantraniliprole in farm animals on the basis of the diets listed in Annex 6 of the 2006 JMPR Report (OECD Feedstuffs Derived from Field Crops). Calculation from highest residue, STMR (some bulk commodities) and STMR-P values provides the levels in feed suitable for estimating MRLs, while calculation from STMR and STMR-P values for feed is suitable for estimating STMR values for animal commodities. The percentage dry matter is taken as 100% when the highest residue levels and STMRs are already expressed as dry weight.

Estimated maximum and mean dietary burdens of farm animals

Dietary burden calculations for beef cattle, dairy cattle, broilers and laying poultry are provided in Annex 6 of the 2008 Report of the JMPR. The calculations were made according to the animal diets from US-Canada, EU and Australia in the OECD Table (Annex 6 of the 2006 JMPR Report).

		Animal dietary burden, chlorantraniliprole, ppm of dry matter diet		
		US-Canada	EU	Australia
Beef cattle	max	0.45	0.18	0.67[a]
	mean	0.35	0.11	0.48[c]
Dairy cattle	max	0.25	0.15	0.63[b]
	mean	0.09	0.074	0.47[d]
Poultry - broiler	max	0.012	0.007	0.007
	mean	0.012	0.007	0.007
Poultry - layer	max	0.011	0.057[e]	0.007
	mean	0.011	0.020[f]	0.007

[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.

[e] Highest maximum poultry dietary burden suitable for MRL estimates for poultry tissues and eggs.

[f] Highest mean poultry dietary burden suitable for STMR estimates for poultry tissues and eggs.

The chlorantraniliprole dietary burdens for animal commodity MRL and STMR estimation (residue levels in animal feeds expressed on dry weight) are: beef cattle 0.67 and 0.48 ppm, dairy cattle 0.63 and 0.47 ppm and poultry 0.057 and 0.020 ppm.

Farm animal feeding studies

The Meeting received information on the residue levels arising in animal tissues and milk when dairy cows were dosed with chlorantraniliprole for 28 days at the equivalent of 1, 3, 10 and 50 ppm in the diet. Average residues of chlorantraniliprole in milk for the 3 ppm dose group were < 0.01 (3) mg/kg. Chlorantraniliprole residues in liver and fat were higher than in other tissues. Average residues for tissues for the 3 ppm dosing level (3 animals per dose group) were all < 0.01 mg/kg for liver, fat, kidney and muscle.

The Meeting also received information on the residue levels arising in tissues and eggs when laying hens were dosed with [^{14}C]chlorantraniliprole for 14 days at the equivalent of 10 ppm in the diet. Residues in eggs were 0.308 mg/kg. Of tissues, residues of chlorantraniliprole were highest in liver at 0.0193 mg/kg, followed by skin and fat at 0.0093 mg/kg and muscle 0.0008 mg/kg at 23 h after the last dose.

Animal commodity maximum residue levels

The maximum dietary burden for beef and dairy cattle is 0.67 and 0.63 ppm respectively, so the levels of residues in tissues can be obtained from the 1 ppm feeding level. Maximum residues expected in tissues are: fat, muscle, liver and kidney are 0.0067 mg/kg (0.01×0.67/1) and the mean residue for milk 0.0063 mg/kg. At the 3 ppm dose level, average residues of chlorantraniliprole were 0.015 mg/kg in cream and < 0.01 mg/kg in whole milk (0.025 and 0.005 mg/kg respectively for cream and whole milk for the 10 ppm dose level at day 14). Expected residues in cream are 5× the residues in whole milk or 5×0.0063 = 0.0315 mg/kg. The fat content of cream is 40–60% and the Meeting estimated the mean residue for milk fat to be 2×0.0315 = 0.063 mg/kg.

The Meeting estimated maximum residue levels for meat (from mammals other than marine mammals) 0.01* mg/kg (fat); edible offal (mammalian) 0.01* mg/kg; milks 0.01* mg/kg and 0.01* mg/kg for milk fat.

As no residues are expected at the maximum dietary burden, estimated STMRs are 0 mg/kg for meat (from mammals other than marine mammals), fat (from mammals other than marine mammals), edible offal mammalian, milk and 0.047 mg/kg for milk fat.

The maximum dietary burden for poultry is 0.057 ppm. Maximum residues expected at 23 h after last feeding are: muscle, skin/fat, liver and eggs are 0.0000016, 0.000019, 0.000039 and 0.000616 mg/kg.

The maximum residue levels for poultry meat 0.01* mg/kg (fat); poultry offal 0.01* and eggs 0.01* mg/kg.

The mean dietary burden for poultry is 0.02 ppm. No residues are expected in poultry tissues and eggs of birds at the mean dietary burden. STMRs for poultry meat, skin/fat, edible offal and eggs are all 0 mg/kg.

FURTHER WORK OR INFORMATION

Desirable

Information on residues in follow crops, especially for brassica vegetables, stalk and stem vegetables, legume vegetables, bulb vegetables, pulses, oilseeds, grass/pasture and legume animal feeds.

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 MRL and for estimation of dietary intake) for plants and animal commodities: *chlorantraniliprole*

The residue is fat-soluble

CCN	Commodity Name	MRL mg/kg New	MRL mg/kg Prev	STMR or STMR-P
VS 0624	Celery	7		2.1
GC 0080	Cereal grains	0.02		0.01
HS 0444	Chilli peppers, (dry)	5		0.46
SO 0691	Cotton seed	0.3		0.049
PE 0112	Eggs	0.01*		0
VC 0045	Fruiting vegetables, Cucurbits	0.3		0.065
VO 0050	Fruiting vegetables, other than Cucurbits (except mushrooms and sweet corn)	0.6		0.066

CCN	Commodity Name	MRL New	mg/kg Prev	STMR or STMR-P
JF 0448	Tomato juice			0.0589
	Tomato ketchup			0.0691
	Tomato purée			0.102
VW 0448	Tomato paste			0.109
FB 0269	Grapes	1		0.119
	Juice			0.0869
	Raisin			0.411
	White wine			0.0262
	Red wine			0.140
VL 0053	Leafy vegetables	20		7.3
MO 0105	Edible offal (Mammalian)	0.01*		0
ML 0106	Milks	0.01*		0
FM 0183	Milk fats	0.1		0.047
MM 0095	Meat (from mammals other than marine mammals)	0.01* (fat)		0M 0F
FP 0009	Pome fruits	0.4		0.07
JF 0226	Apple juice			0.0098
	Apple purée			0.0063
	Apple sauce			0.0189
PM 0110	Poultry meat	0.01* fat		0M 0F
PO 0111	Poultry, edible offal of	0.01*		0
VR 0075	Root and tuber vegetables	0.02		0.01
FS 0012	Stone fruits	1		0.2
AS 0081	Straw and fodder (dry) of cereal grains	0.3		0.051
AB 0226	Apple pomace, dry			0.805
AB 0269	Grape pomace, dry			1.07

* the MRL is estimated at or about the LOQ

DIETARY RISK ASSESSMENT

Long-term intake

The evaluation of chlorantraniliprole has resulted in recommendations for MRLs and STMRs for raw and processed commodities. Consumption data were available for 19 food commodities and were used in the dietary intake calculation. The results are shown in Annex 3 of the 2008 Report of the JMPR.

The International Estimated Daily Intakes for the 13 GEMS/Food regional diets, based on estimated STMRs were 0% (0–0.3%) of the maximum ADI of 2 mg/kg bw (Annex 3). The Meeting concluded that the long-term intake of residues of chlorantraniliprole from uses that have been considered by the JMPR is unlikely to present a public health concern.

Short-term intake

The 2008 JMPR decided that an ARfD is unnecessary. The Meeting therefore concluded that the short-term intake of chlorantraniliprole residues is unlikely to present a public health concern.

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