CYANTRANILIPROLE (263)

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EXPLANATION

Cyantraniliprole is a diamide insecticide with a mode of action (ryanodine receptor activation) similar to chlorantraniliprole and flubendiamide. It has root systemic activity with some translaminar movement and is effective against the larval stages of lepidopteran insects; and also on thrips, aphids, and some other chewing and sucking insects.

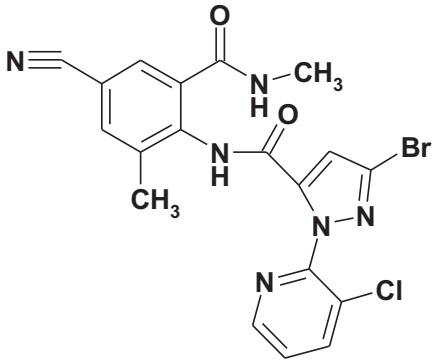
Authorisations exist for the use of cyantraniliprole in Canada, Columbia, Malaysia, New Zealand, Vietnam and the CLISS countries in West Africa. Authorisations are also being progressed in Australia, Europe and USA under an OECD Joint Review exercise.

Cyantraniliprole was scheduled by the Forty-fourth Session of the CCPR as a new compound for consideration by the 2013 JMPR. Residue and analytical aspects of cyantraniliprole were considered for the first time by the present meeting. The manufacturer submitted studies on metabolism, analytical methods, supervised field trials, processing, freezer storage stability, environmental fate in soil and rotational crop residues.

In this evaluation, the values presented in the tables are as reported in the various studies, but in the accompanying text, they have generally been rounded to two significant digits. Abbreviations have also been used for the various cyantraniliprole metabolites mentioned in the study reports. These include:

IN-F6L99	3-Bromo-N-methyl-1*H*-pyrazole-5-carboxamide
IN-HGW87	N-[2-(Aminocarbonyl)-4-cyano-6-methylphenyl]-3-bromo-1-(3-chloro-2-pyridinyl)-1*H*-pyrazole-5-carboxamide
IN-J9Z38	2-[3-Bromo-1-(3-chloro-2-pyridinyl)-1*H*-pyrazol-5-yl]-3,4-dihydro-3,8-dimethyl-4-oxo-6-quinazolinecarbonitrile
IN-JCZ38	4-[[[3-Bromo-1-(3-chloro-2-pyridinyl)-1*H*-pyrazol-5-yl]carbonyl]amino]-N′3′,5-dimethyl-1,3-benzenedicarboxamide
IN-K7H19	4-[[[3-Bromo-1-(3-chloro-2-pyridinyl)-1*H*-pyrazol-5-yl]carbonyl]amino]-5-methyl-1,3-benzenedicarboxamide
IN-MLA84	2-[3-Bromo-1-(3-chloro-2-pyridinyl)-1*H*-pyrazol-5-yl]-1,4-dihydro-8-methyl-4-oxo-6-quinazolinecarbonitrile
IN-MYX98	3-Bromo-1-(3-chloro-2-pyridinyl)-N-[4-cyano-2[[(hydroxymethyl)amino]carbonyl]-6-methylphenyl]-1*H*-pyrazole-5-carboxamide
IN-N5M09	6-Chloro-4-methyl-11-oxo-11*H*-pyrido[2,1-*b*]quinazoline-2-carbonitrile
IN-N7B69	3-Bromo-1-(3-chloro-2-pyridinyl)-N-[4-cyano-2-(hydroxymethyl)-6-[(methylamino)carbonyl]phenyl]-1*H*-pyrazole-5-carboxamide

IDENTITY

ISO common name:	Cyantraniliprole
Code number	DPX-HGW86, SYN545377
IUPAC name:	3-bromo-1-(3-chloro-2-pyridyl)-4′ -cyano-2′ -methyl-6′ -(methylcarbamoyl)pyrazole-5-carboxanilide
Chemical Abstracts name:	3-bromo-1-(3-chloro-2-pyridinyl)-N-[4-cyano-2-methyl-6-[(methylamino)carbonyl] phenyl]-1H-pyrazole-5-carboxamide
CAS number	736994-63-1
CIPAC number	not allocated
Molecular mass:	473.72 g/mol
Molecular formula	C19H14BrClN6O2
Structural formula:	

PHYSICAL AND CHEMICAL PROPERTIES

Pure active ingredient

Property	Findings			Reference
Test Item	DPX-HGW86-209 (98.4% purity)	DPX-HGW86-412 (97% purity)	DPX-HGW86-141 (93.3% purity)	
Melting point	217-219 °C			DP-27440
Boiling point	232 °C			DP-27440
Relative density (20°C)	1.497	1.384	1.496	DP-18863 DP-27440
Vapour pressure (extrapolated)	5.133×10^{-15} Pa (20 °C) 1.787×10^{-14} Pa (25 °C) $< 6.15 \times 10^{-8}$ Pa (80 °C)			DP-17052
Henry's law constant (calculated)	1.7×10^{-13} Pa m^3 mol^{-1} (20 °C)			DP-18861
Appearance	white powder, no noticeable odour			DP-18863 DP-27440
pH (1% dispersion)	5.61			DP-19177

Property	Findings	Reference
Test Item	DPX-HGW86-209 (98.4% purity); DPX-HGW86-412 (97% purity); DPX-HGW86-141 (93.3% purity)	
Solubility in water (20 °C)	pH 4 17.43 mg/L pH 7 12.33 mg/L pH 9 5.94 mg/L) In pure (Milli-RO) water: pH 7.2 14.23 mg/L	DP-17050
Solubility in organic solvents (20°C)	acetone 6.54 g/L; ethyl acetate 1.96 g/L; dichloromethane 5.05 g/L; n-octanol 0.79 g/L; methanol 4.73 g/L; n-hexane 0.00007 g/L; o-xylene 0.29 g/L; acetonitrile 2.45 g/L	DP-27447
n-Octanol/water partition coefficient	log P_{ow} 1.94 at 22 °C (shake flask method) Not pH-dependant (pH 4–9)	DP-17054
Hydrolysis (sterile buffer solutions, [cyano-^{14}C] and [pyrazole carbonyl-^{14}C] radiolabels	Hydrolysis of cyantraniliprole (99% purity) is pH and temperature dependant: DT_{50} (days): pH 4: 362 / 212 / 55.2; pH 7: 126 / 30.3 / 7.51; pH 9: 3.1 / 0.85 / 0.58 (15 °C / 25 °C / 35 °C) DT_{90} (days): pH 4: 1204 / 705 / 183; pH 7: 417 / 101 / 25; pH 9: 10.3 / 2.82 / 1.91 (15 °C / 25 °C / 35 °C) IN-J9Z38 is the major hydrolysis product accounting for up to 28% AR (pH 4), 89% AR (pH 7) and 98% AR (pH 9). Estimated degradation rates: pH 7: DT_{50} 227 @ 35 °C; DT_{90} 755 @ 35 °C pH 9: DT_{50} 376 @ 35 °C; DT_{90} 1248 @ 35 °C	DP-17058
Photolysis [CN-^{14}C] and [PC-^{14}C] radiolabels purity > 98%	Cyantraniliprole is rapidly degraded by photolysis. Degradation rates in natural water and pH 4 sterile buffer at 25 °C, exposed to continuous artificial sunlight for 15 days are: pH 4 buffer: DT_{50} 0.17; DT_{90} 0.57 natural water: DT_{50} 0.22; DT_{90} 0.72 Degradation products include four major (> 5% AR) degradation products (IN-NXX69, IN-QKV54, IN-NXX70, IN-QKV55). Quantum yield: (φ)=1.195 × 10^{-4} molecules degraded/photon	DP-17060
Dissociation constant	pKa=8.8 at 20 °C (pH range of 2–11, wavelength of 316 nm)	DP-19176

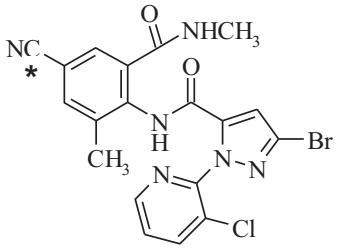
Formulations

Formulations of cyantraniliprole are available for use as foliar applications and soil or seed treatments.

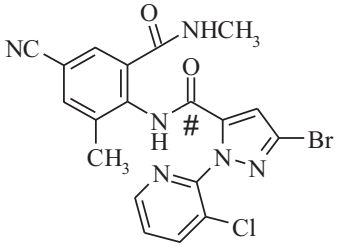
Formulation type	Active substance/s and content	Application type
OD (Oil Dispersion)	Cyantraniliprole 100 g/L	Foliar applications
SC (Soluble Concentrate)	Cyantraniliprole 200 g/L	Soil and seed treatments
FS (Flowable suspension	Cyantraniliprole 625 g/L	Seed treatment

METABOLISM AND ENVIRONMENTAL FATE

The Meeting received cyantraniliprole metabolism studies on animals (rats, lactating goats and laying hens), plants, soil and rotational crops. Cyantraniliprole radiolabelled on the 4-cyano or the pyrazole carbonyl groups were used in these studies. The label positions are given below:



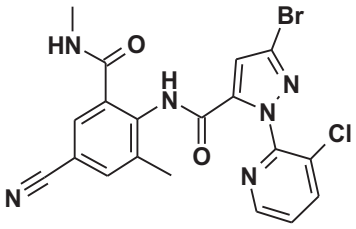
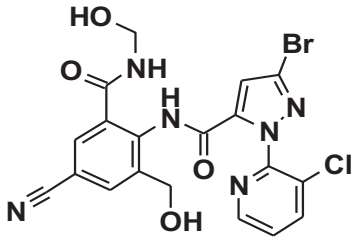
[CN-^{14}C]-cyantraniliprole (CN-label)
*=location of the radiolabel

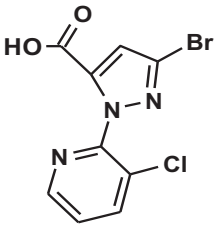
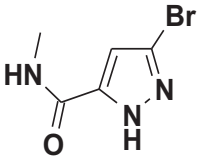
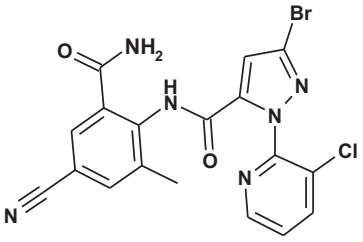
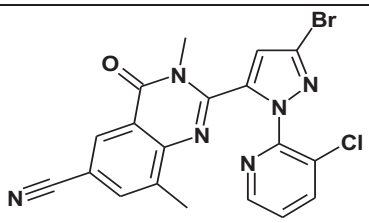
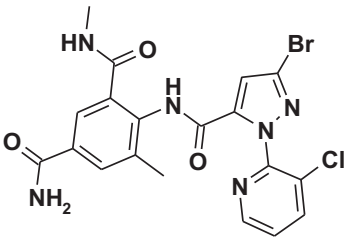
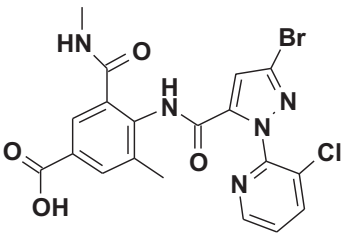


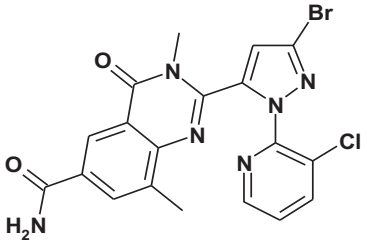
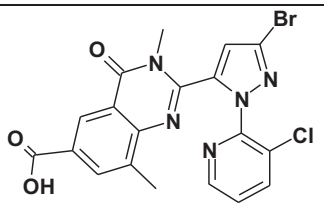
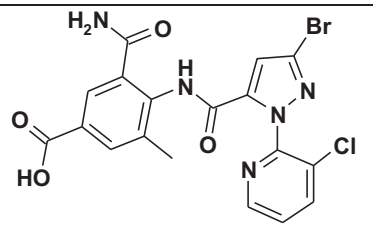
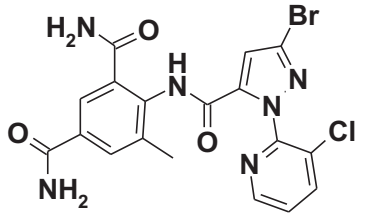
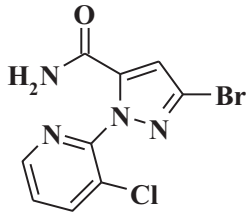
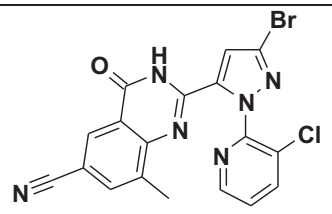
[PC-^{14}C]-cyantraniliprole (PC-label)
#=location of the radiolabel

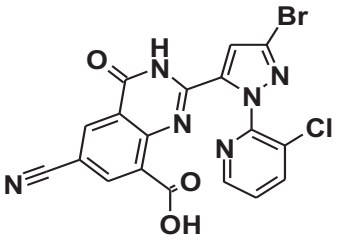
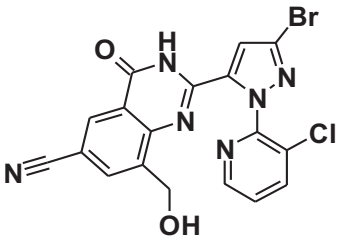
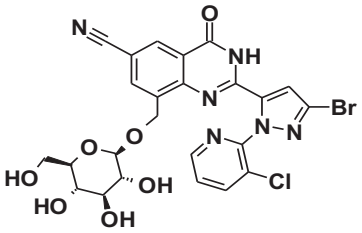
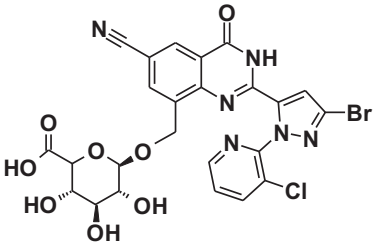
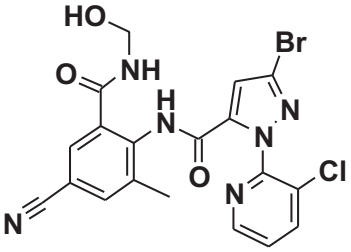
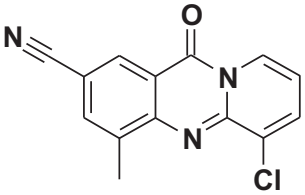
Major metabolites identified in these studies and discussed in this evaluation are listed below.

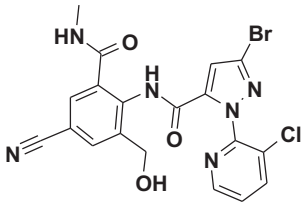
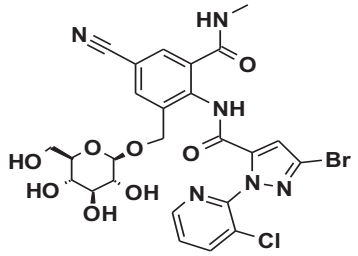
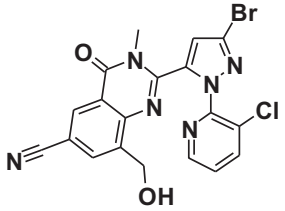
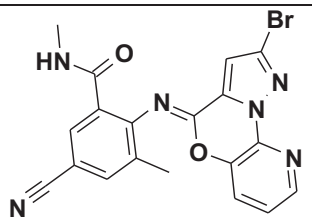
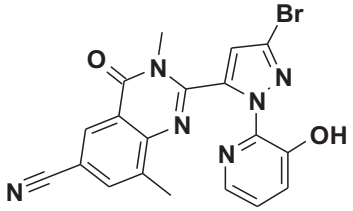
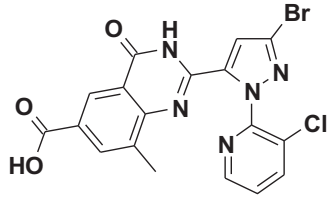
Table 1 Major cyantraniliprole metabolites identified in plant, animal and soil matrices

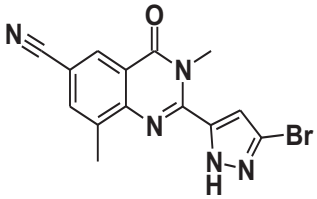
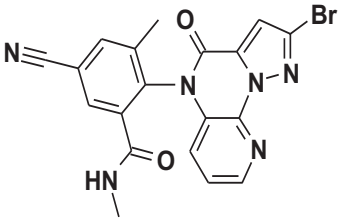
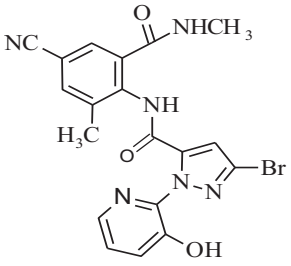
Codes	Names	Molecular formula	Occurrence
DPX-HGW86	3-Bromo-1-(3-chloro-2-pyridinyl)-N-[4-cyano-2-methyl-6-[(methylamino)carbonyl]phenyl]-1H-pyrazole-5-carboxamide		sediment soil hydrolysis photolysis plants rat livestock water
Bis hydroxy DPX-HGW86	3-Bromo-1-(3-chloro-2-pyridinyl)-N-[4-cyano-2-(hydroxymethyl)-6-[[(hydroxymethyl)amino]carbonyl]phenyl]-1*H*-pyrazole-5-carboxamide		rat poultry

Codes	Names	Molecular formula	Occurrence
IN-DBC80	3-Bromo-1-(3-chloro-2-pyridinyl)-1*H*-pyrazole-5-carboxylic acid		Soil rat urine livestock
IN-F6L99	3-Bromo-N-methyl-1*H*-pyrazole-5-carboxamide		soil high temperature hydrolysis
IN-HGW87	N-[2-(Aminocarbonyl)-4-cyano-6-methylphenyl]-3-bromo-1-(3-chloro-2-pyridinyl)-1*H*-pyrazole-5-carboxamide		Plants rat livestock
IN-J9Z38	2-[3-Bromo-1-(3-chloro-2-pyridinyl)-1*H*-pyrazol-5-yl]-3,4-dihydro-3,8-dimethyl-4-oxo-6-quinazolinecarbonitrile		Sediment degradation soil hydrolysis photolysis plants livestock rat
IN-JCZ38	4-[[[3-Bromo-1-(3-chloro-2-pyridinyl)-1*H*-pyrazol-5-yl]carbonyl]amino]-N′3′,5-dimethyl-1,3-benzenedicarboxamide		Sediment degradation soil plants livestock
IN-JSE76	4-[[[3-Bromo-1-(3-chloro-2-pyridinyl)-1*H*-pyrazol-5-yl]carbonyl]amino]-3-methyl-5-[(methylamino)carbonyl]benzoic acid		Sediment degradation soil plants livestock

Codes	Names	Molecular formula	Occurrence
IN-K5A77	2-[3-Bromo-1-(3-chloro-2-pyridinyl)-1*H*-pyrazol-5-yl]-3,4-dihydro-3,8-dimethyl-4-oxo-6-quinazolinecarboxamide		Sediment degradation soil plants livestock
IN-K5A78	2-[3-Bromo-1-(3-chloro-2-pyridinyl)-1*H*-pyrazol-5-yl]-3,4-dihydro-3,8-dimethyl-4-oxo-6-quinazolinecarboxylic acid		Sediment degradation soil plants livestock
IN-K5A79	3-(Aminocarbonyl)-4-[[[3-bromo-1-(3-chloro-2-pyridinyl)-1*H*-pyrazol-5-yl]carbonyl]amino]-5-methylbenzoic acid		Sediment degradation soil plants livestock
IN-K7H19	4-[[[3-Bromo-1-(3-chloro-2-pyridinyl)-1*H*-pyrazol-5-yl]carbonyl]amino]-5-methyl-1,3-benzenedicarboxamide		Soil plants livestock
IN-M2G98	3-Bromo-1-(3-chloro-2-pyridinyl)-1H-pyrazole-5-carboxylic acid, amide		Soil degradation
IN-MLA84	2-[3-Bromo-1-(3-chloro-2-pyridinyl)-1*H*-pyrazol-5-yl]-1,4-dihydro-8-methyl-4-oxo-6-quinazolinecarbonitrile		Plants livestock rat

Codes	Names	Molecular formula	Occurrence
IN-MLA84 carboxylic acid	2-[3-Bromo-1-(3-chloro-2-pyridinyl)-1*H*-pyrazol-5-yl]-6-cyano-1,4-dihydro-4-oxo-8-quinazolinecarboxylic acid		Plants rat poultry
Hydroxy-IN-MLA84	2-[3-Bromo-1-(3-chloro-2-pyridinyl)-1*H*-pyrazol-5-yl]-3,4-dihydro-8-(hydroxymethyl)-4-oxo-6-quinazolinecarbonitrile		Poultry
Hydroxy-IN-MLA84 glucoside	2-[3-Bromo-1-(3-chloro-2-pyridinyl)-1*H*-pyrazol-5-yl]-8-[(β-D-glucopyranosyloxy)methyl]-1,4-dihydro-4-oxo-6-quinazolinecarbonitrile		Plants
Hydroxy-IN-MLA84 glucuronide	[2-[3-bromo-1-(3-chloro-2-pyridinyl)-1*H*-pyrazol-5-yl]-6-cyano-1,4-dihydro-4-oxo-8-quinazolinyl]methyl β-D-glucopyranosiduronate		Rats
IN-MYX98	3-Bromo-1-(3-chloro-2-pyridinyl)-N-[4-cyano-2[[(hydroxymethyl)amino]carbonyl]-6-methylphenyl]-1*H*-pyrazole-5-carboxamide		Plants livestock rat
IN-N5M09	6-Chloro-4-methyl-11-oxo-11*H*-pyrido[2,1-*b*]quinazoline-2-carbonitrile		High temperature hydrolysis

Codes	Names	Molecular formula	Occurrence
IN-N7B69	3-Bromo-1-(3-chloro-2-pyridinyl)-N-[4-cyano-2-(hydroxymethyl)-6-[(methylamino)carbonyl]phenyl]-1*H*-pyrazole-5-carboxamide		rat plants livestock
IN-N7B69 glucoside	3-Bromo-1-(3-chloro-2-pyridinyl)-N-[4-cyano-2-[(β-D-glucopyranosyloxy)methyl]-6-[(methylamino)carbonyl]phenyl]-1*H*-pyrazole-5-carboxamide		plants
IN-NBC94	2-[3-Bromo-1-(3-chloro-2-pyridinyl)-1*H*-pyrazole-5-yl]-3,4-dihydro-8-(hydroxymethyl)-3-methyl-4-oxo-6-quinazolinecarbonitrile		plants rat poultry
IN-NXX69	2-[[(4Z)-2-bromo-4*H*-pyrazolo[1,5-*d*]pyrido[3,2-b][1,4]oxazin-4-ylidene]amino]-5-cyano-N,3-dimethylbenzamide		aqueous photolysis soil photolysis
IN-NXX70	2-[3-Bromo-1-(3-hydroxy-2-pyridinyl)-1*H*-pyrazol-5-yl]-3,4-dihydro-3,8-dimethyl-4-oxo-6-quinazolinecarbonitrile		plants aqueous photolysis soil photolysis
IN-PLT97	2-[3-Bromo-1-(3-chloro-2-pyridinyl)-1*H*-pyrazol-5-yl]-1,4-dihydro-8-methyl-4-oxo-6-quinazolinecarboxylic acid		soil sediment

Codes	Names	Molecular formula	Occurrence
IN-QKV54	2-(3-Bromo-1*H*-pyrazol-5-yl)-1,4-dihydro-3,8-dimethyl-4-oxo-6-quinazolinecarbonitrile		rat plants aqueous photolysis, soil photolysis
IN-RNU71	2-(2-Bromo-4-oxopyrazolo[1,5-a]pyrido[3,2-e]pyrazin-5(4H)-yl)-5-cyano-N,3-dimethylbenzamide		soil photolysis
IN-QKV55	3-bromo-*N*-[4-cyano-2-methyl-6-(methylcarbamoyl)phenyl]-1-(3-hydroxypyridin-2-yl)-1*H*-pyrazole-5-carboxamide		aqueous photolysis

Animal metabolism

The Meeting received animal metabolism studies on rats, lactating goats and laying hens, following oral dosing with [3-^{14}C-thienyl] dimethenamid (the racemic mixture).

Rats

The metabolism of cyantraniliprole in rats was evaluated by the WHO Core Assessment Group of the 2013 JMPR, where it was concluded that majority of the dose was excreted within 24 to 48 hours, with about 1-5.5% TRR being recovered in tissues. Tissue elimination half-lives ranged from 2.6 days (fat) to 5.1 days (kidney).

The metabolic pathway was primarily through hydroxylation (to form IN-N7B69 and IN-MYX98), with IN-N7B69 being further metabolized to a glucuronide. Cyantraniliprole undergoes ring closure to generate IN-J9Z38 which is then in turn hydroxylated to form IN-NBC94, its carboxylic acid, and its glucuronide conjugate. IN-MYX98 is also metabolized to the closed-ring metabolite IN-MLA84, which, like IN-NBC94, is further oxidized to a hydroxylated metabolite, a carboxylic acid, and the glucuronide of the hydroxyl metabolite. Further, the hydroxylated metabolite IN-MYX98 can be N-dealkylated to form IN-HGW87 as well as being hydroxylated a second time to form bis-hydroxy-HGW86. Cyantraniliprole can also be hydroxylated on the pyridine ring, followed by a ring closure analogous to the conversion of cyantraniliprole to IN-J9Z38. Cyantraniliprole can also be N-dealkylated and cleaved at the carbonyl bridge to form IN-DBC80glutathione conjugation, with other pathways involving reductive dechlorination, oxidation, hydroxylation, *O*-demethylation and cyclization as well as conjugation with glucuronic acid.

Lactating goats

One lactating goat was orally administered [CN-^{14}C]-cyantraniliprole in a gelatine capsule at a dose rate equivalent to 13.7 ppm in the diet for seven consecutive days and a second goat was similarly

dosed with the dietary equivalent of 12.5 ppm [PC-^{14}C]-cyantraniliprole. The results of this study have been reported by McLellan, Vance and Lowry, 2008 [Ref: DP-16987].

Urine and faeces were collected before the first dose and daily thereafter. Milk samples were collected twice daily, in the morning before each dosing and in the afternoon, about 7 hours after dosing, with the afternoon milk being combined with the milk collected the next morning. The animals were sacrificed 23 hours after last of the seven daily doses.

Samples were double or triple-extracted with acetonitrile/water mixtures, centrifuged and radioactivity was measured by LSC with the liver samples being further processed by enzyme (protease) digestion. Milk samples were extracted with acetonitrile and partitioned with hexane. Extracts were analysed using reversed phase HPLC (UV detection at 254 nm) to detect reference standards, with quantification either by on-line radiodetection with peak integration or by fraction collection and LSC. LC-MS/MS (electrospray positive ion mode) was used to confirm the presence of cyantraniliprole and major metabolites. All samples were stored at –20 °C and were extracted within 25 days and analysed within 33 days of sampling.

Overall ^{14}C recoveries were 96–97% of the administered dose, primarily in faeces (84–88%), urine (7%), milk (1–2%), liver (0.3%) and 0.01% or less in kidney, muscle and fat.

Table 2 Total radioactive residues in dissected tissues, excreta and milk of the lactating goat following 7 daily oral administrations of [^{14}C]-cyantraniliprole

Matrix	[CN-^{14}C]-cyantraniliprole		[PC-^{14}C]-cyantraniliprole	
	% administered dose	TRR (mg/kg)	% administered dose	TRR (mg/kg)
Urine	6.7	NA	6.9	NA
Faeces	87.5	NA	84.3	NA
Total excreted [a]	95.5		93.5	
Liver	0.3	0.46	0.3	0.495
Kidney	0.01	0.117	0.01	0.177
Total body muscle	NA	0.02	NA	0.043
Total body fat	NA	0.137	NA	0.336
Milk (day 1)	0.123	0.074	0.154	0.099
Milk (day 2)	0.16	0.089	0.239	0.148
Milk (day 3)	0.132	0.082	0.289	0.181
Milk (day 4)	0.151	0.078	0.27	0.156
Milk (day 5)	0.15	0.092	0.259	0.171
Milk (day 6)	0.135	0.093	0.314	0.18
Milk (day 7)	0.186	0.109	0.287	0.164
Milk composite (day 1-7)	1.04	0.08	1.81	0.147
Total	96.8			95.6

[a] Total excreted includes bile and cage wash

Solvent extraction was able to retrieve 99% TRR from milk, 90–98% TRR from fat, 61–81% TRR from muscle, 63–79% TRR from kidney and while the solvent extractable TRR in the liver were lower (54–60% TRR), an additional 21–27% TRR were recovered following digestion of the post extracted solids (PES) with protease.

Characterization and identification of residues used HPLC and/or LC/MS/MS with 88–90% TRR in milk, 69–70% TRR in liver and 75–80% TRR in fat being characterised and/or identified. In kidney and muscle, 42–43% of the CN-^{14}C label and 71% of the PC-^{14}C label was able to be characterised and/or identified.

In milk from the goat dosed with [CN-^{14}C]-cyantraniliprole, TRR on Day 1 were 0.07 mg/kg, reaching a plateau about Day 2 (0.09 mg/kg) and up to 0.11 mg/kg on Day 7. The major

radiochemical residue was cyantraniliprole, accounting for 40% TRR (0.03 mg/kg) with IN-MYX98 (15% TRR–0.01 mg/kg) being the significant metabolite found.

IN-N7B69 (12% TRR), IN-JCZ38 (7.2% TRR), IN-K7H19 (2.6% TRR), IN-HGW87 (1% TRR) and IN-J9Z38 (0.48% TRR) were identified as minor metabolites in milk, all present at less than 0.01 mg/kg.

In the goat dosed with [PC-^{14}C]-cyantraniliprole, results were similar, with TRR in milk on Day 1 being 0.1 mg/kg, reaching a plateau about Day 3 (0.18 mg/kg). Cyantraniliprole was the major residue, representing 50% TRR (0.07 mg/kg). The major metabolite was IN-MYX98, comprising 18% of TRR (0.03 mg/kg).

Other metabolites were identified as IN-K7H19, IN-JCZ38, IN-N7B69, IN-HGW87, IN-K5A78, IN-MLA84 and IN-J9Z38, each representing ≤ 0.01 mg/kg and < 4% TRR.

Table 3 Distribution of radioactive residues in goat milk following administration of [CN-^{14}C]-cyantraniliprole

Component	Milk [a]		Milk [a]	
	[CN-^{14}C]-cyantraniliprole		[PC-^{14}C]-cyantraniliprole	
	% TRR	mg/kg	% TRR	mg/kg
TRR	100.0	0.076	100.0	0.14
Extracted	99.3	0.075	99	0.139
Cyantraniliprole	39.5	0.03	49.6	0.07
IN-K7H19	2.6	0.00133	0.57	0.001
IN-JCZ38	7.2	0.005	1.32	0.002
IN-N7B69	11.8	0.008	2.01	0.003
IN-MYX98	15.1	0.011	18.3	0.026
IN-HGW87	1.1	< 0.001	0.69	0.001
IN-K5A78	ND		1.26	0.002
IN-MLA84	ND		2.24	0.003
IN-J9Z38	0.48	< 0.001	3.72	0.005
Unknowns	12.7	0.006	8.81	0.012
Total Char./Ident.	90.5	0.061	88.4	0.125
Total Unextracted	0.7	0.001	0.83	0.001

[a] Day 1–7 composite sample

Liver from the goat dosed with [CN-^{14}C]-cyantraniliprole contained 0.43 mg/kg TRR with cyantraniliprole being the major residue, accounting for 17% TRR (0.07 mg/kg). IN-K5A77 and IN-MLA84 were also found at up to 0.02 mg/kg (5% TRR).

Other minor components (0.6–2.6% TRR) were IN-K7H19, IN-JCZ38, IN-N7B69, IN-K5A79, IN-MYX98, IN-HGW87 and IN-K5A78, each at ≤ 0.01 mg/kg. About 32% TRR was made up of a large number of unknown components, none of which were found at levels greater than 0.006 mg/kg (1.5% TRR).

In the goat dosed with [PC-^{14}C]-cyantraniliprole, liver contained 0.5 mg/kg TRR, the major component being cyantraniliprole at 27% TRR (0.14 mg/kg). IN-K5A77 and IN-MLA84 were each present at 0.02–0.03 mg/kg (5–6% TRR) with IN-K5A78 and IN-MYX98 also found at about 3.5% TRR (< 0.02 mg/kg).

Other minor components were IN-K7H19, IN-HGW87, IN-J9Z38, IN-K5A79, IN-JCZ38 and IN-N7B69, each at ≤ 0.01 mg/kg and not more than 1% TRR. About 18% TRR was made up of a number of unknown components, none of which were found at levels above 0.009 mg/kg (1.8% TRR).

In kidney from the goat dosed with [CN-^{14}C]-cyantraniliprole, TRR were 0.14 mg/kg with the major residue being cyantraniliprole, accounting for 13% TRR (0.02 mg/kg).

Minor metabolites, present at up to 7% TRR (0.01 mg/kg or less) were IN-MYX98, IN-MLA84, IN-K7H19, IN-J9Z38 and IN-HGW87. Unknown peaks 13% TRR) individually accounted for ≤ 0.01 mg/kg.

In the goat dosed with [PC-^{14}C]-cyantraniliprole, kidney contained TRR of 0.21 mg/kg, 19% of which was cyantraniliprole (0.04 mg/kg). The IN-MYX98 metabolite (7.1% TRR–0.014 mg/kg) and one unknown component, more polar than parent, (present at 11% TRR–0.02 mg/kg) were the only residues found at levels above 0.01 mg/kg.

IN-K7H19, IN-JCZ38, IN-N7B69, IN-HGW87, IN-K5A77 and IN-MLA84 were identified as minor metabolites (< 2.5% TRR) each representing ≤ 0.01 mg/kg parent equivalents.

In muscle from the [CN-^{14}C]-cyantraniliprole dosed goat, TRR were 0.03 mg/kg with the only significant residue being cyantraniliprole, this making up 30% TRR (0.01 mg/kg).

IN-K7H19 was also identified as a minor metabolite (< 0.01 mg/kg). Minor unknowns totalled 7.6% TRR for three peaks each < 0.01 mg/kg parent equivalents.

In the goat dosed with [PC-^{14}C]-cyantraniliprole, muscle contained 0.04 mg/kg TRR the principal ^{14}C-residue was IN-MYX98, accounting for 33% TRR (0.01 mg/kg parent equivalents) with cyantraniliprole making up 15% TRR (0.01 mg/kg).

IN-K5A77 and IN-J9Z38 each represented ≤ 0.01 mg/kg and unidentified components accounted for a total of 17% TRR (0.01 mg/kg).

For fat, omental, subcutaneous and renal fat were sampled and analyzed separately. TRR levels were similar in the three types of fat, with the respective PC-label residues of 0.117, 0.119 and 0.115 mg/kg and 0.047, 0.046 and 0.049 mg/kg for the CN-label. Results are reported as the mean values of the three types of fat in this evaluation.

For the goat dosed with [CN-^{14}C]-cyantraniliprole, the calculated mean TRR in fat was 0.05 mg/kg with cyantraniliprole (31% TRR–0.02 mg/kg) being the major residue. In the three types of fat, cyantraniliprole residues were 23% TRR (omental), 42% TRR (subcutaneous) and 34% TRR (renal). The metabolite IN-J9Z38 accounted for an average of 27% TRR (0.01 mg/kg), present at 24% TRR in omental fat, 22% TRR in subcutaneous fat and 37% TRR (0.02 mg/kg) in renal fat.

All other individual metabolites accounted for ≤ 0.01 mg/kg each (IN-K7H19, IN-N7B69, IN-K5A79, IN-MYX98, IN-NBC94, IN-K5A78, and IN-MLA84. Unknown metabolite peaks individually accounted for ≤ 0.01 mg/kg.

For the goat dosed with [PC-^{14}C]-cyantraniliprole, the calculated mean TRR in fat was 0.12 mg/kg. Cyantraniliprole was the major residue, representing 45% TRR (0.05 mg/kg) on average, with 58% TRR (0.07 mg/kg) in omental fat, 43% TRR in subcutaneous fat and 36% TRR in renal fat. The IN-J9Z38 metabolite was present at 24% TRR (0.03 mg/kg) on average with 0.5% TRR in omental fat, 17% TRR in subcutaneous fat and 56% TRR (0.06 mg/kg) in renal fat.

IN-K7H19, IN-DBC80, IN-N7B69, IN-K5A79, IN-MYX98, IN-K5A77, IN-NBC94, IN-K5A78, and IN-MLA84 were all identified as minor metabolites, each accounted for < 0.01 mg/kg. Individual unknowns accounted for < 0.01 mg/kg parent equivalents.

Table 4 Distribution of radioactive residues in goat tissues following administration of [CN-^{14}C]-cyantraniliprole

Component	Liver				Kidney		Muscle		Fat [a]	
	Solvent Extract		Protease Digest							
	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
TRR	100.0	0.43	100.0	0.43	100.0	0.136	100.	0.027	100.	0.052
Extracted	54.3	0.233	26.8	0.115	62.7	0.085	61.0	0.016	90.4	0.047
Cyantraniliprole	17.1	0.073	0.3	0.002	12.7	0.017	30.3	0.009	30.8	0.016
IN-K7H19	1.0	0.004	0.9	0.005	3.0	0.004	4.6	0.001	1.9	< 0.001
IN-JCZ38	0.83	0.004	0.4	0.003	ND		ND		ND	
IN-N7B69	0.64	0.002	1.8	0.008	ND		ND		1.9	< 0.001

Component	Liver				Kidney		Muscle		Fat [a]	
	Solvent Extract		Protease Digest							
	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
IN-K5A79	0.61	0.003	ND		ND		ND		1.9	< 0.001
IN-MYX98	2.5	0.011	0.1	< 0.001	7.1	0.01	ND		1.9	< 0.001
IN-HGW87	1.1	0.004	0.1	0.001	0.61	0.001	ND		ND	
IN-K5A77	5.4	0.023	ND		ND		ND		ND	
IN-NBC94	ND		ND		ND		ND		1.9	< 0.001
IN-K5A78	0.83	0.003	0.2	0.001	ND		ND		1.9	< 0.001
IN-MLA84	3.7	0.017	0.2	0.001	4.1	0.005	ND		1.9	0.001
IN-J9Z38	ND		ND		1.1	0.001	ND		26.9	0.014
Unknowns	13.2 [b]	0.053 [b]	18.6 [c]	0.080 [c]	13.2	0.018	7.6	0.003	11.5	0.006
Total Char./Ident.	46.9	0.202	22.6	0.101	41.8	0.056	42.5	0.013	75.0	0.039
Total Unextracted	45.7	0.197	18.9 [d]	0.082 [d]	37.4	0.051	39.2	0.011	9.6	0.005

[a] Concentrations are a mean of omental, renal, and subcutaneous fat samples analysed

[b] 13 components, none greater than 0.006 mg/kg (1.4% TRR)

[c] 32 components, none greater than 0.006 mg/kg (1.5% TRR)

[d] Terminal unextracted residues following protease digestion

Table 5 Distribution of radioactive residues in goat tissues following administration of [PC-^{14}C]-cyantraniliprole

Component	Liver				Kidney		Muscle		Fat [a]	
	Solvent Extract		Protease Digest							
	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
TRR	100.0	0.5	100.0	0.5	100.0	0.206	100.0	0.040	100.0	0.119
Extracted	59.5	0.296	20.7	0.103	78.6	0.162	80.9	0.032	98.3	0.117
Cyantraniliprole	27.3	0.136	ND		18.9	0.04	15.3	0.006	45.4	0.054
IN-K7H19	0.95	0.005	0.5	0.003	2.3	0.004	ND		< 0.8	< 0.001
IN-JCZ38	0.42	0.002	ND		0.64	0.001	ND		ND	
IN-DBC80	ND		ND		ND		ND		< 0.8	< 0.001
IN-N7B69	0.32	0.002	ND		1.7	0.003	ND		< 0.8	< 0.001
IN-K5A79	0.61	0.003	ND		ND		ND		2.5	< 0.003
IN-MYX98	3.6	0.017	ND		7.1	0.014	32.8	0.014	< 0.8	< 0.001
IN-HGW87	1.0	0.005	ND		1.1	0.002	ND		ND	
IN-K5A77	5.7	0.028	ND		0.70	0.001	4.4	0.001	< 0.8	< 0.001
IN-NBC94	ND		ND		ND		ND		1.7	0.002
IN-K5A78	3.4	0.017	0.3	0.002	ND		ND		< 0.8	< 0.001
IN-MLA84	5.3	0.026	0.4	0.002	1.8	0.004	ND		1.7	0.002
IN-J9Z38	0.9	0.004	ND		ND		1.1	< 0.001	24.4	0.029
Unknowns	6.2 [b]	0.031 [b]	11.6 [c]	0.059 [c]	36.9 [d]	0.076 [d]	17.1	0.006	5.0	0.006
Total Char./Ident.	55.7	0.279	12.8	0.066	71.0	0.145	70.9	0.027	79.8	0.095
Total Unextracted	40.5	0.204	19.9 [e]	0.101	21.5	0.044	19.2	0.008	1.7	0.002

[a] Concentrations are a mean of omental, renal and subcutaneous fat samples analysed

[b] 7 components, none greater than 0.008 mg/kg (1.6% TRR)

[c] 19 components, none greater than 0.009 mg/kg (1.8% TRR)

[d] 10 components, none greater than 0.023 mg/kg (11.4% TRR)

[e] Terminal unextracted residues following protease digestion

Cyantraniliprole was rapidly eliminated by the lactating goat, primarily in the excreta (94–96% of the applied dose). There was no significant transfer of residues of cyantraniliprole and its metabolites to fat, meat or edible offal as these tissues contained only ca. 1–2% of the total administered dose. Approximately 1–2% of the total administered dose was eliminated via the milk.

Significant metabolites found at more than 10% TRR or 0.01 mg/kg were IN-MYX98 in milk and muscle and IN-J9Z38 in fat.

The biotransformation of cyantraniliprole in the goat is consistent with pathways proposed for poultry (laying hens) and the rat and is outlined below.

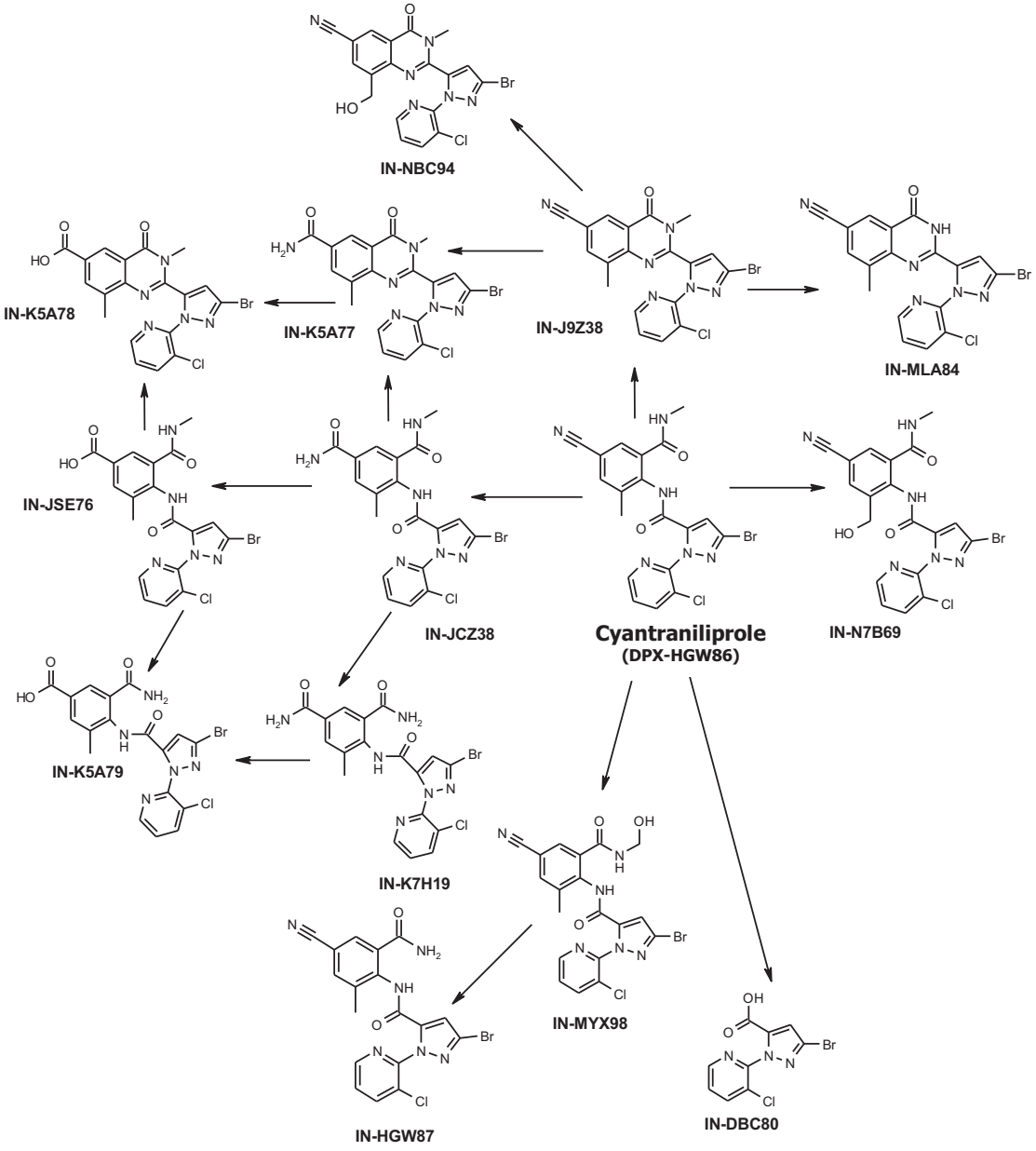


Figure 1 Proposed metabolic pathway of cyantraniliprole in lactating goats

Laying hens

In a study reported by McLellan Lowry, 2008 [Ref: DP-16988], one group of five hens was orally administered [CN-^{14}C]-cyantraniliprole in gelatine capsules at a dose rate equivalent to 11.6 ppm in the diet for 14 consecutive days and a second group of five hens was similarly dosed with the dietary equivalent of 11.2 ppm [PC-^{14}C]-cyantraniliprole.

For each group, eggs were collected twice daily, excreta were collected daily, and tissues were collected after sacrifice, approximately 23 hours after the last dose.

Samples were triple-extracted with acetonitrile:water (9:1) centrifuged and the radioactive content was determined by LSC analysis of duplicate aliquots. Liver samples were further processed by enzyme digestion using protease. Extracts were analysed using reversed phase HPLC (UV detection at 254 nm) eluted with a gradient of acetonitrile and water containing 0.1% formic acid and quantification of radioactive residues was accomplished either by on-line radio detection with peak integration or by fraction collection and LSC. Identification of radioactive residues was based on comparison of HPLC retention times with those of authentic reference standards. LC-MS/MS in electrospray positive ion mode was conducted to confirm the presence of parent and selected metabolites. All samples were stored frozen (-20 °C) for no more than 55 days before extraction and analyzed within 62 days after sampling.

The overall ^{14}C recoveries of the administered dose for both radiolabels were consistent with 97–100% of the total administered dose found in the excreta. Based on composite Day 1–14 samples, egg whites accounted for 0.4–0.5% (0.20–0.26 mg/kg) while egg yolks contained 0.07% (0.09 mg/kg) of the total administered dose. The highest concentrations of total radioactivity in tissues were observed in liver (0.2 mg/kg PC-^{14}C label, 0.14 mg/kg CN-^{14}C label). Abdominal fat, skin with fat and muscle each contained ≤ 0.1% of the total administered dose for either label, each equivalent ≤ 0.01 mg/kg.

The concentration of radioactivity in egg whites was variable, increasing to 0.26 mg/kg (PC-^{14}C label) and 0.56 mg/kg (CN-^{14}C label) in eggs sampled 2 days after the first dose, decreasing to a steady state of about 0.2–0.24 mg/kg after 7 days but with concentrations of the PC-^{14}C label dropping to 0.12 mg/kg on day 13 and increasing to 0.38 mg/kg on day 14. Radioactivity in egg yolks increased steadily reaching a plateau concentration of about 0.1 mg/kg after 5 days (CN-^{14}C label) and 7 days (PC-^{14}C label).

Table 6. Total radioactive residues in dissected tissues, excreta and eggs of laying hens following 14 daily oral administrations of [^{14}C]-cyantraniliprole

Matrix	[CN-^{14}C]-cyantraniliprole				[PC-^{14}C]-cyantraniliprole			
	% administered dose		TRR (mg/kg)		% administered dose		TRR (mg/kg)	
Excreta	96.95		NA		99.72		NA	
Cage wash	3.83		NA		2.52		NA	
Total excreted	100.8							
Fat	0.004		0.004		0.01		0.005	
Liver	0.026		0.141		0.04		0.205	
Muscle	0.006		0.003		0.01		0.005	
Skin with fat	0.01		0.005		0.01		0.007	
Eggs	white	yolk	white	yolk	white	yolk	white	yolk
Day 1	0.01	0.0	0.081	0.004	0.017	0.0	0.127	0.008
Day 2	0.066	0.002	0.56	0.051	0.035	0.001	0.259	0.027
Day 3	0.064	0.005	0.356	0.066	0.034	0.002	0.244	0.038
Day 4	0.057	0.005	0.388	0.084	0.034	0.003	0.256	0.062
Day 5	0.05	0.006	0.329	0.106	0.016	0.004	0.117	0.065
Day 6	0.037	0.006	0.241	0.101	0.026	0.005	0.185	0.084
Day 7	0.031	0.007	0.207	0.114	0.02	0.005	0.186	0.098
Day 8	0.035	0.006	0.242	0.111	0.03	0.006	0.218	0.109
Day 9	0.036	0.006	0.237	0.11	0.024	0.006	0.168	0.106
Day 10	0.027	0.006	0.174	0.1	0.034	0.009	0.195	0.112
Day 11	0.022	0.005	0.191	0.1	0.022	0.006	0.159	0.099
Day 12	0.023	0.005	0.155	0.087	0.03	0.006	0.221	0.103
Day 13	0.03	0.006	0.198	0.092	0.01	0.004	0.12	0.1
Day 14	0.05	0.008	0.233	0.099	0.065	0.009	0.377	0.126
Eggs—composite (Days 1–14)	0.54	0.07	0.259	0.092	0.4	0.07	0.203	0.087
Total	101.4				102.7		95.6	

Solvent extraction was able to retrieve more than 99% TRR from egg white, 79–83% TRR in egg yolk and 17–23% TRR in liver. An additional 38% TRR were recovered following digestion of the post extracted solids (PES) with protease. Low levels of radioactivity < 0.001 mg/kg) were

detected in muscle, abdominal fat and skin + fat, with solvent extraction recovering 100% TRR in muscle, 63% TRR (PC-^{14}C label) and 120% TRR (CN-^{14}C label) in abdominal fat and 53% TRR (PC-^{14}C label) and 72% TRR (CN-^{14}C label) in skin + fat.

Characterization and identification of residues by HPLC (with LC-MS confirmation) was conducted on composite egg white and egg yolk samples (day 1–14) and liver. No analysis was carried out with muscle, abdominal fat or skin + fat samples because of the low residues (< 0.004 mg/kg) present. About 95% TRR in egg white, 71% TRR in egg yolk and 35–42% TRR in liver was able to be characterised and/or identified.

In egg white, TRRs were 0.26 mg/kg (CN-^{14}C label) and 0.2 mg/kg (PC-^{14}C label). Cyantraniliprole was the major ^{14}C-residue representing 32–42% TRR (0.09 mg/kg). IN-MLA84 (18–19% TRR) and IN-J9Z38 (17–29% TRR) were principal egg white metabolites, found at 0.04–0.05 mg/kg and 0.03–0.08 mg/kg respectively.

IN-HGW87, IN-K7H19, IN-MYX98, and IN-NBC94 were also identified in each composite sample, present at ≤ 8.1% TRR (≤ 0.017 mg/kg).

In egg yolk, TRRs were 0.09 mg/kg, with cyantraniliprole making up about 9–10% TRR (0.01 mg/kg). Significant metabolites were IN-MLA84, IN-HGW87 and IN-J9Z38, each present at 12–17% TRR (up to 0.015 mg/kg).

IN-JSE76, IN-K5A77, IN-K5A79, IN-MYX98, and IN-NBC94 were also found in egg yolks as minor metabolites each representing 1–6% TRR (≤ 0.006 mg/kg).

In liver, TRRs were 0.14 mg/kg (CN-^{14}C label) and 0.17 mg/kg (PC-^{14}C label). Cyantraniliprole was not found any samples. IN-JCZ38, IN-K5A78, IN-K5A79, IN-K7H19, IN-MLA84, IN-MYX98 and IN-N7B69 were found as minor metabolites, each representing < 4% TRR (< 0.01 mg/kg).

Table 7 Distribution of radioactive residues in hen eggs and liver following administration of [CN-^{14}C]-cyantraniliprole

Component	Egg white (Day 1–14)		Egg yolk (Day 1–14)		Liver			
					Aqueous ACN		Protease digest	
	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
Total	100.0	0.259	100.0	0.092	100.0	0.137	100.0	0.137
Total extracted	99.01	0.256	79.3	0.073	17.07	0.023	38.09	0.052
Cyantraniliprole	32.48	0.084	10.28	0.009	ND		ND	
IN-HGW87	0.61	0.002	11.98	0.011	ND		ND	
IN-J9Z38	29.21	0.075	7.42	0.006	2.08	0.003	ND	
IN-MLA84	18.66	0.049	11.56	0.011	0.89	0.001	ND	
IN-MYX98	6.4	0.017	5.42	0.006	0.42	0.001	1.1	0.002
IN-NBC94	0.96	0.002	0.86	0.001	ND		0.4	0.001
Total characterized unknowns [a]	7.75	0.021	23.62	0.025	7.58	0.010	22.33	0.042
Total characterized/identified [b]	96.07	0.25	71.14	0.068	10.97	0.015	23.82	0.045
Total uncharacterized/ unidentified [c]	2.94	0.008	8.16	0.005	6.1	0.008	16.94	0.023
Total unextracted	0.99	0.003	20.7	0.019	82.93	0.114	44.97 [d]	0.062 [e]

[a] Up to 32 components, each <5% TRR and < 0.005 mg/kg (except one liver component at 0.008 mg/kg)

[b] Total characterized/identified=Sum of identified metabolites + Total characterized unknowns

[c] Total uncharacterized/unidentified=Total extracted–Total characterized/identified

[d] Does not include 16.9% TRR unaccounted for during sample processing

[e] Does not include 0.023 mg/kg unaccounted for during sample processing

ND=Not Detected

Table 8 Distribution of radioactive residues in hen eggs and liver following administration of [PC-^{14}C]-cyantraniliprole

Residue	Egg white (Day 1–14)		Egg yolk (Day 1–14)		Liver			
					Aqueous ACN		Protease digest	
	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
Total	100.0	0.203	100.0	0.087	100.0	0.174	100.0	0.174
Total extracted	99.7	0.202	83.1	0.072	23.0	0.04	37.6	0.065
Cyantraniliprole	41.7	0.085	9.33	0.008	ND		ND	
IN-HGW87	0.74	0.001	ND		ND		ND	
IN-J9Z38	17.1	0.034	13.1	0.011	ND		ND	
IN-JSE76	ND		1.9	0.002	ND		ND	
IN-JCZ38	ND		ND		ND		0.23	< 0.001
IN-K5A77	ND		6.19	0.005	ND		ND	
IN-K5A78	ND		ND		0.23	< 0.001	ND	
IN-K5A79	3.9	0.007	1.52	0.001	0.27	< 0.001	0.45	0.001
IN-K7H19	ND		ND		ND		0.46	< 0.001
IN-MLA84	18.2	0.037	16.8	0.015	3.98	0.007	ND	
IN-MYX98	8.13	0.016	2.09	0.002	0.27	< 0.001	ND	
IN-N7B69	ND		ND		ND		0.55	0.001
IN-NBC94	2.86	0.005	1.24	0.001	ND		ND	
Total characterized unknowns [a]	0.98	0.002	17.4	0.015	10.5	0.02	25.0	0.045
Total characterized/ identified [b]	93.6	0.187	69.6	0.06	15.2	0.027	26.7	0.047
Total uncharacterized /unidentified [c]	6.09	0.014	13.5	0.012	7.8	0.013	10.9	0.018
Total unextracted	0.33	0.001	16.9	0.015	77.0	0.134	39.5 [d]	0.053 [e]

[a] Up to 24 components, each < 5% TRR and < 0.005 mg/kg (except one liver component at 7% TRR–0.007 mg/kg)

[b] Total characterized/Identified=Sum of identified metabolites + Total characterized unknowns

[c] Total uncharacterized/unidentified=Total extracted–Total characterized/identified

[d] Does not include 23.0% TRR unaccounted for during sample processing

[e] Does not include 0.056 mg/kg unaccounted for during sample processing

ND=Not Detected

Cyantraniliprole was rapidly eliminated in laying hens with more than 97% being excreted, mostly as unchanged parent compound (68–74%). About 0.5–0.6% of the total administered dose was observed in eggs, fat, liver, and muscle, with unchanged cyantraniliprole being the principal (0.08 mg/kg) residue found in egg whites and found at lower levels in yolks (0.01 mg/kg). Cyantraniliprole was not detected in any of the liver samples.

A number of metabolites were identified in egg whites and egg yolks. IN-J9Z38 and IN-MLA84 were the principal metabolites (0.03–0.08 mg/kg) in egg whites but accounted for ≤ 0.02 mg/kg in yolks. In liver, about 35–40% TRR was characterized/identified with most of the TRR remaining bound following solvent and protease extractions.

The proposed metabolic pathway in laying hens was consistent with that proposed for rats and lactating goats, and is outlined below:

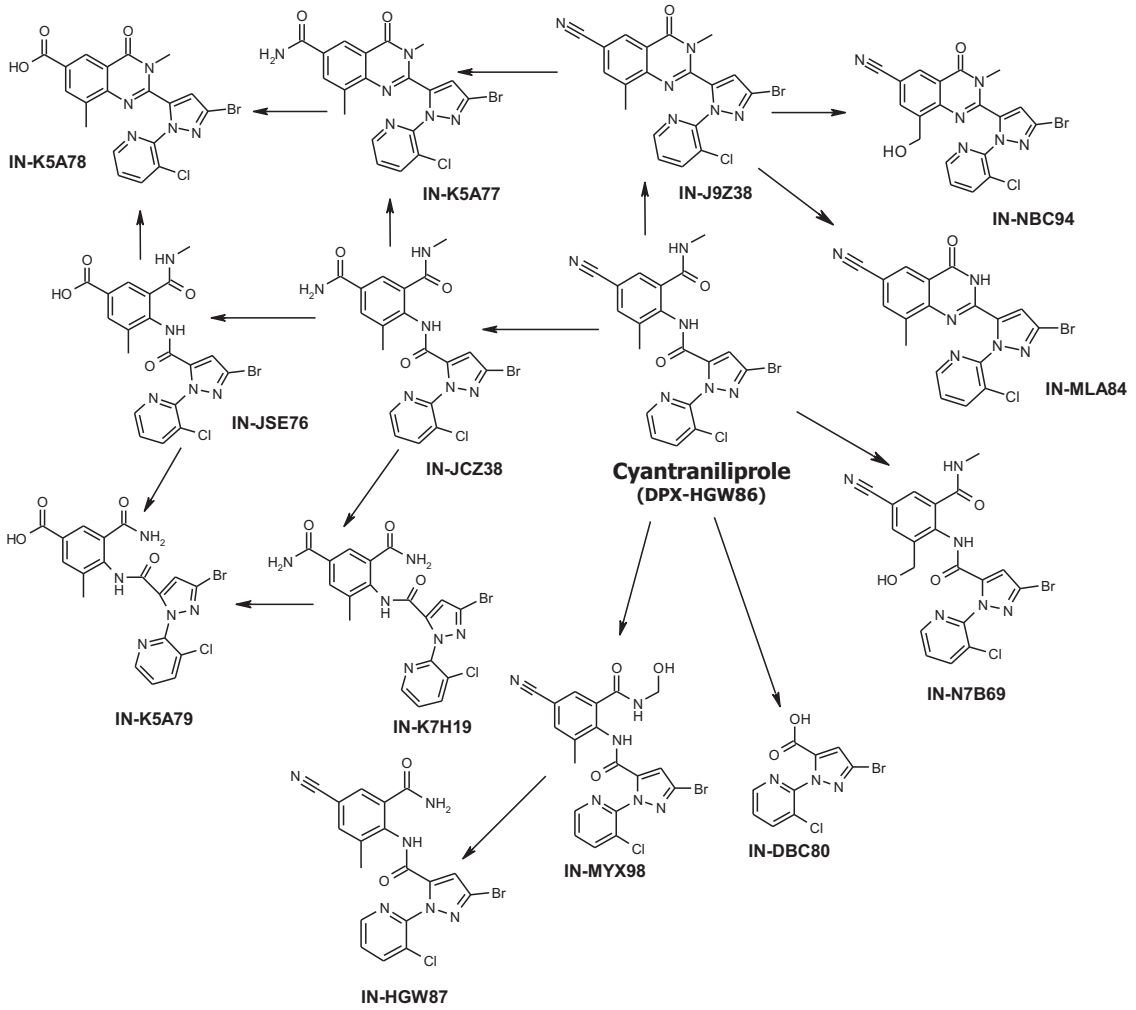


Figure 2 Proposed metabolic pathway of cyantraniliprole in laying hens

Plant metabolism

The Meeting received plant metabolism studies on cotton, lettuce, tomato and rice seedlings following foliar and soil treatments with [^{14}C]-cyantraniliprole. A 1:1 (μCi/μCi ratio) mixture of [CN-^{14}C]-cyantraniliprole and [PC-^{14}C]-cyantraniliprole was used in the foliar treatments and separate treatments of the two radiolabels were applied as soil treatments.

The Meeting noted that the IN-DBC80 metabolite was the only cleavage product containing just the PC-^{14}C label and as this was found only at low concentrations in some metabolism studies, it was agreed that the use of the mixed radiolabels in the foliar treatment studies did not significantly impact on the results of these studies.

Cotton

In a study reported by MacDonald, MacKinnon & Chapleo (2008) [Ref: DP-16984], three applications of [^{14}C]-cyantraniliprole equivalent to 150 g ai/ha/treatment, were made to cotton plants grown in 30 cm pots containing an acidic (pH 6.4) commercial growing medium (since abiotic degradation of cyantraniliprole occurs at alkaline pH). Foliar applications (SC formulation) were made to plants 3 weeks post-emergence and at 7 and 14 days after the initial application. Soil drench

applications (SC formulation), pipetted onto wetted soil were made 7 weeks post-emergence and at 7 and 14 days after the first application.

Leaves were collected from immature plants after the first application (foliar treatment only), immediately prior to and following the second and third applications, and at ca. 7 and 14 days after the third application. Cotton bolls (seed plus lint) and the remaining aerial portions (cotton-gin by-products, gin trash) were sampled at crop maturity (ca. 125 days after the third application). The bolls were separated into undelinted (fuzzy) seed and lint.

Selected samples from the foliar-treatment regime were surface washed with acetonitrile: water (7:3, v/v) immediately after sampling. Plant samples from both regimes were pulverized and total tissue residues determined by oxidative combustion analysis. Where characterization of radioactivity was warranted, samples were extracted three times using acetone:1N formic acid (9:1). The unextracted radioactivity in the post-extracted solids (PES) was determined by combustion analysis. Extracts containing significant radioactivity (≥ 0.01 mg/kg) were analysed by high performance liquid chromatography (HPLC) and identification of ^{14}C-residues was accomplished by HPLC and liquid chromatography mass spectrometry (LC-MS), with reference to authenticated reference standards. Samples were stored at ca -20 °C for up to 28 days before analysis.

To assess if storage at –20 °C resulted in degradation of radioactive residues, a subsample of stored tissue (7 days post-application 3, foliar treatment) was extracted with acetone:formic acid following a storage period of 392 days. The TRR and extractability values after prolonged storage (0.449 mg/kg, 74.4% TRR) were similar to those obtained originally (0.505 mg/kg, 81.5% TRR). The residue composition also remained unchanged with cyantraniliprole being the principle component (30.8% TRR, 0.138 mg/kg) and minor components individually making up ≤ 4.6% TRR (≤ 0.021 mg/kg).

A comparison of the extraction efficiency of acetone:formic acid and acetonitrile:water showed similar results, with the extracted radioactivity from unwashed leaves from the foliar-treated plants being 76% TRR in the acetonitrile extract and 74% TRR in the acetone extract. HPLC analysis of the two extracts also reported comparable results, with cyantraniliprole making up 27% of the TRR in the acetone extract and 21% of the TRR in the acetonitrile extract.

In plants from the soil drench treatments, TRRs in leaves from immature plants, lint and undelinted seed were all ≤ 0.005 mg/kg with TRR values in cotton gin by-products being 0.1 mg/kg (CN-label) and 0.02 mg/kg (PC-label).

The majority of the radioactivity in cotton gin by-products (79.6–99.9% TRR) was extracted into acetone:formic acid. Additional amounts of radioactivity were extracted from the CN-label sample using enzymes (7.4% TRR), 1 M HCl at 60 °C (3.9% TRR), 6 M HCl at 90 °C (2.2% TRR) and 6 M NaOH at 90 °C (1.8% TRR), each releasing < 0.01 mg/kg.

Cyantraniliprole was the major radioactive residue in gin by-products at concentrations of 25.6% TRR (0.02 mg/kg) for the CN-label and 46.8% TRR (0.01 mg/kg) for the PC-label.

Gin by-products from [CN-^{14}C]-treated cotton also contained IN-J9Z38, IN-JCZ38, IN-JSE76, IN-HGW87, IN-QKV54 and unresolved IN-MLA84/IN-NXX70, each at ≤ 0.01 mg/kg and less than 8% TRR. The only identified metabolite in gin by-products from [PC-^{14}C]-treated cotton was IN-J9Z38 (< 0.01 mg/kg).

Table 9 Total radioactive residues (mg/kg) in cotton samples following soil drench treatments with [^{14}C]-cyantraniliprole

Treatment	Leaves				Lint	Seed	Gin by-products
Sample point	7 DAT1	7 DAT2	7–8 DAT3	13–14 DAT3	124–125 DAT3		
[CN-^{14}C]-cyantraniliprole	< 0.001	0.005	< 0.001	0.002	< 0.001	< 0.001	0.095
[PC-^{14}C]-cyantraniliprole	0.002	0.002	< 0.001	0.002	< 0.001	< 0.001	0.023

Table 10 Distribution of radioactive residues in cotton gin by-products 124 days after the last of three soil drench treatments with [^{14}C]-cyantraniliprole

^{14}C-Residue	[CN-^{14}C]-cyantraniliprole		[PC-^{14}C]-cyantraniliprole	
	%TRR	mg/kg	%TRR	mg/kg
TRR, mg/kg		0.095		0.023
Extracted	95.7	0.09	99.9	0.023
Cyantraniliprole	25.6	0.025	46.8	0.011
IN-HGW87	2.6	0.003		ND
IN-J9Z38	7.5	0.006	4.7	0.001
IN-JCZ38	1.2	< 0.001		ND
IN-JSE76	1.7	< 0.001		ND
IN-MLA84/ IN-NXX70	5.7	0.004		ND
IN-QKV54	6.4	0.005		ND
Unidentified	16.4 [a]	0.007	11.6 [b]	0.003

[a] 48 components; none greater than 0.002 mg/kg (2.6%TRR)

[b] One component

TRRs in cotton leaves following foliar treatment with the 1:1 mixture of [CN-^{14}C]-cyantraniliprole + [PC-^{14}C]-cyantraniliprole were 2.7 mg/kg immediately after the first application, 14.5 mg/kg immediately after the second application and 7.9 mg/kg just after the last application. Residues decreased to 0.43 mg/kg at 13 days after the last application. At harvest, TRRs in cotton gin by-products, lint and undelinted seed were 0.13, 0.01, and < 0.01 mg/kg, respectively.

The majority of the radioactivity (74–87%TRR) in immature leaves was removed by surface washing (56–70% TRR) and extraction into acidified aqueous acetone (27–33% TRR). Low concentrations of radioactivity were subsequently extracted using enzymes (4.1–7.7% TRR), 1 M HCl at 60 °C (2.9–4.7% TRR), 6 M HCl at 90 °C (0.6–4.5% TRR) and 6 M NaOH at 90 °C (1.1–8.0% TRR).

Cyantraniliprole was the major residue in leaves, decreasing from 69.7% TRR immediately after the first application to 19.7% TRR seven days later and accounted for 37.3 and 27.1% TRR, respectively, 7 and 13 days after the final application.

The photodegradate, IN-NXX70, found predominately in the surface wash, accounted for 21.7% TRR seven days after the first application, declining to 1.1% TRR 7 days after the third application.

The metabolite IN-QKV54 accounted for 10.3% TRR in leaf extracts 7 days after the first application. Other metabolites identified in leaf samples (IN-DBC80, IN-J9Z38, IN-JCZ38, IN-JSE76, IN-K5A77, IN-K7H19, IN-MYX98 and IN-N7B69) each did not exceed 5% TRR. Numerous other metabolites detected but not identified were each present at less than 10% TRR (0.04 mg/kg). Unextracted leaf residues accounted for ≤ 13.2% TRR (≤ 0.38 mg/kg).

Surface washing of cotton gin by-products removed about 19% TRR (0.025 mg/kg) with a further 40% TRR being extracted using acidified aqueous acetone. Additional radioactivity was extracted using enzymes (7.4% TRR), 1 M HCl at 60 °C (5.4% TRR), 6 M HCl at 90 °C (7.5% TRR) and 6 M NaOH at 90 °C (4.2% TRR), giving a total extraction efficiency of 83.8% TRR (0.11 mg/kg).

Cyantraniliprole was the main component, accounting for 34.4% TRR (0.04 mg/kg). Other metabolites including IN-J9Z38 (5.7% TRR), IN-JCZ38 (6.1% TRR) and IN-N7B69 (1.2% TRR) were present at ≤ 0.01 mg/kg. Other metabolites detected but not identified did not individually exceed 0.9% TRR or 0.001 mg/kg. Unextracted residues accounted for 16.2% TRR (0.02 mg/kg).

Table 11 Distribution of radioactive residues in leaves and cotton gin by-products from plants treated with foliar applications of a 1:1 mixture of [CN-^{14}C]-cyantraniliprole and [PC-^{14}C]-cyantraniliprole

Sampling point	0 DAT1 Leaves	7 DAT1 Leaves	7 DAT2 Leaves	7 DAT3 Leaves	13 DAT3 Leaves	124 DAT3 Gin by-products

TRR, mg/kg	2.709		5.414		2.654		0.505		0.425		0.131	
	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg
Surface wash	55.9	1.514	66.6	3.606	69.9	1.855					19.1	0.025
Tissue extracts [a]	30.9	0.837	33.4	1.808	26.8	0.711					64.7	0.086
Total Extracted	86.8	2.35	100	5.414	96.7	2.566	97.8	0.494	97.9	0.416	83.8	0.111
Unextracted	13.2	0.358	< 0.1	< 0.001	3.5	0.093	2.2	0.011	2	0.009	16.2	0.021
Cyantraniliprole	69.7	1.89	19.7	1.066	70.7	1.873	37.3	0.187	27.1	0.115	34.4	0.043
IN-DBC80	ND		1.2		ND		ND		ND		ND	
IN-J9Z38	1.0	0.028	3.5	0.19	2.9	0.078	2.3	0.011	1.5	0.006	5.7	0.008
IN-JCZ38	ND		1.3	0.069	ND		ND		ND		6.1	0.007
IN-JSE76	ND		0.7	0.039	0.1	0.003	ND		ND		ND	
IN-K5A77	ND		0.9	0.05	ND		ND		ND		ND	
IN-K7H19	ND		1.7	0.089	0.7	0.018	4.1	0.019	4.9	0.021	ND	
IN-MLA84	3.3	0.091	0.8	0.049	0.5	0.013	3.5	0.017	3.3	0.014	ND	
IN-MYX98	ND		2.0	0.106	ND		ND		ND		ND	
IN-N7B69	ND		0.3	0.017	ND		ND		1.1	0.005	1.2	0.001
IN-NXX70	--		21.7	1.172	--		1.1	0.006	--		--	
IN-QKV54	ND		10.3	0.577	1.5	0.039	5.0	0.025	3.8	0.016	ND	
Unidentified	4.4 [b]	0.12	13.9 [c]	0.749	6.9 [d]	0.167	25.2 [e]	0.122	41.3 [f]	0.176	7.3 [g]	0.002

Note: TRR in leaves at 0 DAT2 were 14.5 mg/kg and were 7.9 mg/kg at 0 DAT3

[a] Sequential extraction with acetone:1 N formic acid (3×); a mixture of cellulase, beta-glucosidase and driselase (2× for 24 hours at 37 °C); 1 M HCl (6 hours at 60 °C); 6 M HCl (6 hours at 90 °C) and 6 M NaOH (6 hours at 60 °C)

[b] 8 components; none greater than 0.035 mg/kg (1.3%TRR)

[c] 47 components; none greater than 0.163 mg/kg (3.0%TRR)

[d] 28 components; none greater than 0.047 mg/kg (1.8%TRR)

[e] 32 components; none greater than 0.031 mg/kg (6.2%TRR)

[f] 31 components; none greater than 0.042 mg/kg (9.9%TRR)

[g] 20 components; none greater than 0.001 mg/kg (0.9%TRR)

The proposed pathway is outlined below.

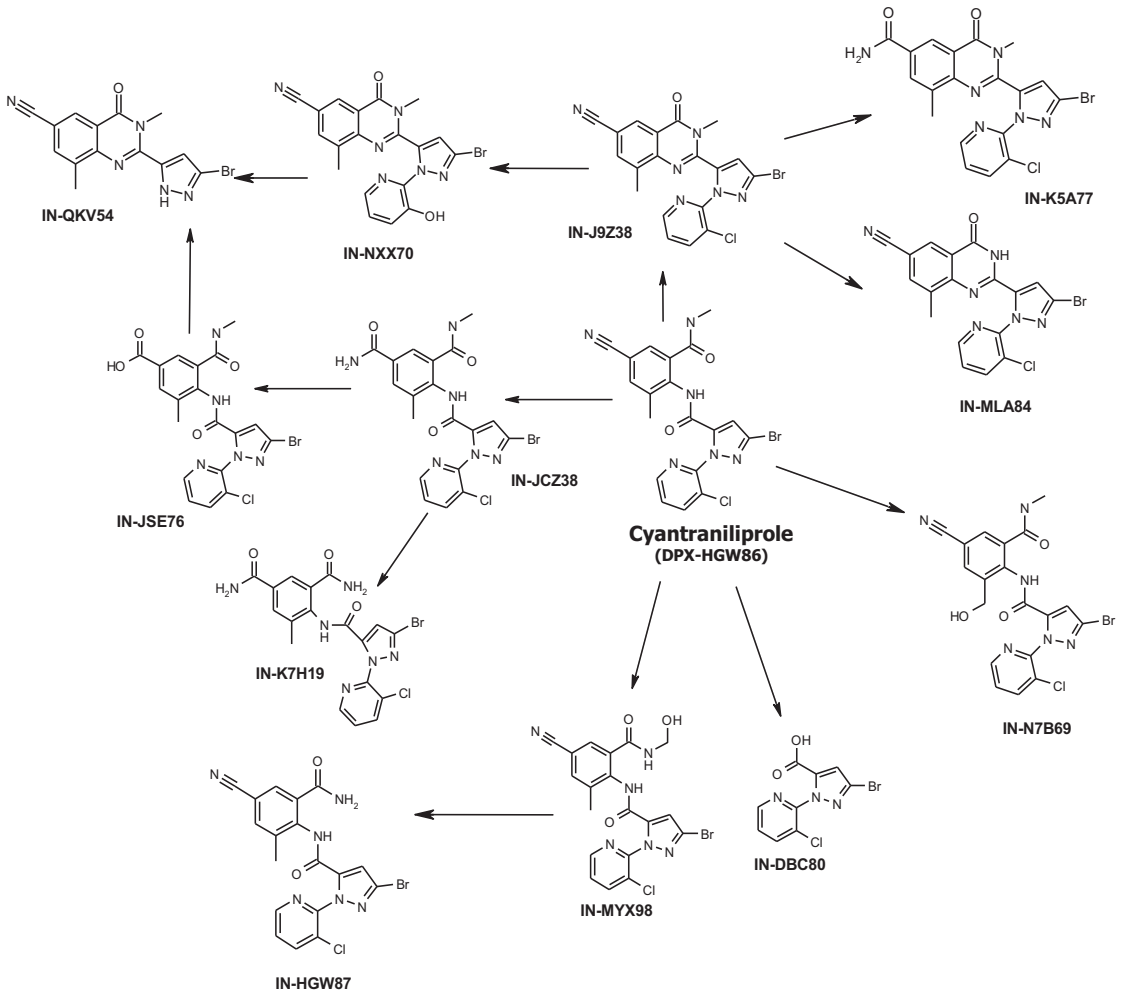


Figure 3 Proposed metabolic pathway for [^{14}C]-cyantraniliprole in cotton plants

Lettuce

In a study reported by MacKinnon (2008) [Ref: DP-16986], three applications of [^{14}C]-cyantraniliprole equivalent to 150 g ai/ha/treatment, were made to lettuce plants grown in 1 square metre outdoor sandy loam plots (pH 6.5) commercial growing medium (since abiotic degradation of cyantraniliprole occurs at alkaline pH).). Foliar applications (SC formulation) were made to plants 3 weeks post-emergence and at 7 and 14 days after the initial application. Soil drench applications were made 7 weeks post-emergence and at 7 and 14 days after the first application.

Leaves were collected from immature plants after the first application (foliar treatment only), immediately before and after the second and third applications, and 7 and 14 days after the third application and mature plants were collected 32 days after the last application.

Selected samples from the foliar-treatment regime were surface washed with acetonitrile: water (7:3, v/v) immediately after sampling. Plant samples from both regimes were pulverized and total tissue residues determined by oxidative combustion analysis. Where characterization of radioactivity was warranted, samples were extracted three times using acetone:1 N formic acid (9:1). The unextracted radioactivity in the post-extracted solids (PES) was determined by combustion analysis. Extracts containing significant radioactivity (≥ 0.01 mg/kg) were analysed by high performance liquid chromatography (HPLC) and identification of ^{14}C-residues was accomplished by HPLC and liquid chromatography mass spectrometry (LC-MS), with reference to authenticated

reference standards. Samples were stored at ca -20 °C for up to 72 days before analysis (up to 28 days before extraction).

To assess if storage at –20 °C resulted in degradation of radioactive residues, a subsample of stored tissue (7 days after the third foliar treatment) was extracted with acetone:formic acid following a storage period of 405 days. Frozen storage did not influence the extractability (%) of TRR (97.3% TRR in stored samples and 98.4% TRR in the original samples). The residue composition also remained unchanged with cyantraniliprole making up 77.2% TRR (1.55 mg/kg) compared to 78.5% TRR (1.56 mg/kg) in the original samples. Minor components in the stored samples individually accounted for < 10% TRR (0.01–0.05 mg/kg).

A comparison of the extraction efficiency of acetone:formic acid and acetonitrile:water showed similar results, with the extracted radioactivity from unwashed leaves from the foliar-treated plants being 97.8% TRR in the acetonitrile extract and 98.4% TRR in the acetone extract. HPLC analysis of the two extracts also reported comparable results, with cyantraniliprole making up 72.1% of the TRR in the acetone extract and 71.6% of the TRR in the acetonitrile extract.

In plants from the soil drench treatments, TRR in leaves from the [CN-^{14}C]-cyantraniliprole plots declined from 0.14 mg/kg (7 days after the first application) to 0.01 mg/kg at final harvest. TRRs following PC-label soil treatment were ≤ 0.06 mg/kg in immature leaves and at crop maturity. Higher residues were observed following foliar application.

The majority (76.7–98.4% TRR) of the radioactivity in lettuce leaves was extracted into acetone:formic acid.

Cyantraniliprole was the major radioactive component in leaves from the [CN-^{14}C]-cyantraniliprole treated plants, accounting for up to 76.7% TRR in young leaves and 37.1% TRR in mature leaves. Minor components (IN-K5A79 and unresolved IN-MLA84/IN-NXX70) were detected at ≤ 0.01 mg/kg in samples taken prior to the second application. Cyantraniliprole was the only component detected (< 0.01 mg/kg) at maturity.

Following the PC-label soil drench treatment, cyantraniliprole was the only detectable component in immature lettuce samples accounting for 75.3% to 84.1.0% TRR and was the major residue (69% TRR–0.039 mg/kg) in mature leaves. Low level metabolites (< 10% TRR) detected in mature leaves were unresolved IN-MLA84/IN-NXX70 and IN-J9Z38, both present at < 0.01 mg/kg.

Table 12 Total radioactive residues (mg/kg) in lettuce leaves following soil drench treatments with [^{14}C]-cyantraniliprole

Treatment	Leaves				
Sample point	7 DAT1	7 DAT2	7 DAT3	14 DAT3	32 DAT3
[CN-^{14}C]-cyantraniliprole	0.144	0.049	0.046	0.035	0.012
[PC-^{14}C]-cyantraniliprole	0.017	0.035	0.009	0.007	0.057

Table 13 Distribution of radioactive residues in lettuce leaves following soil drench treatments with [^{14}C]-cyantraniliprole

Sample point	7 DAT1		7 DAT2		7 DAT3		14 DAT3		32 DAT3	
	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg
^{14}C-Residue	[CN-^{14}C]-cyantraniliprole									
TRR, mg/kg		0.144		0.049		0.045		0.035		0.012
Extracted	98.4	0.142	96.7	0.047	92.9	0.042	89.2	0.031	76.7	0.009
Cyantraniliprole	67.9	0.099	76.8	0.037	72.1	0.033	51	0.018	37.1	0.004
IN-K5A79	7.1	0.011		ND		ND		ND		ND
IN-MLA84/ IN-NXX70	3	0.004		ND		ND		ND		ND
Unidentified	11.4 [a]	0.017	1.9	< 0.001	5.1	0.004	6.9	< 0.001	7.8	< 0.001
^{14}C-Residue	[PC-^{14}C]-cyantraniliprole									
TRR, mg/kg		0.017		0.035						0.057
Extracted	96.4	0.016	96	0.034					85.9	0.049
Cyantraniliprole	75.3	0.014	84.1	0.03					69	0.039

Sample point	7 DAT1		7 DAT2		7 DAT3		14 DAT3		32 DAT3	
	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg
IN-J9Z38		ND		ND					10	0.005
IN-MLA84/ IN-NXX70		ND		ND					3.4	0.002
Unidentified	6.6 [b]	< 0.001		ND						ND

[a] 13 components; none greater than 0.003 mg/kg (2.7% TRR)

[b] 4 components; none greater than 0.001 mg/kg (2% TRR)

In plants from the foliar treatments of a 1:1 mixture of [CN-^{14}C]-cyantraniliprole + [PC-^{14}C]-cyantraniliprole, TRRs in leaves declined from 10.8 mg/kg (immediately after the first foliar application) to 0.03 mg/kg at maturity (32 days after the final application).

The majority of the radioactivity (more than 92% TRR) was removed from leaves by surface washing and/or extraction into acidified aqueous acetone.

Cyantraniliprole was the major residue in leaves, decreasing from about 98% TRR immediately after the first application to 50% TRR in mature leaves, 32 days after the last application.

The only metabolite accounting for more than 5% TRR was IN-J9Z38, present in mature leaves at about 23% TRR (0.011 mg/kg). Other metabolites identified in immature leaf samples (IN-MLA84/IN-NXX70, IN-DBC80, IN-QKV54, IN-K5A77, IN-K5A79, IN-N7B69, IN-HGW87 and IN-JSE76) each did not exceed 5% TRR or 0.06 mg/kg. Numerous other metabolites detected but not identified were each present at less than 1% TRR (0.01 mg/kg). Unextracted leaf residues accounted for ≤ 1.5% TRR (≤ 0.03 mg/kg).

The main HPLC method did not resolve the two components IN-MLA84 and the photodegradate IN-NXX70. Subsequent analysis indicated that the photodegradate, IN-NXX70, accounted for 1.9% TRR (0.03 mg/kg) seven days after the first application and was found predominantly in the surface wash. At 7 days after the first application, IN-MLA84 was present in tissue extracts at much lower levels (0.4% TRR, 0.01 mg/kg).

Table 14 Distribution of radioactive residues in lettuce leaves from plants treated with foliar applications of a 1:1 mixture of [CN-^{14}C]-cyantraniliprole and [PC-^{14}C]-cyantraniliprole

Sampling point	0 DAT1		7 DAT1		0 DAT2	7 DAT2		0 DAT3	7 DAT3		14 DAT3		32 DAT3	
TRR, mg/kg	10.844		1.669		9.622	2.801		7.789	1.993		0.983		0.032	
	%TRR	mg/kg	%TRR	mg/kg		%TRR	mg/kg		%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg
Surface wash	90.7	9.84	82.9	1.38		57.3	1.61				65.6	0.645	12.5	0.004
Tissue extracts [a]	9.2	0.1	16.1	0.27		41.3	1.16		98.4	1.96	32.1	0.315	79.3	0.026
Total Extracted	99.9	10.83	99	1.65		98.6	2.76		98.4	1.96	97.7	0.96	91.7	0.029
Unextracted	< 0.1	< 0.001	1.1	0.018		1.3	0.036		1.5	0.03	2.3	0.023	8.3	0.003
Cyantraniliprole	97.9	10.6	79.1	1.32		87.3	2.45		78.5	1.56	72.6	0.716	50.3	0.016
IN-DBC80	ND		1.2			3.8			3.8		ND		ND	
IN-N7B69	ND		1	0.017		0.4	0.012				ND		ND	
IN-K5A79	ND		1.2	0.018		ND			ND		ND		ND	
IN-JSE76	ND		0.7	0.012		ND			0.7	0.014	ND		ND	
IN-HGW87	ND		0.6	0.01		ND			0.9	0.018	ND		ND	
IN-K5A77	ND		ND			1.2	0.036		ND		ND		ND	
IN-QKV54	ND		ND			1.4	0.042		0.9	0.017	ND		ND	
IN-MLA84/ IN-NXX70	0.3	0.034	3	0.05		2	0.058		1.4	0.027	2.6	0.027	4.9	0.001
IN-J9Z38	0.4	0.042	ND			1	0.028		0.8	0.021	2.3	0.023	23.3	0.011
Unidentified	0.3 [b]	0.035	6.6 [c]	0.103		4.1 [d]	0.117		3.8 [e]	0.078	6.4 [f]	0.067	0.7	0.001

[a] Triple extraction with acetone:1 N formic acid

[b] 7 components; none greater than 0.006 mg/kg (0.1% TRR)

[c] 46 components; none greater than 0.009 mg/kg (0.6% TRR)

[d] 29 components; none greater than 0.011 mg/kg (0.4% TRR)

[e] 27 components; none greater than 0.009 mg/kg (0.4% TRR)

[f] 13 components; none greater than 0.007 mg/kg (0.7% TRR)

The metabolic fate of cyantraniliprole in lettuce grown outdoors was complex, with the formation of many low level metabolites. The proposed metabolic pathway is outlined below.

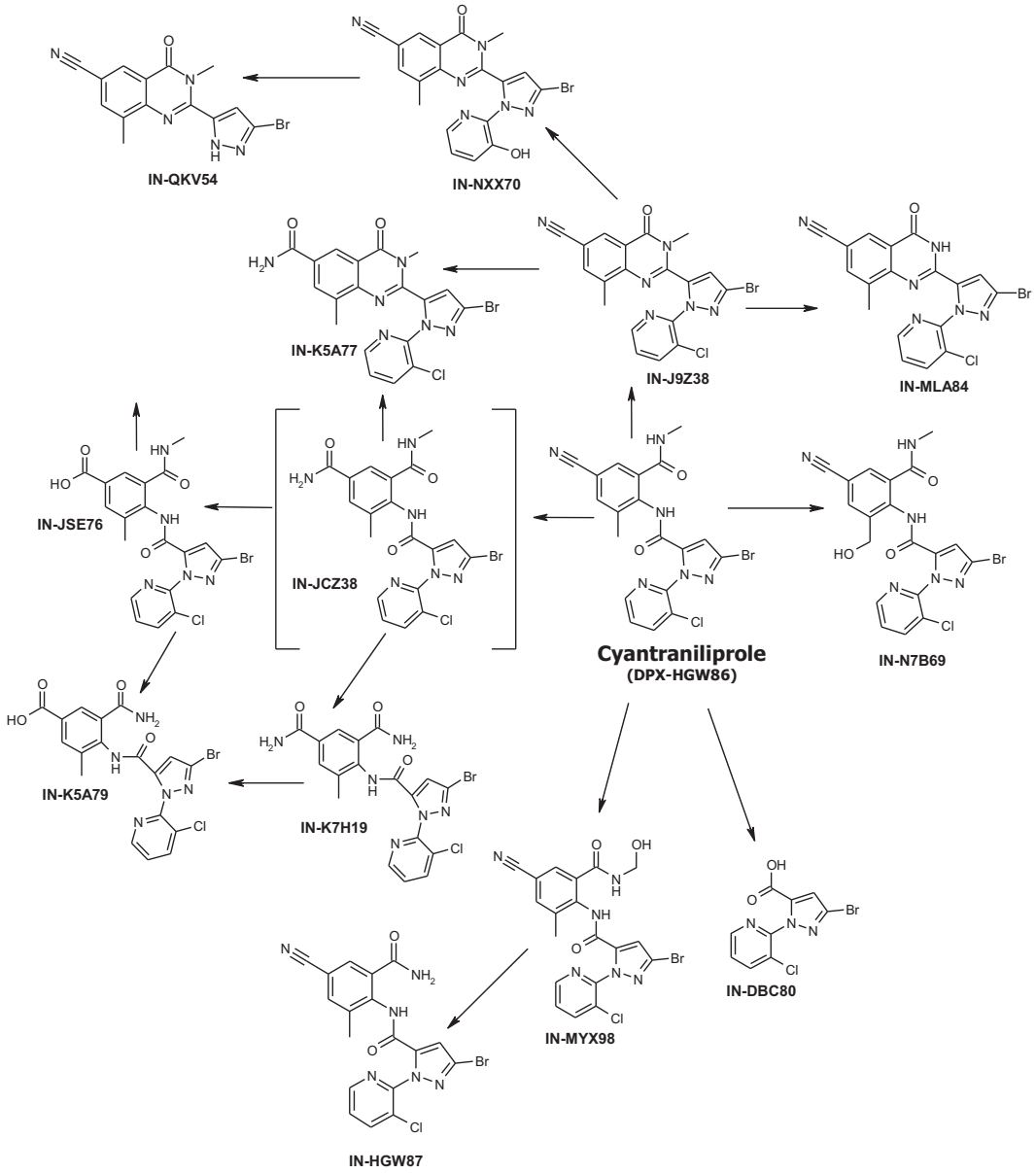


Figure 4 Proposed metabolic pathway for cyantraniliprole in lettuce

Tomatoes

In a study reported by MacKinnon (2008) [Ref: DP-16985], three applications of [^{14}C]-cyantraniliprole equivalent to 150 g ai/ha/treatment, were made to tomato plants grown in 24 cm pots containing an acidic (pH 6.4) commercial growing medium.). Foliar applications (SC formulation) were made to plants 3 weeks post-emergence and at 7 and 14 days after the initial application. Soil drench applications were made 7 weeks post-emergence and at 7 and 14 days after the first application.

Leaves were collected from immature plants after the first application (foliar treatment only), immediately prior to and following the second and third applications, and at ca. 7 and 14 days after the third application. Leaves and fruit were also sampled at crop maturity (ca. 125 days after the third application).

Selected samples from the foliar-treatment regime were surface washed with acetonitrile: water (7:3, v/v) immediately after sampling. Plant samples from both regimes were pulverized and total tissue residues determined by oxidative combustion analysis. Where characterization of radioactivity was warranted, samples were extracted three times using acetone:1 N formic acid (9:1). Insoluble residues from immature leaves were subjected to further sequential extraction using enzyme digestion (2 × 24 hours with cellulose, glucosidase, driselase), 1 M HCl (6 hours, 60 °C), 6 M HCl (6 hours, 90 °C) and 6 M NaOH (6 hours, 90 °C) hydrolysis. The unextracted radioactivity in the post-extracted solids (PES) was determined by combustion analysis.

Extracts containing significant radioactivity (≥ 0.01 mg/kg) were analysed by high performance liquid chromatography (HPLC) and identification of ^{14}C-residues was accomplished by HPLC and liquid chromatography mass spectrometry (LC-MS), with reference to authenticated reference standards. Samples were stored at ca -20 °C for up to 69 days before analysis.

To assess if storage at –20 °C resulted in degradation of radioactive residues, a subsample of stored leaf tissue (7 days post-application 3, foliar treatment) was extracted with acetone:formic acid following a storage period of 392 days. The TRR and extractability values after prolonged storage (1.86 mg/kg, 84.4% TRR) were similar to those obtained originally (2.21 mg/kg, 87.1% TRR). The residue composition also remained unchanged with cyantraniliprole being the principle component (55.3% TRR, 1.03 mg/kg) and minor components individually making up ≤ 5.4% TRR (< 0.01 mg/kg).

A comparison of the extraction efficiency of acetone:formic acid and acetonitrile:water showed similar results, with the extracted radioactivity from unwashed leaves from the foliar-treated plants being 84% TRR in the acetonitrile extract and 82.8% TRR in the acetone extract. HPLC analysis of the two extracts also reported comparable results, with cyantraniliprole making up 43.4% of the TRR in the acetone extract and 36.6% of the TRR in the acetonitrile extract.

In plants from the soil drench treatments, TRRs in leaves from immature plants reached a maximum of 0.03 mg/kg seven days after the last CN-label application and 0.01 mg/kg 14 days after the last PC-label application. In mature leaves (125 days after the last application), TRRs were < 0.01 mg/kg in leaves and 0.001 mg/kg in fruit.

Only extractable residues from immature leaves taken 7 and 14 days after the last CN-label application were sufficiently high to permit HPLC analysis. Cyantraniliprole was the major radioactive component ranging from 22.2% to 26.1% TRR (0.01 mg/kg). Minor components (unresolved IN-MLA84/IN-NXX70 and IN-J9Z38) each at < 0.01 mg/kg, were also detected in leaves taken 7 days after the last application.

Table 15 Total radioactive residues (mg/kg) in tomato samples following soil drench treatments with [^{14}C]-cyantraniliprole

Treatment	Leaves				Leaves	Fruit
Sample point	7 DAT1	7 DAT2	7 DAT3	14 DAT3	125 DAT3	
[CN-^{14}C]-cyantraniliprole	0.005	0.023	0.03	0.026	0.008	0.001
[PC-^{14}C]-cyantraniliprole	0.002	0.012	0.014	0.014	0.009	0.001

Table 16 Distribution of radioactive residues in tomato leaves, 7 and 14 days after the last of three soil drench treatments with [CN^{14}C]-cyantraniliprole

Sample point	7 DAT3		14 DAT3	
	%TRR	mg/kg	%TRR	mg/kg
TRR, mg/kg		0.03		0.026
Extracted	86	0.026	78.4	0.02
Cyantraniliprole	22.2	0.006	26.1	0.008
IN-MLA84/ IN-NXX70	6.4	0.002	5.8	0.002
IN-J9Z38	5.4	0.002		ND
Unidentified	11.8	0.006	13.7	0.002

TRRs in tomato leaves immediately following foliar treatments with the 1:1 mixture of [CN-^{14}C]-cyantraniliprole + [PC-^{14}C]-cyantraniliprole ranged between 2.55 and 4.81 mg/kg and decreased to 1.3 mg/kg at 14 days after the last application. At harvest, 125 days after the last application, TRRs in leaves and fruit were < 0.01 mg/kg.

In immature leaves, 74–87% TRR was removed by surface washing and/or extraction into acidified aqueous acetone. Low concentrations of radioactivity were subsequently extracted using enzymes (1.2–5.6% TRR), 1 M HCl at 60 °C (1–3.4% TRR), 6 M HCl at 90 °C (0.2–3.7% TRR) and 6 M NaOH at 90 °C (0.7–2.9% TRR).

Cyantraniliprole was the major residue in leaves, decreasing from 95% TRR immediately after the first application to 61% TRR seven days later and accounted for 62 and 43% TRR, respectively, 7 and 14 days after the final application.

Concentrations of the unresolved radioactivity corresponding to both IN-MLA84 and IN-NXX70 were highest 7 days after the first application (11.5% TRR), decreasing to 5.8% TRR thereafter. Further analysis indicated that the photodegradate IN-NXX70 accounted for 12% TRR (0.23 mg/kg) 7 days after the first application, mostly (10% TRR) in the surface wash and declined to 3.8% TRR, 0.08 mg/kg, 7 days after final application. Seven days after the first application, IN-MLA84 was present in tissue extracts at lower levels (≤ 2.3% TRR, ≤ 0.02 mg/kg).

Other minor metabolites were identified as IN-J9Z38, IN-K7H19, IN-JCZ38, IN-DBC80, IN-N7B69, IN-MYX98, IN-JSE76, IN-K5A77 and IN-QKV54, with no single metabolite exceeding 5% TRR. Numerous other metabolites were detected which did not correspond to known reference standards, none exceeding 5.6% TRR (0.07 mg/kg). The unextracted residue accounted for ≤ 2.3% TRR (≤ 0.03 mg/kg).

In leaves taken at maturity, low levels of radioactivity were present in the surface wash (< 0.01 mg/kg) and the acidified aqueous acetone extract (0.01 mg/kg). The unextracted residues also accounted for < 0.01 mg/kg.

Table 17 Distribution of radioactive residues in leaves from tomato plants treated with foliar applications of a 1:1 mixture of [CN-^{14}C]-cyantraniliprole and [PC-^{14}C]-cyantraniliprole

Sampling point	0 DAT1		7 DAT1		7 DAT2		7 DAT3		14 DAT3	
TRR, mg/kg	2.546		1.849		4.806		2.216		1.298	
	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg
Surface wash	65.5	1.668	72.3	1.337	85	4.085				
Tissue extracts	33.6	0.854	27.6 [a]	0.509	15 [a]	0.723	99.2 [a]	2.197	97.7 [a]	1.269
Total Extracted	99	2.52	99.8	1.845	100	4.807	99.2	2.197	97.7	1.269
Unextracted	1.0	0.025	0.1	0.002	0.1	0.005	0.8	0.018	2.3	0.03
Cyantraniliprole	95.3	2.429	61.1	1.132	86.5	4.154	62.2	1.377	43.4	0.562
IN-MLA84/ IN-NXX70	0.9	0.023	11.5	0.213	3.1	0.149	6.6	0.147	5.8	0.074
IN-J9Z38	0.9	0.022	1.6	0.029	1.2	0.051	5.1	0.114	4.4	0.055

Sampling point	0 DAT1		7 DAT1		7 DAT2		7 DAT3		14 DAT3	
TRR, mg/kg	2.546		1.849		4.806		2.216		1.298	
	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg
IN-QKV54	ND		ND		0.8	0.04	2.8	0.062	4.3	0.054
IN-N7B69	ND		ND		0.3	0.016	0.8	0.019	2.9	0.037
IN-JCZ38	ND		1.0	0.019	0.2	0.01	1.0	0.022	2.8	0.036
IN-K7H19	ND		ND		ND		1.2	0.025	1.4	0.019
IN-DBC80	ND		3.8		1.0		3.0		ND	
IN-MYX98	ND		ND		ND		1.0	0.023	1.5	0.018
IN-JSE76	ND		ND		ND		ND		1.0	0.012
IN-HGW87	ND		ND		0.6	0.028	ND		ND	
Unidentified	5.0 [b]	0.102	10.7 [c]	0.197	2.6 [d]	0.137	15.9 [e]	0.352	22.2 [f]	0.265

[a] Sequential extraction with acetone:1 N formic acid (3×); a mixture of cellulase, beta-glucosidase and driselase (2× for 24 hours at 37 °C); 1 M HCl (6 hours at 60 °C); 6 M HCl (6 hours at 90 °C) and 6 M NaOH (6 hours at 60 °C)

[b] 32 components; none greater than 0.008 mg/kg (0.4%TRR)

[c] 23 components; none greater than 0.025 mg/kg (1.3%TRR)

[d] 28 components; none greater than 0.02 mg/kg (0.4%TRR)

[e] 51 components; none greater than 0.07 mg/kg (3.1%TRR)

[f] 45 components; none greater than 0.07 mg/kg (5.6%TRR)

The proposed pathway is outlined below.

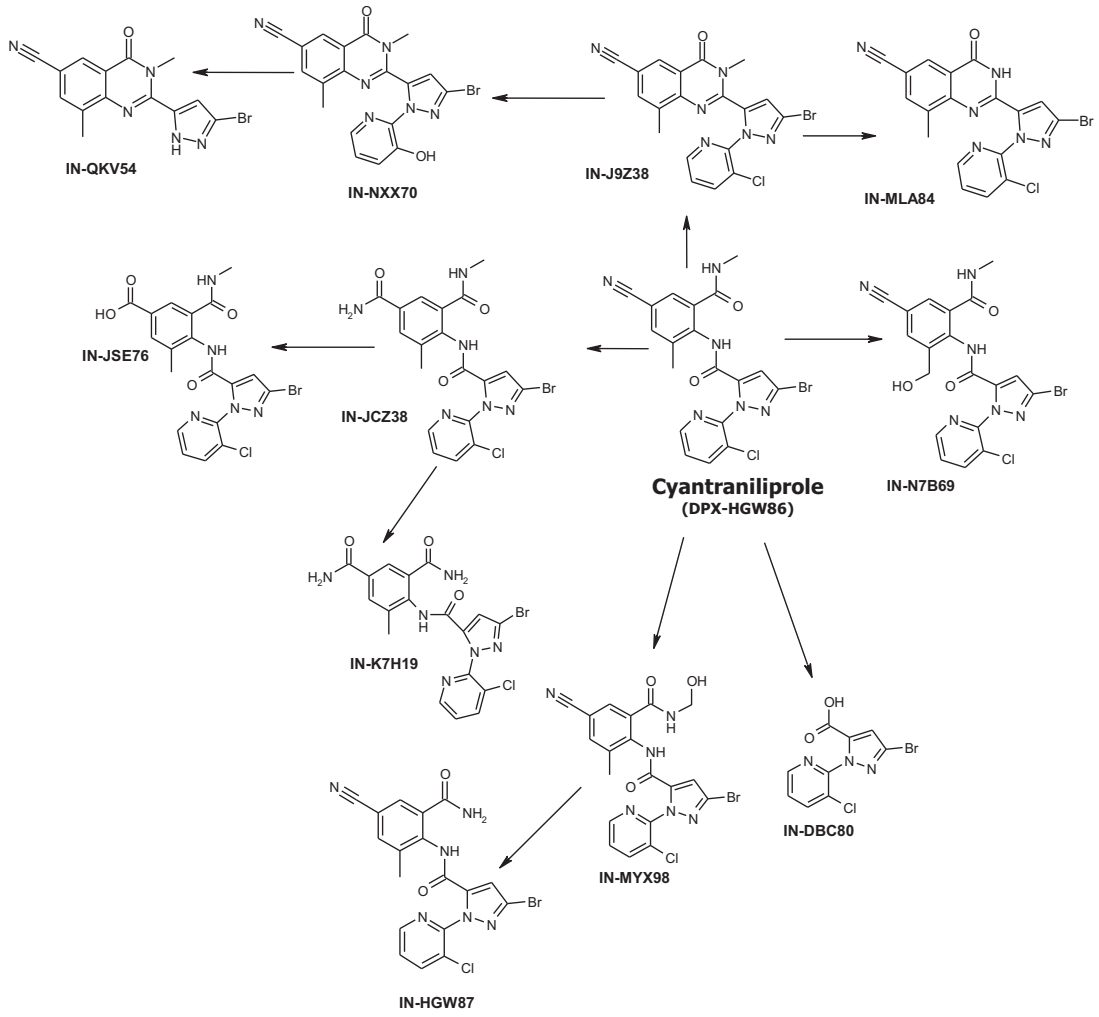


Figure 5 Proposed metabolic pathway for [^{14}C]-cyantraniliprole in tomatoes

Rice

In a study reported by Chapleo, Hobbs & Grant-MacDonald (2010) [Ref: DP-18780], single soil applications of [^{14}C]-cyantraniliprole (granular formulations), equivalent to 300 g ai/ha, was applied at the 3–4 leaf stage to rice plants grown in a glasshouse in a sandy-loam soil (pH 6.2) and three foliar applications (SC formulation) of 0.15 kg ai/ha were made to other plants at the 3–4 leaf stage and 7 and 14 days later. In both cases the plants were grown under flooded conditions (pots immersed in about 3 cm water) from 2 days after the initial treatment until 2–3 days before harvest.

Leaves and shoots from foliar-treated plants were sampled 0 and 7 days after the first treatment, 7 days after the second treatment and 7, 14 days after the last treatment with roots being collected 7 days after each treatment. At maturity, 140 days after the last treatment, whole plants were separated into straw, roots, and panicles.

Leaves and shoots from plants grown in treated soil were sampled 3, 7, 14 and 56 days after treatment and roots were collected 7 and 56 days after treatment. At maturity (175 days after treatment), whole plants were separated into straw, roots, and panicles.

Selected samples from the foliar-treatment regime were surface washed with acetonitrile: water (9:1) immediately after sampling. Plant samples from both regimes were homogenised and residues extracted in acetonitrile:water (9:1 then 7:3). Where required, samples were subjected to

further sequential extraction using enzyme digestion (2 × 24 hours with cellulose, glucosidase, drisealse), 1 M HCl (6 hours, 60 °C), 6 M HCl (6 hours, 90 °C) and 6 M NaOH (6 hours, 90 °C). The unextracted radioactivity in the post-extracted solids (PES) was determined by combustion analysis.

Extracts containing significant radioactivity (≥ 0.01 mg/kg) were analysed by reversed phase HPLC and identification of ^{14}C-residues was accomplished by HPLC and liquid chromatography mass spectrometry (LC-MS), with reference to authenticated reference standards. Samples were stored at ca –20 °C for up to 37 days before analysis.

In plants grown in treated soil, TRRs in immature leaves ranged from 0.4–0.42 mg/kg 56 days after treatment and were 0.28–0.37 mg/kg in roots. At maturity (175 days after treatment), TRRS were 0.28–0.3 mg/kg in straw and 0.01–0.03 mg/kg in grain.

Table 18 Total radioactive residues (mg/kg) in rice samples following granular soil treatments with [^{14}C]-cyantraniliprole

	Foliage				Roots		Roots	Straw	Grain
Sample point	3 DAT	7 DAT	14 DAT	56 DAT	7 DAT	56 DAT	175 DAT		
[CN-^{14}C]-cyantraniliprole	ND	0.076	0.15	0.404	< 0.017	0.253	0.282	0.278	0.012
[PC-^{14}C]-cyantraniliprole	0.153	0.068	0.145	0.42	< 0.04	0.297	0.367	0.297	0.029

The majority of the radioactivity was extracted into aqueous acetonitrile with 85–87% TRR extracted from immature foliage, 79–80% TRR from straw and 61–67% TRR from grain.

Cyantraniliprole was the major radioactive residue in immature 56 DAT foliage extracts, found at up to 49% TRR and 0.2 mg/kg (PC-label), 57% TRR and 0.23 mg/kg (CN-label). The IN-J9Z78 metabolite was found in these samples at 22% TRR (PC-label) and 16% TRR (CN-label). Other metabolites (including IN-JCZ78, IN-N7B69, IN-K5A77, IN-MLA84 and IN-DBC80) did not exceed 0.011 mg/kg or 3% TRR. The unextracted residue accounted for 4.1–9.8% TRR (< 0.02 mg/kg).

The major residue in straw was also cyantraniliprole (42–45% TRR, 0.125 mg/kg), with IN-J9Z38 (14–18% TRR) being the only significant metabolite, found at 14–18% TRR. Other minor metabolites, each present at less than 4% TRR or 0.01 mg/kg were IN-JCZ38, IN-K5A78, IN-K5A77, IN-MLA84 and IN-DBC80. The unextracted residue accounted for 8.9 % TRR (0.03 mg/kg).

In grain, cyantraniliprole accounted for 46–63% TRR, 0.007–0.014 mg/kg), with IN-J9Z38 (5.9–10.2 % TRR) being the only significant metabolite

Table 19 Distribution of radioactive residues in rice samples following granular soil treatments with [^{14}C]-cyantraniliprole

	Foliage								Straw		Grain	
Sample point	3 DAT		7 DAT		14 DAT		56 DAT		175 DAT		175 DAT	
	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg
CN-label												
Extracted	ND		96.1	0.073	90.9	0.136	86.8	0.351	79.7	0.222	61.1	0.007
Unextracted	ND		3.9	0.003	9.1	0.014	13.2 [a]	0.053	20.3 [b]	0.056	38.9	0.005
Cyantraniliprole			102	0.077	67.1	0.101	57.4	0.232	44.9	0.125	62.7	0.007
IN-J9Z38	ND		ND		16.9	0.025	16.2	0.066	18.4	0.051	10.2	0.001
IN-JCZ38			ND		ND		1.5	0.006	3.6	0.01	ND	
IN-K5A77			ND		ND		ND		3.0	0.008	ND	
IN-K5A78			ND		ND		ND		0.3	0.001	ND	
IN-MLA84			ND		ND		ND		1.4	0.004	ND	
IN-N7B69			ND		ND		1.7	0.007	ND		ND	
Unidentified			ND		ND		ND		8.0	0.023	6.9	< 0.001
PC-label												
Extracted	100	0.153	94.7	0.064	90.2	0.131	85.5	0.359	78.5	0.233	67	002
Unextracted	ND		5.3	0.004	9.8	0.014	14.5 [c]	0.061	21.5 [d]	0.064	32.9	0.01
Cyantraniliprole	107.8	0.165	86.2	0.059	58.6	0.085	48.7	0.205	42.1	0.125	46.2	0.014
IN-J9Z38	ND		12.3	0.008	27.9	0.041	22.1	0.093	14.3	0.042	5.9	0.002

	Foliage								Straw		Grain	
Sample point	3 DAT		7 DAT		14 DAT		56 DAT		175 DAT		175 DAT	
	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg
IN-JCZ38	ND		ND		ND		2.3	0.01	2.8	0.008	ND	
IN-K5A77	ND		ND		ND		0.8	0.003	3.7	0.011	ND	
IN-DBC80	ND		ND		ND		0.7	0.003	2.8	0.008	1.1	< 0.001
IN-K5A78	ND		ND		ND		ND		0.6	0.002	ND	
IN-MLA84	ND		ND		ND		0.8	0.003	1.2	0.004	ND	
Unidentified	7.7	0.012	ND		ND		ND		3.0	0.01	3.4	< 0.001

[a] Further extraction released a total of 9.6% TRR (0.038 mg/kg)

[b] Further extraction released a total of 11.7% TRR (0.034 mg/kg)

[c] Further extraction released a total of 10.4% TRR (0.043 mg/kg)

[d] Further extraction released a total of 10.7% TRR (0.032 mg/kg)

TRRs in foliage following foliar treatments with the 1:1 mixture of [CN-^{14}C]-cyantraniliprole + [PC-^{14}C]-cyantraniliprole were 2.13 mg/kg immediately after the first application, decreasing to 0.38 mg/kg after 7 days. TRRS 7 and 14 days after the last application were 1.56 mg/kg and 1.21 mg/kg respectively. At harvest, 140 days after the last application, TRRs in straw were 0.45 mg/kg and 0.02 mg/kg in grain. In roots, TRRs increased from 0.24 mg/kg seven days after the first application to 0.68 mg/kg seven days after the last application and were 0.45 mg/kg at harvest (140 DAT3).

In immature leaves, 92–99% TRR was removed by surface washing and/or extraction into aqueous acetonitrile (75% TRR in the 7 DAT3 surface wash and 47% TRR in the 14 DAT3 wash). In straw, aqueous acetonitrile extraction removed 87% TRR and further 10.9% TRR was recovered in enzyme, acid and alkali digests. In grain, 49% TRR (0.012 mg/kg) was extracted in aqueous acetonitrile.

Cyantraniliprole was the major residue in immature foliage, making up more that 75% TRR and 0.35-2.13 mg/kg). IN-J9Z38 was the predominant metabolite found at 0.6% TRR immediately after the first application and increasing to 10.9% TRR 14 days after the last application. IN-JCZ38, IN-JSE76, IN-K5A77, IN-K5A78 IN-MLA84, and IN-N7B69 were also detected, none exceeding 2% TRR. Multiple unknown metabolites were detected, none exceeding 2.3% TRR. Unextracted residues accounted for 0.1–7.8% TRR.

In straw, cyantraniliprole was the major extractable component, accounting for 24.4% TRR (0.11 mg/kg). Significant metabolites included IN-JCZ38 (9.4% TRR) and IN-JSE76 (9.0% TRR). Other metabolites were identified as IN-K5A77 (5.3% TRR), IN-DBC80 (3.2% TRR), IN-J9Z38 (4.0% TRR), and IN-K7H19 (2.4% TRR) and seven unidentified metabolites were present (including 2 highly polar metabolites) at 6.6–8.1% TRR. 5.5% TRR (0.02 mg/kg) were not extracted.

Cyantraniliprole was the main extractable component in grain, accounting for 21% TRR (0.01 mg/kg). Metabolites included IN-J9Z38, IN-N7B69 and IN-K5A78 made up 1.5–2.6% TRR and 17 unidentified metabolites were also detected, each at less than 8.0% TRR.). Unextracted residues accounted for 51.3% TRR (0.01 mg/kg).

Table 20 Distribution of radioactive residues in rice samples following three foliar applications of a 1:1 mixture of [CN-^{14}C]-cyantraniliprole and [PC-^{14}C]-cyantraniliprole

	Foliage										Straw		Grain	
Sample point	0 DAT1		7 DAT1		7 DAT2		7 DAT3		14 DAT3		140 DAT3		140 DAT3	
TRR (mg/kg)	2.126		0.383		1.0		1.56		1.207		0.446		0.024	
	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg
Surface wash							74.5	1.162	46.8	0.565				
Tissue extracts	99.9	2.124	92.1	0.353	99	0.99	20.0	0.311	51.5	0.623	83.7	0.374	48.7	0.012
Total extracted	99.9	2.124	92.1	0.353	99	0.99	96.6	1.506	95.3	1.151	83.7	0.374	48.7	0.012
Unextracted	0.1	0.002	7.8	0.03	1.0	0.01	3.4	0.053	4.7	0.057	16.3[(a)]	0.073	51.3	0.012

	Foliage										Straw		Grain	
Sample point	0 DAT1		7 DAT1		7 DAT2		7 DAT3		14 DAT3		140 DAT3		140 DAT3	
TRR (mg/kg)	2.126		0.383		1.0		1.56		1.207		0.446		0.024	
	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg
Cyantraniliprole	100	2.127	90.7	0.347	95.5	0.956	75.6	1.179	81.1	0.98	24.4	0.109	20.9	0.005
IN-J9Z38	0.6	0.014	2.5	0.01	6.3	0.063	7.2	0.112	10.9	0.131	4.0	0.018	1.5	< 0.001
IN-JCZ38	ND		ND		ND		0.6	0.009	0.8	0.009	9.4	0.042	ND	
IN-JSE76	ND		ND		ND		ND		0.2	0.002	9.0	0.04	ND	
IN-K5A77	0.6	0.012	ND		ND		ND		0.8	0.01	5.3	0.024	ND	
IN-K5A78	ND		ND		ND		ND		0.2	0.005	ND		2.6	0.001
IN-DBC80	ND		ND		ND		ND		ND		3.2	0.014	ND	
IN-K7H19	ND		ND		ND		ND		ND		2.4	0.011	ND	
IN-MLA84	ND		ND		0.6	0.006	1.5	0.024	1.9	0.023	ND		ND	
IN-N7B69	ND		ND		ND		1.7	0.027	ND		ND		2.2	0.001
Unidentified	ND		ND		0.5	0.005	5.3[b]	0.084	0.9	0.011	29.4[c]	0.131	15.7	0.003

[a] Further extraction released a total of 10.9% TRR (0.048 mg/kg)

[b] 8 components; none greater than 0.035 mg/kg (2.3%TRR)

[c] 7 components; none greater than 0.036 mg/kg (8.1%TRR)

The metabolic fate of cyantraniliprole in rice growing under flooded conditions was complex with the formation of many low level metabolites. Metabolism following foliar and soil applications appear to be similar. The proposed metabolic pathway is outlined below.

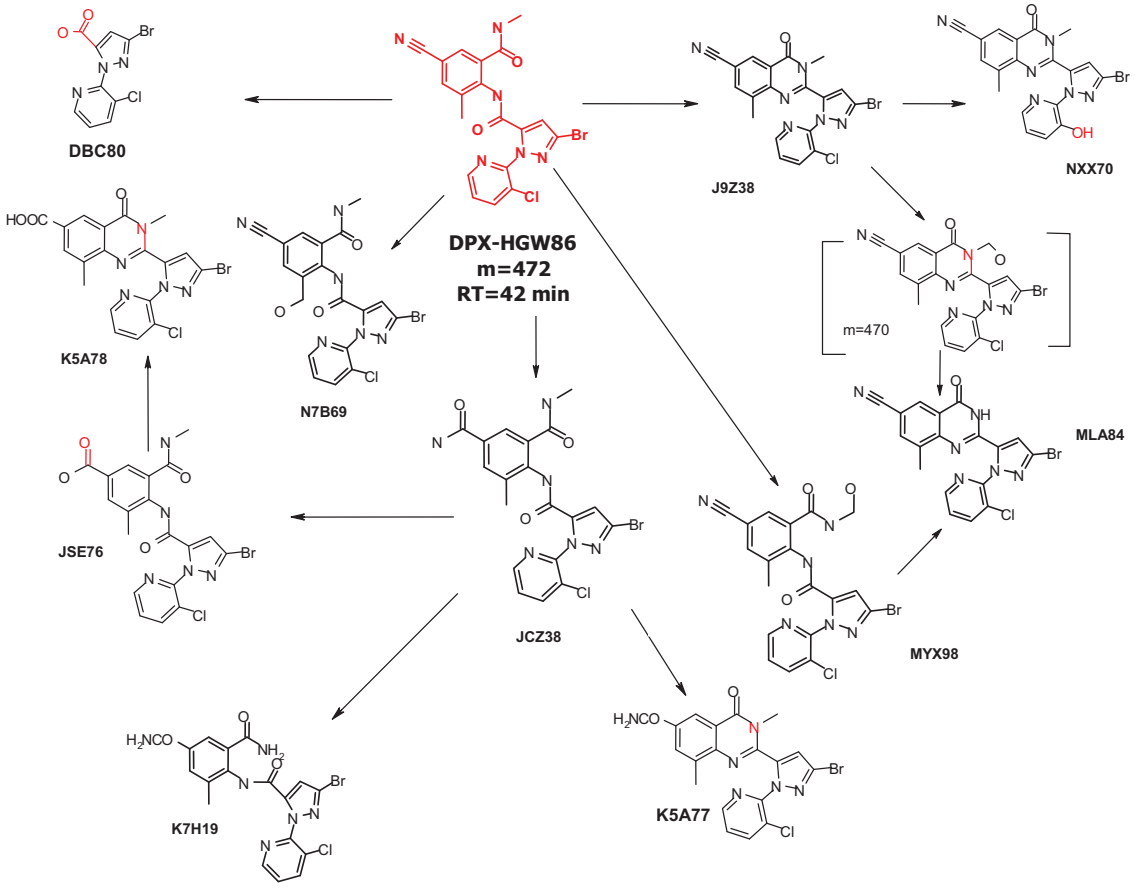


Figure 6 Proposed metabolic pathway for [^{14}C]-cyantraniliprole in rice

Environmental fate

The Meeting received information on the environmental fate and behaviour of cyantraniliprole, including hydrolytic stability, aerobic metabolism, photolysis and degradation in soil, water/sediment degradation and rotational crop metabolism and field studies.

Hydrolysis

The hydrolytic stability of cyantraniliprole in sterile buffer solutions was reported by Lowrie (2005) [Ref: DP-17058]. Sterile buffered solutions of 1 µg/ml radiolabelled cyantraniliprole at pH 4, 7, and 9 were incubated in the dark at 15, 25 and 35 °C for up to 30 days and sampled for analysis by LSC and HPLC at selected time intervals.

Hydrolysis of cyantraniliprole was pH and temperature dependant. At lower pH and temperature, the rate of hydrolysis was significantly less than at high pH and temperature. At all pHs, the only hydrolysis product detected at more than 5% of applied radioactivity (AR) was the cyclisation product IN-J9Z38, which accounted for a maximum of 28% AR (pH 4), 89% AR (pH 7) and 98% AR in the pH 9 samples. Calculated DT_{50} and DT_{90} values for cyantraniliprole and the IN-J9Z38 metabolite are summarised below.

Table 21 Calculated DT_{50} and DT_{90} values for cyantraniliprole and the IN-J9Z38 metabolite

		Cyantraniliprole			IN-J9Z38		
pH	Temperature (°C)	Rate constant, K (days)	DT_{50} (days)	DT_{90} (days)	Rate constant, $_K$ (days)	DT_{50} (days)	DT_{90} (days)
4	15	0.0019	362	1204			
4	25	0.0033	212	705			
4	35	0.0126	55.2	183			
7	15	0.0055	126	417			
7	25	0.0229	30.3	101			
7	35	0.0923	7.51	25	0.0031	227	755
9	15	0.2234	3.1	10.3			
9	25	0.8152	0.85	2.82			
9	35	1.203	0.576	1.91	0.0018	376	1248

Aerobic soil metabolism

In a study reported by Malekani & Mattson (2006) [Ref: DP-15775], a loam soil (pH 7.9, 2% organic matter) and a silty clay loam soil (pH 6.2, 2.7 % organic matter) were treated with the equivalent of 0.4 kg ai/ha [CN-^{14}C]-cyantraniliprole or [PC-^{14}C]-cyantraniliprole and incubated at 22 ± 3 °C in the dark for 358 days. The moisture content of the soils was maintained at 40–60% maximum water holding capacity (MWHC).

Samples were taken immediately following application and at selected intervals up to 358 days after application, extracted with $CaCl_2$ (to assess aged sorption of cyantraniliprole in conjunction with degradation) and then triple-extracted with acetone:formic acid (9:1) for analysis by LSC and HPLC.

The extractability of cyantraniliprole from both soils was quantitative at Day 0, but extractability decreased while the non-extractable residues (NER) increased as the study progressed, reaching a peak of approximately 16 to 22% in both soils.

Volatile organics were not produced in significant amounts in either soil. The amount of $^{14}CO_2$ evolved was significant, up to 11.8% AR for systems involving with [CN-^{14}C]-cyantraniliprole and 4.1% AR for [PC-^{14}C]-cyantraniliprole systems.

Cyantraniliprole levels declined to less than 5% AR in the loam soil and to 30–40% of AR in the silty clay loam by Day 121. Degradation continued past Day 121, with the silty clay loam containing 11–12% AR as cyantraniliprole on Day 358 and was not detected in the loam soil.

Seven major transformation products were observed in the study, with IN-JSE76 being the major component (43% AR) at the end of the study period, with IN-K5A78 and IN-PLT97 also present at 23–29% AR at day 358.

Table 22 Maximum percent of each metabolite in route of degradation study

Component	Maximum% AR [CN-^{14}C]-cyantraniliprole		Maximum% AR [PC-^{14}C]-cyantraniliprole	
	Loam (Nambsheim)	Silty clay loam (Tama)	Loam (Nambsheim)	Silty clay loam (Tama)
Cyantraniliprole	100.0 (Day 0)	104.9 (Day 0)	101.7 (Day 0)	109.1 (Day 0)
IN-J9Z38	16.0 (Day 16)	11.6 (Day 300)	12.9 (Day 100)	10.8 (Day 300)
IN-JCZ38	16.6 (Day 7)	13.1 (Day 22)	16.1 (Day 22)	12.5 (Day 16)
IN-JSE76	40.4 (Day 41)	42.6 (Day 358)	42.3 (Day 41)	42.9 (Day 358)
IN-K5A77	8.6 (Day 100)	3.8 (Day 358)	8.9 (Day 100)	3.3 (Day 300)
IN-K5A78	28.8 (Day 358)	5.4 (Day 358)	27.2 (Day 358)	4.0 (Day 358)
IN-K5A79 [a]	8.4 (Day 63)	ND	9.3 (Day 41)	ND
IN-PLT97 [a]	22.7 (Day 358)	ND	26.3 (Day 358)	ND

[a] IN-K5A79 and IN-PLT97, observed as significant metabolites in Nambsheim soil, were not detected (ND) in Tama soil

In a similar study reported by Malekani (2008) [Ref: DP-17055], three additional soils (a silt loam—pH 7, 2.1% organic matter; a sandy loam soil—pH 4.6, 1.6 % organic matter and a silty clay loam—pH 8.05, 4.5% organic matter) were treated with the equivalent of 0.4 kg ai/ha [CN-^{14}C]-cyantraniliprole or [PC-^{14}C]-cyantraniliprole, the moisture content was adjusted to 45% MWHC and the soils were incubated at 20 ± 2 °C in the dark for 120 days. One soil was also incubated at 10 ± 2 °C for 120 days.

Observed degradation in all soils was similar to that described in the above study, with the major metabolites present at the end of the 120-day study period being IN-JSE76 and IN-JCZ38 (26-33% AR).

Table 23 Maximum percent of each metabolite in the route of degradation study

Component	Maximum% AR [CN-^{14}C]-cyantraniliprole				Maximum% AR [PC-^{14}C]-cyantraniliprole			
	Silt loam (Gross-Umstadt)	Sandy loam (Sassafras)	Silty clay loam (Lleida)	Silt loam (Gross-Umstadt)	Silt loam (Gross-Umstadt)	Sandy loam (Sassafras)	Silty clay loam (Lleida)	Silt loam (Gross-Umstadt)
Cyantraniliprole	98.5 (Day 0)	97.6 (Day 0)	101.3 (Day 0)	104.6 (Day 0)	101.1 (Day 0)	98 (Day 0)	97 (Day 0)	102 (Day 0)
IN-J9Z38	16.7 (Day 45)	11.9 (Day 45)	16.4 (Day 45)	19.4 (Day 120)	17.9 (Day 60)	6.2 (Day 91)	18.3 (Day 60)	13.3 (Day 90)
IN-JCZ38	18.2 (Day 45)	11.9 (Day 45)	16.4 (Day 45)	19.4 (Day 120)	14.3 (Day 120)	32.8 (Day 120)	11.5 (Day 115)	13.7 (Day 60)
IN-JSE76	25.6 (Day 120)	6.2 (Day 120)	33.3 (day 120)	9.7 (Day 120)	28.5 (Day 120)	4.5 (Day 120)	32.5 (Day 120)	13.7 (Day 120)
IN-K5A77	6.2 (Day 30)	1.6 (Day 120)	5.7 (Day 60)	1.9 (Day 30	6.1 (Day 120)	4.0 (Day 91)	5.1 (Day 120)	1.4 (Day 30)
IN-K5A78	2.4 (Day 120)	ND	9.7 (Day 90)	ND	3.1 (Day 120)	0.15 (Day 30)	10.5 (Day 120)	ND
IN-K5A79	ND	ND	2.4 (Day 120)	ND	ND	ND	3.0 (Day 120)	2.4 (Day 120)
IN-K7H19	1.3 (Day 120	ND	ND	ND	1.6 (Day 120)	ND	ND	ND

The residue decline data for the various soils were analyzed using a number of kinetics models. The FOMC model provided the best visual and statistical fit for the observed data across the entire data range.

Table 24 Summary of cyantraniliprole DT_{50} and DT_{90} values in soils

Soil	Site	DT_{50}-P (days)	DT_{90}-P (days)	pH(water)	% OC	CEC	%Clay
Silt loam	Gross-Umstadt	43.7	235	7.04	1.2	8.7	6
Silty clay loam	Lleida	20.9	156	8.05	2.6	17.4	32
Loam	Nambsheim	8.7	66.2	7.9	1.2	6.7	10
Sandy loam	Sassafras	91.9	376	4.62	0.9	6.1	9
Silty clay loam	Tama	39.2	580	6.2	1.6	21.4	34

Degradation rates for all significant metabolites (found at more than 10% of total residue or exceeded 5% at multiple sampling intervals) were investigated in a series of studies. DT_{50} and DT_{90} values from these studies are summarised below.

Table 25 Summary of cyantraniliprole metabolite DT_{50} and DT_{90} values in soils

Compound	Site	DT_{50}-P	DT_{90}-P	pH(water)	% OC	CEC	%Clay
IN-JCZ38	Gross-Umstadt	12.1	63.4	6.98	1.0	11.1	12
IN-JCZ38	Lleida	5.0	29.9	7.93	2.0	17.5	38
IN-JCZ38	Nambsheim	3.6	19.4	8.12	1.2	7.0	15
IN-JCZ38	Sassafras	11.2	77.5	5.95	0.6	9.8	13

Compound	Site	DT_{50}-P	DT_{90}-P	pH(water)	% OC	CEC	%Clay
IN-JCZ38	Tama	9.1	237	6.38	2.2	23.4	34
IN-JSE76	Gross-Umstadt	219	726	6.98	1.0	11.1	12
IN-JSE76	Lleida	157	522	7.93	2.0	17.5	38
IN-JSE76	Nambsheim	86.3	1089	8.12	1.2	7.0	15
IN-JSE76	Sassafras	343	1319	5.95	0.6	9.8	13
IN-JSE76	Tama	1249	5929	6.38	2.2	23.4	34
IN-K5A79	Gross-Umstadt	42.8	142	7.6	1.2	10.2	20
IN-K5A79	Lleida	16.4	54.6	7.64	2	17.6	45
IN-K5A79	Nambsheim	25.4	84.3	7.55	1.6	7.7	13
IN-K5A79	Sassafras	105	350	6.8	1.7	7.1	11
IN-K5A79	Tama	130	432	7.67	1.9	17.1	41
IN-J9Z38	Gross-Umstadt	119	1117	6.99	1.1	8.67	6.8
IN-J9Z38	Lleida	104	346	7.89	1.63	15.74	32.8
IN-J9Z38	Nambsheim	76.8	255	7.95	1.57	7.57	9.6
IN-J9Z38	Sassafras	220	1168	5.37	1.4	7.39	7.6
IN-J9Z38	Tama	177	588	6.17	1.86	19.42	29.6
IN-K5A77	Gross-Umstadt	202	1216	6.99	1.1	8.67	6.8
IN-K5A77	Lleida	27.4	154	7.89	1.63	15.74	32.8
IN-K5A77	Nambsheim	24.0	109	7.95	1.57	7.57	9.6
IN-K5A77	Sassafras	328	1680	5.37	1.4	7.39	7.6
IN-K5A77	Tama	79	1460	6.17	1.86	19.42	29.6
IN-K5A78	Gross-Umstadt	305	1013	6.99	1.1	8.67	6.8
IN-K5A78	Lleida	500	1662	7.89	1.63	15.74	32.8
IN-K5A78	Nambsheim	977	3244	7.95	1.57	7.57	9.6
IN-K5A78	Sassafras	97.8	929	5.37	1.4	7.39	7.6
IN-K5A78	Tama	237	788	6.17	1.86	19.42	29.6
IN-PLT97	Gross-Umstadt	1744	5792	7.6	1.16	10.2	20
IN-PLT97	Lleida	439	1458	7.64	2	17.6	45
IN-PLT97	Nambsheim	711	2363	7.99	1.5	6.7	12
IN-PLT97	Sassafras	1837	6103	6.25	0.93	5.8	17
IN-PLT97	Tama	429	1426	6.71	2.5	22.5	35
IN-QKV54	Gross-Umstadt	215	714	6.9	1.4	9.8	13
IN-QKV54	Lleida	74	247	7.8	1.7	15.0	45
IN-QKV54	Nambsheim	138	457	7.3	2.0	11.7	11
IN-QKV54	Sassafras	179	595	5.3	0.8	5.4	3
IN-QKV54	Tama	161	536	6.7	3.3	17.3	31
IN-RNU71	Gross-Umstadt	107	356	6.9	1.4	9.8	13
IN-RNU71	Lleida	43	142	7.8	1.7	15.0	45
IN-RNU71	Nambsheim	42	139	7.3	2.0	11.7	11
IN-RNU71	Sassafras	175	580	5.3	0.8	5.4	3
IN-RNU71	Tama	46	152	6.7	3.3	17.3	31

Results for IN-JCZ38 reported by Wardrope (2007) [Ref: DP-17597]

Results for IN-JSE76 reported by McCorquodale & Wardrope (2007) [Ref: DP-17598]

Results for IN-K5A79 reported by Pedersen (2008) [Ref: DP-18868]

Results for IN-J9Z38 reported by McCorquodale (2006) [Ref: DP-17596]

Results for IN-K5A77 reported by Morriss (2006) [Ref: DP-17602]

Results for IN-K5A78 reported by McCorquodale & Wardrope (2007) [Ref: DP-17599]

Results for IN-PLT97 reported by Malekani (2008) [Ref: DP-19077]

Results for IN-QKV54 reported by Wardrope (2010) [Ref: DP-29555]

Results for IN-RNU71 reported by McCorquodale (2010) [Ref: DP-29801]

The aerobic degradation of cyantraniliprole in soil is outlined below.

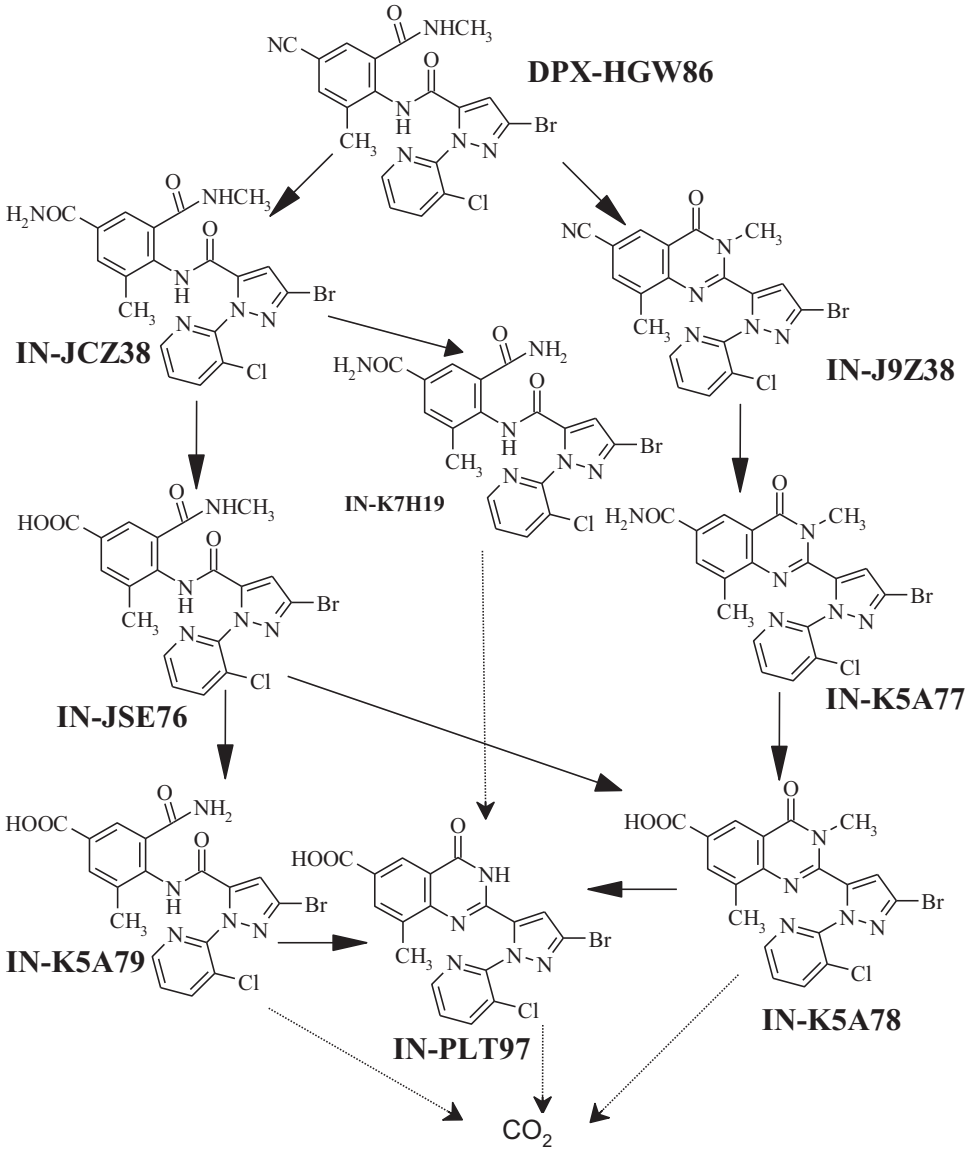


Figure 7 Proposed degradation pathway for cyantraniliprole in aerobic soil

Soil photolysis

The meeting received two studies investigating the soil photolysis of cyantraniliprole on a sandy loam soil (Nambsheim, pH 7.51, 1.22% organic carbon) under continuous irradiation for up to 15 days at 20 ± 2 °C in the study on air-dried soil reported by Morriss (2005) [Ref DP-17056] and up to 30 days at 20 ± 2 °C in the study on moist soil reported by Wardrope (2010) [Ref: DP-28730].

In the 2005 study [Ref DP-17056] the moisture content of the air dried soil was about 14% and the radiolabels were applied to achieve a nominal concentration of 0.4 µg ai/g soil (equivalent to 0.4 kg ai/ha soil) while in the 2010 study [Ref: DP-28730] a higher treatment rate equivalent to 1 kg ai/ha and the moisture level was maintained at about 75% field capacity throughout the study.

The experimental design of both studies were similar, with [CN-^{14}C]-cyantraniliprole and [PC-^{14}C]-cyantraniliprole being applied by pipette to thinly-layered soil and irradiated continuously in artificial irradiation (filtered to eliminate wavelengths of < 290 nm). Both studies included an NaOH trap for the collection of $^{14}CO_2$ and soil samples were extracted twice with acetone:formic acid (9:1)

followed by an extraction with acetone. The extracts were combined, concentrated to a small volume and analysed by reverse-phase HPLC. Identification of the degradation products was performed by HPLC and LC/MS/MS or TLC analysis.

No significant photolysis of cyantraniliprole was observed in the air-dried soil (2005) study, as the results from the irradiated and non-irradiated systems were very similar. One major degradation product, IN-J9Z38, was formed in both irradiated and non-irradiated systems, accounting for < 10% AR and three minor transformation products were observed, each of which were only observed at one sampling occasion and accounted for < 5% AR.

Using a first-order kinetic model, the DT_{50} and DT_{90} values for cyantraniliprole in non-irradiated samples were 115 days and 381 days, respectively. In irradiated samples the DT_{50} and DT_{90} values were 84 days and 278 days, respectively. Levels of IN-J9Z38 were rising at the end of the study.

To isolate the rate of degradation of cyantraniliprole due to photolysis only, the first order rate of degradation constant (k) in the non-irradiated samples was subtracted from the first order rate of degradation constant in the irradiated samples. The resulting corrected DT_{50} and DT_{90} values, extrapolated from this 15-day study, were 308 days and 1024 days, respectively.

In the 2010 moist soil study [Ref: DP-28730], cyantraniliprole residues decreased from a mean value of 102% AR at Day 0, to a mean of 1.2% AR after 30 days in the irradiated soil and to a mean of 33% AR in non-irradiated soil. Three significant degradation products were detected over the course of the study. One of these degradation products was identified as IN-J9Z38, reaching a maximum of 49% AR at Day 10 then declining to 34% AR in the irradiated soil and 49% AR in the non-irradiated soil by the end of the study. IN-J9Z38 was likely a result of non-photolytic reactions and has been seen earlier in all soil degradation studies in dark. Two additional degradation products were attributed to photo-degradations, and were identified as IN-RNU71 and IN-QKV54. IN-RNU71 reached a maximum mean value of 13% AR by the end of the study, while IN-QKV54 reached a maximum mean value of 14% AR at Day 10. A further six minor degradation products were also found at < 5% AR and with the exception of IN-NXX70 (3.8% AR), are likely due to non-photolytic reactions.

Using a simple first-order (SFO) model, the calculated DT_{50} and DT_{90} values for cyantraniliprole in irradiated soil were 6.9 days and 23 days, respectively and 16 days and 53.3 days, respectively in non-irradiated (dark control) samples.

To calculate the rate of degradation due to photolysis only, the first-order rate of degradation constant in the non-irradiated samples was subtracted from the first-order rate of degradation constant in the irradiated samples. The dark corrected DT50 and DT90 values were 12.2 days and 40.5 days respectively.

Table 26 Photodegradation, DT_{50} and DT_{90} for [^{14}C]-cyantraniliprole in moist soil

System	Kinetic model	Optimized parameters ± standard error	χ^2 error	r^2	DT_{50} (days)	DT_{90} (days)
Parent only irradiated soil system	SFO	M0=97.01 ± 4.4 k_SFO=0.1001 ± 0.001	12	0.943	6.9	23.0
	SFO [a]	k_photolysis=0.0567 [b]	–	–	12.2	40.6
Dark control system	SFO–SFO	M0 (AR)=95.44 ± 5.27 k_cyantraniliprole (d^{-1})=0.0434 ± 0.0056 k_J9Z38=0.0234 ± 0.0117	8	0.829	10.8	36
Parent plus four metabolites irradiated system	All SFO	M0= 96.2 ± 2.6 Fr_cyantraniliprole to J9Z38=0.66 ± 0.058 k_cyantraniliprole=0.0976 ± 0.0059	12	0.94	7.1	23.6
		k_IN-J9Z38=0.0269 ± 0.0072	25	0.716	25.8	85.6
		k_IN-RNU71=0.065 ± 0.044	18	0.918	10.7	35.4
		k_IN-NXX70=0.442 ± 0.217	36	0.360	1.6	5.0
		k_IN-QKV54=0.091 ± 0.034	16	0.834	7.7	25.4

[a] Corrected first-order rate of degradation (k) calculated by subtracting the dark control sample

[b] Obtained by subtraction (0.1001–0.0434)

Kinetic modelling suggested that nearly 34% of the parent compound degraded via the photolysis pathway while ca 64% degraded via the soil degradation pathway, indicating that photolysis on the surface of moist soil may be a significant dissipation pathway for cyantraniliprole in the environment.

Significant degradation of cyantraniliprole in irradiated moist soil samples occurred primarily via the non-photolytic soil degradation transformations observed in the absence of light. IN-J9Z38 observed as a major metabolite along with IN-JCZ38, IN-JSE76, IN-K5A77, IN-K5A79, and IN-K7H19 were all identified in the aerobic soil metabolism study. Photodegradation products observed, IN-NXX70 and IN-QKV54, were derived sequentially from photolysis of cyantraniliprole; while IN-RNU71 is proposed to be generated from photolysis of IN-J9Z38. Similar light induced transformations have been observed in the aqueous photolysis and the outdoor water sediment study.

Degradation in soil

Ten field studies were conducted to investigate the degradation and mobility of cyantraniliprole under field conditions. In all of the trials a single application of 0.3 kg ai/ha or 0.45 kg ai/ha was made to bare soil in late spring or early summer.

Soil samples were collected, down to a maximum depth of 90 cm, immediately prior to application and at pre-determined intervals over an 18 month period. Samples were extracted in aqueous acetone, cleaned up via SPE columns and analysed using reverse phase HPLC separation coupled to tandem mass spectrometry (LC-MS/MS). The Limit of Quantification (LOQ) for all analytes was 0.001 mg/kg and mean recovery rates ranged from 74–96%. Analytes included the major soil metabolites (IN-J9Z38, IN-JCZ38, IN-JSE76, IN-K5A77, IN-K5A78, IN-K5A79, and IN-PLT97) in addition to the parent compound. In order to determine the extent of formation of photodegradation products, analysis of IN-NXX70, IN-QKV54, and IN-RNU71 was also carried out in a number of the studies and reported by Vogl & Sharma (2010) [Refs: DP-29556 and DP-29557].

The field studies demonstrated rapid degradation of the cyantraniliprole in field soils (17–51 day half lives). While laboratory studies suggested that pH had some effect on degradation rates, degradation rate under field conditions does not appear to be pH-dependent. Approximately 75% or more of the applied mass dissipated in most of the field studies during the study duration. Downward mobility of the parent compound as well as its metabolites was limited, with residues rarely found below 15 cm. Uptake of the test compound or its metabolites by the crop was generally very low, despite the systemic nature of cyantraniliprole. Soil metabolites, especially those from the IN-JCZ38 side of the degradation pathway (IN-JCZ38, IN-JSE76, and IN-K5A79) were not observed in the field studies to the extent that was expected based on the lab studies. Photochemical degradation was only a minor degradation mechanism (<< 5%) in the field.

Table 27 Maximum formation [%] of all metabolites in soil dissipation studies with cyantraniliprole

Study Ref Location	kg ai/ha [a]	IN-J9Z38	IN-JCZ38	IN-JSE76	IN-K5A77	IN-K5A78	IN-K5A79	IN-PLT97
DP-17061 Spain	0.4	35.8	5.2	4.0	7.7	3.7	0.0	1.5
DP-17062 France	0.45	41.7	11.1	11.6	11.0	13.4	8.2	7.7
DP-17063 Germany	0.35	16.0	2.7	0.4	1.7	0.4	1.4	0.0
DP-17458 Italy	0.22	28.4	13.2	11.7	9.2	7.2	6.1	2.4
DP-17065 USA (WA)	0.43	33.3	9.8	5.6	10.2	7.9	3.5	4.4

Study Ref Location	kg ai/ha [a]	IN-J9Z38	IN-JCZ38	IN-JSE76	IN-K5A77	IN-K5A78	IN-K5A79	IN-PLT97
DP-17066 USA (CA)	0.41	21.8	8.0	5.6	6.1	5.1	1.0	1.9
DP-17067 (USA (MO)	0.45	23.1	8.0	3.3	6.0	2.7	1.4	1.2
DP-17457 USA (TX)	0.23	36.2	5.5	13.7	7.0	16.8	2.1	6.7
DP-17064 USA (NY)	0.27	20.1	7.2	2.7	3.1	1.4	3.5	0.6
DP-17068 Canada (MB)	0.25	12.7	3.4	2.7	1.6	0.3	0.9	0.4
Overall max % -bare soil		41.7	13.2	13.7	11.0	16.8	8.2	7.7
DP-17065 USA (WA)^{CS}	0.45	26.9	10.9	8.7	6.4	4.0	3.3	2.4
DP-17066 USA (CA)^{CS}	0.45	15.6	13.1	5.6	5.1	2.4	1.3	0.9
DP-17067 USA (MO)^{CS}	0.45	14.7	8.2	5.3	3.1	2.4	3.8	2.3
Overall max % cropped soil		26.9	13.1	8.7	6.4	4.0	3.8	2.4

[a] kg ai/ha used to compute % metabolite. Value is lower of either nominal applied or actual found on Day 0.

CS=cropped soil plot, all others bare soil plots

The mean temperature-normalized field DT_{50} value from all studies was 32.4 days, consistent with the mean laboratory derived value of 31.8 days.

Table28 Mean temperature-normalized field DT_{50} values from soil dissipation studies. (Snyder & White, 2010 [Ref: DP-31454].

Study location	Sand (%)	Silt (%)	Clay (%)	Organic matter (%)	pH	Soil Texture	DT_{50} (days)	Ref
Washington, USA	72	20	9	1.1	7.44	Sandy loam	31.4	DP-17065
California, USA	63	30	8	0.9	7.81	Sandy loam	20.9	DP-17066
Missouri, USA	16	60	24	2.3	6.2	Silt loam	50.8	DP-17067
Texas, USA	54	19	27	1.2	7.9	Sandy clay loam	22.9	DP-17457
New York, USA	80	16	4	3.2	6.5	Loamy sand	51.3	DP-17064
Manitoba, Canada	30	42	28	6.6	7.8	Clay loam	38.5	DP-17068
Nambsheim, France	22	48.8	29.2	3.6	7.93	Clay loam	16.9	DP-17062
Sevilla, Spain	84.4	6.8	8.8	2.5	8.22	Loamy sand	31.5	DP-17061
Goch, Germany	39	52	9	2.5	6.3	Silt loam	46.7	DP-17063
Milan, Italy	45	38	17	1.8	6	Loam	33.8	DP-17458
Geometric mean for all field studies(based on normalized best fit kinetics)							32.4	

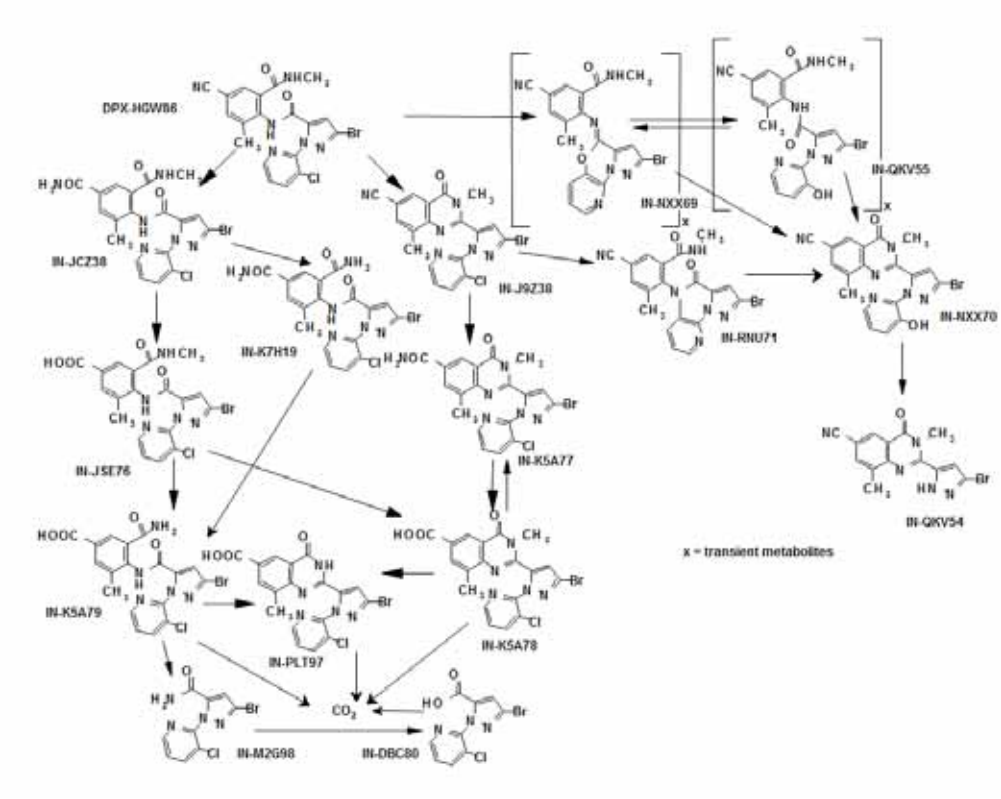


Figure 8 Overall degradation pathway for cyantraniliprole in soil

Degradation in water/sediment systems

A study on the fate of [^{14}C]-cyantraniliprole in the absence of light under anaerobic conditions in two aquatic sediment test systems was reported by McCorquodale & Wardrope, 2006 [Ref: DP-17059]. The sand sediment system, at a pH of 5.6 contained 2% organic carbon with overlying water at pH 6.1 and 56.4 mg/L organic carbon and the silt loam sediment system, at a pH of 7.7 contained 4.9% organic carbon with overlying water at pH 7.6 and 21.2 mg/L organic carbon. [CN-^{14}C]-cyantraniliprole and [PC-^{14}C]-cyantraniliprole were separately applied to the two sediment systems at a rate of 0.4 μg ai/g and incubated for up to 100 days in the dark at 20 ± 2 °C.

Under anaerobic conditions, cyantraniliprole degraded in the water phase and also partitioned to the sediment where it was further degraded to other metabolites that eventually were incorporated into the sediment organic fraction.

The major metabolite in both systems was IN-J9Z38 which increased to about 23% AR (0.09 ppm) at Day 28 before declining to 7.3% AR (0.03 ppm) in the sand system at the end of the study period (Day 100) and increased to about 40% AR (0.16 ppm) at Day 7 before declining to 5.1% AR (0.02 ppm) by day 100 in the silt loam system.

The IN-K5A77 metabolite accounted for greater than 5% AR at two non-subsequent sampling occasions in the silt loam sediment, but was a minor metabolite in the sand system and silt loam surface water. Additional minor metabolites IN-K5A78 and IN-JSE76 were also observed at ≤ 1.6% and 0.8% AR, respectively. The remaining, combined, unidentified metabolites did not exceed *ca* 1.3% of the applied radioactivity.

The first-order DT_{50} and DT_{90} values for dissipation from the water phase and degradation in the water phase, sediment and total system are summarised below.

Table 29 Cyantraniliprole and metabolite DT_{50} and DT_{90} in anaerobic water sediment systems (in the absence of light)

Compound	System	Endpoint type	DT_{50} (days)	DT_{90} (days)
Loamy sand sediment				
Cyantraniliprole	Water	Dissipation	15.9	53.0
	Total system	Degradation	25.1	83.4
	Water	Degradation	37.7	125
	Sediment	Dissipation	76.6	254
IN-J9Z38	Water	Dissipation	39.0	130
	Total system	Degradation	155	515
	Water	Degradation	324	1077
	Sediment	Degradation	122	405
Silt loam sediment				
Cyantraniliprole	Water	Dissipation	2.74	9.09
	Total system	Degradation	3.87	12.8
	Water	Degradation	3.34	11.1
	Sediment	Dissipation	35.91	1191
IN-J9Z38	Water	Dissipation	17.8	59.1
	Total system	Degradation	272	905
	Water	Degradation	544	1807
	Sediment	Degradation	312	1036
IN-K5A77	Total system	Degradation	19.7	65.4

The aerobic degradation of [^{14}C]-cyantraniliprole in two irradiated water/sediment systems was studied by Wardrope & Sharma, 2010 [Ref: DP-24798]. A sand sediment system (pH 5.6 containing 2% organic carbon with overlying water at pH 6.1 and 56.4 mg/L organic carbon) and a silt loam sediment system (pH 7.7 containing 4.9% organic carbon with overlying water at pH 7.6 and 21.2 mg/L organic carbon) were treated with [PC-^{14}C]-cyantraniliprole at a rate of 0.5 µg ai/g and incubated outdoors for 14 days under natural sunlight at 23 ± 2 °C.

At zero time the majority of the radioactivity was detected in the surface water (92–94% AR) decreasing to about 16% AR (silt loam system) and 33% AR (sand system) by Day 14. Solvent extractable radioactivity in the sediment increased from 1% AR at Day 0, to a maximum value of 64% AR (silt loam system) and 55% AR (sand system) by Day 14. Non-extractable residues increased to a maximum value of 5–10% AR by the end of the study period. No radioactivity associated with $^{14}CO_2$ was detected throughout the study period.

HPLC analysis of irradiated water sediment systems demonstrated that [^{14}C]-cyantraniliprole degraded rapidly in the water phase, from 90% AR at Day 0 to 1–2% AR at Day 14, through a combination of degradation and transfer into the sediment. One significant metabolite was identified as IN-J9Z38, found in the surface water at a maximum of about 15% AR at Day 3 (silt loam system) and 27% AR at Day 5 (sand system) then declining to 4.5% AR and 12% AR respectively on Day 14. Metabolite IN-RNU71 was present at about 4–10% AR and but not observed in dark controls, implying that it was a photolysis product of IN-J9Z38. Minor degradation products (14) accounted for a combined maximum of 6–11% AR and individually less than 3% AR.

The amount of [^{14}C]-cyantraniliprole in the sediment of the irradiated silt loam system increased initially due to transfer from the water phase, from about 1% AR at Day 0 to a maximum of 22% AR at Day 2 (silt loam system) and 15% AR at Day 3 (sand system) before declining to 9% and 6% AR respectively at 14 days. Six to seven metabolites were detected in the sediment extracts including IN-J9Z38 which increased from about 2% AR at Day 1 to about 42% AR at Day 14. The IN-RNU71 metabolite was present at up to 5% AR and the remaining components each accounted for less than 3% AR.

Table 30 Aerobic degradation of cyantraniliprole in an irradiated silt-loam sediment/water system

Compound	System	% Applied Radioactivity							
		Sampling Intervals (days)							
		0	1	2	3	5	7	10	14
Cyantraniliprole	Water	90.33	68.12	56.14	23.65	10.39	3.77	2.49	2.00
	Sediment	1.10	16.52	22.10	21.94	19.46	15.82	17.26	9.03
	Total	91.43	84.64	78.24	45.59	29.85	19.59	19.75	11.03
	Water (dark)						17.55		9.67
	Sediment (dark)						37.80		30.85
	Total (dark)						55.35		40.52
IN-J9Z38	Water	1.67	4.50	3.11	14.75	14.53	13.42	8.65	4.50
	Sediment	ND	1.57	3.16	22.48	32.57	35.28	41.65	43.29
	Total	1.67	6.07	6.27	37.23	47.10	48.70	50.30	47.79
	Water (dark)						4.06		2.78
	Sediment (dark)						21.04		39.01
	Total (dark)						25.10		41.79
IN-RNU71	Water	ND	ND	ND	1.53	4.27	3.71	3.78	2.52
	Sediment	ND	ND	ND	ND	1.81	2.16	4.52	3.73
	Total	ND	ND	ND	1.53	6.08	5.87	8.30	6.25
Unidentified Radioactivity	Water	ND	1.96	2.76	1.89	3.04	4.00	6.20	6.11
	Sediment	ND	1.09	ND	2.02	2.94	1.69	ND	8.11
	Total	ND	3.05	2.76	3.91	5.98	5.69	6.20	14.22
	Water (dark)						1.65		2.61
	Sediment (dark)						1.93		2.46
	Total (dark)						3.58		5.07
Total extracted residues	Water	92.00	74.56	62.00	41.82	32.24	24.91	21.12	15.70
	Sediment	1.10	19.19	25.26	46.44	56.78	54.96	63.44	64.15
	Water (dark)						23.26		15.05
	Sediment (dark)						60.77		72.31

Table 31 Aerobic degradation of cyantraniliprole in an irradiated sand sediment/water system

Compound	System	% Applied Radioactivity							
		Sampling Intervals (days)							
		0	1	2	3	5	7	10	14
Cyantraniliprole	Water	91.45	78.32	60.40	61.02	25.34	31.61	6.27	1.22
	Sediment	0.88	7.61	9.80	14.78	8.00	14.31	9.72	6.30
	Total	92.33	85.93	70.20	75.80	33.34	45.92	15.99	7.52
	Water (dark)						50.68		7.53
	Sediment (dark)						18.92		22.84
	Total (dark)						69.90		30.37
IN-J9Z38	Water	2.80	4.47	13.78	8.28	26.78	13.42	20.26	11.86
	Sediment	ND	2.06	7.31	7.16	20.88	15.05	34.86	41.71
	Total	2.80	6.53	21.09	15.44	47.66	28.47	55.12	53.57
	Water (dark)						9.91		8.03

Compound	System	% Applied Radioactivity							
		Sampling Intervals (days)							
		0	1	2	3	5	7	10	14
	Sediment (dark)						13.78		48.27
	Total (dark)						23.69		56.30
IN-RNU71	Water	ND	ND	0.36	1.11	4.55	3.28	9.71	9.92
	Sediment	ND	ND	ND	ND	ND	ND	2.50	5.14
	Total	ND	ND	0.36	1.11	4.55	3.28	12.21	15.06
Unidentified Radioactivity	Water	ND	1.92	2.49	2.89	4.85	11.37	6.56	10.16
	Sediment	ND	1.86	0.73	ND	2.91	2.64	1.54	1.59
	Total	ND	3.78	3.22	2.89	7.76	14.01	8.10	11.75
	Water (dark)						ND		1.75
	Sediment (dark)						0.60		2.85
	Total (dark)						0.60		4.60
Total extracted residues	Water	94.25	84.71	77.03	73.31	61.53	59.66	43.19	33.17
	Sediment	0.88	11.53	17.84	21.94	31.79	32.01	48.63	54.75
	Water (dark)						60.59		17.32
	Sediment (dark)						33.30		73.96

The % AR for the silt loam and sandy irradiated sediment/water systems, the dark control systems and the pH 7 buffer system, were analyzed using ModelMaker to derive various degradation rates. The calculated DT_{50} and DT_{90} values for cyantraniliprole, IN-J9Z38 and IN-RNU71 are summarized below.

Table 32 Cyantraniliprole and metabolite DT_{50} and DT_{90}'s in irradiated aerobic water sediment systems

Sediment/water system	Model (All SFO)	Component	DT_{50} (days)	DT_{90} (days)
Silt loam outdoor	Parent only, total system	cyantraniliprole	3.5	11.6
	Parent + 2 metabolites, total system	cyantraniliprole	3.6	11.9
		IN-J9Z38	40.1	133
		IN-RNU71	13.5	45.0
	Parent + 2 metabolites, two phase system	cyantraniliprole _sediment	2.6	8.3
		cyantraniliprole _water	6.7	
		IN-J9Z38_sediment	74.7	
		IN-J9Z38_water	3.9	
		IN-RNU71_water	3.1	
Sandy, outdoor	Parent only, total system	cyantraniliprole	4.4	14.8
	Parent + 2 metabolites, total system	cyantraniliprole	4.5	14.9
		IN-J9Z38	40.8	135
	Parent + 2 metabolites, two phase system	cyantraniliprole _sediment	2.3	7.6
		cyantraniliprole _water	10.1	33.4
		IN-J9Z38_sediment	49.4	
		IN-J9Z38_water	11.3	
		IN-RNU71_water	42.9	
Silt loam and sandy [a] dark control	Parent + 1 metabolite	cyantraniliprole	7.7	25.6
Buffer control outdoor	Parent + 3 metabolites	cyantraniliprole	14.1	47
		IN-J9Z38	2.5	8.4
		IN-RNU71	12.1	40.3

[a] Both sediments modelled as replicates

In summary, cyantraniliprole degrades rapidly in outdoor water sediment systems, with total system DT_{50} values of 3.5 and 4.4 days in the silt loam and sand systems, respectively. The corresponding parent only total system DT_{90} values were 11.6 and 14.8 days in the silt loam and sand systems, respectively. The degradation rate was significantly faster than corresponding systems shielded from sunlight. The degradation rate was also faster than degradation observed in sterilized buffer exposed to sunlight.

Significant metabolites formed in the water/sediment system exposed to sunlight were IN-J9Z38 and IN-RNU71. While IN-J9Z38 was also identified in the silt loam and sand system dark controls, degradation was faster in the presence of light, suggesting that photodegradation is an important pathway. The formation of IN-RNU71, a photodegradate of IN-J9Z38 supports this conclusion.

Cyantraniliprole degradation in water/sediment systems involved a number of pathways and is outlined below.

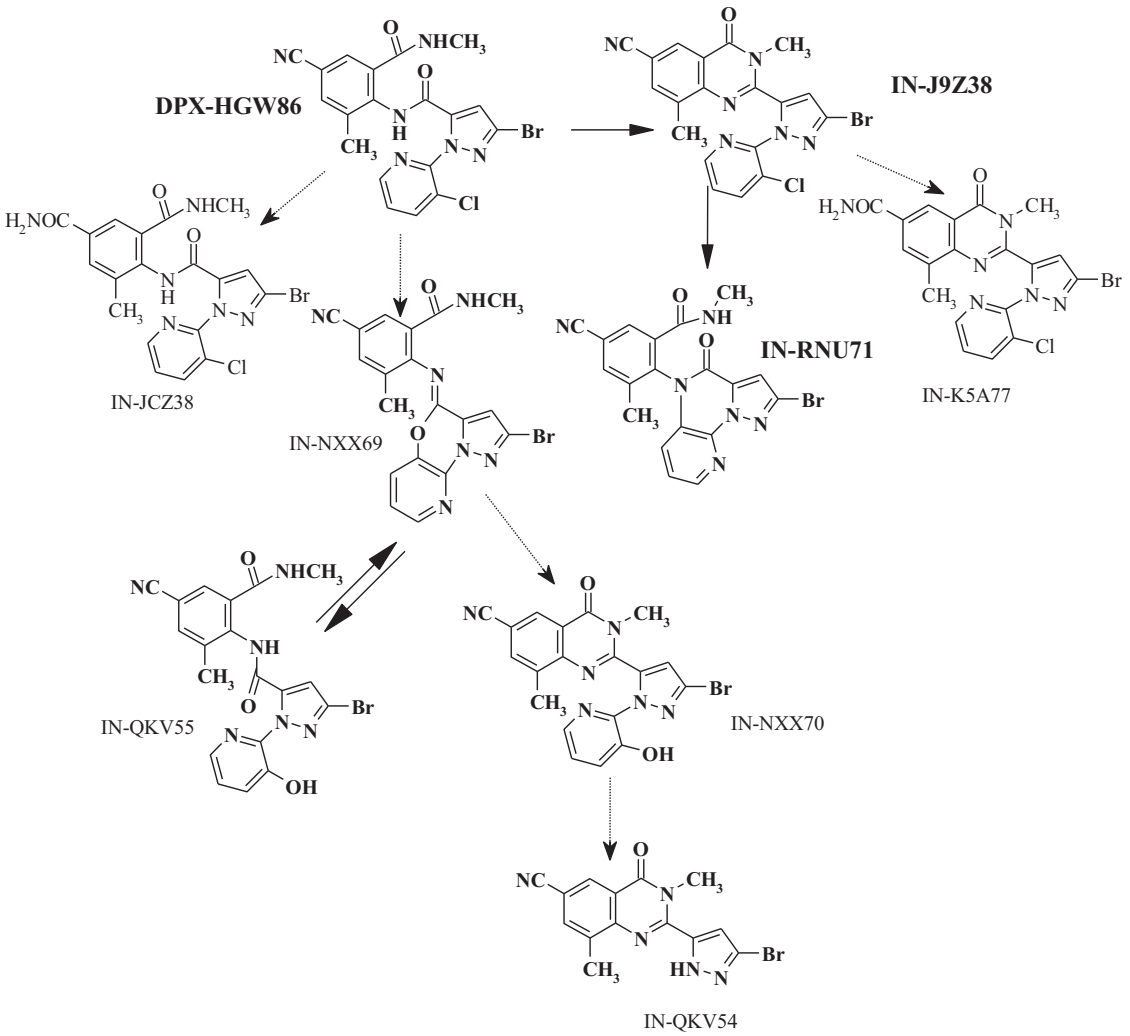


Figure 9 Degradation of cyantraniliprole in outdoor water-sediment

Residues in rotational crops

Rotational crops metabolism

The Meeting received information on the fate of residues in lettuce, red beet, wheat, soya bean and wheat grown as rotational crops in cyantraniliprole-treated soil.

In a preliminary glasshouse study reported by Scott, Swain & Young (2006) [Ref: DP-15513], a sandy loam soil was treated with the equivalent of 0.3 kg ai/ha of [PC-^{14}C]-cyantraniliprole (SC formulation) and wheat, soya bean, and red beet seeds were sown 25 and 120 days after treatment.

Crop samples from each soil-aging interval were taken, pulverised in dry ice and total radioactive residues (TRR) determined by combustion analysis and LSC. Samples were extracted using acetonitrile:water (9:1) followed by acetonitrile:water (7:3 or 1:1), cleaned up by SPE and analysed by HPLC, with confirmation by LC-MS and LC-MS/MS.

TRRs in wheat straw were 1.15 mg/kg and 0.75 mg/kg (cyantraniliprole equivalents) from the 25 and 120 day soil aging intervals, respectively. Lower TRRs were observed in the corresponding wheat chaff (0.37 and 0.21 mg/kg) and grain (0.02 mg/kg). Cyantraniliprole was the predominant residue in wheat straw (0.52 mg/kg) and chaff (0.23 mg/kg) after the 25-day soil aging period and 0.36 and 0.1 mg/kg respectively after the 120-day aging period. Components in straw from the 25-day aging period included IN-N7B69 (0.01 mg/kg), IN-N7B69 glucoside (0.07 mg/kg), IN-JCZ38 (0.08 mg/kg), IN-K5A77 (0.03 mg/kg), IN-JSE76 (0.1 mg/kg), IN-J9Z38 (0.05 mg/kg), IN-MLA84 (0.01 mg/kg), IN-K5A78 (0.01 mg/kg), IN-K5A79 (0.01 mg/kg) and polars (0.03 mg/kg). In general all the components (with the exception of IN-K5A78) were observed in the straw from the 120-day soil aging period but at lower concentrations. In grain, residues included cyantraniliprole, IN-N7B69, IN-N7B69 glucoside, IN-JSE76 and polars, each at ≤ 0.01 mg/kg.

Table 33 Distribution of residues in wheat planted as a rotational crop 25 days and 120 days after soil treatment with [PC-^{14}C]-cyantraniliprole (0.3 kg ai/ha)

Aging period	25 day						120 days					
	Grain		Chaff		Straw		Grain		Chaff		Straw	
Residue	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg
TRR		0.021		0.374		1.149		0.019		0.201		0.749
Extracted	90.5	0.019	87.7	0.328	94.6	0.966	97.6	0.019	106	0.213	92.4	0.692
Cyantraniliprole	9.7	0.002	61.6	0.23	45.3	0.521	14.6	0.003	47.4	0.101	47.9	0.359
Polars [a]	37.9	0.008	3.1	0.012	3.0	0.034	39.8	0.008	7.0	0.015	23.8	0.028
IN-K5A77	ND		ND		0.91	0.011	ND		ND		1.7	0.013
IN-K5A78	ND		ND		0.87	0.010	ND		5.9	0.013	ND	
IN-K5A79	ND		ND		2.5	0.029	ND		ND		ND	
IN-J9Z38	ND		4.1	0.015	4.6	0.053	ND		ND		5.7	0.043
IN-JCZ38	ND		5.0	0.019	6.5	0.075	ND		6.9	0.015	7.4	0.056
IN-JSE76	ND		3.3	0.012	9.2	0.105	3.5	0.001	8.3	0.018	9.4	0.07
IN-MLA84	ND		4.2	0.016	3.7	0.014	ND		ND		5.7	0.043
IN-N7B69	13.2	0.003	ND		1.1	0.013	25.1	0.005	ND		3.1	0.023
IN-N7B69gluc [b]	22.0	0.005	ND		6.7	0.07	ND		ND		1.9	0.014

[a] Polars are one or more components individually present at ≤ 0.02 mg/kg

[b] IN-N7B69 glucoside

TRRs in soya bean foliage, pods, and seed grown in soil aged 25 days were 0.18, 0.09, and 0.04 mg/kg, respectively. TRRs in the corresponding soya bean fractions from the 120-day interval were 0.13, 0.07, and 0.02 mg/kg, respectively. Cyantraniliprole accounted for 0.07 and 0.02 mg/kg in soya bean foliage from the 25-day and 120-day soil aging periods, respectively. Other foliage components included IN-JCZ38 (0.01 mg/kg), IN-JSE76, IN-MLA84 (≤ 0.03 mg/kg) and IN-J9Z38 (0.02 mg/kg). Soya bean (seed) components included IN-JCZ38, IN-K5A78, IN-N7B69, and polar components, each at ≤ 0.01 mg/kg. No cyantraniliprole was detected in the soya bean seed.

Table 34 Distribution of residues in soya beans planted as a rotational crop 25 days and 120 days after soil treatment with [PC-^{14}C]-cyantraniliprole (0.3 kg ai/ha)

Aging period	25 day						120 days					
	Seed		Foliage		Pod		Seed		Foliage		Pod	
Residue	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg
TRR		0.038		0.185		0.094		0.018		0.127		0.073
Extracted	98.1	0.037	94.1	0.174	86.4	0.081	101	0.018	72.5	0.092	88.6	0.065
Cyantraniliprole	ND		36.3	0.067	14.0	0.013	ND		14.8	0.019	14.2	0.01
Polars [a]	ND		ND		ND		33.6	0.006	ND		11.2	0.008
IN-K5A77	ND		ND		ND		ND		ND		ND	
IN-K5A78	ND		ND		ND		11.1	0.002	ND		ND	
IN-K5A79	ND		ND		ND		ND		ND		ND	
IN-J9Z38	ND		ND		ND		ND		15.6	0.02	ND	
IN-JCZ38	23.6	0.009	7.4	0.014	24.1	0.023	10.9	0.002	ND		9.4	0.007
IN-JSE76	ND		14.4	0.027	ND		ND		9.5	0.012	ND	
IN-MLA84			15.0	0.028	ND		ND		15.6	0.02	ND	
IN-N7B69	23.3	0.009	ND		ND		ND		ND		ND	
IN-N7B69gluc [b]	ND		ND		ND		ND		ND		ND	

[a] Polars are one or more components individually present at ≤ 0.02 mg/kg

[b] IN-N7B69 glucoside

TRRs in red beet were 0.11 and 0.04 mg/kg for foliage samples; 0.02 mg/kg and 0.01 mg/kg in root samples taken from red beets grown in soil aged 25 and 120 days, respectively. No cyantraniliprole was found in beet foliage from either aging period. Foliage components included IN-N7B69 glucoside (≤ 0.05 mg/kg) and polars (0.01 mg/kg). Low levels (≤ 0.01 mg/kg) of cyantraniliprole, IN-N7B69 glucoside, IN-N7B69, IN-J9Z38, and IN-JCZ38 were found in beet roots.

Table 35 Distribution of residues in red beet planted as a rotational crop 25 days and 120 days after soil treatment with [PC-^{14}C]-cyantraniliprole (0.3 kg ai/ha)

Aging period	25 day				120 days			
	Roots		Foliage		Roots		Foliage	
Residue	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg
TRR		0.021		0.107		0.008		0.039
Extracted	99	0.021	92	0.098	99	0.008	94	0.037
Cyantraniliprole	26.8	0.006	ND		16.9	0.001	ND	
Polars [a]	ND		ND		ND		12.9	0.005
IN-K5A77	ND		ND		ND		ND	
IN-K5A78	ND		ND		ND		ND	
IN-K5A79	ND		ND		ND		ND	
IN-J9Z38	11.5	0.002	ND		13.9	0.001	ND	
IN-JCZ38	ND		ND		9.6	0.001	ND	
IN-JSE76	ND		ND		ND		ND	
IN-MLA84	ND		ND		ND		ND	
IN-N7B69	24.8	0.005	ND		14.7	0.001	ND	
IN-N7B69gluc [b]	16.3	0.003	49.5	0.053	ND		40.0	0.015

[a] Polars are one or more components individually present at ≤ 0.02 mg/kg

[b] IN-N7B69 glucoside

In a glasshouse study reported by Chapleo & Green (2012) [Ref: DP-15778], a sandy loam soil (pH 6.2, 1.4% organic matter) was treated with the equivalent of 0.45 kg ai/ha of either [CN-^{14}C]-cyantraniliprole or [PC-^{14}C]-cyantraniliprole (SC formulation) and wheat, lettuce, and red beet seeds were sown 30 and 120 days after treatment. After 365 days aging, a further sowing of spring wheat was made into the soil which had received the 30 day sowing.

Crop samples from each soil-aging interval were taken, pulverised in dry ice and total radioactive residues (TRR) determined by combustion analysis and LSC. Samples were extracted using either acetone:1N formic acid (9:1) or acetonitrile:water (9:1) followed by acetonitrile:water

(7:3) under ambient conditions, with further successive extractions conducted as necessary with enzymes (a mixture including β-glucosidase, cellulase and driselase), 1 M HCl (60 °C, 6 hours); 6 M HCl (90 °C, 6 hours), and 6 M NaOH (90 °C, 24 hours).

Samples were extracted and analysed within 4 weeks of harvest. Subsequent analysis of samples stored for (64 to 73 weeks at –20 °C) reported TRR values similar to the original levels and the residue compositions were also generally comparable.

Soil cores were taken from each soil-aging interval at the time of sowing and maturity (wheat), separated into 0 to 15 cm and 15 to 30 cm portions and each sample extracted with acetone:formic acid (9:1) followed by acetone.

Extracts were analysed using reversed phase HPLC eluted with a gradient of acetonitrile and water containing 0.1% formic acid. The effluent was passed through an UV detector (254 nm) to detect reference standards followed by fraction collection of the column eluate and subsequent LSC to quantify the radiolabelled materials present. LC/MS and also LC/MS/MS were conducted to confirm the presence of cyantraniliprole, IN-J9Z38 and IN-MLA84. LC/MS with multiple reaction monitoring (MRM) was conducted to confirm the presence of cyantraniliprole, IN-JCZ38, IN-JSE76, IN-K7H19, IN-MYX98, and IN-N7B69. Solid phase extraction and enzyme hydrolysis techniques were used to characterise the radioactivity in red beet foliage extracts.

Total radioactive residues in wheat from soil treated with [^{14}C]-cyantraniliprole ranged from < 0.01 to 0.06 mg/kg for wheat grain, 0.09 to 0.31 mg/kg for early forage, 0.31 to 1.62 mg/kg for hay, and 0.27 to 0.97 mg/kg for straw. In beet roots, TRRs ranged from 0.01 to 0.03 mg/kg and were 0.01 to 0.14 mg/kg in foliage. In lettuce, TRRs ranged from 0.02 to 0.11 mg/kg.

Table 36 Total radioactive residues (TRRs), expressed as parent compound equivalents in wheat, lettuce and red beet from three rotations in soil treated with [CN-^{14}C]-cyantraniliprole

TRR	Wheat				Lettuce	Red beet	
[mg/kg]	forage	hay	straw	grain		foliage	roots
[CN-^{14}C]-cyantraniliprole							
1st rotation (30 days)	0.313	1.463	0.973	0.056	0.114	0.063	0.021
2nd rotation (120 days)	0.129	0.312	0.347	0.004	0.036	0.013	0.007
3rd rotation (365 days)	0.128	0.561	0.429	0.014			
[PC-^{14}C]-cyantraniliprole							
1st rotation (30 days)	0.287	1.625	0.966	0.054	0.078	0.145	0.031
2nd rotation (120 days)	0.103	0.449	0.272	0.008	0.023	0.045	0.009
3rd rotation (365 days)	0.089	0.555	0.482	0.018			

Concentrations of total radioactivity in soil from the upper 0 to 15 cm horizon varied throughout the study but remained similar to values measured at 30 days post-application (0.21 to 0.38 mg/kg). Concentrations of total radioactivity in soil from the lower 15 to 30 cm horizon were < 0.01 mg/kg.

Table 37 Total radioactive residues in soil sampled from containers sown with wheat following single applications of 0.45 kg ai/ha [^{14}C]-cyantraniliprole, mg/kg (% TRR)

	Sowing	Maturity	Sowing	Maturity	Sowing	Maturity
Days after soil treatment	30	146	120	263	365	493
[CN-^{14}C]-cyantraniliprole						
Extracted	0.353 (93.2%)	0.092 (79.0%)	0.166 (81.6%)	0.218 (70.9%)	0.217 (73.5%)	0.236 (67.4%)
Unextracted	0.026 (6.8)	0.025 (20.9)	0.037 (18.3)	0.089 (29.0)	0.078 (26.5)	0.114 (32.6)
Total	0.379	0.117	0.203	0.307	0.295	0.350
[PC-^{14}C]-cyantraniliprole						

	Sowing	Maturity	Sowing	Maturity	Sowing	Maturity
Days after soil treatment	30	146	120	263	365	493
Extracted	0.195 (93.5%)	0.158 (81.3%)	0.167 (83.1)	0.150 (74.2%)	0.109 (66.7%)	0.141 (63.3%)
Unextracted	0.014 (6.5)	0.036 (18.7)	0.034 (16.9)	0.052 (25.8)	0.054 (33.3)	0.081 (36.7)
Total	0.209	0.194	0.201	0.202	0.163	0.222

The predominant residue was cyantraniliprole, contributing 14–36% TRR (≤ 0.02 mg/kg) in wheat grain, 21–24% TRR (< 0.01mg/kg) in red beet roots, and 40–69% TRR (0.01 to 0.08 mg/kg) in lettuce. Cyantraniliprole was also the principal radioactive component (0.01 to 0.85 mg/kg, 6 to 73% TRR) in wheat forage (71–73% TRR, 0.2–0.23 mg/kg), hay (52–53% TRR, 0.78–0.85 mg/kg) and straw 941–45% TRR, 0.4–0.44 mg/kg). Unchanged cyantraniliprole in the beet foliage accounted for no more than 0.005 mg/kg (4.1% TRR).

The IN-MYX98 metabolite was found in lettuce at up to 16% TRR (0.007 mg/kg in the 120 day rotation. Minor components individually present at ≤ 0.01 mg/kg (< 10% TRR) included IN-J9Z38, IN-JCZ38, IN-K5A79, IN-K7H19, IN-MLA84, and IN-N7B69 in grain; IN-DBC80, IN-J9Z38, IN-K5A77, IN-MYX98, and IN-N7B69 in red beet roots; and IN-J9Z38, IN-JCZ38, IN-MLA84 in lettuce. Uncharacterised fractions accounted for ≤ 0.01 mg/kg.

The main metabolites detected in wheat forage, hay and straw were IN-J9Z38 (≤ 0.18 mg/kg, 12% TRR), IN-K7H19 (≤ 0.06 mg/kg, 11% TRR), and IN-MLA84 (≤ 0.11 mg/kg, 6.6% TRR) and IN-JCZ38 (≤ 0.05 mg/kg),; concentrations of minor metabolites (IN-DBC80, IN-HGW87 IN-JSE76, IN-K5A77, IN-K5A78, IN-K5A79, IN-MYX98, IN-N7B69, and IN-NBC94) were individually ≤ 0.02 mg/kg . Unextracted residues in the wheat feed samples accounted for no more than 10% TRR.

Red beet foliage contained low level (≤ 0.01 mg/kg) metabolites, IN-DBC80, IN-HGW87, IN-J9Z38, IN-JCZ38, IN-JSE76, IN-K5A77, IN-K5A78, IN-K5A79, IN-K7H19, IN-MLA84, IN-MYX98, IN-N7B69, and IN-NBC94. Unidentified components accounted for up to 0.03 mg/kg. Following enzyme treatment, these unidentified components decreased and levels of cyantraniliprole and IN-N7B69 increased, indicating that both IN-N7B69 and cyantraniliprole were present in conjugated form or associated with soluble natural products. Unextracted residues accounting for no more 9.1% TRR and did not exceed 0.01 mg/kg.

Table 38 Nature of residue in wheat grain from plants sown in soil treated with 0.45 kg ai/ha [^{14}C]-cyantraniliprole

Planting interval:	[CN-^{14}C]-cyantraniliprole				[PC-^{14}C]-cyantraniliprole			
	30 DAT		365 DAT		30 DAT		365 DAT	
	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
TRR (mg/kg) [a]	0.056		0.014		0.054		0.018	
Solvent extractable	43.3	0.024	56.5	0.008	54.5	0.03	71.2	0.013
Cyantraniliprole	20.0	0.011	33.0	0.005	36.3	0.02	13.6	0.003
IN-J9Z38	2.9	0.002	2.8	< 0.001	5.1	0.003	ND	
IN-JCZ38	5.7	0.003	ND		0.8	< 0.001	ND	
IN-K5A79	ND		ND		ND		3.4	0.001
IN-K7H19	ND		ND		ND		7.2	0.002
IN-MLA84	1.9	0.001	ND		1.2	0.001	ND	
IN-N7B69	ND		ND		ND		5.2	0.001
Total unidentified metabolites	1.3	< 0.001	ND		2.3	0.001	23.8	0.005
Total solubilized fractions [b]	48.2	0.026			35.1	0.019		
Total extractable [c]	91.5	0.050	56.5	0.008	89.6	0.049	71.2	0.013
Terminal unextracted [d]	14.1	0.008	43.6	0.006	8.1	0.004	28.8	0.005

[a] Solvent extractable + solubilised fractions + unextracted

[b] Sum of residues released in enzyme, acid and alkaline digests; no digest contained > 0.01 mg/kg

[c] Values include losses or gains incurred during processing of samples.

[d] Residues remaining in the PES after exhaustive extraction

Table 39 Nature of residue in red beet roots from plants sown in soil 30 days after treatment with 0.45 kg ai/ha [^{14}C]-cyantraniliprole

	[CN-^{14}C]-cyantraniliprole		[PC-^{14}C]-cyantraniliprole	
	% TRR	mg/kg	% TRR	mg/kg
TRR (mg/kg [a]	0.021		0.031	
Solvent extractable [b]	83.3	0.017	87.2	0.027
Cyantraniliprole	21.0	0.005	23.8	0.007
IN-DBC80			4.1	< 0.001
IN-J9Z38	3.2	< 0.001	5.5	0.002
IN-K5A77	1.2	< 0.001	2.2	< 0.001
IN-MYX98	ND	ND	2.4	0.001
IN-N7B69	6.3	< 0.001	2.9	< 0.001
Total unidentified metabolites	10.3	< 0.001	8.6	0.001
Unextracted	16.4	0.003	15.4	0.005

[a] Solvent extractable + unextracted

[b] Values include losses or gains incurred during processing of samples.

Table 40 Nature of residue in lettuce foliage from plants sown in soil treated with 0.45 kg ai/ha [^{14}C]-cyantraniliprole

	[CN-^{14}C]-cyantraniliprole				[PC-^{14}C]-cyantraniliprole			
Planting interval:	30 DAT		120 DAT		30 DAT		120 DAT	
	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
TRR (mg/kg) [a]	0.114		0.036		0.078		0.023	
Solvent extractable [b]	89.4	0.103	97.9	0.035	90.2	0.070	102.4	0.024
Cyantraniliprole	68.1	0.079	69.1	0.025	60.0	0.047	39.6	0.009
IN-J9Z38	4.5	0.005	1.4	0.001	1.2	0.001	ND	ND
IN-JCZ38	3.7	0.005	1.6	0.001	ND	ND	ND	ND
IN-MLA84	4.3	0.005	2.3	0.001	3.5	0.003	2.1	< 0.001
IN-MYX98	0.7	0.001	16.4	0.007	ND	ND	9.1	0.003
Total unidentified metabolites	0.6	0.001	1.5	0.001	6.7	0.002	15.8	0.004
Unextracted	6.5	0.007	4.7	0.002	8.2	0.006	4.1	0.001

[a] Solvent extractable + unextracted

[b] Values include losses or gains incurred during processing of samples.

Table 41 Nature of residue in wheat forage from plants sown in soil treated with 0.45 kg ai/ha [^{14}C]-cyantraniliprole

Planting interval:	30 DAT		120 DAT		365 DAT	
	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
[CN-^{14}C]-cyantraniliprole						
TRR (mg/kg) [a]	0.313		0.129		0.128	
Solvent extractable	84.4	0.263	95.3	0.122	81.8	0.105
Cyantraniliprole	72.7	0.226	70.5	0.090	25.2	0.032
IN-J9Z38	4.0	0.013	11.0	0.015	3.9	0.005
IN-JCZ38	1.8	0.006	1.1	0.001	6.2	0.008
IN-MLA84	3.9	0.012	4.8	0.006	6.8	0.008
Total unidentified metabolites	ND		ND		5.1	0.006
Total solubilized fractions [b]					4.7	0.006
Total extractable [c]	84.4	0.263	95.3	0.122	86.5	0.111
Terminal unextracted [d]	7.6	0.024	9.8	0.013	8.5	0.011
[PC-^{14}C]-cyantraniliprole						
TRR (mg/kg) [a]	0.287		0.103		0.089	
Solvent extractable	93.0	0.267	88.7	0.092	86.7	0.077
Cyantraniliprole	71.0	0.205	34.6	0.036	6.4	0.006
IN-J9Z38	3.8	0.011	11.5	0.011	ND	
IN-JCZ38	1.6	0.004	0.5	0.001	ND	

Planting interval:	30 DAT		120 DAT		365 DAT	
	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
IN-JSE76	0.2	0.001	6.2	0.007	1.2	0.001
IN-HGW87	0.5	0.002	ND		3.4	0.003
IN-K7H19	1.4	0.005	ND		1.2	0.001
IN-K5A79	ND		ND		2.1	0.002
IN-MYX98	ND		ND		1.5	0.001
IN-MLA84	5.0	0.015	1.5	0.002	ND	
IN-N7B69	ND		3.3	0.004	4.4	0.004
Total unidentified metabolites	2.9	0.008	12.3	0.012	27.0	0.023
Total solubilized fractions [b]			4.2	0.004	4.7	0.004
Total extractable [c]	93.0	0.267	92.9	0.096	91.4	0.081
Terminal unextracted [d]	9.0	0.026	7.1	0.007	8.7	0.008

[a] Solvent extractable + solubilised fractions + unextracted

[b] Total solubilized fractions= residues released by enzymes

[c] Values include losses or gains incurred during processing of samples.

[d] Residues remaining in the PES after enzyme extraction

Table 42 Nature of residue in wheat hay from plants sown in soil treated with 0.45 kg ai/ha [^{14}C]-cyantraniliprole

Planting interval:	[CN-^{14}C]-cyantraniliprole						[PC-^{14}C]-cyantraniliprole					
	30 DAT		120 DAT		365 DAT		30 DAT		120 DAT		365 DAT	
	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg
TRR (mg/kg) [a]	1.463		0.312		0.561		1.625		0.449		0.555	
Solvent extracted	60.0	0.88	77.4	0.241	79.4	0.447	83.8	1.361	81.5	0.366	77.2	0.429
Cyantraniliprole	53.4	0.78	50.0	0.155	40.8	0.23	52.2	0.848	35.8	0.161	29.5	0.163
IN-DBC80							1.5	0.025	ND		1.5	0.008
IN-HGW87	ND		ND		0.6	0.004	0.3	0.005	ND		ND	
IN-J9Z38	12.3	0.18	7.2	0.022	12.9	0.073	10.9	0.178	6.6	0.03	7.1	0.039
IN-JCZ38	2.3	0.03	ND		3.7	0.021	2.7	0.044	5.1	0.022	4.0	0.022
IN-JSE76	ND		1.7	0.005	1.0	0.006	0.6	0.01	ND		2.6	0.015
IN-K5A77	ND		ND		0.8	0.004	ND		ND		0.2	0.001
IN-K5A78	0.1	0.001	ND		0.2	0.001	ND		ND		0.3	0.001
IN-K5A79	ND		ND		0.7	0.004	ND		0.8	0.004	0.2	0.001
IN-K7H19	ND		ND		1.3	0.007	0.9	0.014	ND		10.9	0.060
IN-MLA84	6.1	0.09	3.8	0.012	5.5	0.031	6.6	0.108	2.7	0.012	2.9	0.016
IN-MYX98	0.8	0.01	1.2	0.004	3.3	0.018	ND		5.1	0.024	ND	
IN-N7B69	1.3	0.02	ND		1.6	0.01	0.3	0.005	ND		0.7	0.004
IN-NBC94	ND		ND		0.3	0.002	ND		ND		ND	
Total unidentified	1.4	0.02	4.9	0.015	3.3	0.017	3.5	0.057	6.2	0.027	9.7	0.054
Total solubilized [b]	35.4	0.52	11.5	0.036	12.6	0.08	9.8	0.152	10.9	0.049	7.0	0.038
Total extractable [(c)]	95.4	1.4	88.9	0.277	92.0	0.517	93.6	1.521	92.4	0.415	84.2	0.467
Unextracted [(d)]	2.1	0.03	7.6	0.024	7.0	0.039	4.3	0.07	5.6	0.025	7.9	0.044

[a] Solvent extractable + solubilised fractions + unextracted

[b] Sum of residues released by enzyme, acid and/or alkali digestion

[c] Values include losses or gains incurred during processing of samples.

[d] Residues remaining in the PES after exhaustive extraction

Table 43 Nature of residue in wheat straw from plants sown in soil treated with 0.45 kg ai/ha [^{14}C]-cyantraniliprole

Planting interval	[CN-^{14}C]-cyantraniliprole						[PC-^{14}C]-cyantraniliprole					
	30 DAT		120 DAT		365 DAT		30 DAT		120 DAT		365 DAT	
	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg
TRR (mg/kg) [a]	0.973		0.347		0.429		0.966		0.272		0.482	
Solvent extracted	81.9	0.797	80.2	0.278	66.9	0.288	79.8	0.771	72.2	0.196	93.2	0.448
Cyantraniliprole	44.8	0.437	38.4	0.133	33.0	0.141	41.3	0.399	22.9	0.062	35.1	0.153
IN-DBC80							ND		ND		1.8	0.008
IN-HGW87	ND		ND		ND	0.6	0.006	8.1	0.021	ND		
IN-J9Z38	10.9	0.108	4.5	0.016	8.9	0.038	9.5	0.091	2.2	0.006	8.1	0.035
IN-JCZ38	4.3	0.040	4.7	0.017	1.8	0.007	5.5	0.053	0.6	0.001	5.5	0.024
IN-JSE76	1.5	0.013	ND	ND	4.8	0.021	0.8	0.008	0.2	< 0.001	3.9	0.017
IN-K5A77	0.3	0.002	0.6	0.002	1.2	0.005	0.3	0.003	ND		1.3	0.007
IN-K5A78	0.2	0.002	ND		ND		ND		ND		0.4	0.002
IN-K5A79	1.6	0.015	6.4	0.024	1.5	0.007	ND		1.1	0.003	ND	
IN-K7H19	0.3	0.003	ND	ND	0.8	0.003	ND		0.1	< 0.001	10.1	0.043
IN-MLA84	5.8	0.056	3.6	0.012	5.1	0.022	5.5	0.053	0.6	0.002	4.5	0.019
IN-MYX98	1.2	0.012	5.8	0.02	ND		1.5	0.014	0.1	< 0.001	1.6	0.007
IN-N7B69	ND	ND	0.5	0.002	4.2	0.018	ND		2.2	0.006	ND	
IN-NBC94	ND		ND		ND		0.5	0.004	ND		0.1	0.001
Total unidentified	1.5	0.013	4.4	0.018	1.7	0.007	5.9	0.054	12.4	0.029	12.7	0.057
Total solubilised [b]	10.7	0.103	10.7	0.037	13.3	0.057	9.9	0.095	4.2	0.011	6.4	0.031
Total extractable [c]	92.6	0.9	90.9	0.315	80.2	0.345	89.7	0.866	75.6	0.205	99.6	0.436
Unextracted [d]	6.9	0.067	8.2	0.028	8.0	0.034	5.8	0.056	8.1	0.022	9.7	0.047

[a] Solvent extractable + solubilised fractions + unextracted

[b] Sum of residues released by enzyme, acid and/or alkali digestion

[c] Values include losses or gains incurred during processing of samples.

[d] Terminal unextracted=Residues remaining in the PES after exhaustive extraction

Table 44 Nature of residue in red beet foliage from plants sown in soil treated with 0.45 kg ai/ha [^{14}C]-cyantraniliprole

Planting interval:	[CN-^{14}C]-cyantraniliprole				[PC-^{14}C]- cyantraniliprole			
	30 DAT		120 DAT		30 DAT		120 DAT	
	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
TRR (mg/kg) [a]	0.063		0.013		0.145		0.045	
Solvent extractable [b]	94.1	0.059	95.3	0.012	94.0	0.136	85.5	0.038
Cyantraniliprole	3.0	0.003	2.1	< 0.001	4.1	0.005	0.8	< 0.001
IN-DBC80	ND		ND		1.7	0.002	ND	
IN-HGW87	ND		ND		0.2	< 0.001	ND	
IN-J9Z38	ND		ND		ND		0.7	< 0.001
IN-JCZ38	1.5	0.001	2.0	< 0.001	1.8	0.003	ND	
IN-JSE76	ND		ND		1.0	0.001	3.8	0.002
IN-K5A77	ND		ND		0.2	< 0.001	ND	
IN-K5A78	ND		ND		0.6	< 0.001	ND	
IN-K5A79	1.1	0.001	ND		5.0	0.007	ND	
IN-K7H19	0.7	< 0.001	ND		3.2	0.006	ND	
IN-MLA84	ND		ND		1.7	0.002	ND	
IN-MYX98	ND		ND		0.5	< 0.001	ND	
IN-N7B69	0.7	< 0.001	ND		1.4	0.002	ND	
IN-NBC94	ND		ND		ND		0.7	< 0.001
Total unidentified metabolites	79.9 [(c)]	0.048	52.3	0.006	66.2 [(d)]	0.095	37.2	0.012
Unextracted	7.7	0.005	9.1	0.001	6.8	0.010	5.6	0.003

[a] Solvent extractable + unextracted

[b] Values include losses or gains incurred during processing of samples for HPLC.

[c] Comprised of 12 components, none > 0.01 mg/kg

[d] Comprised of 12 components, individually ≤ 0.03 mg/kg

The metabolic fate of cyantraniliprole in the confined rotational crop study was complex with the formation of over 20 metabolites. A proposed pathway for the metabolism of cyantraniliprole in confined rotational crops is presented below:

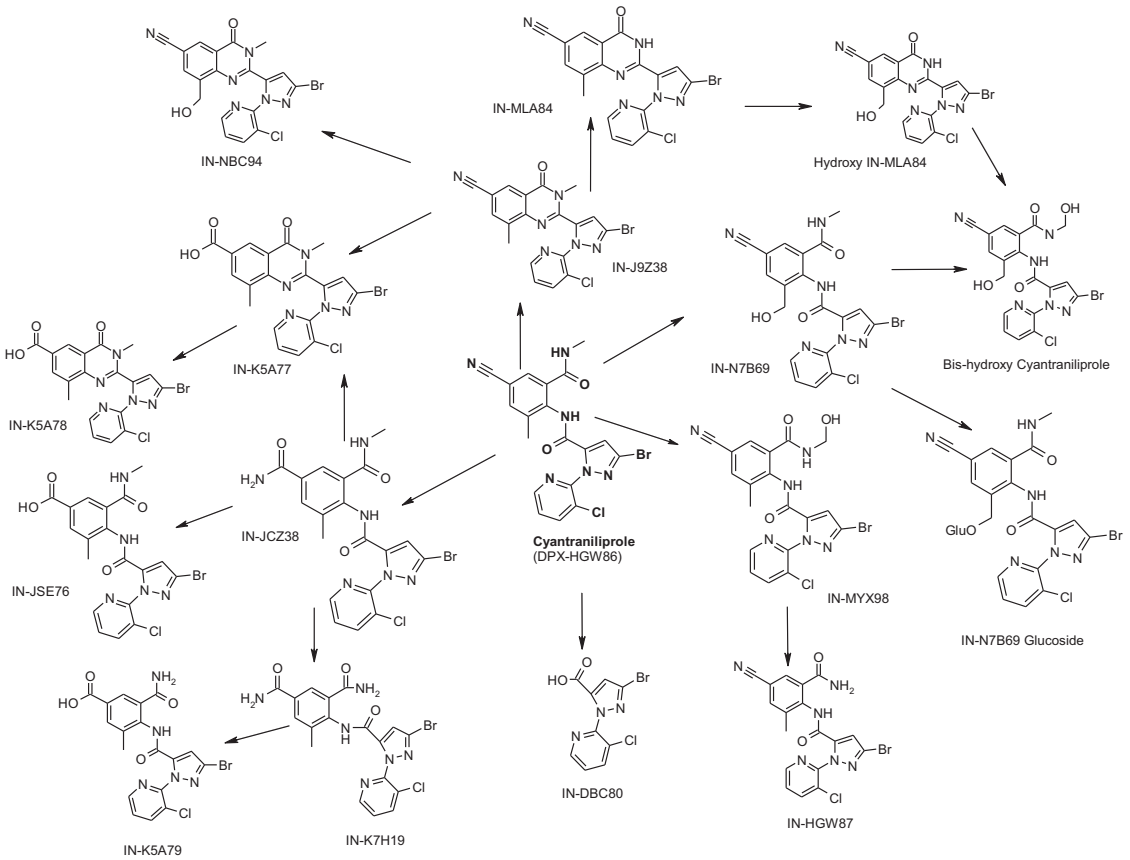


Figure 10 Proposed metabolic pathway for cyantraniliprole in confined rotational crops

Rotational crop field studies

In one European (Scotland) study reported by Haigh & Woodmansey, 2010 [Ref: DP-21447], bare soil plots were treated with two applications of 0.1 kg ai/ha cyantraniliprole (OD formulation) about 7 days apart to achieve a total application rate of 0.2 kg ai/ha and spinach, spring barley and radish were planted as rotational crops, 14, 30, 120, and 270 days after the last application.

In a further European study (Spain), reported by Old & Woodmansey, 2010 [Ref: DP-19678], bare soil plots were treated with three applications of 0.15 kg ai/ha cyantraniliprole (OD formulation) about 7 days apart to achieve a total application rate of 0.45 kg ai/ha and lettuce, spring oats, soya bean and radish were planted as rotational crops, 14, 30, 120, and 365 days after the last application.

All crop samples were harvested at normal maturity, with immature samples also being taken for some leafy crops. Samples were stored for up to 39 months before extraction and analysis (within 72 days after extraction) for cyantraniliprole and six metabolites using analytical method DP15736, with reported LOQs of 0.01 mg/kg. Mean recovery rates ranged from 76–102% (cyantraniliprole) and 71–109% (metabolites) in samples spiked with 0.01 to 0.5 mg/kg.

With the exception of soya bean (seeds and forage) and radish tops, residues of cyantraniliprole and metabolites were not found in succeeding crops following bare ground applications of 0.2–0.45 kg ai/ha.

Table 45 Cyantraniliprole residues in rotational crops (30-day plantback interval) following bare ground applications of cyantraniliprole in Europe

Commodity		Total application rate (kg ai/ha)	Plant back interval (days)	cyantraniliprole	IN-N7B69
Root/tubers	Radish roots	0.2–0.45	14	ND	ND
			30	ND	ND
			120	ND	ND
			275-365	ND	ND
Root/tubers	Radish tops	0.2–0.45	14	0.005	ND
			30	0.007	ND
			120	0.006	ND
			275-365	ND	ND
Legume	Soya bean seeds	0.45	14	ND	0.006
			30	ND	0.008
			120	ND	ND
			365	ND	ND
Legume	Soya bean forage	0.45	14	0.027	ND
			30	0.024	ND
			120	0.018	ND
			365	ND	ND
Cereal	Oat, barley ears	0.45	14	ND	ND
			30	ND	ND
			120	ND	ND
			365	ND	ND
Cereal	Oat, barley grain	0.2–0.45	14	ND	ND
			30	ND	ND
			120	ND	ND
			275-365	ND	ND
Cereal	Oat, barley forage	0.2–0.45	14	ND	ND
			30	ND	ND
			120	ND	ND
			275-365	ND	ND
Cereal	Oat, barley hay	0.2	14	ND	ND
			30	ND	ND
			120	ND	ND
			275	ND	ND
Cereal	Oat, barley straw	0.2–0.45	14	ND	ND
			30	ND	ND
			120	ND	ND
			275-365	ND	ND
Leafy vegetables	Immature leaves (spinach, lettuce)	0.2–0.45	14	ND	ND
			30	ND	ND
			120	ND	ND
			275-365	ND	ND
Leafy vegetables	Mature leaves (spinach, lettuce)	0.2–0.45	14	ND	ND
			30	ND	ND
			120	ND	ND
			275-365	ND	ND

Note: Residues of other metabolites (IN-J9Z38, IN-JCZ38, IN-K7H19, IN-MLA84 and IN-MYX98) not detected in any samples

Five field rotational crop studies were conducted in North America and two studies were conducted in Europe, all involving bare soil treatments with cyantraniliprole prior to planting a wide range of representative rotational crops.

In three North American studies, bare soil plots were treated with 2–3 applications of cyantraniliprole (OD formulation) about 5 days apart to achieve a total application rate of 0.45 kg ai/ha and four rotational crops (lettuce/spinach, oat, radishes and soya bean) were planted 14, 30, 120, and 365 days after the last application. These studies were reported by Thiel, 2009 and 2010 [Ref: DP-19640][Ref: DP-19639][Ref: DP-21446].

Table 46 Cyantraniliprole residues in rotational crops following bare ground applications of 0.45 kg ai/ha cyantraniliprole in North America. [Ref DP-19640, DP-19639, DP-21446]

Commodity	PBI (days)	Residues (mg/kg)			
		Cyantraniliprole	IN-J9Z38	IN-JCZ38	IN-MLA84
Study DP-19640 (Loamy sand, 1.0% OM, pH 7.4, CEC 12.4)					
Lettuce, mature leaves	14	0.033	ND	0.007	0.004
	30	0.011	ND	0.008	ND
	120	ND	ND	ND	ND
	365	ND	ND	ND	ND
Lettuce, immature leaves	14	0.039	ND	0.006	0.005
	30	0.029	ND	0.006	ND
	120	ND	ND	ND	ND
	365	ND	ND	ND	ND
Oat forage	14	0.036	0.007	0.014	ND
	30	0.032	0.008	0.019	ND
	120	0.048	0.003	0.015	ND
	365	ND	ND	ND	ND
Oat grain	14	0.007	ND	ND	ND
	30	0.005	ND	ND	ND
	120	ND	ND	ND	ND
	365	ND	ND	ND	ND
Oat hay	14	0.13	0.035	0.04	ND
	30	0.063	0.019	0.056	ND
	120	0.021	0.008	0.029	ND
	365	ND	ND	ND	ND
Oat straw	14	0.045	0.029	0.018	ND
	30	0.022	0.01	0.019	ND
	120	0.015	0.015	0.029	ND
	365	ND	ND	ND	ND
Radish root	14	0.003	ND	ND	ND
	30	ND	ND	ND	ND
	120	ND	ND	ND	ND
	365	ND	ND	ND	ND
Radish top	14	0.046	0.006	0.005	ND
	30	0.041	0.005	0.006	ND
	120	ND	ND	ND	ND
	365	ND	ND	ND	ND
Soya bean forage	14	0.18	0.008	0.022	0.023
	30	0.14	0.009	0.024	0.02
	120	0.048	ND	0.025	0.005
	365	ND	ND	ND	ND
Soya bean hay	14	0.023	ND	0.023	0.012
	30	0.012	ND	0.037	0.007
	120	0.018	ND	0.013	0.008
	365	ND	ND	0.007	ND
Soya bean seed	14	ND	ND	ND	ND
	30	ND	ND	ND	ND
	120	ND	ND	ND	ND
	365	ND	ND	ND	ND
DP-19639 (Loamy sand, 0.6% OM, pH 7.5)					
Spinach, mature leaves	14	ND	ND	ND	ND
	30	ND	ND	ND	ND
	119	ND	ND	ND	ND
	367	ND	ND	ND	ND
Spinach, immature leaves	14	ND	0.003	ND	ND

Commodity	PBI (days)	Residues (mg/kg)			
		Cyantraniliprole	IN-J9Z38	IN-JCZ38	IN-MLA84
	30	0.003	0.005	ND	ND
	119	ND	0.004	ND	ND
	367	ND	ND	ND	ND
Oat grain	14	0.008	ND	ND	ND
	30	0.01	ND	ND	ND
	119	ND	ND	ND	ND
	365	ND	ND	ND	ND
Oat hay	14	0.066	0.014	0.039	ND
	30	0.096	0.021	0.048	ND
	119	0.006	0.008	0.004	ND
	365	ND	ND	ND	ND
Oat spring forage	14	0.018	ND	0.005	ND
	30	0.015	ND	0.007	ND
	119	ND	ND	ND	ND
	365	ND	ND	ND	ND
Oat straw	14	0.052	0.02	0.023	ND
	30	0.073	0.033	0.029	ND
	119	0.008	0.017	0.007	ND
	365	ND	ND	ND	ND
Oat winter forage	14	0.014	ND	0.006	ND
	30	0.022	0.003	0.01	ND
	119	ND	ND	ND	ND
	365	ND	ND	ND	ND
Radish root	14	ND	0.003	ND	ND
	31	ND	0.003	ND	ND
	119	ND	0.004	ND	ND
	367	ND	ND	ND	ND
Radish top	14	0.019	0.008	0.007	ND
	31	0.008	0.008	0.005	ND
	119	ND	0.005	ND	ND
	367	ND	ND	ND	ND
Soya bean forage	14	0.043	0.005	0.014	0.015
	31	0.024	ND	0.015	0.01
	119	ND	ND	ND	ND
	365	ND	ND	ND	ND
Soya bean hay	14	0.1	0.023	0.054	0.062
	31	0.058	0.01	0.049	0.028
	119	ND	ND	ND	ND
	365	ND	ND	ND	ND
Soya bean seed	14	ND	ND	ND	ND
	31	ND	ND	ND	ND
	119	ND	ND	ND	ND
	365	ND	ND	ND	ND
DP-21446 (Loam, 1.8% OM, pH 6.9, CEC 12.8)					
Lettuce, immature leaves	16	0.026	0.006	0.005	ND
	29	0.018	0.003	0.003	ND
	366	ND	ND	ND	ND
Lettuce, mature leaves	16	0.011	0.003	ND	ND
	29	0.018	0.007	0.004	ND
	366	ND	ND	ND	ND
Radish top	16	0.039	0.005	0.007	ND
	29	0.011	0.007	0.005	ND
	121	0.01	0.007	0.003	ND
	366	ND	ND	ND	ND
Radish root	16	0.005	ND	ND	ND
	29	0.015	0.01	0.003	ND
	121	0.009	0.01	0.003	ND
	366	ND	ND	ND	ND
Oat forage	14	0.011	ND	0.004	ND
	31	0.006	ND	ND	ND

Commodity	PBI (days)	Residues (mg/kg)			
		Cyantraniliprole	IN-J9Z38	IN-JCZ38	IN-MLA84
	188	0.004	ND	ND	ND
	365	ND	ND	ND	ND
Oat hay	14	0.034	0.011	0.012	ND
	31	0.02	0.006	0.01	ND
	188	0.012	0.009	0.006	ND
	365	0.003	0.003	ND	ND
Oat grain	14	0.021	0.016	0.009	ND
	31	0.011	0.014	0.005	ND
	188	0.007	0.01	0.004	ND
			0.004	ND	ND
Oat straw	14	0.003	ND	ND	ND
	31	ND	ND	ND	ND
	188	ND	ND	ND	ND
	365	ND	ND	ND	ND
Soya bean forage	15	0.023	ND	0.003	ND
	29	0.007	ND	ND	ND
	195	ND	ND	ND	ND
	366	ND	ND	ND	ND
Soya bean hay	15	0.061	0.004	0.01	0.01
	29	0.017	ND	0.005	ND
	195	0.011	ND	0.003	ND
	366	0.004	ND	ND	ND
Soya bean seed	15	ND	ND	ND	ND
	29	ND	ND	ND	ND
	195	ND	ND	ND	ND
	366	ND	ND	ND	ND

Note: Residues of other metabolites (IN-K7H19, IN-MYX98 and IN-N7B69) not detected in any samples

In the fourth North American study reported by Thiel, 2010 [Ref: DP-27972], bare soil plots were treated with three applications of cyantraniliprole (SE formulation), 5 days apart to achieve a total application rate of 0.45 kg ai/ha and seventeen rotational crops (turnip, sugar beet, garden beet, carrot, bean, pea, soya bean, field corn, sweet corn, sorghum, rice, wheat, Bermuda grass, alfalfa, brome-grass, clover, and bluegrass) were planted 30 days after the last application.

Table 47 Cyantraniliprole residues in rotational crops (30-day plantback interval) following bare ground applications of 0.45 kg ai/ha cyantraniliprole in North America. [Ref DP-27972]

Commodity	Residues (mg/kg)				
	Cyantraniliprole	IN-J9Z38	IN-JCZ38	IN-MLA84	IN-N7B96
Alfalfa forage	0.054	0.006	0.006	0.02	ND
Alfalfa hay	0.14	0.02	0.011	0.051	ND
Bean dry seed	ND	ND	ND	ND	ND
Bean forage	0.029	ND	ND	0.003	ND
Bean hay	0.14	0.014	0.008	0.017	0.007
Bean seed	0.003	ND	ND	ND	ND
Bean succulent seed	ND	ND	ND	ND	ND
Bermuda grass forage	0.011	0.005	0.006	ND	ND
Bermuda grass hay	0.025	0.008	0.01	ND	ND
Bluegrass forage	0.092	0.008	ND	0.004	ND
Bluegrass hay	0.23	0.028	0.007	0.006	ND
Brome grass forage	0.011	ND	0.006	ND	ND
Brome grass hay	0.024	0.005	0.008	ND	ND
Carrot root	0.011	ND	ND	ND	ND
Clover forage	0.009	0.011	0.003	0.005	ND
Clover hay	0.034	0.028	0.019	0.012	0.01
Field corn forage	0.006	0.003	ND	ND	ND
Field corn stover	0.012	0.012	ND	ND	ND
Garden beet root	0.003	ND	ND	ND	ND
Garden beet top	0.008	ND	ND	ND	ND

Commodity	Residues (mg/kg)				
	Cyantraniliprole	IN-J9Z38	IN-JCZ38	IN-MLA84	IN-N7B96
Pea dry seed	ND	ND	ND	ND	ND
Pea forage	0.035	0.003	0.004	ND	ND
Pea hay	0.13	0.01	0.016	ND	ND
Pea seed	ND	ND	ND	ND	ND
Pea succulent seed	ND	ND	ND	ND	ND
Rice straw	0.005	0.009	0.003	ND	ND
Sorghum forage	0.01	0.005	0.004	0.004	ND
Sorghum stover	0.008	0.009	ND	0.006	ND
Soya bean forage	0.091	ND	0.009	0.014	ND
Soya bean hay	0.63	0.012	0.029	0.069	ND
Soya bean seed	ND	ND	ND	ND	ND
Sugar beet root	ND	ND	ND	ND	ND
Sugar beet top	ND	ND	ND	ND	ND
Sweet corn forage	0.009	ND	ND	ND	ND
Sweet corn stover	0.01	0.013	ND	ND	ND
Turnip root	ND	0.005	ND	ND	ND
Turnip top	ND	ND	ND	ND	ND
Wheat forage	0.11	0.007	0.006	0.004	ND
Wheat hay	0.21	0.017	0.014	0.012	ND
Wheat straw	0.081	0.012	0.015	0.012	ND

Note: Residues of other metabolites (IN-K7H19, IN-MYX98, and IN-N7B69) not detected in any samples

In the fifth North American study, reported by Thiel, 2011 [Ref: DP-29456], bare soil plots were treated with three applications of cyantraniliprole (SE formulation), 5 days apart to achieve a total application rate of 0.45 kg ai/ha and ten rotational crops (turnip, sugar beet, radish, carrot, pea, soya bean, alfalfa, clover, peanut, and strawberries) were planted 30 days after the last application.

Table 48 Cyantraniliprole residues in rotational crops (30-day plantback interval) following bare ground applications of 0.45 kg ai/ha cyantraniliprole in North America. [Ref DP-29456]

Commodity	Residues (mg/kg)					
	Cyantraniliprole	IN-J9Z38	IN-JCZ38	IN-K7H19	IN-MLA84	IN-N7B96
Alfalfa forage	0.008	ND	ND	ND	ND	ND
Alfalfa hay	0.021	0.004	0.008	0.006	ND	ND
Carrot root	0.005	0.01	ND	ND	ND	ND
Clover forage	0.023	ND	0.013	ND	ND	0.007
Clover hay	0.046	0.013	0.034	0.011	0.008	ND
Pea forage	ND	0.005	ND	ND	ND	ND
Pea hay	0.008	0.017	ND	ND	ND	ND
Pea seed	ND	ND	ND	ND	ND	ND
Peanut hay	0.009	0.015	0.004	0.005	ND	ND
Peanut nutmeat	ND	ND	ND	ND	ND	ND
Radish root	0.004	ND	ND	ND	ND	ND
Soya bean forage	0.039	ND	0.005	0.005	ND	ND
Soya bean hay	0.19	0.021	0.026	0.025	ND	ND
Soya bean pod	ND	ND	ND	ND	ND	ND
Soya bean seed	ND	ND	ND	ND	ND	ND
Strawberry fruit	0.005	ND	ND	ND	ND	ND
Sugar beet root	ND	ND	ND	ND	ND	ND
Sugar beet top	ND	ND	ND	ND	ND	ND
Turnip top	0.02	0.008	0.004	ND	ND	ND

Residues of IN-MYX98 were not detected in any samples

In the North American studies, residues of cyantraniliprole and metabolites in the first rotation crops (30 day plant-back interval were below 0.05 mg/kg in commodities for human consumption (cereal grains, root crops, legumes and pulses, leafy vegetables). Higher residues were

reported in animal feed commodities, up to 0.2 mg/kg in forage crops and 0.3 mg/kg in most hays and straws. Highest residues were found in soya bean hay, up to 0.63 mg/kg.

Table 49 Summary of highest residues of cyantraniliprole and metabolites in rotational crops (30-day plantback interval) following bare ground applications of 0.45 kg ai/ha cyantraniliprole (North America)

Commodity group	Sample	Cyantraniliprole	Total (parent plus metabolites)
Root/tubers (roots)	carrot roots	0.01	0.02
	beet roots	< 0.01	< 0.01
	radish roots	0.02	0.03
	sugar beet roots	ND	ND
	turnip roots	ND	ND
Root/tubers (tops)	beet tops	< 0.01	< 0.01
	radish tops	0.04	0.05
	sugar beet tops	ND	ND
	turnip tops	0.02	0.03
Legume seeds	bean seed (dry)	ND	ND
	pea seed (dry)	ND	ND
	bean seed (succulent)	< 0.01	< 0.01
	pea seed (succulent)	ND	ND
	soya bean seed	ND	ND
Legume forage	alfalfa forage	0.05	0.09
	bean forage	0.03	0.03
	clover forage	0.02	0.04
	pea forage	0.04	0.04
	soya bean forage	0.14	0.19
Legume hay	alfalfa hay	0.14	0.22
	bean hay	0.14	0.19
	clover hay	0.05	0.11
	pea hay	0.13	0.16
	peanut hay	< 0.01	0.03
	soya bean hay	0.63	0.74
Leafy vegetables	Lettuce (immature leaves)	0.03	0.04
	Spinach (immature leaves)	< 0.01	< 0.01
	Lettuce (mature leaves)	0.02	0.03
	spinach (mature leaves)	ND	ND
Cereal grains	oat grain	0.01	0.03
Cereal forage	corn forage	< 0.01	< 0.01
	oat forage	0.03	0.06
	sorghum forage	0.01	0.02
	wheat forage	0.11	0.13
Cereal hay	oat hay	0.1	0.17
	wheat hay	0.21	0.25
Cereal stover	corn stover	0.01	0.02
	sorghum stover	< 0.01	0.02
Cereal straw	oat straw	0.07	0.14
	rice straw	< 0.01	0.02
	wheat straw	0.08	0.12
Grass forage	Bermuda grass forage	0.01	0.02
	Bluegrass forage	0.09	0.1
	Brome grass forage	0.01	0.02
Grass hay	Bermuda grass hay	0.03	0.04
	Bluegrass hay	0.23	0.27
	Brome grass hay	0.02	0.04
Peanut	nutmeat	ND	ND
Strawberry	fruit	< 0.01	< 0.01

Total residues are the sum of cyantraniliprole and metabolites IN-J9Z38, IN-JCZ38, IN-MLA84 and IN-N7B96.

METHODS OF RESIDUE ANALYSIS

Analytical methods

The meeting received analytical method descriptions and validation data for cyantraniliprole and major metabolites in crop and animal commodities and in soil and water. A summary of the analytical methods for plant and animal commodities is provided below.

Table 50 Summary of cyantraniliprole analytical methods developed for plant and animal matrices

Matrix	Analyte	Method	Principle	LOQ	Reference
tomato wheat grain orange almond	cyantraniliprole	21328	DFG S19 extraction (aqueous acetone) LC-MS/MS analysis	0.01 mg/kg	DP-21328
cucumber tomato lime almond wheat straw grape apple peach almond (nutmeat) lettuce wheat grain potato lemon oilseed rape onion tomato (paste) tomato (dried)	cyantraniliprole IN-J9Z38 IN-JCZ38 IN-K5A79 IN-K7H19 IN-MLA84 IN-MYX98 IN-N7B69 IN-F6L99	15736 [1187]	Aqueous acetonitrile extraction LC-MS/MS analysis	0.05 mg/kg (wheat straw) 0.01 mg/kg (other matrices)	DP-15736 DP-18846 DP-2600 DP-15736-S1
meat liver milk eggs	cyantraniliprole	25544	DFG S19 extraction (aqueous acetone) SPE clean-up LC-MS/MS	0.01 mg/kg	DP-25544 DP17942
milk	cyantraniliprole IN-HGW87 IN-J9Z38 IN-JCZ38 IN-K5A79 IN-K7H19 IN-MLA84 IN-MYX98 IN-N7B69	25544	DFG S19 extraction (aqueous acetone) SPE clean-up LC-MS/MS	0.01 mg/kg	DP-25544
muscle fat liver kidney milk skim milk heavy cream eggs	cyantraniliprole IN-J9Z38 IN-JCZ38 IN-K5A79 IN-K7H19 IN-MLA84 IN-MYX98 IN-N7B69	18844 [1552]	aqueous acetonitrile extraction SPE clean-up LC-MS/MS analysis		DP-18844 DP-25543 DP-26458

Data collection methods

Method 21328 (plant matrices)

The analytical method 21328, based on the German method DFG S19, was described and reported by Bacher, 2009 [Ref: DP-21328] as suitable for as a data-collection method to measure residues of cyantraniliprole in plant commodities, with an LOQ of 0.01 mg/kg.

Cyantraniliprole was extracted from the plant tissue according to extraction module E 1 (tomato), E 2 (wheat grain), E 3 (orange), and E 7 for (almond) of the DFG Method S 19 method. Extracts of tomato, orange, wheat grain, and almond were analyzed by liquid chromatography with electrospray ionization and tandem mass spectrometry detection. The report noted that degradation of cyantraniliprole was observed under GC/MS conditions.

The E1 extraction module involved the addition of water and acetone prior to homogenization for 2 minutes and then the addition of 1:1 ethyl acetate:cyclohexane and NaCl. The sample was homogenized a second time for 1 minute, allowed to separate and the organic phase was removed and filtered through a cellulose folded filter, which was coated with Na_2SO_4. The extract was evaporated until only aqueous remained and a mixture of ethyl acetate and 1:1 Na_2SO_4:NaCl was added. An additional aliquot of cyclohexane was added to adjust the volume of the organic layer.

The E2 extraction module involved the addition of water and a 30-minute soaking period before the addition of acetone and homogenization. After the addition of 1:1 ethyl acetate:cyclohexane and 35 g NaCl, the sample was homogenized a second time for 1 minute and the phases were allowed to separate. The organic phase was removed and filtered through a cellulose folded filter coated with Na_2SO_4. The extract was evaporated until only aqueous remained and a mixture of ethyl acetate and 1:1 Na_2SO_4:NaCl was added. An additional aliquot of cyclohexane was added to adjust the volume of the organic layer.

The E3 extraction module involved mixing with $NaHCO_3$, the addition of water and after allowing the sample to stand for about 30 minutes, the addition of acetone prior to homogenization. This was followed by an addition of 1:1 ethyl acetate:cyclohexane and 35 g NaCl and a second homogenization. After the phases were allowed to separate, the organic phase was removed and filtered through a cellulose folded filter coated with Na_2SO_4. The extract was evaporated until only aqueous remained and a mixture of ethyl acetate and 1:1 Na_2SO_4:NaCl was added. An additional aliquot of cyclohexane was added to adjust the volume of the organic layer.

The E7 extraction module involved the addition of acetone, acetonitrile, Calflo E and Celite before homogenization and vacuum filtration through a Buchner porcelain funnel. The filtrate was then passed through a dry fluted cellulose filter paper coated with Calflo E and rotary evaporated into an oil residue and bathed at 40 °C. The samples were then mixed with ethyl acetate, sonicated until completely dissolved and after the addition of a 1:1 mixture of Na_2SO_4:NaCl, cyclohexane was added to adjust the volume of the organic layer.2

The extracts were purified using gel permeation chromatography using 1:1 ethyl acetate:cyclohexane as the eluant. The eluate was evaporated to dryness using a rotary evaporator, re-constituted in ethyl acetate, evaporated to dryness in a gentle stream of nitrogen and re-constituted in methanol/water.

The final extracts were analysed by liquid chromatography with tandem mass spectrometric detection (LC-MS/MS) using a C18 column gradient elution using 0.1% formic acid in water to 0.1% formic acid in acetonitrile as the mobile phase. MS/MS detection in the positive ionization mode was used to monitor ion transitions 475→286 and 475→112 for cyantraniliprole.

Average (n=2) recovery rates in tomato, orange, wheat grain and almond (nutmeat) ranged from 92–104% (RSD ≤ 8%) in samples spiked with 0.01 mg/kg cyantraniliprole and from 93–115% (RSD ≤ 7%) in samples spiked with 0.1 mg/kg. The LOQ was 0.01 mg/kg.

Method 21328 was independently validated in a study by Weber, 2010 [Ref: DP-21326] with similar average (n=5) recovery rates (94–107%, ≤ 11% RSD) in tomatoes, oranges, wheat grain and almond nutmeat spiked with 0.01 mg/kg or 0.1 mg/kg cyantraniliprole, demonstrating the reproducibility of this method.

While the DFG S 19 extraction procedures used in Method 21328 include a pre-soak in water followed by an acetone extraction, the cyantraniliprole metabolism study [Ref: DP-16986] demonstrated that incurred residues could be completely extracted using an acetonitrile:water, 9:1 extraction. Based on the physicochemical properties of cyantraniliprole, including the higher

solubility in acetone (6.45 g/L) than in acetonitrile (2.45 g/L), acetone is considered a suitable solvent for the extraction of incurred cyantraniliprole residues.

Method 15736 (plant matrices)

The analytical method 15736, described and reported by McClory, Stevens-Shreve & Hense, 2011, [Ref DP-15736] was developed to measure residues of cyantraniliprole and seven metabolites (IN-J9Z38, IN-JCZ38, IN-K5A79, IN-K7H19, IN-MLA84, IN-MYX98 and IN-N7B69) in representative commodities with a high water content (cucumber and tomato), a high acid content (lime), a high oil content (almond nutmeat) and a low water content (wheat straw). This method is also known as DuPont method 1187.

A supplement to this study, reported by McClory, Stevens-Shreve & Hense, 2011, [Ref DP-15736-S1] also validated this method for tomato paste and sun-dried tomato for the above analytes and for the IN-N5M09 and IN-F6L99 metabolites as potential breakdown products formed during processing.

Samples were extracted twice with 9:1 acetonitrile:water by high-speed shaking (2 minutes at 700 cycles/minute in the presence of ball bearings) and centrifugation. The supernatants from the two extracts were combined, diluted with acetonitrile:water and mixed vigorously. Aliquots of these extracts were filtered (0.2 μm PTFE) and after the addition of methanol and de-ionized water, were analyzed using LC/MS/MS.

The final extracts were analysed by liquid chromatography with tandem mass spectrometric detection (LC-MS/MS) using a C18 column gradient elution using formic acid/ammonuim formate in water to formic acid/ammonium formate in methanol as the mobile phase. MS/MS detection in the positive ionization mode was used to monitor the following ion transitions:

Cyantraniliprole	475→286	and	475→443.9
IN-J9Z38	457→188	and	457→290
IN-JCZ38	493→286	and	493→462
IN-K5A79	480→286	and	480→463
IN-K7H19	479→286	and	479→462
IN-MLA84	443→298	and	443→407.4
IN-MYX98	491→444	and	491→473
IN-N7B69	491→285.7	and	491→473
IN-F6L99	204→173	and	204→66

Average (n=2) recovery rates in samples spiked with 0.01 mg/kg ranged from 74–121% (RSD ≤ 15) and in samples spiked with 0.1 mg/kg were 67–96% (RSD ≤ 13). The method LOQ was 0.01 mg/kg.

This method was validated under GLP by two additional laboratories. One of the additional validations reported by Kinney, 2008 [Ref: DP-18846] investigated the performance of Method 15376 for grapes, apples, peaches, tomatoes, almonds (nutmeat), lettuce, wheat grain, wheat straw, potatoes, lemons, and rape seed and the second validation, reported by Seal, 2010 [Ref: DP-26000] evaluated the performance of the method for almonds, onions, tomato paste, and sun-dried tomatoes.

Average (n=5) recovery rates in all matrices and for all analytes in samples spiked with 0.01 mg/kg, 0.05 mg/kg (wheat straw), 0.1 mg/kg or 0.5 mg/kg (wheat straw) were within the range 70–111% (RSD ≤ 13).

Extraction efficiency of this method was demonstrated in a study reported by Lowrie and MacKinnon, 2009 [Ref: DP-24754] where samples of lettuce leaf, beet foliage, wheat grain, and wheat hay from the confined rotational crop metabolism study [Ref: DP-15778] containing weathered residues of radiolabelled parent and metabolites were extracted using the Method 15736 extraction procedure. The levels of the parent and metabolites obtained using the residue method were within 89–100% of those found in the metabolism study, which indicates that the residue method provides acceptable extraction efficiency.

Method 15736 was shown to be suitable for the purposes of residue data collection and enforcement of MRLs, with an LOQ of 0.05 mg/kg for wheat straw and 0.01 mg/kg for all other matrices.

Method 25544 (animal matrices)

The analytical Method 25544, based on the German multi-residue method DFG S19 (module E1 extraction and module GPC clean-up) with LC-MS/MS detection, was described and reported by Bacher, 2010 [Ref: DP-25544] as suitable for use as a data-collection method to measure residues of cyantraniliprole in animal commodities, with an LOQ of 0.01 mg/kg and was also suitable to measure residues of the metabolites IN-J9Z38, IN-JCZ38, IN-HGW87, IN-K7H19, IN-MLA84, IN-MYX98, and IN-N7B69 in milk (but not other animal commodities.

Samples of meat, milk, egg and liver were mixed with water and after the addition of acetone, were homogenized for 2 minutes before the addition of 1:1 ethyl acetate:cyclohexane and NaCl and further homogenization for 1 minute. The phases were allowed to separate and the organic phase was filtered through cellulose folded filter coated with Na_2SO_4. After repeat rinsing with ethyl acetate:cyclohexane the filtrate was evaporated to an aqueous remainder (not to dryness), re-dissolved in ethyl acetate, mixed with 1:1 Na_2SO_4:NaCl and the volume adjusted with cyclohexane.

Extracts were cleaned up by gel permeation chromatography using a 1:1 mixture of ethyl acetate:cyclohexane as eluant (5.0 mL/min) and an automated gel permeation chromatograph. The collected eluate was evaporated to dryness, reconstituted in ethyl acetate, evaporated to dryness in a gentle stream of nitrogen and re-constituted in 1:1 methanol:water.

The final extracts were analysed by liquid chromatography with electrospray ionisation and tandem mass spectrometric detection (LC-MS/MS) using a C18 column gradient elution using formic acid in water to formic acid in methanol as the mobile phase. MS/MS detection in the positive ionization mode was used to monitor the following ion transitions:

Cyantraniliprole	475→444	and	475→286
IN-J9Z38	457→188	and	457→112
IN-JCZ38	493→462	and	493→286
IN-K7H19	479→462	and	479→286
IN-MLA84	443→298	and	443→407
IN-MYX98	491→444	and	491→442
IN-N7B69	491→286	and	491→473
IN-HGW87	459→284	and	459→112

Average (n=5) recovery rates in samples spiked with 0.01 mg/kg ranged from 74–107% (RSD ≤ 14%) and in samples spiked with 0.1 mg/kg were 70–103% (RSD ≤ 14%). The method LOQ was 0.01 mg/kg.

This method was independently validated for the detection, quantitative analysis, and confirmation of cyantraniliprole in milk, eggs, meat, and liver in a study reported by Weber, 2010

[Ref: DP-17942]. Average (n=5) recovery rates in all matrices spiked with 0.01 mg/kg ranged from 98–106% (RSD < 6%) and were 95–108% (RSD < 7%) in the 0.1 mg/kg spiked samples.

While the DFG S 19 extraction procedures used in Method 25544 include a pre-soak in water followed by an acetone extraction, the cyantraniliprole metabolism study [Ref: DP-16987] demonstrated that incurred residues could be completely extracted using an acetonitrile:water, 9:1 extraction. Based on the physicochemical properties of cyantraniliprole, including the higher solubility in acetone (6.45 g/L) than in acetonitrile (2.45 g/L), acetone is considered a suitable solvent for the extraction of incurred cyantraniliprole residues in animal commodities.

The residue Method 25544, is suitable for the determination of cyantraniliprole in milk, eggs, meat, and liver, with an LOQ of 0.01 mg/kg for animal commodities.

Method 18844 (animal matrices)

The analytical method 18844, also reported as DuPont method 1552, was described and reported by Henze and McClory, 2010 [Ref: DP-18844] as suitable for use as a data-collection method to measure residues of cyantraniliprole and the metabolites IN-J9Z38, IN-JCZ38, IN-HGW87, IN-K5A79, IN-K7H19, IN-MLA84, IN-MYX98, and IN-N7B69 in muscle, fat, kidney, liver, egg, whole milk, skim milk and heavy cream, with an LOQ of 0.01 mg/kg.

In this method, samples were extracted twice with acetonitrile, cleaned up by shaking with hexane and centrifugation, with the hexane fractions then being discarded. The extracts were then evaporated to near-dryness in a gentle stream of nitrogen and after the addition of methanol, made up to volume with water, vortexed, sonicated and filtered.

Residues of cyantraniliprole, IN-HGW87, IN-J9Z38, IN-JCZ38, IN-K5A79, IN-K7H19, IN-N7B69, IN-MLA84, and IN-MYX98 were separated from co-extracts by reversed phase liquid chromatography (C18 column gradient elution using formic acid/ammonium formate in water to formic acid/ammonium formate in methanol as the mobile phase) and detected by mass spectrometry/mass spectrometry (MS/MS) using positive ion atmospheric pressure chemical ionization to monitor the following ion transitions:

Cyantraniliprole	475→443	and	475→286
IN-J9Z38	457→298	and	457→188
IN-JCZ38	493→462		
IN-K7H19	479→462	and	479→286
IN-K5A79	480→463	and	480→286
IN-MLA84	443→298	and	443→407
IN-MYX98	491→444		
IN-N7B69	491→286		
IN-HGW87	459→284	and	459→112

Average (n=5) recovery rates in samples spiked with 0.01 mg/kg ranged from 80–116% (RSD ≤ 20% except 34% RSD for IN-JCZ38 in eggs) and in samples spiked with 0.1 mg/kg were 74–116% (RSD < 13%). The method LOQ was 0.01 mg/kg.

This method was validated in a study reported by Ward, 2010 [Ref: DP-25543] on the detection and measurement of cyantraniliprole and metabolites (except IN-K5A79) in bovine muscle, fat, kidney, liver, milk and in eggs. An independent validation study was also conducted by Schwartz, 2010 [Ref: DP-26458] for cyantraniliprole, IN-J9Z38, IN-MLA84 and IN-N7B69 in whole milk, kidney and muscle.

In these studies, average (n=5) recovery rates in all matrices spiked with 0.01 mg/kg ranged from 72–131% (RSD < 13%) and were 71–112% (RSD < 12%) in the 0.1 mg/kg spiked samples.

Extraction efficiency of this method was demonstrated in a study reported by Kinney and Melville, 2010 [Ref: DP-27356] where samples of muscle, milk, liver, egg yolks, and egg whites from the cattle metabolism study [Ref: DP-16987] containing weathered residues of radiolabelled parent and metabolites were extracted using the Method 18844 procedure. The levels of extracted radioactivity obtained using the residue method were equivalent to those found in the metabolism extraction; 75.5% (liver), 92.7% (muscle), 103.7% (milk), 99.5% (egg white), and 125.1% (egg yolk), indicating that the method 18844 provides acceptable extraction efficiency.

The residue method 18844 for the determination of cyantraniliprole, IN-N7B69, IN-K7H19, IN-K5A79, IN-JCZ38, IN-MYX98, IN-HGW87, IN-J9Z38, and IN-MLA84 in muscle, fat, kidney, liver, heavy cream, skim milk, whole milk, and eggs involves simple extraction, clean-up, and analytical determination by HPLC/MS/MS detection. A limit of quantification of 0.01 mg/kg can be achieved consistently for all tissues.

Method 25544 (soil)

The analytical Method 15440, involving acetone extraction, SPE clean-up and LC-MS/MS analysis was reported by Henze, Devine and McClory, 2006 [Ref: DP-15440] as suitable for use as a data-collection method to measure residues of cyantraniliprole and metabolites IN-J9Z38, IN-JCZ38, IN-JSE76, IN-K5A77, IN-K5A78 IN-K5A79, IN-K7H19, and IN-PLT97 in soil.

Soil samples were extracted twice with 9:1 acetone:formic acid by shaking at high speed on a platform shaker. After dilution with water, extracts were passed through an NH_2 and an ENV SPE column. The NH_2 removed co-extracts with cyantraniliprole and or its photoproducts being retained on the ENV SPE column. Cyantraniliprole and its photoproducts were eluted from the ENV column with ammonium hydroxide in acetonitrile. The extracts were evaporated to dryness, reconstituted in methanol:formic acid, filtered and analyzed by LC/MS/MS.

Average (n=5) recovery rates in samples spiked with 0.001 mg/kg ranged from 75–102% (RSD < 17% and in samples spiked with 0.01 mg/kg were 72–96% (RSD < 9%). The method LOQ was 0.001 mg/kg.

The method 15440 was independently validated in a study reported by Schwartz, 2010 [Ref: DP-18847] with average (n=5) recovery rates in sandy loam soil samples spiked with 0.001 mg/kg ranging from 81–99% (RSD < 10%) and were 78–118% (RSD < 15%) in samples spiked with 0.01 mg/kg. The method LOQ was 0.001 mg/kg.

Method 29747 (soil)

Method 29747, an extension of the above method 15440 to include the measurement of the cyantraniliprole photoproducts IN-NXX70, IN-QKV54, and IN-RNU71, was described in a report by Vogl, 2010 [Ref: DP-29747], with average (n=5) recovery rates of 93–116% (RSD ≤ 12%) in a sandy clay loam soil spiked with 0.001 mg/kg or 0.01 mg/kg.

Enforcement methods

Multi-residue method DFG S19

Method 21328, the German method DFG S19 with LC-MS/MS analysis is suitable for enforcement of the MRL for cyantraniliprole in plant commodities, based on the method validation for tomato (representing high water content), orange (high acid content), wheat grain (high starch content) and almond (high oil content).

Method 26459, a GC/ECD procedure for crops was developed and validated in DuPont-26459. This method is intended for monitoring purposes in regions where LC/MS/MS is not readily available.

FDA PAM Multi-residue method

Rockwell, 2010 [Ref: DP-21329] evaluated the suitability of the FDA PAM Multi-residue methods for measuring residues of cyantraniliprole and six metabolites. The test substances were naturally fluorescent (with the exception of IN-MLA84-000, which was not soluble in methanol) but were not capable of being evaluated further without modifying the methods outlined in Protocol A. IN-JCZ38-004 and IN-K7H19-001 were found to chromatograph according to one Module DG 18, Level II, in Protocol C. However the two analytes were insoluble in the solvents required for the methods outlined in Protocols D, E, and F and further testing was suspended. Based on these results, none of the FDA multi-residue method test procedures are suitable for the regulatory analysis of DPX-HGW86-307 and its metabolites.

Analytical (concurrent) recoveries in supervised crop trials

Analytical recovery rates were measured in all the supervised crop field trials, with control samples being fortified with cyantraniliprole and metabolites at 0.01 mg/kg and at higher levels that generally reflected the range of expected residues. In most trials analysis was with Method 15736.

For each study, average recoveries per fortification level generally fell within the 70–120% range, with a relative standard deviation of 20% or less). Information on the concurrent recovery rates for individual commodities are summarised in the relevant supervised crop field trial sections.

Stability of residues in stored analytical samples

Plant matrices

The Meeting received information on the stability of residues of cyantraniliprole and metabolites in various substrates with a high water content (apple), a high starch content (potato), a high protein content (dry bean seed), a high oil content (peanut) and a high acid content (grape) stored at freezer temperatures for 24 months.

In a study by Rodgers, 2010, [Ref: DP-16990], samples of the test commodities were fortified at 0.2 mg/kg of each analyte. Spiked and the control samples were sealed and stored in the dark at -20 ± 10 °C. Samples were taken for extraction and analysis after 0, 1, 3, 6, 12, 18 and 24 months (and also peanuts after 63 days), with the stored control samples being freshly fortified with each analyte and analysed concurrently to determine the procedural recovery efficiency. Analysis was by LC-MS/MS (Method 15736), with mean procedural recovery rates of 83–94% for all analytes and matrices (fortified at 0.2 mg/kg and also at 0.01 mg/kg in peanuts.

After 24 months storage the measured residues of cyantraniliprole and major metabolites in stored samples of representative plant matrices with high water, acid, starch and protein content were greater than 80% of the spiked levels.

For peanut (high oil content), 24-month stability was demonstrated for cyantraniliprole, IN-F6L99, IN-J9Z38, IN-MLA84, IN-MYX98 and IN-N5M09, with measured residues also greater than 80% of the spiked levels. However, for the IN-JCZ38, IN-K7H19, and IN-N7B69 metabolites, the results showed reduced recoveries, attributed to extractability issues associated with the higher lipid content of this matrix.

Table 51 Stability of cyantraniliprole residues in plant matrices spiked at 0.2 mg/kg and stored at -20 ± 10 °C

Commodity	Storage interval (months)	Residues remaining (mg/kg)	% Residues remaining	Procedural recovery (%)
Apples	0			100.5
	1	0.18, 0.18	89.5	96.5
	3	0.17, 0.18	87	89
	6	0.16, 0.16	81.5	86.5
	12	0.17, 0.18	86	81.5
	18	0.18, 0.18	88.5	94
	24	0.19, 0.2	96	98.5

Commodity	Storage interval (months)	Residues remaining (mg/kg)	% Residues remaining	Procedural recovery (%)
Peanuts	0			93.5
	1	0.16, 0.15	78.5	93.5
	2	0.17, 0.17	84.5	87
	3	0.16, 0.16	80.5	100
	6	0.16, 0.15	77.5	82.5
	12	0.14, 0.14	71.5	75
	18	0.14, 0.16	75	88.5
	24	0.13, 0.13	66	79.5
Dried beans	0			94
	1	0.19, 0.17	88	89
	3	0.19, 0.18	91	92.5
	6	0.16, 0.14	75.5	88.5
	12	0.15, 0.15	75	78.5
	18	0.19, 0.19	94.5	103.5
	24	0.13, 0.13	65.5	71
Potatoes	0			98.5
	1	0.16, 0.17	83.5	87.5
	3	0.19, 0.19	96.5	97.5
	6	0.16, 0.17	83.5	87
	12	0.17, 0.17	85.5	87.5
	18	0.19, 0.18	92	93
	24	0.18, 0.19	91.5	94.5
Grapes	0			107
	1	0.17, 0.17	85	83
	3	0.18, 0.19	93.5	93.5
	6	0.17, 0.18	86	86
	12	0.18, 0.17	87	80
	18	0.19, 0.18	92.5	95
	24	0.2, 0.19	98	105.5

Table 52 Stability of cyantraniliprole metabolite IN-F6L99 residues in plant matrices spiked at 0.2 mg/kg and stored at -20 ± 10 °C.

Commodity	Storage interval (months)	Residues remaining (mg/kg)	% Residues remaining	Procedural recovery (%)
Apples	0			106.5
	1	0.16, 0.16	79.5	85
	3	0.15, 0.16	77	80
	6	0.16, 0.17	83	83.5
	12	0.16, 0.17	82.5	78.5
	18	0.19, 0.19	94	94.5
	24	0.17, 0.17	83	84.5
Peanuts	0			89
	1	0.19, 0.18	92	107.5
	2	0.19, 0.19	93	96.5
	3	0.18, 0.17	86.5	103
	6	0.18, 0.17	87.5	90.5
	12	0.17, 0.17	83.5	84.5
	18	0.15, 0.15	75	85
	24	0.15, 0.13	73	82.5
Dried beans	0			94
	1	0.2, 0.18	93.5	96.5
	3	0.18, 0.17	89.5	92
	6	0.19, 0.17	91	95
	12	0.17, 0.16	83	87.5
	18	0.17, 0.17	86	86
	24	0.14, 0.14	70	90.5

Commodity	Storage interval (months)	Residues remaining (mg/kg)	% Residues remaining	Procedural recovery (%)
Potatoes	0			103
	1	0.19, 0.21	100	96.5
	3	0.18, 0.18	91	89.5
	6	0.18, 0.18	90.5	90
	12	0.17, 0.17	85.5	87
	18	0.21, 0.21	104	103.5
	24	0.17, 0.17	85.5	89
Grapes	0			103
	1	0.18, 0.19	93.5	92
	3	0.18, 0.18	91	89
	6	0.17, 0.18	88.5	83
	12	0.17, 0.17	85.5	80
	18	0.2, 0.19	96.5	97
	24	0.16, 0.16	81	85

Table 53 Stability of cyantraniliprole metabolite IN-J9Z38 residues in plant matrices spiked at 0.2 mg/kg and stored at -20 ± 10 °C

Commodity	Storage interval (months)	Residues remaining (mg/kg)	% Residues remaining	Procedural recovery (%)
Apples	0			95.5
	1	0.2, 0.19	96.5	102.5
	3	0.17, 0.17	85	85
	6	0.16, 0.15	77	83.5
	12	0.15, 0.16	79.5	73
	18	0.16, 0.16	80.5	89.5
	24	0.17, 0.17	85	85
Peanuts	0			88.5
	1	0.18, 0.17	86.5	103
	2	0.15, 0.16	78.5	82
	3	0.14, 0.15	72	94
	6	0.15, 0.13	75	77
	12	0.14, 0.14	70.5	73
	18	0.14, 0.15	76	92.5
	24	0.15, 0.17	80.5	90
Dried beans	0			88.5
	1	0.19, 0.16	88	92
	3	0.18, 0.17	86	87
	6	0.14, 0.12	72	80.5
	12	0.15, 0.14	72.5	73
	18	0.16, 0.16	78.5	88
	24	0.18, 0.18	91	105.5
Potatoes	0			92
	1	0.17, 0.17	84.5	87.5
	3	0.18, 0.19	93.5	94.5
	6	0.14, 0.15	74	77.5
	12	0.16, 0.17	83	80.5
	18	0.18, 0.18	90	94
	24	0.16, 0.16	81.5	82.5
Grapes	0			102
	1	0.18, 0.18	88.5	88
	3	0.18, 0.18	89.5	86.5
	6	0.16, 0.17	81.5	80
	12	0.17, 0.16	80.5	73
	18	0.18, 0.18	89.5	92
	24	0.18, 0.17	86.5	93

Table 54 Stability of cyantraniliprole metabolite IN-JCZ38 residues in plant matrices spiked at 0.2 mg/kg and stored at -20 ± 10 °C

Commodity	Storage interval (months)	Residues remaining (mg/kg)	% Residues remaining	Procedural recovery (%)

Commodity	Storage interval (months)	Residues remaining (mg/kg)	% Residues remaining	Procedural recovery (%)
Apples	0			99
	1	0.18, 0.18	89.5	97.5
	3	0.17, 0.18	86	88
	6	0.16, 0.17	81.5	86
	12	0.16, 0.16	81	76.5
	18	0.17, 0.17	86.5	91.5
	24	0.18, 0.18	90.5	93
Peanuts	0			88.5
	1	0.13, 0.12	62.5	94.5
	2	0.15, 0.15	75.5	88.5
	3	0.13, 0.12	62	98
	6	0.14, 0.13	66.5	82
	12	0.12, 0.11	59	78
	18	0.11, 0.12	58	89.5
	24	0.11, 0.097	51.5	86.5
Dried beans	0			92.5
	1	0.19, 0.18	92	92.5
	3	0.17, 0.17	86.5	90
	6	0.16, 0.14	76.5	89.5
	12	0.14, 0.14	70.5	76.5
	18	0.17, 0.17	86.5	97
	24	0.14, 0.14	71.5	89.5
Potatoes	0			94.5
	1	0.18, 0.18	90.5	94
	3	0.19, 0.19	96	98
	6	0.16, 0.16	80.5	91
	12	0.16, 0.16	81	85.5
	18	0.19, 0.2	95.5	97.5
	24	0.17, 0.17	86.5	92.5
Grapes	0			106
	1	0.17, 0.18	86	86
	3	0.18, 0.19	93.5	91
	6	0.17, 0.17	85	85
	12	0.16, 0.16	80	76
	18	0.18, 0.18	89.5	92.5
	24	0.19, 0.18	92	100

Table 55 Stability of cyantraniliprole metabolite IN-K7H19 residues in plant matrices spiked at 0.2 mg/kg and stored at -20 ± 10 °C

Commodity	Storage interval (months)	Residues remaining (mg/kg)	% Residues remaining	Procedural recovery (%)
Apples	0			99
	1	0.18, 0.18	91	96.5
	3	0.16, 0.18	85	86.5
	6	0.16, 0.16	81	83.5
	12	0.16, 0.18	85	79
	18	0.17, 0.17	84	90.5
	24	0.19, 0.19	94	96
Peanuts	0			90
	1	0.12, 0.1	55.5	92
	2	0.14, 0.14	70.5	89.5
	3	0.12, 0.1	55	98.5
	6	0.13, 0.12	63	82.5
	12	0.12, 0.11	56	77.5
	18	0.097, 0.097	49	85.5
	24	0.087, 0.072	39.5	76

Commodity	Storage interval (months)	Residues remaining (mg/kg)	% Residues remaining	Procedural recovery (%)
Dried beans	0			93
	1	0.18, 0.17	87	87.5
	3	0.17, 0.17	84.5	90
	6	0.16, 0.14	76	89.5
	12	0.14, 0.15	73	78.5
	18	0.19, 0.19	94	103.5
	24	0.2, 0.21	103.5	111.5
Potatoes	0			97.5
	1	0.17, 0.18	88	89
	3	0.19, 0.18	93	95
	6	0.16, 0.16	79	92
	12	0.17, 0.17	83.5	85
	18	0.17, 0.18	88.5	92
	24	0.18, 0.17	87.5	91.5
Grapes	0			107
	1	0.17, 0.18	87.5	87
	3	0.18, 0.19	93	92
	6	0.16, 0.17	82.5	83
	12	0.18, 0.18	91.5	83.5
	18	0.18, 0.18	90	92
	24	0.19, 0.19	93.5	96.5

Table 56 Stability of cyantraniliprole metabolite IN-MLA84 residues in plant matrices spiked at 0.2 mg/kg and stored at -20 ± 10 °C

Commodity	Storage interval (months)	Residues remaining (mg/kg)	% Residues remaining	Procedural recovery (%)
Apples	0			92
	1	0.19, 0.17	88.5	95
	3	0.16, 0.17	83.5	86
	6	0.15, 0.15	74	80
	12	0.15, 0.16	78	77
	18	0.17, 0.16	81.5	90
	24	0.16, 0.16	79	82
Peanuts	0			90
	1	0.16, 0.15	78	97
	2	0.17, 0.17	86	88
	3	0.15, 0.15	75	100
	6	0.15, 0.14	72.5	75
	12	0.13, 0.13	63.5	68c
	18	0.14, 0.16	74.5	96
	24	0.14, 0.16	75	90
Dried beans	0			87
	1	0.18, 0.16	86	86
	3	0.17, 0.17	84	84
	6	0.15, 0.13	72	74
	12	0.14, 0.14	70	75
	18	0.16, 0.16	80.5	86
	24	0.2, 0.19	97.5	113
Potatoes	0			86
	1	0.16, 0.17	82.5	87
	3	0.18, 0.18	88.5	92
	6	0.17, 0.18	88	85
	12	0.15, 0.16	77.5	80
	18	0.18, 0.19	93.5	94
	24	0.15, 0.15	74	75
Grapes	0			98
	1	0.16, 0.17	81	77
	3	0.18, 0.18	89	91
	6	0.15, 0.16	78	82
	12	0.16, 0.16	80	76
	18	0.18, 0.17	87.5	88
	24	0.17, 0.16	81	83

Table 57 Stability of cyantraniliprole metabolite IN-MYX98 residues in plant matrices spiked at 0.2 mg/kg and stored at -20 ± 10 °C

Commodity	Storage interval (months)	Residues remaining (mg/kg)	% Residues remaining	Procedural recovery (%)
Apples	0			106
	1	0.18, 0.18	89	97
	3	0.17, 0.18	87	90.5
	6	0.17, 0.17	85.5	87
	12	0.19, 0.19	94.5	83
	18	0.22, 0.23	112.5	106
	24	0.22, 0.21	107	97.5
Peanuts	0			102
	1	0.14, 0.14	70	93
	2	0.16, 0.16	80	90.5
	3	0.14, 0.14	72	101
	6	0.16, 0.15	79	84.5
	12	0.16, 0.15	77	80.5
	18	0.16, 0.18	85.5	99.5
	24	0.14, 0.12	67	81
Dried beans	0			99
	1	0.19, 0.17	88	90.5
	3	0.18, 0.17	87.5	94
	6	0.17, 0.15	81	90
	12	0.17, 0.17	84	83.5
	18	0.24, 0.24	121	114
	24	0.14, 0.15	73	71
Potatoes	0			103
	1	0.17, 0.18	85.5	89.5
	3	0.19, 0.19	96.5	96
	6	0.18, 0.18	89.5	93
	12	0.19, 0.19	95.5	90
	18	0.22, 0.23	112	103.5
	24	0.2, 0.2	101.5	95
Grapes	0			111
	1	0.16, 0.17	83	84
	3	0.19, 0.19	94	96.5
	6	0.18, 0.19	91	89
	12	0.19, 0.19	96.5	83.5
	18	0.23, 0.23	114.5	109.5
	24	0.23, 0.22	112	105.5

Table 58 Stability of cyantraniliprole metabolite IN-N7B69 residues in plant matrices spiked at 0.2 mg/kg and stored at -20 ± 10 °C

Commodity	Storage interval (months)	Residues remaining (mg/kg)	% Residues remaining	Procedural recovery (%)
Apples	0			105.5
	1	0.19, 0.18	92	97
	3	0.17, 0.17	87	90.5
	6	0.17, 0.17	83.5	88
	12	0.18, 0.19	92.5	85
	18	0.18, 0.18	90	96.5
	24	0.18, 0.19	92.5	100
Peanuts	0			97.5
	1	0.14, 0.13	68.5	93.5
	2	0.15, 0.16	77.5	88
	3	0.14, 0.14	70.5	99.5
	6	0.15, 0.13	69	77
	12	0.15, 0.15	75.5	81
	18	0.13, 0.14	67	78.5
	24	0.11, 0.081	46.5	67

Commodity	Storage interval (months)	Residues remaining (mg/kg)	% Residues remaining	Procedural recovery (%)
Dried beans	0			96.5
	1	0.18, 0.16	85	88
	3	0.18, 0.18	89.5	94
	6	0.15, 0.13	71	83
	12	0.17, 0.17	86	86.5
	18	0.23, 0.23	117	119.5
	24	0.17, 0.18	86	89.5
Potatoes	0			104.5
	1	0.17, 0.17	85	91
	3	0.19, 0.2	98.5	101
	6	0.16, 0.16	78	86.5
	12	0.19, 0.2	97.5	99.5
	18	0.17, 0.14	77.5	79
	24	0.16, 0.17	82	91
Grapes	0			116
	1	0.17, 0.17	84.5	84.5
	3	0.19, 0.2	97.5	95.5
	6	0.17, 0.17	86	87
	12	0.19, 0.2	98.5	85.5
	18	0.19, 0.2	97	98.5
	24	0.2, 0.2	99.5	105.5

Table 59 Stability of cyantraniliprole metabolite IN-N5M09 residues in plant matrices spiked at 0.2 mg/kg and stored at -20 ± 10 °C

Commodity	Storage interval (months)	Residues remaining (mg/kg)	% Residues remaining	Procedural recovery (%)
Apples	0			97
	1	0.18, 0.18	90	92
	3	0.17, 0.18	87.5	89
	6	0.16, 0.17	84	87
	12	0.16, 0.18	84.5	79.5
	18	0.19, 0.19	95.5	97
	24	0.2, 0.2	102	100
Peanuts	0			88
	1	0.16, 0.16	80.5	94
	2	0.19, 0.19	93.5	95
	3	0.15, 0.15	76	96.5
	6	0.15, 0.14	72.5	79.5
	12	0.14, 0.14	71	77
	18	0.15, 0.16	79.5	96
	24	0.15, 0.15	75.5	92
Dried beans	0			88
	1	0.18, 0.16	84.5	89
	3	0.18, 0.17	86	91
	6	0.15, 0.13	72	89.5
	12	0.14, 0.14	70	78.5
	18	0.18, 0.17	87.5	101.5
	24	0.16, 0.16	79	96.5
Potatoes	0			91.5
	1	0.17, 0.17	86	89.5
	3	0.19, 0.19	94.5	99
	6	0.18, 0.17	87	83.5
	12	0.17, 0.17	84	86.5
	18	0.2, 0.2	100.5	101
	24	0.17, 0.19	91	97.5

Commodity	Storage interval (months)	Residues remaining (mg/kg)	% Residues remaining	Procedural recovery (%)
Grapes	0			102.5
	1	0.16, 0.16	80	80
	3	0.18, 0.19	91	90
	6	0.16, 0.18	87	86.5
	12	0.17, 0.16	84	79
	18	0.2, 0.2	99	97.5
	24	0.21, 0.2	100.5	102.5

Animal matrices

The stability of residues in animal matrices during frozen storage (at ca. –80 °C) was measured in the both milk and egg matrices as part of the cattle feeding study, reported by Ward & Vance, 2011 [Ref: DP-27180] in support of analyses conducted as part of the cattle and hen feeding studies

Cyantraniliprole and its seven metabolites (IN-HGW87, IN-J9Z38, IN-JCZ38, IN-K7H19, IN-MLA84, IN-MYX98, and IN-N7B69) were shown to be stable for at least 3 months in milk and eggs. Storage stability testing was not performed for residues in other cow or hen tissues given that all analysis was carried out within 30 days of sampling.

Table 60 Stability of cyantraniliprole and metabolite residues in milk stored at -80 °C

Analyte	% Residues remaining		Procedural recovery (%)
	Day 0	Day 99	
Cyantraniliprole	96	99	100
IN-HGW87	102	113	108
IN-J9Z38	94	69	77
IN-JCZ38	90	93	96
IN-K7H19	98	99	100
IN-MLA84	93	56	58
IN-MYX98	103	98	96
IN-N7B69	116	87	93

Table 61 Stability of cyantraniliprole and metabolite residues in eggs stored at -80 °C

Analyte	% Residues remaining		Procedural recovery (%)
	Day 0	Day 99	
Cyantraniliprole	98	101	100
IN-HGW87	99	104	96
IN-J9Z38	98	84	85
IN-JCZ38	92	91	93
IN-K7H19	95	95	95
IN-MLA84	103	81	73
IN-MYX98	105	98	97
IN-N7B69	109	79	81

USE PATTERNS

Information on GAP in Canada, Columbia, Malaysia, New Zealand, Vietnam and West Africa was provided to the Meeting. Proposed uses in Australia, Europe, and USA that are currently being progressed under an OECD Joint Review exercise and in Brazil, China and India were also provided but are not reported.

Cyantraniliprole is intended for use to control of larval stages of lepidopteran insects, thrips, aphids, and some other chewing and sucking insects in a wide range of fruit and vegetable crops, tree crops and oil seed crops as foliar applications and as seed treatments (potato seed pieces, oil seed crops) and as pre-plant soil ‘in-furrow’ treatments (potatoes and brassica vegetables).

Table 62 Registered uses of cyantraniliprole (foliar applications)

Crop (Group)	Country	Application				Max/season		PHI (days)	Comments
		kg ai/ha	g ai/hL	water L/ha	RTI (days)	no	kg ai/ha		
Pome fruits (002)									
Pome fruit	Canada	0.05–0.15		min 450	7	4	0.45	3	
Stone fruits (003)									
Stone fruit	Canada	0.05–0.15		min 450	7	4	0.45	3	
Bush berries (004B)									
Bush berries	Canada	0.05–0.15		200 50 (air)	5–7	4	0.45	3	
Bulb vegetables (009)									
Bulb vegetables	Canada	0.1–0.15		200 50 (air)	5	4	0.45	1	
Onions (bulb)	Columbia	0.1–0.125				2	0.3	1	
Welsh onion	Columbia	0.1–0.125				2	0.3	1	
Brassica (cole or cabbage) vegetables (010)									
Brassica vegetables	Canada	0.025–0.15		100 50 (air)	5–7	4	0.45	1	
Fruiting vegetables, Cucurbits (011)									
Cucurbit vegetables	Canada	0.025–0.15		100 50 (air)	5–7	4	0.45	1	
Cucumber	Malaysia	0.075–0.1	10–13	750	7	3		1	
Fruiting Vegetables, other than Cucurbits (012)									
Fruiting Vegetables, (except Cucurbits)	Canada	0.025–0.15		100 50 (air)	5–14	4	0.45	1	
Tomatoes	Malaysia	0.075–0.1	10–13	750	7	3		1	
Eggplant (Brinjal)	Malaysia	0.04–0.01	5–13	750	7	3		1	
Chili pepper	Malaysia	0.04–0.1	5–13	750	7	3		1	
Leafy vegetables (including Brassica leafy vegetables) (013)									
Leafy vegetables Brassica vegetables	Canada	0.025–0.15		100 50 (air)	5–7	4	0.45	1	
Legume vegetables (014)									
Long bean	Malaysia	0.04–0.075	5–10	750	7	2		1	
Root and tuber vegetables (016)									
Tuber & corm vegetables	Canada	0.1–0.15		100 50 (air)	5–14	4	0.45	7	
Potato	Canada	0.05–0.15		100 50 (air)	7–14	4	0.45	7	
Stalk and stem vegetables (017)									
Leafy vegetables	Canada	0.025–		100	5–7	4	0.45	1	includes celery

Crop (Group)	Country	Application				Max/season		PHI (days)	Comments
		kg ai/ha	g ai/hL	water L/ha	RTI (days)	no	kg ai/ha		
		0.15		50 (air)					
Cereal grains (020)									
Rice	Vietnam	0.05–0.1		500–600				5	
Tree nuts (022)									
Tree nuts	Canada	0.05–0.1		min 450	7	4	0.45	5	
Oilseed (023)									
Oil seed group	Canada	0.025–0.1		100 50 (air)	7	4	0.11	7	includes rape seed, sunflower
Cotton	CILSS	0.05		150	14	3	0.15	7	
Seeds for beverages and sweets (024)									
Coffee	Columbia	0.125–0.175	50–70	50 ml/tree		1	0.3	28	based on 5000 trees/ha @ 2.5–3.5 g ai/100 trees
Miscellaneous Fodder and Forage crops (052)									
Fodder brassicas	New Zealand	0.015			14–21	3		28 (stock)	with surfactant

Canada: GAP includes a general recommendation to not apply cyantraniliprole following a soil or seed treatment of a Group 28 Insecticide (including cyantraniliprole).

CILSS: Regional (Sahel) group, of countries—Benin, Côte d'Ivoire, Gambia, Guinea Bissau, Mauritania, Senegal, Togo, Burkina Faso, Mali, Niger, Chad and Cape Verde.

Table 63 Registered uses of cyantraniliprole (soil and seed treatments)

Crop (Group)	Country	Application				Max/season		Comments
		form	type	rate	kg ai/ha	no	kg ai/ha	
Brassica (cole or cabbage) vegetables (010)								
Brassica vegetables	Canada	200SC	furrow		0.15–0.2	1	0.2	Band spray in-furrow
Brassica vegetables	Canada	200SC	drench	45 mL drench solution/plant	0.15–0.2	1		At transplanting
Brassica vegetables	Canada	200SC	surface		0.15–0.2	1		5 cm band spray at planting with soil incorporation by irrigation
Leafy vegetables (including Brassica leafy vegetables) (013)								
Leafy vegetables Brassica vegetables	Canada	200SC	furrow		0.15–0.2	1	0.2	Band spray in-furrow
Leafy vegetables Brassica vegetables	Canada	200SC	drench	45 mL drench solution/plant	0.15–0.2	1		At transplanting
Leafy vegetables Brassica vegetables	Canada	200SC	surface		0.15–0.2	1		5 cm band spray at planting with soil incorporation by irrigation
Root and tuber vegetables (016)								
Potato	Canada	200SC	seed	9 g ai/100 kg seed	0.2 (equiv)	1	0.3	Based on sowing rate of 2200 kg seed/ha
Potato	Canada	200SC	furrow	1.35–1.8 g ai/100 m row	0.15–0.2 (equiv)	1		Band spray in-furrow to cover seed pieces Based on 90 cm row spacing

Crop (Group)	Country	Application				Max/season		Comments
		form	type	rate	kg ai/ha	no	kg ai/ha	
Oilseed (023)								
Oilseed rape rapeseed Oilseed mustard	Canada	625SC	seed	0.3– 1.0 kg ai/100 kg seed				

Canada: GAP includes a general recommendation to not apply any subsequent soil or foliar treatments of a Group 28 Insecticide (including cyantraniliprole).

RESIDUES RESULTING FROM SUPERVISED TRIALS

The Meeting received information on supervised field trials involving foliar, drip irrigation or seed treatment applications of cyantraniliprole to the following crops.

Group	Crop	Countries	Table no
Citrus	Orange	Brazil, USA, Europe	64–67
	Mandarin	Europe	68
	Grapefruit	USA	69
	Lemons	USA	70–71
Pome fruit	Apple	Europe, Nth America	72–73
	Pear	Europe, Nth America	74–75
Stone fruit	Apricot,	Europe	76
	Cherry	Nth America	77
	Peach	Nth America, Europe	78–79
	Plum	Nth America, Europe	80–81
Small fruit	Blueberry	Nth America	82
	Grape	Europe	83
Other fruit	Olive	Europe	84
	Pomegranate	India	85
Bulb vegetables	Onion, bulb	North America	86
	Spring onion		87
Brassica vegetables	Broccoli	Nth America	88
	Cauliflower	Nth America	89
	Cabbage	Nth America	90
Cucurbits	Cucumber	Nth America, Europe, Australia	91–94
	Summer squash	Nth America, Europe, Australia	95–98
	Melon	Nth America, Europe, Australia	99–102
Fruiting vegetables	Tomato	Nth America, Europe, Australia	103–107
	Peppers	Nth America, Europe, Australia	108–113
Leafy vegetables	Head lettuce	Nth America, Europe	114–119
	Leaf lettuce	Nth America, Europe	120–125
	Lamb's lettuce	Europe	126–127
	Spinach	USA	128–129
	Mustard greens	USA	130
	Scarole	Europe	131–134
Legume vegetables	Common bean	Europe	135–136

Group	Crop	Countries	Table no
Tuber vegetables	Potato	North America, Europe	137–139
Stem vegetables	Celery	Nth America	140–141
Cereals	Rice	China	142
Tree nuts	Almond	USA	143
	Pecan	USA	144
Oilseeds	Cotton	Australia, Nth America	145–146
	Oil-seed rape	Australia, Nth America	147–148
	Sunflower	North America	149
Others	Coffee	Brazil	150

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

Results from replicated field plots are presented as individual values. When residues were not detected they are shown as ND. Residues and application rates have been reported as provided in the study reports, although the results from trials used for the estimation of maximum residue levels (underlined) have been rounded to two significant digits (or if close to the LOQ, rounded to one significant digit) in the Appraisal.

In some trials, samples were taken just before the final application and then, again on the same day after the spray had dried. In the data tables the notation for these two sampling times is '–0' and '0' respectively.

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

The analytical methods used in the field trials were capable of analysing both cyantraniliprole and from 1 to 7 metabolites. In most cases, residues of these metabolites were not detected (LOD of 0.003 mg/kg in most trials) or in some cases were reported at levels below the LOQ of 0.01 mg/kg. Where metabolite residues were present at levels above the LOQ, these values are recorded in the following tables using the abbreviations listed below:

M1	IN-J9Z38	2-[3-Bromo-1-(3-chloro-2-pyridinyl)-1*H*-pyrazol-5-yl]-3,4-dihydro-3,8-dimethyl-4-oxo-6-quinazolinecarbonitrile
M2	IN-MYX98	3-Bromo-1-(3-chloro-2-pyridinyl)-N-[4-cyano-2[[(hydroxymethyl)amino]carbonyl]-6-methylphenyl]-1*H*-pyrazole-5-carboxamide
M3	IN-N7B69	3-Bromo-1-(3-chloro-2-pyridinyl)-N-[4-cyano-2-(hydroxymethyl)-6-[(methylamino)carbonyl]phenyl]-1*H*-pyrazole-5-carboxamide

M4	IN-MLA84	2-[3-Bromo-1-(3-chloro-2-pyridinyl)-1*H*-pyrazol-5-yl]-1,4-dihydro-8-methyl-4-oxo-6-quinazolinecarbonitrile
M5	IN-JCZ38	4-[[[3-Bromo-1-(3-chloro-2-pyridinyl)-1*H*-pyrazol-5-yl]carbonyl]amino]-N′3′,5-dimethyl-1,3-benzenedicarboxamide
M6	IN-N5M09	6-Chloro-4-methyl-11-oxo-11*H*-pyrido[2,1-*b*]quinazoline-2-carbonitrile
M7	IN-F6L99	3-Bromo-N-methyl-1*H*-pyrazole-5-carboxamide

Citrus fruits

Results from supervised trials from Brazil, Europe and USA on oranges, grapefruit, lemons and mandarins were provided to the Meeting.

Brazil

In four Brazilian trials on oranges, one plot was treated with two soil drench applications of cyantraniliprole (200 g ai/L SC), 30 days apart, using 1.4 g ai in 200 mL water/tree (equivalent to 0.7 kg ai/ha for a tree density of 500 trees/ha) and a second plot was treated with two foliar applications of cyantraniliprole (100 g/L OD) using 0.3 kg ai/ha in 1500 L water/ha. No adjuvants were used.

Samples were frozen within 4 hours of sampling, stored at –20 °C for up to 84 days before whole fruit analysis for cyantraniliprole and metabolite IN-J9Z38 using an adaptation of method DP-15736, with reported LOQs of 0.01 mg/kg. Average concurrent recoveries were 108% (cyantraniliprole) and 99% (IN-J9Z38) in samples spiked with 0.01 and 0.1 mg/kg.

North America

In trials conducted in the USA on oranges, grapefruit and lemons, three foliar applications of 0.15 kg ai/ha cyantraniliprole (SE formulation) were applied at 7 day intervals, either as high volume, concentrate or ultra-low-volume treatments, with adjuvant added. Band treatment plots also included in three trial sites, where band spray applications (200 SC formulation) were made on one side of the tree rows, targeting 950 mL spray solution per tree.

Duplicate samples were frozen within 5 hours of sampling, stored at –20 °C for up to 5 months 12 months for Test 2 peel) before extraction and analysis of pulp and peel for cyantraniliprole and metabolites (within 46 days of extraction) using an adaptation of method DP-15736, with reported LOQs of 0.01 mg/kg. Average concurrent recoveries in peel and pulp were 91–96% (cyantraniliprole) and 75–110% (metabolites) in samples spiked with 0.01, 0.1 and 1.0 mg/kg (peel). Whole fruit residues were calculated from the pulp and peel analysis results.

Europe

In trials conducted in Europe on oranges and mandarins, 2–3 foliar applications of 0.15 kg ai/ha cyantraniliprole (SE formulation) were applied at 7 day intervals, either as high volume or concentrate treatments, with added surfactant.

Samples were stored at –18 °C for up to 12 months before extraction and analysis of pulp and peel for cyantraniliprole and six metabolites (within 7 days of extraction) using method DP-15736, with reported LOQs of 0.01 mg/kg. Average concurrent recoveries in peel and pulp were 88–98% (cyantraniliprole) and 80–102% (metabolites) in samples spiked with 0.01, 0.1 and 1.0 mg/kg (peel). Whole fruit residues were calculated from the pulp and peel analysis results.

Table 64 Residues in oranges from supervised trials in Brazil involving soil drench applications of cyantraniliprole (OD formulation)

ORANGE Country, year Location (variety)	Application				RTI (days)	DAT, (days)	Matrix	Residues (mg/kg)		Reference & Comments
	no	kg ai/ha [a]	g ai/tree	water (mL/tree)				cyantraniliprole	metabolites	
Jaguariuna, SP Brazil, 2010 (Valencia)	2	0.7	1.4	200	28	7 14 21 28	whole fruit	ND ND ND ND		BRI-09/10-014 Test A (357 trees/ha)
Conchal, SP Brazil, 2010 (Valencia)	2	0.7	1.4	200	28	7 14 21 28	whole fruit	ND < 0.01 ND ND		BRI-09/10-014 Test B (357 trees/ha)
Botucatu (Valencia)	2	0.7	1.4	200	28	21	whole fruit	ND		BRI-09/10-014 Test C (332 trees/ha)
Espirito Santo do Pinhal, SP Brazil, 2010 (Valencia)	2	0.7	1.4	200	28	21	whole fruit	< 0.01		BRI-09/10-014 Test D (446 trees/ha)

[a] Calculated application rate/ha based on a planting density of 500 trees/ha

Table 65 Residues in oranges from supervised trials in Brazil involving foliar applications of cyantraniliprole (SC formulation)

ORANGE Country, year Location (variety)	Application				RTI (days)	DAT, (days)	Matrix	Residues (mg/kg)		Reference & Comments
	no	kg ai/ha	g ai/hL	water (L/ha)				cyantraniliprole	metabolites	
Jaguariuna, SP Brazil, 2010 (Valencia)	2	0.3	20	1500	28	7 14 21 28	whole fruit	0.15 0.05 0.05 0.04		BRI-09/10-014 Test A (357 trees/ha)
Conchal, SP Brazil, 2010 (Valencia)	2	0.3	21	1430	28	7 14 21 28	whole fruit	0.62 0.33 0.33 0.23		BRI-09/10-014 Test B (357 trees/ha)
Botucatu Brazil, 2010 (Valencia)	2	0.3	23	1330	28	21	whole fruit	0.1		BRI-09/10-014 Test C (332 trees/ha)
Espirito Santo do Pinhal, SP Brazil, 2010 (Valencia)	2	0.3	21	1430	28	21	whole fruit	0.23		BRI-09/10-014 Test D (446 trees/ha)

Table 66 Residues in oranges from supervised trials in the USA involving foliar applications of cyantraniliprole (SE formulation)

ORANGE Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Clermont, FL USA, 2009 (Hamlin)	3	0.15	535	28	7	1	peel pulp whole	0.37, 0.19 0.033, 0.028 0.19, 0.11	0.28 0.03 0.15		DP-27554 Test 01
Clermont, FL USA, 2009 (Mid Sweet)	3	0.15	535	28	7	1	peel pulp whole	0.56, 0.69 0.054, 0.074 0.31, 0.38	0.63 0.064 0.35		DP-27554 Test 02
Mascotte, FL USA, 2009 (Valencia—Early)	3	0.15	535	28	7	1	peel pulp whole	0.54, 0.39 0.08, 0.092 0.31, 0.24	0.46 0.086 0.28		DP-27554 Test 03
Oviedo, FL USA, 2009 (Navel)	3	0.15	11	1400	7	1	peel pulp whole	0.36, 0.36 0.053, 0.039 0.17, 0.17	0.36 0.046 0.17		DP-27554 Test 04
Oviedo, FL USA, 2009 (Hamlin)	3	0.15	11	1400	7	1	peel pulp whole	0.27, 0.14 0.026, 0.029 0.15, 0.085	0.21 0.027 0.12		DP-27554 Test 05
Mims, FL USA, 2009 (Hamlin)	3	0.15	20	700	7	1	peel pulp whole	0.48, 0.64 0.036, 0.043 0.26, 0.35	0.56 0.04 0.3		DP-27554 Test 06
Holopaw, FL USA, 2009 (Valencia)	3	0.15	21	700	7	1	peel pulp whole	0.34, 0.47 0.041, 0.045 0.18, 0.24	0.41 0.043 0.21		DP-27554 Test 07
Chuluota, FL USA, 2009 (Hamlin)	3	0.15	11	1400	7	1	peel pulp whole	0.69, 0.7 0.081, 0.092 0.37, 0.4	0.7 0.086 0.39		DP-27554 Test 08
Alamo, TX USA, 2009 (Valencia)	3	0.15	25	610	7	1	peel pulp whole	0.86, 0.91 0.071, 0.066 0.22, 0.23	0.88 0.069 0.22		DP-27554 Test 09
Sanger, CA USA, 2009 (Fisher)	3	0.15	25	610	7	1	peel pulp whole	0.23, 0.28 0.016, 0.02 0.087, 0.11	0.25 0.018 0.098	M1=0.01	DP-27554 Test 10
Sanger, CA USA, 2009) (Campbell)	3	0.15	25	610	7	1	peel pulp whole	0.45, 0.35 0.017, 0.01 0.14, 0.1	0.4 0.013 0.12		DP-27554 Test 14
Sanger, CA USA, 2009) (Navel)	3	0.15	8	1870	7	1	peel pulp whole	0.21, 0.21 0.038, 0.035 0.1, 0.1	0.21 0.036 0.1		DP-27554 Test 25
Sanger, CA USA, 2009) (Washington Navel)	3	0.15	0.01	1550	7	1	peel pulp whole	0.7, 0.64 0.019, 0.024 0.2, 0.2	0.67 0.021 0.2		DP-27554 Test 26

M1: Average residues of metabolite IN-J9Z38 reported in peel

Table 67 Residues in oranges from supervised trials in Europe involving foliar applications of cyantraniliprole (SE formulation)

ORANGE Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	kg ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Andalucia Spain, 2010 (Navelina)	2	0.15	0.01	1500	7	-0	peel	0.27			DP-27716 Test 14 Orange
							pulp	0.023			
							whole	0.093			
						0	peel	0.33			
							pulp	0.017			
							whole	0.11			
						1	peel	0.074			
							pulp	0.021			
							whole	0.036			
						3	peel	0.84			
							pulp	0.012			
							whole	0.26			
						7	peel	0.51			
							pulp	ND			
							whole	0.16			
Andalucia Spain, 2010 (Navelina)	2	0.15	0.01	1500	7	-0	peel	0.24			DP-27716 Test 15 Orange
							pulp	0.012			
							whole	0.067			
						0	peel	0.56			
							pulp	0.046			
							whole	0.17			
						1	peel	0.73			
							pulp	0.034			
							whole	0.18			
						3	peel	0.56			
							pulp	ND			
							whole	0.13			
						7	peel	0.86			
							pulp	0.018			
							whole	0.22			
Kostaki Greece, 2010 (Salustiana)	2	0.15	0.01	1500	7	7	peel	0.26			DP-27716 Test 08 Orange
							pulp	0.023			
							whole	0.09			

ORANGE Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	kg ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Sicily Italy, 2010 (Tarocco)	2	0.15	0.01	1500	7	-0	peel pulp whole	0.49 ND 0.1			DP-27716 Test 16 Orange
						0	peel pulp whole	0.96 0.011 0.21			
						1	peel pulp whole	1.2 0.01 0.26			
						3	peel pulp whole	0.73 ND 0.15			
						7	peel pulp whole	0.66 ND 0.14			
Tortosa Spain, 2009 (Navelina)	2	0.15	0.01	1500	7	-0	peel pulp whole	0.28 0.018 0.091			DP-27716 Test 06 Orange
						0	peel pulp whole	0.53 0.041 0.17			
						1	peel pulp whole	0.51 0.032 0.16			
						3	peel pulp whole	0.47 0.032 0.15			
						7	peel pulp whole	0.76 0.023 0.21			
Xerta Spain, 2010 (Navelate)	2	0.15	0.01	1500	7	7	peel pulp whole	0.54 0.016 0.13			DP-27716 Test 07 Orange
Kostaki Greece, 2009 (Salustiana)	3	0.15	0.01	1500	7	-0	peel pulp whole	0.65 0.043 0.23			DP-27716 Test 01 Orange
						1	peel pulp whole	0.79 0.041 0.26			

ORANGE Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	kg ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Sicily Italy, 2009 (Tarocco)	3	0.15	0.01	1500	7	-0	peel	0.85			DP-27716 Test 02 Orange
							pulp	0.004			
							whole	0.2			
						1	peel	0.9			
							pulp	0.007			
							whole	0.23			

Table 68 Residues in mandarins from supervised trials in Europe involving foliar applications of cyantraniliprole (SE formulation)

MANDARIN Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg			Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)	RTI (days)			cyantraniliprole	mean	metabolites	
Andaluci Spain, 2009 (Clementina)	2	0.15	0.01	1500	7	–0	peel	0.37			DP-27716 Test 04 Mandarin
							pulp	0.01			
							whole	0.079			
						0	peel	0.6			
							pulp	0.025			
							whole	0.14			
						1	peel	0.78			
							pulp	ND			
							whole	0.17			
						3	peel	0.61			
							pulp	0.008			
							whole	0.15			
						7	peel	0.14			
							pulp	0.006			
							whole	0.036			
Andalucia, Spain, 2010 (Clementina)	2	0.15	0.01	1500	7	–0	peel	0.56			DP-27716 Test 11 Mandarin
							pulp	0.041			
							whole	0.16			
						0	peel	1.2		M1=0.013	
							pulp	0.086			
							whole	0.35			
						1	peel	1.0		M1=0.013	
							pulp	0.063			
							whole	0.3			
						3	peel	0.73			
							pulp	0.026			
							whole	0.22			
						7	peel	0.54		M1=0.011	
							pulp	0.028			
							whole	0.16			

MANDARIN Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg			Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)	RTI (days)			cyantraniliprole	mean	metabolites	
Kostaki Greece, 2010 (Nova)	2	0.15	0.01	1500	7	7	peel pulp whole	0.54 0.017 0.14		M1=0.01	DP-27716 Test 09 Mandarin
Sant Jordi Spain, 2010 (Ortanike)	2	0.15	0.03	510		–0	peel pulp whole	0.24 0.023 0.073			DP-27716 Test 10 Mandarin
						0	peel pulp whole	0.54 0.11 0.19			
						1	peel pulp whole	0.67 ND 0.15			
						3	peel pulp whole	0.74 0.035 0.19			
						7	peel pulp whole	0.55 0.033 0.16			
Sicily Italy, 2010 (Clementine Comune)	2	0.15	0.01	1500	7	–0	peel pulp whole	0.58 0.004 0.17			DP-27716 Test 12 Mandarin
						0	peel pulp whole	1.4 0.014 0.39			
						1	peel pulp whole	0.9 0.007 0.25			
						3	peel pulp whole	1.2 0.009 0.33			
						7	peel pulp whole	0.81 0.006 0.23			

MANDARIN Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg			Reference & Comments
	no	kg ai/ha	kg ai/hL	water (L/ha)	RTI (days)			cyantraniliprole	mean	metabolites	
Sicily Italy, 2010 (Nova)	2	0.157	0.01	1560	7	–0	peel pulp whole	0.57 0.006 0.13			DP-27716 Test 13 Mandarin
						0	peel pulp whole	1.0 0.013 0.24			
						1	peel pulp whole	1.1 0.043 0.29			
						3	peel pulp whole	0.84 0.009 0.2			
						7	peel pulp whole	0.64 0.008 0.16			
Tivenys Spain, 2009 (Nules)	2	0.15	0.03	500	7	–0	peel pulp whole	1.5 0.06 0.42			DP-27716 Test 05 Mandarin
						0	peel pulp whole	1.6 0.1 0.49			
						1	peel pulp whole	1.9 0.07 0.51			
						3	peel pulp whole	2.0 0.09 0.56			
						7	peel pulp whole	2.7 0.03 0.71		M1=0.01	
Kostaki Greece, 2009 (Clementine)	3	0.15	0.01	1500	7	–0	peel pulp whole	1.1 0.08 0.38		M1=0.01	DP-27716 Test 03 Mandarin
						1	peel pulp whole	1.1 0.2 0.47		M1=0.014	

M1: Average residues of metabolite IN-J9Z38 reported in peel

Table 69 Residues in grapefruit from supervised trials in the USA involving foliar applications of cyantraniliprole (SE formulation)

GRAPEFRUIT Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Mims, FL USA, 2009 (White Marsh)	3	0.15	21	700	7	1	peel pulp whole	0.35, 0.34 0.022, 0.019 0.14, 0.14	0.35 0.021 0.14		DP-27554 Test 11
Oviedo, FL USA, 2009 (Flame)	3	0.15	11	1400	7	1	peel pulp whole	0.41, 0.43 0.028, 0.037 0.18, 0.2	0.42 0.032 0.19		DP-27554 Test 12
Holopaw, FL USA, 2009 (White)	3	0.15	10	1500	7	1	peel pulp whole	0.77, 0.67 0.043, 0.055 0.33, 0.3	0.72 0.049 0.31		DP-27554 Test 13
Alamo, TX USA, 2009 (Rio Red)	3	0.15	6	2400	7	1	peel pulp whole	0.45, 0.28 0.032, 0.019 0.11, 0.21	0.36 0.026 0.16	M1=0.015	DP-27554 Test 15
Elderwood, CA USA, 2009 Duncan	3	0.15	32	470	7	1	peel pulp whole	0.26, 0.18 0.035, 0.03 0.11, 0.076	0.22 0.033 0.091		DP-27554 Test 16
Sanger, CA USA, 2009 (Rio Red)	3	0.15	0.025	620	7	1	peel pulp whole	0.32, 0.29 0.02, 0.039 0.12, 0.12	0.3 0.029 0.12		DP-27554 Test 17
Sanger, CA USA, 2009 (Marsh White)	3	0.15	0.01	1560	7	1	peel pulp whole	0.31, 0.34 0.012, 0.016 0.11, 0.13	0.33 0.014 0.12		DP-27554 Test 18

M1: Average residues of metabolite IN-J9Z38 reported in peel

Table 70 Residues in lemons from supervised trials in the USA involving foliar applications of cyantraniliprole (SE formulation.

LEMON Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Newman, CA USA, 2009/2010 (Lisbon)	3	0.15	8	1870	7	1	peel pulp whole	0.42, 0.44 0.11, 0.11 0.21, 0.22	0.43 0.11 0.21		DP-27554 Test 19
Sanger, CA USA, 2009 (Lisbon)	3	0.15	25	610	7	1	peel pulp whole	0.3, 0.45 0.022, 0.024 0.13, 0.2	0.37 0.023 0.16		DP-27554 Test 20
Sanger, CA USA, 2009 (Frost Lisbon)	3	0.15	10	1560	7	1	peel pulp whole	0.62, 0.63 0.068, 0.057 0.31, 0.3	0.63 0.063 0.3		DP-27554 Test 21
Sanger, CA USA, 2009/2010 (Eureka)	3	0.15	33	470	7	1	peel pulp whole	0.34, 0.39 0.069, 0.071 0.18, 0.2	0.36 0.07 0.19		DP-27554 Test 22
Sanger, CA USA, 2009 (Lizbon 8A)	3	0.16	8	1870	7	1	peel pulp whole	0.32, 0.39 0.059, 0.066 0.14, 0.17	0.35 0.063 0.16		DP-27554 Test 23
Elderwood, CA USA, 2009 (Lizbon)	3	0.15	32	470	7	1	peel pulp whole	0.24, 0.42 0.037, 0.077 0.11, 0.21	0.33 0.057 0.16		DP-27554 Test 24

Table 71 Residues in lemons from supervised trials in the USA involving soil band applications of cyantraniliprole (200 SC formulation)

LEMON Location Country, year (variety)	Application					DAT (days)	Cyantraniliprole Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	water L/tree		peel	pulp	whole fruit	metabolites	
Sanger, CA USA, 2009/2010 (Eureka)	1	0.45	117	390	0.95	1 7 14	< 0.01 ND ND	ND ND ND	< 0.01 ND ND		DP-27554 Test 22
Sanger, CA USA, 2009 (Lizbon 8A)	1	0.45	0.16	280	0.95	1 7 14	ND < 0.01 ND	ND ND ND	ND < 0.01 ND		DP-27554 Test 23
Elderwood, CA USA, 2009 (Lizbon)	1	0.45	0.17	260	0.95	1 7 14	ND ND ND	ND ND ND	ND ND ND		DP-27554 Test 24

Pome fruits

Results from supervised trials from Europe and North America on apples and pears were provided to the Meeting.

North America

In trials conducted in North America on apples and pears, three foliar applications of 0.15 kg ai/ha cyantraniliprole (SE formulation) were applied at 7 day intervals, either as high volume or concentrate airblast applications, with adjuvant added (except in the processing study trials).

Duplicate samples were stored at –20 °C for up to 7 months before analysis (within 76 days of extraction) for cyantraniliprole and six metabolites using analytical method DP-15736, with reported LOQs of 0.01 mg/kg. Average concurrent recoveries were 93–97% (cyantraniliprole) and 83–102% (metabolites) in samples spiked with 0.01 and 0.1 mg/kg.

Europe

In trials conducted in Europe on apples and pears, two foliar applications of 6–15 g ai/hL cyantraniliprole (SE or WG formulations) were applied at 7 day intervals, either as high volume or concentrate treatments, with and without added adjuvant. These trials included a number of reverse decline studies, where different plots were treated at staggered intervals so that the sampling dates coincided with normal commercial harvest.

Samples were stored at –18 °C for up to 4–5 months before analysis of whole fruit for cyantraniliprole and 6–7 metabolites (within 2–24 hours after extraction) using analytical method DP-15736, with reported LOQs of 0.01 mg/kg. Average concurrent recoveries were 90–94% (cyantraniliprole) and 88–99% (metabolites) in samples spiked with 0.01 and 0.1 mg/kg.

Table 72 Residues in apples from supervised trials in North America involving foliar applications of cyantraniliprole (SE formulation)

APPLE Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Beldenville, WI USA, 2009 (Connell Red)	3	0.15	31	490	7	3 7	whole fruit	0.13, 0.13 0.08, 0.12	0.13 0.099		DP-27438 Trial 20

APPLE Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Berwick, NS, CAN, 2009 (Coatland)	3	0.15	34	450	6, 9	3 7	whole fruit	0.086, 0.056 0.066, 0.052	0.071 0.059		DP-27438 Test 02
Branchton, ON, CAN, 2009 (Northern Spy)	3	0.15	12	1260	7	3 7	whole fruit	0.2, 0.21 0.19, 0.17	0.21 0.18		DP-27438 Trial 05
Cana, VA USA, 2009 (Yellow Delicious)	3	0.15	27	560	7	3 7	whole fruit	0.25, 0.26 0.29, 0.21	0.26 0.25		DP-27438 Trial 04
Centralia, IL USA, 2009 (Golden Delicious)	3	0.15	38	400	7, 6	3 7	whole fruit	0.2, 0.16 0.13, 0.13	0.18 0.13		DP-27438 Trial 08
Conklin, MI USA, 2009 (Golden Delicious)	3	0.15	8	1900	4, 7	3 7	whole fruit	0.15, 0.19 0.099, 0.12	0.17 0.11		DP-27438 Trial 16
Ephrata, WA USA, 2009 (Red Delicious)	3	0.15	7	1880	7	3 7	whole fruit	0.31, 0.27 0.26, 0.27	0.29 0.27		DP-27438 Trial 10
Hart, MI USA, 2009 (Golden Delicious)	3	0.15	30	500	7	3 7	whole fruit	0.31, 0.31 0.29, 0.31	0.31 0.3		DP-27438 Trial 07
Hood River, OR USA, 2009 (Gingergold)	3	0.15	8	1900	7	3 6	whole fruit	0.15, 0.16 0.1, 0.089	0.15 0.096		DP-27438 Trial 13
Hood River, OR USA, 2009 (Jonagold)	3	0.15	13	550	7	3 7	whole fruit	0.046, 0.057 0.072, 0.055	0.052 0.064		DP-27438 Trial 15
North Rose, NY USA, 2009 (Ida Red)	3	0.15	7	2100	7, 6	3 8	whole fruit	0.15, 0.086 0.073, 0.072	0.12 0.072		DP-27438 Trial 03
Payette, ID USA, 2009 (Early Spur Rome)	3	0.15	13	1180	7	3 7	whole fruit	0.23, 0.28 0.17, 0.13	0.26 0.15		DP-27438 Trial 14
Perry, UT USA, 2009 (Gala)	3	0.15	8	2000	7	3 7	whole fruit	0.13, 0.17 0.11, 0.12	0.15 0.12		DP-27438 Trial 11
Sanger, CA USA, 2009 (Pink Lady)	3	0.15	32	480	7	3 7	whole fruit	0.072, 0.062 0.094, 0.11	0.067 0.1		DP-27438 Trial 12
Santa Maria, CA USA, 2009 (Starkrimson)	3	0.15	7	2250	6	3 7	whole fruit	0.1, 0.15 0.14, 0.12	0.13 0.13		DP-27438 Test 01
Wyoming, IL USA, 2009 (Imperial Gala)	3	0.15	12	1290	7	–0 0 1 3 6	whole fruit	0.066, 0.097 0.16, 0.18 0.2, 0.17 0.16, 0.15 0.12, 0.16	0.081 0.17 0.19 0.16 0.14		DP-27438 Trial 06

Table 73 Residues in apples from supervised trials in Europe involving foliar applications of cyantraniliprole (SE formulation unless specified)

APPLE Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg			Reference & Comments
	no	kg ai/ha	g ai/hL	water (L/ha)	RTI (days)			cyantraniliprole	mean	metabolites	
Bentley, Suffolk, UK South, 2010 (Spartan)	2	0.045	6	750	6–8	–0 0 7 14 27 55	whole fruit	0.042 0.06 0.052 0.041 0.02 ND			DP-27714 Test 21 [reverse decline]
KR Groesbeek, Limburg, Netherlands, 2010 (Elstar)	2	0.05	6	900	6–8	–0 0 7 14 28 56	whole fruit	0.034 0.13 0.06 0.041 0.026 0.015			DP-27714 Test 32 [reverse decline]
Dommartin, Rhone Alpes, S France, 2010 (Golden)	2	0.06	15	400	6	8	whole fruit	0.011			DP-27714 Test 30
Herlies, NordPas de Calais, N France, 2010 (Elstar)	2	0.06	15	400	7	7	whole fruit	0.058			DP-27714 Test 17
Herlies, NordPas de Calais, N France, 2010 (Jonagored)	2	0.06	6	1000	6–7	–0 0 7 13 28 55	whole fruit	0.039 0.14 0.052 0.042 0.013 ND			DP-27714 Test 25 [reverse decline]
Thurins, Rhone Alpes, S France, 2010 (Braeburn)	2	0.06	6	1000	6–8	–0 0 7 15 28 56	whole fruit	0.025 0.059 0.027 0.052 0.021 0.005			DP-27714 Test 23 [reverse decline]
Calatorao, Aragon Spain, 2009 (Starking)	2	0.07	5	1400	10	0 0 1 3 7 14	whole fruit	0.05 0.12 0.13 0.06 0.1 0.07			S09-01572 Test 04 [400 WG]
Meauzac, Midi Pyrénées S. France, 2010 (Pink Lady)	2	0.07	5	1400	10	0 1 3 7 14	whole fruit	0.06 0.07 0.04 0.04 <LOQ			S10-01052 Test 04 [400 WG]
Meauzac, Midi Pyrénées S. France, 2010 (Pink Lady)	2	0.07 (adjuvant)	5	1400	10	0 1 3 7 14	whole fruit	0.12 0.12 0.07 0.08 0.09			S10-01052 Test 04 [400 WG]

APPLE Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg			Reference & Comments
	no	kg ai/ha	g ai/hL	water (L/ha)	RTI (days)			cyantraniliprole	mean	metabolites	
Fuilla, Languedoc Roussillon S. France, 2010 (Golden)	2	0.075	5	1500	10	0 1 3 7 14	whole fruit	0.07 0.12 0.08 0.03 0.05			S10-01052 Test 03 [400 WG]
Hechthausen, Lower Saxony, Germany, 2009 (Delba)	2	0.075	5	1500	11	0 0 1 3 7 14	whole fruit	0.05 0.17 0.15 0.18 0.1 0.08			S09-01571 Test 02 [400 WG]
Heidelberg, BadenWürttemberg Germany, 2010 (Gala)	2	0.075	5	1500	10	0 1 3 7 14	whole fruit	0.08 0.07 0.06 0.05 0.03			S10-01051 Test 01
Heidelberg, BadenWürttemberg Germany, 2010 (Gala)	2	0.075 (adjuvant)	5	1400	10	0 1 3 7 14	whole fruit	0.1 0.07 0.04 0.03 0.02			S10-01051 Test 01 [400 WG]
Innenheim, Alsace N. France, 2009 (Elstar)	2	0.075	5	1500	10	0 1 3 7 14	whole fruit	0.1 0.09 0.08 0.06 0.03			S09-01571 Test 01 [400 WG]
Languedoc Roussillon S. France, 2010 (Golden)	2	0.075 (adjuvant)	5	1500	10	0 1 3 7 14	whole fruit	0.13 0.17 0.08 0.04 0.08			S10-01052 Test 03 [400 WG]
Ressen, Gelderland Netherlands, 2010 (var)	2	0.075	6	1260	7	7	whole fruit	0.045			DP-27714 Test 22
Stotzheim, Alsace N. France, 2010 (Ambassy)	2	0.075	5	1400	10	0 1 3 7 14	whole fruit	0.16 0.13 0.09 0.04 0.04			S10-01051 Test 02 [400 WG]
Stotzheim, Alsace N. France, 2010 (Ambassy)	2	0.075 (adjuvant)	5	1500	10	0 1 3 7 14	whole fruit	0.12 0.1 0.09 0.08 0.02			S10-01051 Test 02 [400 WG]
Castelnaudary, Languedoc Roussillon S. France, 2009 (Fuji)	2	0.08	5	1600	10	0 1 3 7 14	whole fruit	0.04 0.03 0.02 0.01 0.02			S09-01572 Test 03 [400 WG]

APPLE Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg			Reference & Comments
	no	kg ai/ha	g ai/hL	water (L/ha)	RTI (days)			cyantraniliprole	mean	metabolites	
Arbeca, Catalunya, N Spain, 2010 (Golden)	2	0.11	7.6	1500	7	–0 0 7 14 28 56	whole fruit	0.051 0.11 0.079 0.087 0.11 0.025			DP-27714 Test 26 [reverse decline]
Monleale, Piemonte, Italy, 2010 (Stark)	2	0.11	7.6	1500	7	–0 0 7 14 29 56	whole fruit	0.094 0.11 0.13 0.076 0.015 0.017			DP-27714 Test 24 [reverse decline]
Nea Trapezounda, Central Macedonia, Greece, 2010 (Fuji)	2	0.11	7.6	1500	7	7	whole fruit	0.12			DP-27714 Test 18
Calatorao, Aragon Spain, 2009 (Starking)	2	0.125	8.3	1500	10	0 0 1 3 7 14	whole fruit	0.12 0.22 0.13 0.21 0.19 0.1			S09-01572 Test 04 [400 WG]
Calatorao, Aragon Spain, 2009 (Starking)	2	0.125 (adjuvant)	8.3	1500	10	0 0 1 3 7 14	whole fruit	0.06 0.33 0.2 0.23 0.28 0.21			S09-01572 Test 04 [400 WG]
Castelnaudary, Languedoc Roussillon S. France, 2009 (Fuji)	2	0.125	8.6	1600	10	0 1 3 7 14	whole fruit	0.12 0.06 0.04 0.03 0.02			S09-01572 Test 03 [400 WG]
Castelnaudary, Languedoc Roussillon S. France, 2009 (Fuji)	2	0.125 (adjuvant)	8.3	1500	10	0 1 3 7 14	whole fruit	0.06 0.05 0.04 0.02 0.02			S09-01572 Test 03 [400 WG]
Hechthausen, Lower Saxony, Germany, 2009 (Delba)	2	0.125	8.3	1500	11	0 0 1 3 7 14	whole fruit	0.08 0.38 0.31 0.39 0.26 0.16			S09-01571 Test 02 [400 WG]
Hechthausen, Lower Saxony, Germany, 2009 (Delba)	2	0.125 (adjuvant)	8.3	1500	11	0 0 1 3 7 14	whole fruit	0.09 0.29 0.27 0.23 0.22 0.13			S09-01571 Test 02 [400 WG]

APPLE Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg			Reference & Comments
	no	kg ai/ha	g ai/hL	water (L/ha)	RTI (days)			cyantraniliprole	mean	metabolites	
Innenheim, Alsace N. France, 2009 (Elstar)	2	0.125	8.6	1600	10	0 1 3 7 14	whole fruit	0.24 0.14 0.17 0.13 0.08			S09-01571 Test 01 [400 WG]
Innenheim, Alsace N. France, 2009 (Elstar)	2	0.125 (adjuvant)	8.3	1500	10	0 1 3 7 14	whole fruit	0.18 0.18 0.18 0.09 0.09			S09-01571 Test 01 [400 WG]

Table 74 Residues in pears from supervised trials in North America involving foliar applications of cyantraniliprole (SE formulation)

PEAR Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Alton, NY USA, 2009 (Clapps Favorite)	3	0.15	13	1100	6, 7	3 7	whole fruit	0.21, 0.24 0.18, 0.15	0.23 0.16		DP-27438 Trial 17
Beamsville, ON CAN, 2009 (D'Anjou)	3	0.15	15	1000	7	3 7	whole fruit	0.078, 0.074 0.058, 0.071	0.077 0.065		DP-27438 Trial 18
Ephrata, WA USA, 2009 (Bartlett)	3	0.15	32	470	7, 8	3 7	whole fruit	0.56, 0.6 0.45, 0.46	0.58 0.45		DP-27438 Trial 26
Ephrata, WA USA, 2009 (Concord)	3	0.15	32	470	7	3 7	whole fruit	0.39, 0.48 0.37, 0.39	0.44 0.38		DP-27438 Trial 25
Hood River, OR USA, 2009 (Bartlett)	3	0.15	27	550	7	3 6	whole fruit	0.13, 0.11 0.12, 0.12	0.12 0.12		DP-27438 Trial 24
Hood River, OR USA, 2009 (Red Clapp)	3	0.15	7	2200	7	3 6	whole fruit	0.13, 0.15 0.11, 0.098	0.14 0.1		DP-27438 Trial 23
Lindsay, CA USA, 2009 (Olympic)	3	0.15	9	1700	7	3 8	whole fruit	0.16, 0.14 0.14, 0.17	0.15 0.16		DP-27438 Trial 21
Marysville, CA USA, 2009 (Bartlett)	3	0.15	11	1400	7	3 7	whole fruit	0.54, 0.3 0.39, 0.3	0.42 0.35		DP-27438 Trial 22
Shelby, MI USA, 2009 (Bartlett)	3	0.15	31	500	7	3 7	whole fruit	0.42, 0.65 0.59, 0.54	0.54 0.56		DP-27438 Trial 19
Simcoe, ON CAN, 2009 (Bartlett)	3	0.15	26	600	7	3 7	whole fruit	0.1, 0.09 0.034, 0.049	0.095 0.042		DP-27438 Trial 09

Table 75 Residues in pears from supervised trials in Europe involving foliar applications of cyantraniliprole (SE formulation)

PEAR Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg			Reference & Comments
	no	kg ai/ha	g ai/hL	water (L/ha)	RTI (days)			cyantraniliprole	mean	metabolites	
Bentley, Suffolk, UK South, 2010 (Conference)	2	0.045	6	750	6–8	–0 0 7 14 27 55	whole fruit	0.025 0.056 0.075 0.052 0.044 0.01			DP-27714 Test 31 [reverse decline]
Dommartin, Rhone Alpes, S France, 2010 (William)	2	0.06	6	1000	6–8	–0 0 6 14 28 55	whole fruit	0.038 0.084 0.081 0.033 0.02 0.013			DP-27714 Test 34 [reverse decline]
Herlies, NordPas de Calais, N France, 2010 (Angelys)	2	0.06	6	1000	6–7	–0 0 7 13 28 55	whole fruit	0.03 0.083 0.021 0.019 0.008 0.004			DP-27714 Test 33 [reverse decline]
Herlies, NordPas de Calais, N France, 2010 (Comice)	2	0.06	15	400	7	7	whole fruit	0.13			DP-27714 Test 28
Höhnstedt, SaxonyAnhalt, Germany, 2010 (Williams)	2	0.06	6	1000	8	7	whole fruit	0.063			DP-27714 Test 19
KR Groesbeek, Limburg, Netherlands, 2009 (Conference)	2	0.06	7.5	800	7	7	whole fruit	0.055			DP-27714 Test 10
Le Marne, Rhone Alpes, S France, 2010 (Packham)	2	0.06	15	400	6	7	whole fruit	0.081			DP-27714 Test 20
Elne, Languedoc Roussillon S. France, 2010 (Williams)	2	0.07	5	1400	10	0 1 3 7 14	whole fruit	0.12 0.1 0.13 0.11 0.06			S10-01054 Test 07 [400 WG]
Altedo Bologna Italy, 2010 (Decana)	2	0.075	5	1500	10	0 1 3 7 14	whole fruit	0.13 0.13 0.1 0.1 0.05			S10-01054 Test 08 [400 WG]

PEAR Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg			Reference & Comments
	no	kg ai/ha	g ai/hL	water (L/ha)	RTI (days)			cyantraniliprole	mean	metabolites	
Altedo Bologna Italy, 2010 (Decana)	2	0.075 (adjuvant)	5	1500	10	0 1 3 7 14	whole fruit	0.12 0.13 0.09 0.04 0.05			S10-01054 Test 08 [400 WG]
Bradfield Combust, Suffolk UK South, 2009 (Conference)	2	0.075	7.5	1000	6-9	–0 0 7 13 27 56	whole fruit	0.068 0.11 0.076 0.12 0.022 0.015			DP-27714 Test 14 [Reverse decline]
Calatorao, Aragon Spain, 2009 (Conference)	2	0.075	5	1500	10	0 0 1 3 7 14	whole fruit	0.03 0.11 0.12 0.11 0.04 0.05			S09-01574 Test 08 [400 SG]
Elne, Languedoc Roussillon S. France, 2010 (Williams)	2	0.075 (adjuvant)	5	1500	10	0 1 3 7 14	whole fruit	0.15 0.15 0.01 0.1 0.03			S10-01054 Test 07 [400 WG]
Elne, Languedoc Roussillon| S. France, 2009 (Williams)	2	0.075	5	1500	10	0 1 3 7 14	whole fruit	0.04 0.04 0.02 0.02 0.02			S09-01574 Test 07 [400 WG]
Herlies, NordPas de Calais, N France, 2009 (Conference)	2	0.075	19	400	6	9	whole fruit	0.11			DP-27714 Test 09
Herlies, NordPas de Calais, N France, 2009 (Angelys)	2	0.075	7.5	1000	5–7	–0 0 5 12 26 54	whole fruit	0.027 0.044 0.019 0.029 0.01 ND			DP-27714 Test 13 [Reverse decline]
Jork, Lower Saxony Germany, 2009 (Condo)	2	0.075	5	1500	10	0 0 1 3 7 14	whole fruit	0.02 0.13 0.13 0.08 0.05 0.04			S09-01573 Test 06 [400 WG]
Neuenschleuse, Niedersachsen Germany, 2010 (Hollandische Zuckerbirne)	2	0.075 (adjuvant)	5	1500	10	0 1 3 7 14	whole fruit	0.16 0.13 0.14 0.08 0.03			S10-01053 Test 05 [400 WG]

PEAR Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg			Reference & Comments
	no	kg ai/ha	g ai/hL	water (L/ha)	RTI (days)			cyantraniliprole	mean	metabolites	
Stotzheim, Alsace N. France, 2009 (Williams)	2	0.075	5	1500	10	0 1 3 7 14	whole fruit	0.06 0.04 0.03 0.02 0.02			S09-01573 Test 05 [400 WG]
Stotzheim, Alsace N. France, 2010 (Williams)	2	0.075	5	1500	10	0 1 3 7 14	whole fruit	0.12 0.16 0.08 0.04 0.03			S10-01053 Test 06 [400 WG]
Stotzheim, Alsace N. France, 2010 (Williams)	2	0.075 (adjuvant)	5	1500	10	0 1 3 7 14	whole fruit	0.19 0.12 0.08 0.05 0.04			S10-01053 Test 06 [400 WG]
Vergers des 4 Loups, Lot en Garonne, S France, 2009 (Passe Ciassome)	2	0.075	7.5	1000	6–8	–0 0 7 13 27 55	whole fruit	0.031 0.044 0.06 0.035 0.018 0.006			DP-27714 Test 15 [Reverse decline]
Dommartin, Rhône Alpes, S France, 2009 (William)	2	0.076	19	410	7	7	whole fruit	0.092			DP-27714 Test 12
Ablass, Saxony, Germany, 2010 (Williams)	2	0.08	6	1300	7	–0 0 7 14 28 56	whole fruit	0.04 0.097 0.042 0.029 0.023 0.004			DP-27714 Test 27 [reverse decline]
Neuenschleuse, Niedersachsen Germany, 2010 (Hollandische Zuckerbirne)	2	0.08	5	1600	10	0 1 3 7 14	whole fruit	0.08 0.06 0.08 0.06 0.01			S10-01053 Test 05 [400 WG]
Agia Triada, Central Macedonia, Greece, 2009 (Kristalia)	2	0.11	7.5	1450	7	7	whole fruit	0.099			DP-27714 Test 11
Akrolimni, Central Macedonia, Greece, 2010 (Williams)	2	0.11	7.6	1500	7	7	whole fruit	0.18			DP-27714 Test 29

PEAR Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg			Reference & Comments
	no	kg ai/ha	g ai/hL	water (L/ha)	RTI (days)			cyantraniliprole	mean	metabolites	
Bellvis, Catalunya, N Spain, 2010 (Ercolini)	2	0.11	7.6	1500	7	–0 0 7 14 28 56	whole fruit	0.095 0.17 0.16 0.11 0.045 0.009			DP-27714 Test 36 [reverse decline]
Bellvis, Catalunya, N Spain, 2009 (Conference)	2	0.11	7.5	1500	7–8	–0 0 7 14 29 55	whole fruit	0.12 0.26 0.15 0.071 0.067 0.017			DP-27714 Test 16 [Reverse decline]
Carpi, Emilia Romagna, Italy, 2010 (William)	2	0.11	7.6	1500	7	–0 0 7 14 28 56	whole fruit	0.037 0.1 0.048 0.032 0.057 0.004			DP-27714 Test 35 [reverse decline]
Calatorao, Aragon Spain, 2009 (Conference)	2	0.125	8.3	1500	10	0 0 1 3 7 14	whole fruit	0.02 0.2 0.19 0.08 0.09 0.03			S09-01574 Test 08 [400 SG]
Calatorao, Aragon Spain, 2009 (Conference)	2	0.125 (adjuvant)	8.3	1500	10	0 0 1 3 7 14	whole fruit	0.07 0.13 0.29 0.16 0.09 0.13			S09-01574 Test 08 [400 SG]
Elne, Languedoc Roussillon| S. France, 2009 (Williams)	2	0.125	8.3	1500	10	0 1 3 7 14	whole fruit	0.08 0.08 0.02 0.02 0.05			S09-01574 Test 07 [400 WG]
Elne, Languedoc Roussillon| S. France, 2009 (Williams)	2	0.125 (adjuvant)	8.3	1500	10	0 1 3 7 14	whole fruit	0.15 0.08 0.07 0.06 0.04			S09-01574 Test 07 [400 WG]
Jork, Lower Saxony Germany, 2009 (Condo)	2	0.125	8.3	1500	10	0 0 1 3 7 14	whole fruit	0.04 0.24 0.17 0.18 0.11 0.12			S09-01573 Test 06 [400 WG]

PEAR Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg			Reference & Comments
	no	kg ai/ha	g ai/hL	water (L/ha)	RTI (days)			cyantraniliprole	mean	metabolites	
Jork, Lower Saxony Germany, 2009 (Condo)	2	0.125 (adjuvant)	8.3	1500	10	0 0 1 3 7 14	whole fruit	0.06 0.21 0.26 0.17 0.16 0.11			S09-01573 Test 06 [400 WG]
Stotzheim, Alsace N. France, 2009 (Williams)	2	0.125	8.3	1500	10	0 1 3 7 14	whole fruit	0.09 0.06 0.07 0.06 0.04			S09-01573 Test 05 [400 WG]
Stotzheim, Alsace N. France, 2009 (Williams)	2	0.125 (adjuvant)	8.3	1500		0 1 3 7 14	whole fruit	0.09 0.07 0.06 0.07 0.04			S09-01573 Test 05 [400 WG]

Stonefruit

Results from supervised trials from Europe and North America on peaches, plums and cherries were provided to the Meeting.

North America

In trials conducted in North America, three foliar applications of 0.15 kg ai/ha cyantraniliprole (SE formulation) were applied at 7 day intervals, either as high volume (1000–2000 L/ha) or concentrate (100–800 L/ha) airblast applications, with adjuvant added.

Duplicate samples were stored at –20 °C for up to 7 months before analysis of flesh (within 101 days of extraction) for cyantraniliprole and six metabolites using analytical method DP-15736, with reported LOQs of 0.01 mg/kg. Average concurrent recoveries were 100–104% (cyantraniliprole) and 91–109% (metabolites) in samples spiked with 0.01, 0.1, 0.5 and/or 2 mg/kg. Residues in whole fruit were calculated from the flesh residue results and the relative flesh:stone weights.

Europe

In trials conducted in Europe on apricots, peaches and plums, two foliar applications of 0.15 kg ai/ha (10–30 g ai/hL) cyantraniliprole (SE or WG formulations) were applied 7 days apart, using 500–1500 L/ha, with and without added adjuvant. These trials included a number of reverse decline studies, where different plots were treated at staggered intervals so that the sampling dates coincided with normal commercial harvest.

Samples were stored at –18 °C for up to 9 months before analysis for cyantraniliprole and six to seven metabolites (within 1–23 days after extraction) using analytical method DP-15736, with reported LOQs of 0.01 mg/kg. Average concurrent recoveries were 82–96% (cyantraniliprole) and 79–96% (metabolites) in samples spiked with 0.01, 0.1 and 0.5 or 1.0 mg/kg. Residues in whole fruit were calculated from residues measured in the flesh, and the relative weights of flesh and stones.

Table 76 Residues in apricots from supervised trials in Europe involving foliar applications of cyantraniliprole (SE formulation)

APRICOT Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Mandalo Greece, 2009 (Bebeku)	2	0.15	10	1500	8	7	flesh whole fruit	0.36 0.34		M1=0.01 M1=0.01	DP-27717 Test 02
La Granja d'Escarp Spain, 2010 (Pinkoot)	2	0.15	30	500		–0 0 7 14 28 56 –0 0 7 14 28 56	flesh whole fruit 	0.17 0.17 0.34 0.25 0.19 0.065 0.16 0.16 0.32 0.24 0.18 0.062			DP-27717 Test 04 [reverse decline]
Seros Spain, 2010 (Traver)	2	0.15	10	1500		7 15 21 34 63 7 15 21 34 63	flesh whole fruit 	0.083 0.066 0.06 0.05 0.012 0.08 0.064 0.058 0.048 0.012			DP-27717 Test 09 [reverse decline]

M1: Residues of metabolite IN-J9Z38

Table 77 Residues in cherries from supervised trials in North America involving foliar applications of cyantraniliprole (SE formulation).

CHERRY Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Denair, CA USA, 2009 Sweet Cherry (Bing)	3	0.15	10	1500	7, 8	3 7 3 7	flesh whole fruit	0.32, 0.4 0.31, 0.36 0.28, 0.35 0.28, 0.32	0.36 0.34 <u>0.32</u> 0.3	M1=0.03 M1=0.04 M1=0.03 M1=0.04	DP-27437 Trial 28
Hart, MI USA, 2009 Tart Cherry (Montmorency)	3	0.15	28	500	7	3 7 3 7	flesh whole fruit	0.99, 0.79 0.75, 0.91 0.89, 0.71 0.66, 0.81	0.89 0.83 <u>0.8</u> 0.74		DP-27437 Trial 26

CHERRY Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Hood River, OR USA, 2009 Sweet Cherry (Selah)	3	0.15	26	600	7	3 7 3 7	flesh whole fruit	0.37, 0.29 0.21, 0.19 0.33, 0.26 0.19, 0.17	0.33 0.2 <u>0.3</u> 0.18		DP-27437 Trial 29
Orefield, PA USA, 2009 Tart Cherry (Montmorency)	3	0.15	6	2300	7, 6	3 7 3 7	flesh whole fruit	1.1, 0.87 0.96, 0.95 1.0, 0.8 0.87, 0.87	0.96 0.96 <u>0.9</u> 0.87	M1=0.01 M1=0.01 M1=0.01 M1=0.01	DP-27437 Test 23
Pentwater, MI USA, 2009 Tart Cherry (Montmorency)	3	0.15	30	500	7	3 7 3 7	flesh whole fruit	3.9, 3.6 1.3, 3.1 <u>3.5</u>, 3.2 1.2, 2.9	3.8 2.2 <u>3.4</u> 2.1	M1=0.03 M1=0.03 + M2=0.01 M1=0.02 M1=0.03	DP-27437 Trial 25
Perry, UT USA, 2009 Tart Cherry (Montmorency)	3	0.15	7	2000	6, 7	3 7 3 7	flesh whole fruit	1, 0.93 1, 0.73 0.92, 0.85 0.92, 0.67	0.98 0.88 <u>0.89</u> 0.8	M1=0.05 M1=0.05 M1=0.05 M1=0.05	DP-27437 Trial 27
Ridgeville, ON, Canada, 2009 Tart Cherry (Montmorency)	3	0.15	12	1200	7, 6	3 7 3 7	flesh whole fruit	0.93, 0.93 0.72, 0.83 0.67, 0.72 0.58, 0.65	0.93 0.77 <u>0.7</u> 0.62	M1=0.04 M1=0.04 M1=0.03 M1=0.03	DP-27437 Trial 24

M1: Average residues of metabolite IN-J9Z38

M2: Average residues of metabolite IN-MYX98

Table 78 Residues in peaches from supervised trials in North America involving foliar applications of cyantraniliprole (SE formulation)

PEACH Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Alexandria, LA USA, 2009 (June Prince)	3	0.15	9	1750	7	3 7 3 7	flesh whole fruit	0.17, 0.17 0.18, 0.22 0.16, 0.16 0.17, 0.21	0.17 0.2 0.16 <u>0.19</u>	M1=0.01 M1=0.015 M1=0.01	DP-27437 Trial 06
Branchton, ON Canada, 2009 (Reliance)	3	0.15	13	1200	7	3 7 3 7	flesh whole fruit	0.16, 0.21 0.15, 0.11 0.13, 0.18 0.13, 0.1	0.19 0.13 <u>0.16</u> 0.12		DP-27437 Trial 07
Clanton, AL USA, 2009 (Bounty)	1+ 2	0.15+ 0.15	120+ 60	120+ 250	7	3 7 3 7	flesh whole fruit	0.47, 0.66 0.32, 0.4 0.43, 0.58 0.3, 0.38	0.56 0.36 <u>0.51</u> 0.34		DP-27437 Trial 04

PEACH Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Hart, MI	3	0.15	30	500	7	3	flesh	1.4, 0.47	0.94	M1=0.01	DP-27437
USA, 2009						7		0.44, 0.67	0.56		Trial 08
(Baby Gold)											
						3	whole	1.2, 0.41	0.81		
						7	fruit	0.4, 0.61	0.51		
Hickman, CA	3	0.15	23	650	6, 7	3	flesh	0.31, 0.39	0.35		DP-27437
USA, 2009						7		0.35, 0.33	0.34		Trial 10
(Summerset)											
						3	whole	0.3, 0.37	0.34		
						7	fruit	0.34, 0.32	0.33		
Hondo, TX	3	0.15	35	450	7	3	flesh	0.73, 0.47	0.6	M1=0.02	DP-27437
USA, 2009						6		0.85, 0.93	0.89	M1=0.03	Trial 09
(Tex Royal)											
						3	whole	0.66, 0.43	0.55	M1=0.02	
						6	fruit	0.75, 0.82	0.79	M1=0.03	
Live Oak, CA	3	0.15	23	650	7	3	flesh	0.41, 0.27	0.34		DP-27437
USA, 2009						7		0.11, 0.1	0.1		Trial 11
(Halfords)											
						3	whole	0.34, 0.22	0.28		
						7	fruit	0.09, 0.09	0.09		
Madera, CA	3	0.15	11	1400	7	–0	flesh	0.17, 0.17	0.17		DP-27437
USA, 2009						0		0.25, 0.27	0.26		Trial 12
(Springcrest)						1		0.29, 0.21	0.25		
						3		0.22, 0.17	0.19		
						7		0.2, 0.15	0.18		
						–0	whole	0.15, 0.15	0.15		
						0	fruit	0.22, 0.24	0.23		
						1		0.26, 0.19	0.23		
						3		0.2, 0.15	0.18		
						7		0.18, 0.13	0.16		
Monetta, SC	3	0.15	20	700	7	3	flesh	0.25, 0.22	0.23		DP-27437
USA, 2009						7		0.18, 0.19	0.18		Trial 03
(Red Globe)											
						3	whole	0.19, 0.18	0.19		
						7	fruit	0.16, 0.15	0.16		
Oliver, BC,	3	0.15	15	1000	6	3	flesh	0.33, 0.52	0.42	M1=0.01	DP-27437
Canada, 2009						6		0.34, 0.31	0.32		Trial 13
(Red Haven)											
							whole	0.3, 0.47	0.39		
							fruit	0.3, 0.28	0.29		
Orefield, PA,	3	0.15	8	1800	6, 8	3	flesh	0.27, 0.23	0.25		DP-27437
USA, 2009						7		0.24, 0.21	0.23		Trial 01
(Glen Glo)											
						3	whole	0.24, 0.21	0.23		
						7	fruit	0.22, 0.19	0.21		
Pikeville, NC,	3	0.15	13	1200	7, 6	3	flesh	0.52, 0.47	0.49	M1=0.02	DP-27437
USA, 2009						6		0.37, 0.46	0.41	M1=0.02	Trial 02
(New Haven)											
						3	whole	0.47, 0.42	0.45	M1=0.02	
						6	fruit	0.33, 0.41	0.37	M1=0.02	

PEACH Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Ridge Spring, SC USA, 2009 (Contender)	3	0.15	24	630	7	3	flesh	0.3, 0.23	0.27		DP-27437 Trial 05
						7		0.13, 0.2	0.17		
						3	whole fruit	0.27, 0.21	0.24		
						7		0.12, 0.19	0.16		

M1: Average residues of metabolite IN-J9Z38

Table 79 Residues in peaches from supervised trials in Europe involving foliar applications of cyantraniliprole (SE formulation)

PEACH Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Altedo Italy, 2009 (Red Coast)	2	0.075	5	1500	7	0	whole fruit	0.07			S09-01576 Test 04a 400 WG
						1		0.13			
						3		0.08			
						7		0.06			
						14		0.04			
Barbiano Italy, 2010 (Big top)	2	0.075	5	1500	7	0	whole fruit	0.12			S10-01055 Test 2a 400 WG
						1		0.07			
						3		0.05			
						7		0.05			
						14		0.02			
Calatorao Spain, 2009 (Miraflores)	2	0.075	5	1500	7	0	whole fruit	0.08			S09-01576 Test 03a 400 WG
						0		0.13			
						1		0.13			
						3		0.13			
						7		0.13			
						14		0.05			
Calatorao Spain, 2009 (Sudanell)	2	0.075	5	1500	7	0	whole fruit	0.06			S09-01576 Test 02a 400 WG
						0		0.13			
						1		0.12			
						3		0.12			
						7		0.09			
						14		0.05			
Corbère S France, 2010 (Corindon)	2	0.075	5	1500	7	0	whole fruit	0.32			S10-01055 Test 1a 400 WG
						1		0.31			
						3		0.24			
						7		0.13			
						14		0.1			
Corbère S. France, 2009 (Corindon)	2	0.075	5	1500	7	0	whole fruit	0.02			S09-01576 Test 01a 400 WG
						1		0.12			
						3		0.11			
						7		0.05			
						14		0.08			
El Coronil Spain, 2010 (Red Robin)	2	0.075	5	1500	7	0	whole fruit	0.29			S10-01055 Test 4a 400 WG
						1		0.19			
						3		0.16			
						7		0.1			
						14		0.05			

PEACH Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Valdelacalzada Spain, 2010 (Spring lady)	2	0.075	5	1500	7	0 1 3 7 14	whole fruit	0.48 0.23 0.1 0.13 0.09			S10-01055 Test 3a 400 WG
Barbiano Italy, 2010 (Big top)	2	0.125	8.3	1500	7	0 1 3 7 14	whole fruit	0.2 0.19 0.17 0.1 0.04			S10-01055 Test 2b 400 WG
Corbère S France, 2010 (Corindon)	2	0.125	8.3	1500	7	0 1 3 7 14	whole fruit	0.39 0.63 0.5 0.26 0.21			S10-01055 Test 1b 400 WG
El Coronil Spain, 2010 (Red Robin)	2	0.125	8.3	1500	7	0 1 3 7 14	whole fruit	0.42 0.26 0.37 0.26 0.09			S10-01055 Test 4b 400 WG
Valdelacalzada Spain, 2010 (Spring lady)	2	0.125	8.3	1500	7	0 1 3 7 14	whole fruit	0.48 0.34 0.28 0.18 0.14			S10-01055 Test 3b 400 WG
Barbiano Italy, 2010 (Big top)	2	0.125+ surfact	8.3	1500	7	0 1 3 7 14	whole fruit	0.19 0.18 0.15 0.11 0.06			S10-01055 Test 2c 400 WG
Corbère S France, 2010 (Corindon)	2	0.125+ surfact	8.3	1500	7	0 1 3 7 14	whole fruit	0.56 0.29 0.25 0.35 0.14			S10-01055 Test 1c 400 WG
El Coronil Spain, 2010 (Red Robin)	2	0.125+ surfact	8.3	1500	7	0 1 3 7 14	whole fruit	0.3 0.25 0.36 0.22 0.1			S10-01055 Test 4c 400 WG
Valdelacalzada Spain, 2010 (Spring lady)	2	0.125+ surfact	8.3	1500	7	0 1 3 7 14	whole fruit	0.59 0.35 0.33 0.21 0.17			S10-01055 Test 3c 400 WG
Alcarras Spain 2009 (Romestart)	2	0.15	10	1500	6	7	flesh whole fruit	0.2 0.19		M1=0.01 M1=0.01	DP-27717 Test 01

PEACH Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Altedo Italy, 2009 (Red Coast)	2	0.15	10	1500	7	0 1 3 7 14	whole fruit	0.11 0.2 0.14 0.19 0.14			S09-01576 Test 04b 400 WG
Calatorao Spain, 2009 (Miraflores)	2	0.15	10	1500	7	0 0 1 3 7 14	whole fruit	0.12 0.2 0.35 0.3 0.19 0.15			S09-01576 Test 03b 400 WG
Calatorao Spain, 2009 (Sudanell)	2	0.15	10	1500	7	0 0 1 3 7 14	whole fruit	0.17 0.47 0.49 0.23 0.19 0.29			S09-01576 Test 02b 400 WG
Corbère S. France, 2009 (Corindon)	2	0.15	10	1500	7	0 1 3 7 14	whole fruit	0.19 0.36 0.12 0.08 0.1			S09-01576 Test 01b 400 WG
Galatades Greece, 2010 (Katerina)	2	0.15	10	1500		7	flesh whole fruit	0.3 0.28		M1=0.01 M1=0.01	DP-27717 Test 05
Lizac S France, 2009 (Plat)	2	0.15	10	1500		7	flesh whole fruit	0.023 0.02			DP-27717 Test 06
Poulariot S France, 2010 (Plat Sweetcap)	2	0.15	15	1000		–0 0 6 14 28 56 –0 0 6 14 28 56	flesh whole fruit	0.034 0.055 0.032 0.026 0.035 0.014 0.031 0.05 0.029 0.024 0.032 0.013			DP-27717 Test 07 [reverse decline]

PEACH Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Volpedo Italy, 2010 (Cresthaven)	2	0.15	10	1500		–0 0 7 15 29 56 –0 0 7 15 29 56	flesh whole fruit 	0.089 0.31 0.14 0.058 0.078 0.027 0.081 0.28 0.13 0.053 0.07 0.024			DP-27717 Test 08 [reverse decline]
Volpedo Italy, 2009 (Cresthaven)	2	0.15	10	1500	5–7	–0 0 7 14 28 56 –0 0 7 14 28 56	flesh whole fruit 	0.096 0.18 0.18 0.13 0.074 0.046 0.092 0.17 0.17 0.12 0.07 0.044			DP-27717 Test 03 [reverse decline]
Altedo Italy, 2009 (Red Coast)	2	0.15+ surfact	10	1500	7	0 1 3 7 14	whole fruit 	0.15 0.31 0.16 0.18 0.14			S09-01576 Test 04c 400 WG
Calatorao Spain, 2009 (Miraflores)	2	0.15+ surfact	10	1500	7	0 0 1 3 7 14	whole fruit 	0.18 0.16 0.34 0.21 0.31 0.16			S09-01576 Test 03c 400 WG
Calatorao Spain, 2009 (Sudanell)	2	0.15+ surfact	10	1500	7	0 0 1 3 7 14	whole fruit 	0.15 0.33 0.54 0.29 0.25 0.14			S09-01576 Test 02c 400 WG
Corbère S. France, 2009 (Corindon)	2	0.15+ surfact	10	1500	7	0 1 3 7 14	whole fruit 	0.2 0.13 0.09 0.11 0.12			S09-01576 Test 01c 400 WG

M1: Residues of metabolite IN-J9Z38

Table 80 Residues in plums from supervised trials in North America involving foliar applications of cyantraniliprole (SE formulation)

PLUM Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Beamville, ON, Canada, 2009 (Vibrant V70034)	3	0.15	26	600	6, 7	3 7 3 7	flesh whole fruit	0.065, 0.077 0.061, 0.057 0.06, 0.07 0.06, 0.05	0.07 0.06 <u>0.07</u> 0.06		DP-27437 Trial 15
Conklin, MI USA, 2009 (Vision)	3	0.15	7	2000	7	3 7 3 7	flesh whole fruit	0.23, 0.17 0.17, 0.14 0.22, 0.16 0.16, 0.14	0.2 0.15 <u>0.19</u> 0.15		DP-27437 Trial 14
Hughson, CA USA, 2009 (French Plum)	3	0.15	11	1300	7	3 7 3 7	flesh whole fruit	0.3, 0.26 0.29, 0.3 0.28, 0.24 0.27, 0.28	0.28 0.29 0.26 <u>0.28</u>		DP-27437 Trial 16
Live Oak, CA USA, 2009 (French)	3	0.15	9	1700	7	3 7 3 7	flesh whole fruit	0.043, 0.046 0.046, 0.055 0.041, 0.044 0.043, 0.052	0.04 0.05 0.04 <u>0.05</u>		DP-27437 Trial 20
Madera, CA USA, 2009 (Fortune)	3	0.15	23	650	7	2 7 2 7	flesh whole fruit	0.065, 0.063 0.059, 0.062 0.063, 0.059 0.056, 0.058	0.06 0.06 <u>0.06</u> 0.06		DP-27437 Trial 17
Madera, CA USA, 2009 (Red Beauty)	3	0.15	23	650	6	3 7 3 7	flesh whole fruit	0.028, 0.035 0.028, 0.028 0.026, 0.032 0.025, 0.025	0.03 0.03 <u>0.03</u> 0.03		DP-27437 Trial 18
Monmouth, OR USA, 2009 (Moyer)	3	0.15	10	1500	6, 7	3 7 3 7	flesh whole fruit	0.059, 0.073 0.06, 0.051 0.056, 0.07 0.058, 0.049	0.07 0.06 <u>0.06</u> 0.05		DP-27437 Trial 22
Payette, ID USA, 2009 (Empress)	3	0.15	11	1400	6, 7	3 7 3 7	flesh whole fruit	0.13, 0.13 0.11, 0.12 0.12, 0.12 0.11, 0.11	0.13 0.12 <u>0.12</u> 0.11		DP-27437 Trial 21
Sanger, CA USA, 2009 (Santa Rosa)	3	0.15	12	1250	7	3 7 3 7	flesh whole fruit	0.043, 0.06 0.073, 0.048 0.037, 0.054 0.067, 0.045	0.05 0.06 0.05 <u>0.06</u>		DP-27437 Trial 19

Table 81 Residues in plums from supervised trials in Europe involving foliar applications of cyantraniliprole (SE formulation)

PLUM Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Calatorao Spain, 2009 (Anaspar)	2	0.075	5	1500	7	0 0 1 3 7 14	whole fruit	0.01 0.02 0.03 0.01 0.01 0.01			S09-01578 Test 10a 400 WG
Dirmstein Germany, 2010 (Czadczaks Schöne)	2	0.075	5	1500	7	0 1 3 7 14	whole fruit	0.33 0.02 0.03 0.02 0.01			S10-01056 Test 6a 400 WG
Innenheim N France, 2010 (Quetsches d'Alsace)	2	0.075	5	1500	7	0 1 3 7 14	whole fruit	0.12 0.12 0.12 0.09 0.06			S10-01056 Test 7a 400 WG
Innenheim N. France, 2009 (Quetsche)	2	0.075	5	1500	7	0 1 3 7 14	whole fruit	0.13 0.15 0.14 0.13 0.11			S09-01577 Test 05a 400 WG
Jork Germany, 2010 (Bühler)	2	0.075	5	1500	7	0 1 3 7 14	whole fruit	< 0.01 < 0.01 ND ND ND			S10-01056 Test 5a 400 WG
Moissac S. France, 2009 (Royal)	2	0.075	5	1500	7	0 1 3 7 14	whole fruit	0.06 0.03 0.03 0.02 0.02			S09-01578 Test 09a 400 WG
Ortenberg Germany, 2009 (Presenta)	2	0.075	5	1500	7	0 0 1 3 7 14	whole fruit	< 0.01 0.01 0.03 0.02 0.01 0.01			S09-01577 Test 08a 400 WG
Quatretonda La Costera Spain, 2010 (Black Diamond)	2	0.075	5	1500	7	0 1 3 7 14	whole fruit	0.02 0.02 < 0.01 < 0.01 < 0.01			S10-01057 Test 10a 400 WG
Saint Aignan S France, 2010 (Golden Japan)	2	0.075	5	1500	7	0 1 3 7 14	whole fruit	ND ND ND ND ND			S10-01057 Test 9a 400 WG

PLUM Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Stade Germany, 2009 (Schoneberger)	2	0.075	5	1500	7	0 0 1 3 7 14	whole fruit	0.02 0.06 0.06 0.06 0.04 0.02			S09-01577 Test 07a 400 WG
Stotzheim N France, 2010 (Elena)	2	0.075	5	1500	7	0 1 3 7 14	whole fruit	0.06 0.07 0.09 0.06 0.03			S10-01056 Test 8a 400 WG
Stozheim N. France, 2009 (Elena)	2	0.075	5	1500	7	0 1 3 7 14	whole fruit	0.08 0.1 0.11 0.1 0.08			S09-01577 Test 06a 400 WG
Dirmstein Germany, 2010 (Czadczaks Schöne)	2	0.125	8.3	1500	7	0 1 3 7 14	whole fruit	0.15 0.08 0.07 0.04 0.04			S10-01056 Test 6b 400 WG
Innenheim N France, 2010 (Quetsches d'Alsace)	2	0.125	8.3	1500	7	0 1 3 7 14	whole fruit	0.25 0.22 0.23 0.15 0.12			S10-01056 Test 7b 400 WG
Jork Germany, 2010 (Bühler)	2	0.125	8.3	1500	7	0 1 3 7 14	whole fruit	0.02 < 0.01 < 0.01 < 0.01 ND			S10-01056 Test 5b 400 WG
Quatretonda La Costera Spain, 2010 (Black Diamond)	2	0.125	8.3	1500	7	0 1 3 7 14	whole fruit	0.04 0.02 0.02 0.02 0.02			S10-01057 Test 10b 400 WG
Saint Aignan S France, 2010 (Golden Japan)	2	0.125	8.3	1500	7	0 1 3 7 14	whole fruit	ND ND ND ND ND			S10-01057 Test 9b 400 WG
Stotzheim N France, 2010 (Elena)	2	0.125	8.3	1500	7	0 1 3 7 14	whole fruit	0.18 0.16 0.16 0.1 0.08			S10-01056 Test 8b 400 WG
Dirmstein Germany, 2010 (Czadczaks Schöne)	2	0.125+ surfact	8.3	1500	7	0 1 3 7 14	whole fruit	0.05 0.23 0.15 0.11 0.09			S10-01056 Test 6c 400 WG

PLUM Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Innenheim N France, 2010 (Quetsches d'Alsace)	2	0.125+ surfact	8.3	1500	7	0 1 3 7 14	whole fruit	0.43 0.34 0.37 0.18 0.17			S10-01056 Test 7c 400 WG
Jork Germany, 2010 (Bühler)	2	0.125+ surfact	8.3	1500	7	0 1 3 7 14	whole fruit	0.03 0.01 0.02 0.01 0.02			S10-01056 Test 5c 400 WG
Quatretonda La Costera Spain, 2010 (Black Diamond)	2	0.125+ surfact	8.3	1500	7	0 1 3 7 14	whole fruit	0.06 0.06 0.06 0.07 0.05			S10-01057 Test 10c 400 WG
Saint Aignan S France, 2010 (Golden Japan)	2	0.125+ surfact	8.3	1500	7	0 1 3 7 14	whole fruit	< 0.01 ND ND ND ND			S10-01057 Test 9c 400 WG
Stotzheim N France, 2010 (Elena)	2	0.125+ surfact	8.3	1500	7	0 1 3 7 14	whole fruit	0.21 0.24 0.27 0.12 0.17			S10-01056 Test 8c 400 WG
Calatorao Spain, 2009 (Anaspar)	2	0.15	10	1500	7	0 0 1 3 7 14	whole fruit	0.02 0.09 0.06 0.04 0.05 0.06			S09-01578 Test 10b 400 WG
Innenheim N. France, 2009 (Quetsche)	2	0.15	10	1500	7	0 1 3 7 14	whole fruit	0.33 0.4 0.32 0.26 0.3			S09-01577 Test 05b 400 WG
Moissac S. France, 2009 (Royal)	2	0.15	10	1500	7	0 1 3 7 14	whole fruit	0.1 0.1 0.08 0.06 0.06			S09-01578 Test 09b 400 WG
Ortenberg Germany, 2009 (Presenta)	2	0.15	10	1500	7	0 0 1 3 7 14	whole fruit	0.01 0.07 0.05 0.02 0.03 0.05			S09-01577 Test 08b 400 WG
Stade Germany, 2009 (Schoneberger)	2	0.15	10	1500	7	0 0 1 3 7 14	whole fruit	0.05 0.11 0.12 0.15 0.07 0.03			S09-01577 Test 07b 400 WG

PLUM Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Stozheim N. France, 2009 (Elena)	2	0.15	10	1500	7	0 1 3 7 14	whole fruit	0.17 0.17 0.13 0.11 0.19			S09-01577 Test 06b 400 WG
Calatorao Spain, 2009 (Anaspar)	2	0.15+ surfact	10	1500	7	0 0 1 3 7 14	whole fruit	0.04 0.13 0.13 0.13 0.08 0.08			S09-01578 Test 10c 400 WG
Innenheim N. France, 2009 (Quetsche)	2	0.15+ surfact	10	1500	7	0 1 3 7 14	whole fruit	0.57 0.59 0.51 0.42 0.49			S09-01577 Test 05c 400 WG
Moissac S. France, 2009 (Royal)	2	0.15+ surfact	10	1500	7	0 1 3 7 14	whole fruit	0.19 0.17 0.14 0.23 0.16			S09-01578 Test 09c 400 WG
Ortenberg Germany, 2009 (Presenta)	2	0.15+ surfact	10	1500	7	0 0 1 3 7 14	whole fruit	0.04 0.17 0.17 0.13 0.1 0.03			S09-01577 Test 08c 400 WG
Stade Germany, 2009 (Schoneberger)	2	0.15+ surfact	10	1500	7	0 0 1 3 7 14	whole fruit	0.07 0.17 0.23 0.21 0.12 0.09			S09-01577 Test 07c 400 WG
Stozheim N. France, 2009 (Elena)	2	0.15+ surfact	10	1500	7	0 1 3 7 14	whole fruit	0.3 0.39 0.45 0.31 0.17			S09-01577 Test 06c 400 WG

Blueberries

In trials conducted on blueberries in North America, three foliar applications of 0.15 kg ai/ha cyantraniliprole (SE formulation) were applied at 4–7 day intervals, using between 200 and 650 L/ha (with one trial at 50 L/ha), with adjuvant added.

Duplicate samples were stored at –20 °C for up to 6 months before analysis (within 23 days of extraction) for cyantraniliprole and six metabolites using analytical method DP-15736, with reported LOQs of 0.01 mg/kg. Average concurrent recoveries were 101–104% (cyantraniliprole) and 95–109% (metabolites) in samples spiked with 0.01 and 0.1 mg/kg.

Table 82 Residues in blueberries from supervised trials in North America involving foliar applications of cyantraniliprole (SE formulation)

BLUEBERRY Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Aldergrove, BC, CAN, 2009 (Briggitta)	3	0.15	34	440	6, 5	3	berries	0.53, 0.5	0.52		AAFC09-025R Trial 268
East Gove, NS, CAN, 2009 (Wild Clones)	3	0.15	50	300	5, 4	3	berries	1.9, 2.0	2.0		AAFC09-025R Trial 264
Frelighsburg, QC, CAN, 2009 (Bluecrop)	3	0.15	50	300	6, 4	3	berries	0.73, 0.87	0.8		AAFC09-025R Trial 266 early treatments
Frelighsburg, QC, CAN, 2009 (Bluecrop)	3	0.15	50	300	5	4	berries	0.64, 0.85	0.75		AAFC09-025R Trial 267 late treatments
Kinston NC, USA, 2009 (Blue Haven)	3	0.15	330	47	5	3	berries	0.61, 0.43	0.52		DP-27971 Trial 02
Rawdon, NS, CAN, 2009 (Wild Clones)	3	0.15	50	300	5, 4	3	berries	1.4, 1.6	1.5	M1=0.01 M2=0.01	AAFC09-025R Trial 261 early treatments
Rawdon, NS, CAN, 2009 (Wild Clones)	3	0.15	50	300	4, 3	4	berries	0.37, 0.46	0.42	M1=0.02 M2=0.01	AAFC09-025R Trial 263 late treatments
Scotland, ON, CAN, 2009 (Duke)	3	0.15	75	200	6, 4	0 4 7 10	berries	1.0, 1.1 0.47, 0.55 0.31, 0.3 0.25, 0.23	1.1 0.51 0.31 0.24		AAFC09-025R Trial 265
Sheffield Mills, NS, CAN, 2009 (Wild Clones)	3	0.15	50	300	5, 6	0 2 7 8	berries	0.74, 0.58 0.66, 0.55 0.22, 0.25 0.19, 0.18	0.66 0.60 0.23 0.19	 M1=0.01 M1=0.01	AAFC09-025R Trial 262
Wetumpka, AL USA, 2009 (Tifblue)	3	0.15	24	630	7, 8	3	berries	1.5, 1.5	1.5	M1=0.01 M2=0.01	DP-27971 Trial 01

M1: Average residues of metabolite IN-J9Z38

M2: Average residues of metabolite IN-MYX98

Grapes

In trials on wine grapes conducted in Europe, two foliar applications of 0.12–0.15 kg ai/ha cyantraniliprole (SE formulation) were applied at 14 day intervals, using 1500 L/ha (300 L/ha in two trials), with no added adjuvant. These trials were reverse decline studies, where treatments were applied at staggered intervals to align the sampling times with the commercial harvest date.

Samples were stored at –18 °C for up to 10 months before analysis of berries (within 5 days of extraction) for cyantraniliprole and six metabolites using analytical method DP-15736, with reported LOQs of 0.01 mg/kg. Average concurrent recoveries were 92–94% (cyantraniliprole) and 88–96% (metabolites) in samples spiked with 0.01 and 0.1 mg/kg.

Table 83 Residues in grapes from supervised trials in Europe involving foliar applications of cyantraniliprole (SE formulation)

GRAPES Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Boxted UK, 2010 (Baccus)	2	0.12	8	1500	14–15	–0 0 10 22 29 56	grapes	0.053 0.13 0.096 0.059 0.039 0.034			DP-27718 Trial 19 [reverse decline]
Höhnstedt Germany, 2010 (Müller-Thurgau)	2	0.12	8	1500	14	–0 0 10 21 28 56	grapes	0.033 0.34 0.11 0.14 0.12 0.045			DP-27718 Trial 20 [reverse decline]
Kesten Germany, 2010 (Riesling)	2	0.12	8	1500	14–15	–0 0 10 21 28 56	grapes	0.16 0.23 0.24 0.22 0.21 0.12			DP-27718 Trial 21 [reverse decline]
La Roche Vineuse N France, 2010 (Gamay)	2	0.12	40	300	13–15	–0 0 10 21 28 56	grapes	0.061 0.13 0.16 0.16 0.047 0.047			DP-27718 Trial 15 [reverse decline]
Miradolo Terme Italy, 2009 (Bonarda)	2	0.12	8	1500	13–14	–0 0 10 21 28 56	grapes	0.45 0.99 0.8 0.63 0.45 0.21			DP-27718 Trial 16 [reverse decline]
Veldenz Germany, 2010 (Müller-Thurgau)	2	0.12	8	1500	13–15	–0 0 10 21 28 56	grapes	0.22 0.64 0.49 0.19 0.22 0.14			DP-27718 Trial 22 [reverse decline]
Verdu Spain, 2010 (Macabeu)	2	0.12	8	1500	13–14	–0 0 10 21 28 56	grapes	0.06 0.16 0.48 0.14 0.19 0.056			DP-27718 Trial 18 [reverse decline]
Verdu Spain, 2010 (Ull de Llebre)	2	0.12	8	1500	14	–0 0 10 21 28 56	grapes	0.13 0.4 0.13 0.19 0.074 0.024			DP-27718 Trial 17 [reverse decline]

GRAPES Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Villié Morgon S France, 2010 (Gamay)	2	0.12	8	1500	13–15	–0 0 10 21 28 56	grapes	0.021 0.073 0.071 0.036 0.028 0.032			DP-27718 Trial 14 [reverse decline]
Guimera Spain, 2009 (Tempranillo)	2	0.15	10	1500	13–14	–0 0 10 21 28 56	grapes	0.062 0.065 0.21 0.18 0.079 0.077			DP-27718 Trial 07 [reverse decline]
Höhnstedt Germany, 2009 (Gutedel)	2	0.15	10	1500	14	–0 0 10 21 28 56	grapes	0.29 0.42 0.33 0.27 0.15 0.17			DP-27718 Trial 03 [reverse decline]
Kato Milia Greece, 2009 (Muscat)	2	0.15	10	1500	14	–0 0 10 21 28 56	grapes	0.14 0.32 0.28 0.076 0.11 0.029			DP-27718 Trial 01 [reverse decline]
Kato Milia Greece, 2010 (Muscat)	2	0.15	10	1500	14	–0 0 10 21 28 56	grapes	0.15 0.33 0.41 0.31 0.18 0.071			DP-27718 Trial 13 [reverse decline]
Marfaux N France, 2009 (Chardonnay)	2	0.15	50	300	14–15	–0 0 10 21 28 56	grapes	0.023 0.071 0.07 0.034 0.04 0.047			DP-27718 Trial 08 [reverse decline]
Mersea Island UK, 2009 (Reichensteiner)	2	0.15	10	1500	14	–0 0 10 21 28 56	grapes	0.11 0.23 0.3 0.16 0.16 0.1			DP-27718 Trial 04 [reverse decline]
Miradolo Terme Italy, 2009 (Bonarda)	2	0.15	10	1500	13–15	–0 0 10 21 28 56	grapes	0.34 0.65 0.56 0.48 0.67 0.63			DP-27718 Trial 06 [reverse decline]

GRAPES Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Veldenz Germany, 2009 (Kerner)	2	0.15	10	1500	13–15	–0 0 10 21 28 56	grapes	0.16 0.46 0.36 0.3 0.68 0.19			DP-27718 Trial 05 [reverse decline]
Villié Morgon S France, 2009 (Gamay)	2	0.15	10	1500	14	–0 0 10 21 28 56	grapes	0.05 0.24 0.14 0.11 0.096 0.05			DP-27718 Trial 02 [reverse decline]

Olives

In trials on olives conducted in Europe, two foliar applications of 0.15 kg ai/ha cyantraniliprole (SE formulation) were applied at 10 day intervals, using 2000 or 3000 L/ha with added adjuvant.

After the olive samples were weighed and and the stoned removed in the field, flesh samples were stored at –18 °C for up to 17 months before analysis (within 7 days of extraction) for cyantraniliprole and six metabolites using analytical method DP-15736, with reported LOQs of 0.01 mg/kg. Average concurrent recoveries were 88–94% (cyantraniliprole) and 86–108% (metabolites) in samples spiked with 0.01 and 0.1 mg/kg. Whole fruit residues were calculated from the relative flesh:stone weights.

Table 84 Residues in olives from supervised trials in Europe involving foliar applications of cyantraniliprole (SE formulation)

OLIVES Location Country, year (variety)	Application					DAT (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)		cyantraniliprole		metabolites		
							flesh	whole fruit	flesh	whole fruit	
Agios Mamas Greece, 2009 (Chondrolia Chalkidikis)	2	0.15	7.5	2000	9	14	0.47	0.36	M1=0.013	M1=0.01	DP-27709 Trial 1
Aguadulce Spain, 2010 (Hojiblanca)	2	0.15	5	3000	10	14	0.3	0.25			DP-27709 Trial 10
Aguadulce Spain, (Hojiblanca)	2	0.15	7.5	2000	14	–0 0 3 7 14	0.17 0.53 0.53 0.64 0.41	0.11 0.34 0.35 0.41 0.26	 M1=0.012		DP-27709 Trial 4
Bardolino Italy, 2009 (Casaliva)	2	0.15	7.5	2000	9	–0 0 3 7 14	0.39 0.84 0.18 0.19 0.3	0.26 0.53 0.14 0.13 0.21			DP-27709 Trial 3
Bardolino Italy, 2010 (Leccino)	2	0.15	5	3100	13	14	0.39	0.27			DP-27709 Trial 9

OLIVES Location Country, year (variety)	Application					DAT (days)	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)		cyantraniliprole		metabolites		
							flesh	whole fruit	flesh	whole fruit	
Fuente de Piedra Spain, 2010 (Hojiblanca)	2	0.15	5	3000	9	–0	0.26	0.2			DP-27709 Trial 6
						0	0.82	0.57			
						3	0.62	0.47			
						7	0.54	0.41	M1=0.1	M1=0.075	
						14	0.63	0.47	M1=0.02	M1=0.015	
Nea Skion, Greece, 2010 (Chondroelia Chalkidikis)	2	0.15	5	3000	11	–0	0.29	0.21			DP-27709 Trial 5
						0	0.64	0.44			
						3	0.49	0.36	M1=0.011		
						7	0.41	0.31			
						14	0.27	0.21	M1=0.013	M1=0.1	
Venterol S France, 2010 (Tanche)	2	0.15	5	3000	10	14	0.66	0.38			DP-27709 Trial 7
Venterol S France, (Tanche)	2	0.15	7.5	2000	11	–0	0.85	0.49			DP-27709 Trial 2
						0	2.3	1.4			
						3	2.4	1.5			
						7	2.3	1.3			
						14	1.9	1.1			

M1: Residues of metabolite IN-J9Z38

Pomegranate

In trials on pomegranates conducted in India, two to five foliar applications of 0.075–0.18 kg ai/ha cyantraniliprole (OD formulation) were applied at 10 day intervals, using 400–1000 L/ha with added adjuvant

Samples were stored at –20 °C for up to 1 month before analysis for cyantraniliprole and the IN-J9Z38 metabolite using analytical method DP-15736, with reported LOQs of 0.005 mg/kg. Average concurrent recoveries were 83–93% (cyantraniliprole) and 85–92% (IN-J9Z38) in samples spiked with 0.005, 0.025 and 0.05 mg/kg.

Table 85 Residues in pomegranates from supervised trials in India involving foliar applications of cyantraniliprole (OD formulation)

POMEGRANATE Location Country, year (variety)	Application					DAT (days)	Cyantraniliprole residues (mg/kg)				Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)		rind (parent)	rind M1	seed	juice	
Raichur India, 2011	2	0.075	12.5–19	400–600	10	0	0.05		ND	ND	DP-1104829 Trial 1
						1	0.03	0.03	ND	ND	
						3	0.006		ND	ND	
						5	ND		ND	ND	
Raichur India, 2011	2	0.09	15–23	400–600	10	0	0.07		ND	ND	DP-1104829 Trial 1
						1	0.03	M1=0.035	ND	ND	
						3	0.008		ND	ND	
						5	ND		ND	ND	
Raichur India, 2011	2	0.18	30–45	400–600	10	0	0.14		ND	ND	DP-1104829 Trial 1
						1	0.07	M1=0.065	ND	ND	
						3	0.01		ND	ND	
						5	ND		ND	ND	

POMEGRANATE Location Country, year (variety)	Application					DAT (days)	Cyantraniliprole residues (mg/kg)				Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)		rind (parent)	rind M1	seed	juice	
Medhak India, 2011	3	0.075	7.5	1000	10	0	0.04		ND	ND	DP-1104829 Trial 3
						1	0.03	M1=0.02	ND	ND	
						3	0.005		ND	ND	
						5	ND		ND	ND	
Medhak India, 2011	3	0.09	9	1000	10	0	0.05		ND	ND	DP-1104829 Trial 3
						1	0.02	M1=0.02	ND	ND	
						3	0.006		ND	ND	
						5	ND		ND	ND	
Medhak India, 2011	3	0.18	18	1000	10	0	0.09		ND	ND	DP-1104829 Trial 3
						1	0.04	M1=0.04	ND	ND	
						3	0.009		ND	ND	
						5	ND		ND	ND	
Rahuri India, 2011	5	0.075	15	500	10	0	0.07		ND	ND	DP-1104829 Trial 2
						1	0.05	M1=0.02	ND	ND	
						3	0.01		ND	ND	
						5	ND		ND	ND	
Trichy India 2011	5	0.075	12.5	600	10	0	0.06		ND	ND	DP-1104829 Trial 4
						1	0.03		ND	ND	
						3	0.01		ND	ND	
						5	ND		ND	ND	
Rahuri India, 2011	5	0.09	18	500	10	0	0.08		ND	ND	DP-1104829 Trial 2
						1	0.06	M1=0.03	ND	ND	
						3	0.01		ND	ND	
						5	ND		ND	ND	
Trichy India 2011	5	0.09	15	600	10	0	0.08		ND	ND	DP-1104829 Trial 4
						1	0.03		ND	ND	
						3	0.01		ND	ND	
						5	ND		ND	ND	
Rahuri India, 2011	5	0.18	36	500	10	0	0.17		ND	ND	DP-1104829 Trial 2
						1	0.12	M1=0.05	ND	ND	
						3	0.03	M1=0.02	ND	ND	
						5	ND		ND	ND	
Trichy India 2011	5	0.18	30	600	10	0	0.16		ND	ND	DP-1104829 Trial 4
						1	0.06	M1=0.02	ND	ND	
						3	0.03	M1=0.01	ND	ND	
						5	ND		ND	ND	

M1: Residues of metabolite IN-J9Z38

Bulb vegetables

Onion, bulb, Spring onion

In trials conducted in North America on dry bulb onions and on green onions, three foliar applications of 0.15 kg ai/ha cyantraniliprole (OD formulation) were applied at 4–6 day intervals, using 300–400 L/ha, with adjuvant added. In one trial on green onions, an additional plot was treated with two soil treatments of 0.23 kg ai/ha cyantraniliprole (SC formulation) applied through the drip-line irrigation system, 7 days apart using about 100 L/ha, without added surfactant.

Duplicate samples of dry onion bulbs and green onion plants (without roots) were stored frozen at –20 °C for up to 1 year before analysis for cyantraniliprole and six metabolites (within 55 days of extraction) using method DP-15736, with reported LOQs of 0.01 mg/kg. Average concurrent

recoveries were 92% (cyantraniliprole) and 81–97% (metabolites) in samples spiked with 0.01 and 0.1 mg/kg.

Table 86 Residues in bulb onions from supervised trials in North America involving foliar applications of cyantraniliprole (OD formulation)

ONION, BULB Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Arlington, WI USA, 2009 (Yellow Stuttgarten)	3	0.15	38	400	6, 4	1	bulb	0.03, 0.02	0.02		DP-27436 WI09
Freeville, NY USA, 2009 (EX 18368)	3	0.15	42–46	350–320	5, 4	1	bulb	0.01, 0.02	0.01		DP-27436 NY09
Harrow, ON CAN, 2009 (Joliet)	3	0.15	50	300	4, 5	1	bulb	0.03, 0.03	0.03		DP-27436 ON06
Harrow, ON CAN, 2009 (Pulsar)	3	0.15	50	300	4, 5	1	bulb	0.02, 0.02	0.02		DP-27436 ON07
Las Cruces, NM USA, 2009 (NuMex Freedom)	3	0.15	53–44	290–380	4	1	bulb	0.01, 0.02	0.015		DP-27436 NM15
Parma, ID USA, 2009 (Granero)	3	0.15	47	330	6	1	bulb	ND, ND	ND		DP-27436 ID10
Salem, OR USA, 2009 (Red Bull)	3	0.15	42	380	4	1	bulb	ND, ND	ND		DP-27436 OR13
Ste-Clotilde, QC CAN, 2009 (Champlain)	3	0.15	39	370	5, 6	1 4 10 15	bulb	< 0.01, < 0.01 ND, ND ND, ND ND, ND	< 0.01 ND ND ND		DP-27436 QC02
Ste-Clotilde, QC CAN, 2009 (Ricochet)	3	0.15	41	370	5, 6	1	bulb	< 0.01, < 0.01	< 0.01		DP-27436 QC03
Westlaco, TX USA, 2009 (Sweet Sunrise)	3	0.15	40	390	5, 6	1	bulb	0.02, 0.02	0.02		DP-27436 TX07

Table 87 Residues in spring onions (green onion) from supervised trials in North America involving foliar applications of cyantraniliprole (OD formulation) or soil (dripline) treatments (SC formulation)

SPRING ONION Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Willard, OH USA, 2009 (Feast)	3	0.15	47	320	4	1	trimmed plant	0.71, 0.55	0.63		DP-27436 OH11
Westlaco, TX USA, 2009 (Sweet Sunrise)	3	0.15	39	390	4	1	trimmed plant	4.1, 4.1	4.1	M1=0.016 M2=0.01 M3=0.08	DP-27436 TX28
						3		1.4, 1.4	1.4	M1=0.014 M3=0.09	
						7		0.84, 0.85	0.85	M2=0.01 M3=0.04	
						13		0.16, 0.16	0.16	M3=0.04	
Charleston, SC USA, 2009 (Red Baron Bunching)	3	0.15	43	350	4, 5	1	trimmed plant	1.6, 1.6	1.6	M3=0.024	DP-27436 SC08
Salinas, CA USA, 2009 (White Spear)	3	0.15	36	420	5, 6	1	trimmed plant	0.35, 0.42	0.38		DP-27436 CA118
Ste-Clotilde, QC CAN, 2009 (Tokyo Long White)	3	0.15	42	370	6	1	trimmed plant	1.3, 1.4	1.3		DP-27436 QC04
Westlaco, TX USA, 2009 (Sweet Sunrise)	2	0.225	24	94	7	1 3 7 13	trimmed plant	0.04, 0.03 0.03, 0.03 0.05, 0.06 0.06, 0.05	0.03 0.03 0.05 0.05		DP-27436 TX28 Soil dripline treatment

M1: Average residues of metabolite IN-J9Z38

M2: Average residues of metabolite IN-MYX98

M3: Average residues of metabolite IN-N7B69

Brassica vegetables

In trials conducted in North America on broccoli, cauliflower and head cabbage, two or three foliar applications of 0.15 kg ai/ha cyantraniliprole (OD and SE formulations) were applied at 4–6 day intervals, using 200–500 L/ha, with adjuvant added. In several trials, seperate plots were also treated with the equivalent of 0.2 kg ai/ha as a soil injection (soil shank) treatment at planting (without added surfactant).

Duplicate samples of broccoli florets, cauliflower heads, cabbage heads (incuding wrapper leaves) and trimmed cabbage heads (washed after removing the wrapper leaves) were stored at –20 °C for up to 8 months before extraction and analysis for cyantraniliprole and six metabolites (within 5 months of extraction) using method DP-15736, with reported LOQs of 0.01 mg/kg. Average concurrent recoveries were 90–94% (cyantraniliprole) and 80–103% (metabolites) in samples spiked with 0.01, 0.05, 0.1 and 1.0 mg/kg.

Table 88 Residues in broccoli from supervised trials in North America involving two or three foliar applications of cyantraniliprole (OD formulation unless specified)

BROCCOLI Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Guadalupe, CA, USA, 2008 (Bay Meadows) Broccoli	2	0.15	65	230	5	1	florets	0.15, 0.15	0.15		DP-25641 Trial 1
Santa Maria, CA, USA, 2008 (Green Magic) Broccoli/	2	0.15	54	280	5	1	florets	0.28, 0.23	0.26		DP-25641 Trial 2
Terra Bella, CA, USA, 2008-09 (Heritage) Broccoli/	2	0.15	73	210	5	1	florets	0.61, 0.54	0.58		DP-25641 Trial 3
Terra Bella, CA, USA, 2008-09 (Heritage) Broccoli/	2	0.15	73	210	5	-0 0 1 3 5	florets	0.12, 0.13 0.4, 0.63 0.57, 0.34 0.4, 0.24 0.19, 0.23	0.13 0.52 0.45 0.32 0.21		DP-25641 Trial 3
Albany, OR, USA, 2009 (Arcadia) Broccoli/	3	0.15	53	280	5	1	florets	0.59, 0.42	0.51		DP-25641 Trial 17
Branchton, ON, CAN, 2009 (Windsor) Broccoli/	3	0.15	50	300	5	1	florets	0.73, 0.91	0.82	M1=0.01	DP-25641 Trial 14
Branchton, ON, CAN, 2009 (Windsor) Broccoli/	3	0.15	50	300	5	1	florets	0.8, 0.77	0.79	M1=0.015	DP-25641 Trial 14 [100 SE]
Conklin, MI, USA, 2009 (Everest) Broccoli/	3	0.15	53	280	5	1	florets	1.1, 1	1.0	M1=0.02	DP-25641 Trial 16
Conklin, MI, USA, 2009 (Everest) Broccoli/	3	0.15	53	280	5	1	florets	1.1, 1.1	1.1	M1=0.02	DP-25641 Trial 16 [100 SE]
Delavan, WI, USA, 2009 (Patron) Broccoli/	3	0.15	75	200	5	1	florets	0.56, 0.62	0.59		DP-25641 Trial 15
Delavan, WI, USA, 2009 (Patron) Broccoli/	3	0.15	75	200	5	1	florets	0.43, 0.51	0.47		DP-25641 Trial 15 [100 SE]
Guadalupe, CA, USA, 2008 (Bay Meadows) Broccoli	3	0.15	65	230	6, 5	1	florets	0.23, 0.21	0.22		DP-25641 Trial 1

BROCCOLI Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Santa Maria, CA, USA, 2008 (Green Magic) Broccoli/	3	0.15	54	280	5	1	florets	0.3, 0.26	0.28		DP-25641 Trial 2
Terra Bella, CA, USA, 2008-09 (Heritage) Broccoli/	3	0.15	73	210	5	1	florets	0.46, 0.92	0.69		DP-25641 Trial 3
Wyoming, IL, USA, 2009 (Premium Crop) Broccoli/	3	0.15	75	200	5, 4	1	florets	0.51, 0.59	0.55		DP-25641 Trial 13 [100 OD]
Wyoming, IL, USA, 2009 (Premium Crop) Broccoli/	3	0.15	75	200	5, 4	1	florets	0.59, 0.62	0.61		DP-25641 Trial 13 [100 SE]
Terra Bella, CA, USA, 2008-09 (Heritage) Broccoli/	1+ 1+ 1	0.2 (soil) 0.1+ 0.15	97 49 73	210 210	116 5	1	florets	0.49, 0.47	0.48		DP-25641 Trial 3 soil inject at planting+ 2 foliar sprays

M1: Average residues of metabolite IN-J9Z38

Soil injection (shank) treatment rate was 2.13 g ai/100 m of row

Table 89 Residues cauliflowers from supervised trials in North America involving two or three foliar applications of cyantraniliprole (OD formulation)

CAULIFLOWER Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Santa Maria, CA, USA, 2008 (Apex) Cauliflower/	2	0.15	54	280	5	1	flower-head	0.006, 0.012	0.01		DP-25641 Trial 4
Terra Bella, CA, USA, 2008 (Symphony) Cauliflower/	2	0.15	70	220	5	1	flower-head	0.12, 0.14	0.13		DP-25641 Trial 5
Santa Maria, CA, USA, 2008 (Apex) Cauliflower/	3	0.15	54	280	5	1	flower-head	0.007, 0.009	0.01		DP-25641 Trial 4
Terra Bella, CA, USA, 2008 (Symphony) Cauliflower/	3	0.15	70	220	5	1	flower-head	0.079, 0.086	0.08		DP-25641 Trial 5

Table 90 Residues in cabbages from supervised trials in North America involving two or three foliar applications of cyantraniliprole (OD formulation)

CABBAGE Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Bradenton, FL, USA, 2008 (Copenhagen) Cabbage/	2	0.15	50	300	5	1	head trimmed	0.22, 0.22 0.016, 0.013	0.22 0.01		DP-25641 Trial 06
King City, CA, USA, 2008 (Romanov) Cabbage/	2	0.15	65	230	5	1	head trimmed	0.72, 0.64 0.024, 0.005	0.68 0.01		DP-25641 Trial 8
Porterville, CA, USA, 2008 (Supreme Vantage) Cabbage/	2	0.15	70	220	5	1	head	0.82, 0.54	0.68		DP-25641 Trial 9
Raymondville, TX, USA, 2008 (Cheers) Cabbage/	2	0.15	40	380	5	1	head trimmed	0.46, 0.28 0.027, 0.01	0.37 0.02		DP-25641 Trial 7
Alton, NY, USA, 2009 (Fario) Cabbage/	3	0.15	53	280	5	1	heads	0.68, 0.74	0.71		DP-25641 Trial 18
Bradenton, FL, USA, 2008 (Copenhagen) Cabbage/	3	0.15	50	300	5	1	head trimmed	0.29, 0.28 0.035, 0.097	<u>0.29</u> 0.07		DP-25641 Trial 06
Branchton, ON, CAN, 2009 (Red Dynasty) Cabbage/	3	0.15	52	290	4, 5	1	heads	0.78, 0.94	0.86		DP-25641 Trial 21
Carlyle, IL, USA, 2009 (Stonehead) Cabbage/	1+ 1+ 1	0.15	46 52 72	340 290 220	5	1	heads	0.42, 0.52	0.47		DP-25641 Trial 20
Conklin, MI, USA, 2009 (Megaton) Cabbage/	3	0.15	53	280	5	1	heads	0.38, 0.45	0.42		DP-25641 Trial 23
Corvallis, OR, USA, 2009 (Late Flat Dutch) Cabbage/	3	0.15	53	280	5,4	1	heads	0.3, 0.34	0.32		DP-25641 Trial 24
Delavan, WI, USA, 2009 (Vantage Point) Cabbage/	3	0.15	70	220	5, 6	1	heads	0.36, 0.27	0.32		DP-25641 Trial 22
King City, CA, USA, 2008 (Romanov) Cabbage/	3	0.15	70	220	5	1	head trimmed	0.64, 0.67 0.007, 0.006	<u>0.65</u> 0.01		DP-25641 Trial 8

CABBAGE Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Porterville, CA, USA, 2008 (Supreme Vantage) Cabbage/	3	0.15	70	220	5	1	head	0.66, 0.47	0.56		DP-25641 Trial 9
Raymondville, TX, USA, 2008 (Cheers) Cabbage/	3	0.15	40	380	5	1	head trimmed	0.6, 0.54 0.028, 0.015	0.57 0.02		DP-25641 Trial 7
Seven Springs, NC, USA, 2009 (Early Jersey Wakefield) Cabbage/	3	0.15	70	220	6, 4	1	heads	0.93, 0.98	0.95	M1=0.01	DP-25641 Trial 19
Porterville, CA, USA, 2008 (Supreme Vantage) Cabbage/	1+ 1+ 1	0.2 (soil) 0.1 0.15	110 47 70	190 220 220	69 5	1DAA 3	head	0.59, 0.4	0.49		DP-25641 Trial 9 soil inject at planting+ 2 foliar sprays

M1: Average residues of metabolite IN-J9Z38

Soil injection (shank) treatment rate was 2.13 g ai/100 m of row

Trimmed=cabbage heads without wrapper leaves

Fruiting vegetable, Cucurbits

Results from supervised trials from Australia, Europe and North America on cucumber, summer squash and melons were provided to the Meeting.

North America

In trials conducted in North America on cucumber, melons and summer squash, two or three foliar applications of 0.15 kg ai/ha cyantraniliprole (OD formulation) were applied at 5–7 day intervals, using 200–500 L/ha with adjuvant added. Soil treatment plots were also included in three trial sites, where two soil treatments of 0.15 kg ai/ha cyantraniliprole (SC formulation) were applied through drip-line irrigation systems (adjusted to pH 4–5, with no added adjuvant).

Duplicate samples were stored at –20 °C for up to 9 months before analysis of whole fruit or pulp and peel for cyantraniliprole and six metabolites analysis (within 70 days of extraction) using an adaptation of method DP-15736, with reported LOQs of 0.01 mg/kg. Average concurrent recoveries were 90–94% (cyantraniliprole) and 82–100% (metabolites) in samples spiked with 0.01, 0.1, 0.2 and 0.6 mg/kg.

Europe

In trials conducted in Europe on melons, cucumbers and summer squash (courgettes), two applications of 0.09 kg ai/ha cyantraniliprole (OD formulation) were applied to field crops using 800 L/ha or four applications of 0.12 kg ai/ha cyantraniliprole (OD formulation) using 1200 L/ha were applied to crops grown under protection. The retreatment intervals in all trials was 7 days and all foliar treatments included added surfactant. Soil treatment plots were also included in a number of trial sites, where 2–4 soil treatments of 0.075–0.1 kg ai/ha cyantraniliprole (SC formulation) were applied through the drip-line irrigation systems, with no added adjuvants.

Samples were stored at –18 °C for up to 8 months before analysis of whole fruit or pulp and peel for cyantraniliprole and six metabolites analysis (within 10 days of extraction) using an adaptation of method DP-15736, with reported LOQs of 0.01 mg/kg. For melons, the peel and pulp (minus seeds) weights were used to calculate whole fruit residues. Average concurrent recoveries were 91–101% (cyantraniliprole) and 93–104% (metabolites) in samples spiked with 0.01, 0.1, and 0.5 mg/kg.

Australia

In trials conducted in Australia on rockmelons, cucumbers and summer squash (zucchini), two applications of 0.05, 0.075 and 0.15 kg ai/ha cyantraniliprole (OD formulation) were applied to field crops using 400 L/ha. The retreatment intervals in all trials was 7 days and all treatments included added surfactant.

Samples were stored at –20 °C for up to 6 months before analysis of whole fruit for cyantraniliprole and six metabolites analysis (within 1 day of extraction) using method DP-15736, with reported LOQs of 0.01 mg/kg. Average concurrent recoveries were 76–77% (cyantraniliprole) and 75–92% (metabolites) in samples spiked with 0.01–2.0 mg/kg.

Table 91 Residues in field cucumbers from supervised trials in the USA with cyantraniliprole as foliar applications (OD formulation) or applied via dripline irrigation systems (SC formulation)

CUCUMBER Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference& Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Hinton, OK USA, 2008 (Poinsett 76)	2	0.15	75	200	5	1	whole fruit	0.037, 0.035	0.036		DP-25642 Trial 3
Oviedo, FL USA, 2008 (Park's Bush Whopper II)	2	0.15	53	280	5	1	whole fruit	0.032, 0.034	0.033		DP-25642 Trial 1
Clewiston, FL USA, 2009 (Speedway)	2	0.16	58	260	5	1	whole fruit	0.1, 0.12	0.11		DP-25642 Trial 2
(Branchton, ON, Canada, 2009) (Thunder)	3	0.15	75	200	5, 4	1	whole fruit	0.026, 0.032	0.029		DP-25642 Trial 15
(Corvallis, OR USA, 2009 (Genuine F1)	3	0.15	54	280	5	1	whole fruit	0.027, 0.02	0.023		DP-25642 Trial 18
Carlyle, IL USA, 2009 (Talladega)	3	0.15	65	230	5	1	whole fruit	0.082, 0.057	0.07		DP-25642 Trial 14
Conklin, MI USA, 2009 (Dasher II)	3	0.15	68	220	5	1	whole fruit	0.1, 0.13	0.12		DP-25642 Trial 17
Delavan, WI USA, 2009 (Marketmore 76)	3	0.15	75	200	5	1	whole fruit	0.019, 0.024	0.021		DP-25642 Trial 16
Elko, SC USA, 2009 (Talladega)	3	0.15	75	200		1	whole fruit	0.038, 0.052	0.045		DP-25642 Trial 13

CUCUMBER Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference& Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Hinton, OK USA, 2008 (Poinsett 76)	3	0.15	75	200	5, 7	1	whole fruit	0.042, 0.049	0.045		DP-25642 Trial 3
Oviedo, FL USA, 2008 (Park's Bush Whopper II)	3	0.15	53	280	5, 4	1	whole fruit	0.02, 0.016	0.018		DP-25642 Trial 1
Seven Springs, NC USA, 2009 (Ashley)	3	0.15	60	250	5	1	whole fruit	0.039, 0.044	0.041		DP-25642 Trial 12
Clewiston, FL USA, 2009 (Speedway)	2+ 1	0.16 0.115	58+ 50	260+ 230	5	1	whole fruit	0.2, 0.12	0.16		DP-25642 Trial 2
Clewiston, FL USA, 2009 (Speedway)	2+ 1	0.15 0.15	4.4 60	3400 250	7 7	–0 0 1 3 5 6	whole fruit	ND, ND 0.086, 0.095 0.058, 0.12 0.044, 0.053 0.055, 0.064 0.048, 0.048	ND 0.09 0.089 0.049 0.06 0.048		DP-25642 Trial 2 2 drip applications (2.3 g ai/100m row) + 1 foliar spray

Table 92 Residues in field cucumbers from supervised trials in Europe with cyantraniliprole as foliar applications (OD formulation) or applied via dripline irrigation systems (SC formulation)

CUCUMBER Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Torrevecchia Pia Italy, 2009 (Gemini) cucumber	2	0.09	11	800	7	1	whole fruit	0.022			DP-27711 Test 05
Mediglia Italy, 2010 (Unspecified) Cucumber	2	0.09	11	800	7	1	whole fruit	0.099			DP-27711 Test 11
Aguadulce Spain, 2010 (Unspecified) Cucumber	2	0.09	11	800	7	1	whole fruit	0.04			DP-27711 Test 12
Llieda Spain, 2010 (Unspecified) Cucumber	2	0.09	11	800	7	1	whole fruit	0.032			DP-27711 Test 13

CUCUMBER Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Torrevecchia Pia Italy, 2009 (Gemini)	2	0.075	3.8	2000	7	–0 0 1 3 7	whole fruit	ND ND ND ND ND			DP-27711 Test 05 200 SC soil dripline irrigation

Table 93 Residues in cucumbers from supervised trials in Australia involving foliar applications of cyantraniliprole (OD formulation)

CUCUMBER Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference Comments
	no	kg ai/ha	kg ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Bowen, QLD Australia, 2009 (Gremlin) Cucumber	2	0.05	0.0125	400	7	–0 1	whole fruit	0.01 0.05			DP-31413 Site 2
Bowen, QLD Australia, 2009 (Gremlin) Cucumber	2	0.075	0.019	400	7	–0 1	whole fruit	0.04 0.09			DP-31413 Site 2
Bowen, QLD Australia, 2009 (Gremlin) Cucumber	2	0.15	0.0375	400	7	–0 1	whole fruit	0.08 0.22			DP-31413 Site 2

Table 94 Residues in protected cucumbers from supervised trials in Europe with cyantraniliprole as foliar applications (OD formulation) or applied via dripline irrigation systems (SC formulation)

CUCUMBER Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Oirlo Netherlands, 2009 (Cratos) Cucumber	4	0.12	10	1200	7	1	whole fruit	0.089			DP-28201 Test 01
Nea Magnisia Greece, 2009 (Gallileo) Cucumber	4	0.12	10	1200	7	1	whole fruit	0.053			DP-28201 Test 02

CUCUMBER Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Castenray Netherlands, 2009 (Sheilla) Cucumber	4	0.12	10	1200	6–8	–0 0 1 3 7 14	whole fruit	0.11 0.12 0.11 0.091 0.13 0.18			DP-28201 Test 05
Puente del Rio Spain, 2009 (Estrada) Cucumber	4	0.12	10	1200	7–8	–0 0 1 3 7 14	whole fruit	0.019 0.076 0.077 0.18 0.044 0.013			DP-28201 Test 06
Castenray Netherlands, 2009 (Sheilla)	4	0.1	5	2000	6–8	–0 0 1 3 7 14	whole fruit	0.015 0.01 0.003 0.009 0.019 0.019			DP-28201 Test 05 200 SC soil dripline irrigation
Puente del Rio Spain, 2009 (Estrada)	4	0.1	5	2000	7–8	–0 0 1 3 7 14	whole fruit	ND ND ND ND ND ND			DP-28201 Test 06 200 SC soil dripline irrigation

Table 95 Residues in summer squash from supervised trials in North America with cyantraniliprole as foliar applications (OD formulation) or applied via dripline irrigation systems (SC formulation)

SUMMER SQUASH Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Fresno, CA USA, 2008 (Jackpot F1)	2	0.15	54	280	5	1	whole fruit	0.074, 0.1	0.089		DP-25642 Trial 11
Oviedo, FL USA, 2008 (Early Summer Crookneck)	2	0.15	52	290	5	1	whole fruit	0.14, 0.086	0.11		DP-25642 Trial 9
Porterville, CA USA, 2008 (Early Summer Yellow Crookneck)	2	0.15	38	400	6	1	whole fruit	0.1, 0.083	0.094		DP-25642 Trial 10

SUMMER SQUASH Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Alton, NY USA, 2009 (Meteor)	3	0.15	54	280	5	1	whole fruit	0.059, 0.06	0.059		DP-25642 Trial 19
Branchton, ON Canada, 2009 (Senator)	3	0.15	75	200	5	1	whole fruit	0.012, 0.01	0.011		DP-25642 Trial 22
Conklin, MI USA, 2009 (Spineless Beauty) Zucchini	3	0.15	50	300	5, 7	1	whole fruit	0.093, 0.067	0.08		DP-25642 Trial 23
Corvallis, OR USA, 2009 (Noche)	3	0.15	53	280	5	1	whole fruit	0.05, 0.055	0.053		DP-25642 Trial 24
Fresno, CA USA, 2008 (Jackpot F1)	3	0.15	54	280	5	1	whole fruit	0.1, 0.12	0.11		DP-25642 Trial 11
Oviedo, FL USA, 2008 (Early Summer Crookneck)	3	0.15	52	290	5, 4	1	whole fruit	0.091, 0.084	0.088		DP-25642 Trial 9
Porterville, CA USA, 2008 (Early Summer Yellow Crookneck)	3	0.15	38	400	6, 4	1	whole fruit	< 0.01, 0.06	0.034		DP-25642 Trial 10
Seven Springs, NC USA, 2009 (Yellow Straight-Neck)	3	0.15	60	250	5	1	whole fruit	0.071, 0.072	0.072		DP-25642 Trial 20
Wyoming, IL USA, 2009) (Spineless Beauty F1) Zucchini	3	0.15	75	200	5	1	whole fruit	0.044, 0.041	0.043		DP-25642 Trial 21
Porterville, CA USA, 2008 (Early Summer Yellow Crookneck)	2+ 1	0.15 0.15	1.5 38	10200 400	7	1	whole fruit	0.031, 0.029	0.03		DP-25642 Trial 10 2 drip applications (2.3 g ai/100m row) + 1 foliar spray

Table 96 Residues in field summer squash from supervised trials in Europe with cyantraniliprole as foliar applications (OD formulation) or applied via dripline irrigation systems (SC formulation)

SUMMER SQUASH Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
St Vincent S France, 2009 (Mirza) Courgette	2	0.09	11	800	8	1	whole fruit	< 0.01 ND ND			DP-27711 Test 04
Nea Magnisia Greece, 2009 (Ezra) Courgette	2	0.09	11	800	6	1	whole fruit	0.025			DP-27711 Test 06
Le Mas Rillier S France, 2009 (Mirza) Courgette	2	0.09	11	800	6	–0 0 1 3 7	whole fruit	< 0.01 0.046 0.022 0.014 0.025			DP-27711 Test 09
Los Palacios Spain, 2009 (Amalia) Courgette	2	0.09	11	800	6	–0 0 1 3 7	whole fruit	< 0.01 0.054 0.039 < 0.01 < 0.01			DP-27711 Test 10
Perea Greece, 2010 (Unspecified) Courgette	2	0.09	11	800	8	1	whole fruit	0.1			DP-27711 Test 14
Mediglia Italy, 2010 (Unspecified) Courgette	2	0.09	11	800	7	1	whole fruit	0.1			DP-27711 Test 15
Aguadulce Spain, 2010 (Unspecified) Courgette	2	0.09	11	800	7	1	whole fruit	< 0.01			DP-27711 Test 16
Le Mas Rillier S France, 2009 (Mirza)	2	0.075	3.8	2000	7	–0 0 1 3 7	whole fruit	ND ND ND ND ND			DP-27711 Test 09 (soil dripline)
Los Palacios Spain, 2009 (Amalia) Courgette	2	0.075	3.8	2000	7	–0 0 1 3 7	whole fruit	ND ND ND ND ND			DP-27711 Test 10 (soil dripline)
Nea Magnisia Greece, 2009 (Ezra)	2	0.075	3.8	2000	7	–0 0 1 3 7	whole fruit	ND ND ND ND ND			DP-27711 Test 06 (soil dripline)

SUMMER SQUASH Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
St Vincent S France, 2009 (Mirza)	2	0.075	3.8	2000	8	–0 0 1 3 7	whole fruit	ND ND ND ND ND			DP-27711 Test 04 (soil dripline)

Table 97 Residues in summer squash (zucchini) from supervised trials in Australia involving foliar applications of cyantraniliprole (OD formulation)

SUMMER SQUASH Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference Comments
	no	kg ai/ha	kg ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Bowen, QLD Australia, 2009 (Crowbar) Zucchini	2	0.05	0.0125	400	7	–0 1	whole fruit	0.02 0.04			DP-31413 Site 3
Bowen, QLD Australia, 2009 (Crowbar) Zucchini	2	0.075	0.019	400	7	–0 1	whole fruit	0.02 0.05			DP-31413 Site 3
Bowen, QLD Australia, 2009 (Crowbar) Zucchini	2	0.15	0.0375	400	7	–0 1	whole fruit	0.07 0.15			DP-31413 Site 3

Table 98 Residues in protected summer squash from supervised trials in Europe with cyantraniliprole as foliar applications (OD formulation) or applied via dripline irrigation systems (SC formulation)

SUMMER SQUASH Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Dirillo Italy, 2009 (Richgreen) Courgette	4	0.12	10	1200	7	1	whole fruit	0.22			DP-28201 Test 03
Berja Spain, 2009 (Otelo) Courgette	4	0.12	10	1200	7	1	whole fruit	0.033			DP-28201 Test 04
Mediglia Italy, 2009 (President) Courgette	4	0.12	10	1200	7	–0 0 1 3 7 14	whole fruit	ND 0.051 0.035 0.015 0.005 ND			DP-28201 Test 07

SUMMER SQUASH Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Utrera Spain, 2009 (Nieves) Courgette	4	0.12	10	1200	6-7	–0 0 1 3 7 14	whole fruit	0.022 0.084 0.08 0.07 0.059 0.021			DP-28201 Test 08
Puente del Rio Spain, 2009 (Sinatra) Courgette	4	0.12	10	1200	7-8	–0 0 1 3 7 14	whole fruit	0.016 0.051 0.06 ND 0.008 ND			DP-28201 Test 09
Mediglia Italy, 2009 (President)	4	0.1	5	2000	7	–0 0 1 3 7 14	whole fruit	ND ND ND ND ND ND			DP-28201 Test 07 (soil dripline)
Puente del Rio Spain, 2009 (Sinatra)	4	0.1	5	2000	7-8	–0 0 1 3 7 14	whole fruit	0.004 ND ND 0.004 ND ND			DP-28201 Test 09 (soil dripline)
Utrera Spain, 2009 (Nieves)	4	0.1	5	2000	6-7	–0 0 1 3 7 14	whole fruit	ND ND ND ND ND ND			DP-28201 Test 08 (soil dripline)

Table 99 Residues in melons from supervised trials in North America with cyantraniliprole as foliar applications (OD formulation) or applied via dripline irrigation systems (SC formulation).

MELON Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Fresno, CA USA, 2008 (Athena) Cantaloupe	2	0.15	54	280	5	1	flesh skin whole fruit	ND, ND 0.22, 0.22 0.075, 0.091	ND 0.22 0.083		DP-25642 Trial 6
Hinton, OK USA, 2008 (PMR 45) Cantaloupe	2	0.15	80	200	6	1	flesh skin whole fruit	ND, ND 0.17, 0.13 0.106, 0.085	ND 0.15 0.095		DP-25642 Trial 4
Paso Robles, CA USA, 2008 (Hale's Best) Cantaloupe	2	0.15	50	300	5	1	flesh skin whole fruit	ND, ND 0.35, 0.24 0.12, 0.088	ND 0.3 0.11		DP-25642 Trial 8

MELON Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Porterville, CA USA, 2008 (Hale's Best Jumbo) Cantaloupe	2	0.15	38	400	5	1	flesh skin whole fruit	ND, ND 0.12, 0.096 0.061, 0.049	ND 0.11 0.055		DP-25642 Trial 5
Porterville, CA USA, 2008 (Honeydew)	2	0.15	60	250	5	1	flesh skin whole fruit	< 0.01, < 0.01 0.035, 0.054 0.03, 0.049	< 0.01 0.044 0.04		DP-25642 Trial 7
Branchton, ON Canada, 2009 (Primo) Cantaloupe	3	0.15	75	200	5	1	flesh skin whole fruit	< 0.01, < 0.01 0.22, 0.18 0.12, 0.098	< 0.01 0.2 <u>0.11</u>		DP-25642 Trial 27
Conklin, MI USA, 2009 (Aphrodite VIP (8793)) Cantaloupe	3	0.15	52	290	5	1	flesh skin whole fruit	ND, ND 0.17, 0.19 0.096, 0.11	ND 0.18 <u>0.1</u>		DP-25642 Trial 28
Fresno, CA USA, 2008 (Athena) Cantaloupe	3	0.15	54	280	5	1	flesh skin whole fruit	< 0.01, ND 0.3, 0.093 0.14, 0.039	< 0.01 0.2 <u>0.087</u>		DP-25642 Trial 6
Hinton, OK USA, 2008 (PMR 45) Cantaloupe	3	0.15	80	200	6, 4	1	flesh skin whole fruit	ND, ND 0.23, 0.25 0.15, 0.16	ND 0.24 <u>0.15</u>		DP-25642 Trial 4
Paso Robles, CA USA, 2008 (Hale's Best) Cantaloupe	3	0.15	50	300	5, 4	1	flesh skin whole fruit	ND, ND 0.54, 0.43 0.18, 0.15	ND 0.48 <u>0.17</u>		DP-25642 Trial 8
Porterville, CA USA, 2008 (Hale's Best Jumbo) Cantaloupe	3	0.15	38	400	5	1	flesh skin whole fruit	ND, ND 0.19, 0.21 0.081, 0.1	ND 0.2 <u>0.09</u>		DP-25642 Trial 5
Porterville, CA USA, 2008 (Honeydew)	3	0.15	60	250	5	1	flesh skin whole fruit	< 0.01, < 0.01 0.046, 0.023 0.058, 0.024	< 0.01 0.034 <u>0.041</u>		DP-25642 Trial 7
Seven Springs, NC USA, 2009 (Hales Best Jumbo) Cantaloupe	3	0.15	37	400	4, 6	1	flesh skin whole fruit	ND, ND 0.13, 0.12 0.05, 0.047	ND 0.13 <u>0.048</u>		DP-25642 Trial 25
Wyoming, IL USA, 2009 (Atlantis F1) Muskmelon	3	0.15	68	220	5	1	flesh skin whole fruit	ND, ND 0.12, 0.15 0.072, 0.095	ND 0.14 <u>0.084</u>		DP-25642 Trial 26

MELON Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Porterville, CA USA, 2008 (Hale's Best Jumbo) Cantaloupe	2+ 1	0.15 0.15	1.47 38	10200 400	7 7	1	flesh skin whole fruit	ND, ND 0.039, 0.045 0.022, 0.024	ND 0.042 0.023		DP-25642 Trial 5 2 drip applications (4.2 g ai/100m row) + 1 foliar spray

Table 100 Residues in outdoor melons from supervised trials in Europe with cyantraniliprole as foliar applications (OD formulation) or applied via dripline irrigation systems (SC formulation)

MELON Location Country, year (variety)	Application					DAT (days)	Cyantraniliprole Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)		whole fruit	peel	pulp	
Svoronos Greece, 2009 (Lavigal) Melon	2	0.09	11	800	6	1	0.069	0.12	ND	DP-27711 Test 01
Casteldidone Italy, 2009 (Sogno) Melon	2	0.09	11	800	8	1	0.041	0.093	ND	DP-27711 Test 02
Puerto Serrano Spain, 2009 (Anasta) Cantaloupe Melon	2	0.09	11	800	7	1	0.05	0.12	ND	DP-27711 Test 03
Villamartin Spain, 2009 (Alonso) Cantaloupe Melon	2	0.09	11	800	7	–0 0 1 3 7	0.039 0.051 0.015 0.039 0.046	0.086 0.11 0.031 0.096 0.12	ND < 0.01 ND ND ND	DP-27711 Test 07
Los Palacios Spain, 2009 (Linord) Melon	2	0.09	11	800	6	–0 0 1 3 7	0.016 0.021 0.016 0.03 0.013	0.042 0.058 0.04 0.076 0.035	ND ND ND ND ND	DP-27711 Test 08
Svoronos Greece, 2009 (Lavigal)	2	0.075	3.8	2000	7	–0 0 1 3 7	ND ND ND ND ND	ND ND ND ND ND	ND ND ND ND ND	DP-27711 Test 01 (soil dripline)
Casteldidone Italy, 2009 (Sogno)	2	0.075	3.8	2000	7	–0 0 1 3 7	ND ND ND ND ND	ND ND ND ND ND	ND ND ND ND ND	DP-27711 Test 02 (soil dripline)

MELON Location Country, year (variety)	Application					DAT (days)	Cyantraniliprole Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)		whole fruit	peel	pulp	
Puerto Serrano Spain, 2009 (Anasta) Cantaloupe	2	0.075	3.8	2000	7	–0	ND	ND	ND	DP-27711 Test 03 (soil dripline)
						0	ND	ND	ND	
						1	ND	ND	ND	
						3	ND	ND	ND	
						7	ND	ND	ND	
Villamartin Spain, 2009 (Alonso) Cantaloupe	2	0.075	3.8	2000	8	–0	ND	ND	ND	DP-27711 Test 07 (soil dripline)
						0	ND	ND	ND	
						1	ND	ND	ND	
						3	ND	ND	ND	
						7	ND	ND	ND	
Los Palacios Spain, 2009 (Linord)	2	0.075	3.8	2000	7	–0	ND	ND	ND	DP-27711 Test 08 (soil dripline)
						0	ND	ND	ND	
						1	ND	ND	ND	
						3	ND	ND	ND	
						7	ND	ND	ND	

Table 101 Residues in melons (rockmelon) from supervised trials in Australia involving foliar applications of cyantraniliprole (OD formulation)

MELON Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference Comments
	no	kg ai/ha	kg ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Bowen, QLD Australia, 2009 (Northern Sky) Rockmelon	2	0.05	0.0125	400	7	0	whole fruit	0.08			DP-31413 Site 1
						1		0.06			
						7		0.02			
						14		ND			
						21		ND			
Bowen, QLD Australia, 2009 (Northern Sky) Rockmelon	2	0.075	0.019	400	7	0	whole fruit	0.18			DP-31413 Site 1
						1		0.1			
						7		0.03			
						14		< 0.01			
						21		ND			

Table 102 Residues in protected melons from supervised trials in Europe with cyantraniliprole as foliar applications (OD formulation) or applied via irrigation dripline systems (SC formulation)

MELON Location Country, year (variety)	Application					DAT (days)	Cyantraniliprole residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)		whole fruit	peel	pulp	
Contrada Randello Italy, 2009 (Cabrero) Melon	4	0.12	10	1200	7	1	0.044	0.066	ND	DP-28186 Test 01

MELON Location Country, year (variety)	Application					DAT (days)	Cyantraniliprole residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)		whole fruit	peel	pulp	
Contada Randello Italy, 2009 (Cabrero) Melon	4	0.12	10	1200	7	1	0.06	0.11	ND	DP-28186 Test 02
Svoronos Greece, 2009 (Lavigas) Melon	4	0.12	10	1200	6–8	1	0.031	0.064	ND	DP-28186 Test 03
Mea Magnisia Greece, 2009 (Galia F1) Melon	4	0.12	10	1200	7	1	0.038	0.068	ND	DP-28186 Test 04
Mezin S France, 2009 (Charentais) Melon	4	0.12	10	1200	6–8	–0 0 1 3 7 14	0.038 0.1 0.035 0.05 0.029 0.034	0.068 0.19 0.083 0.13 0.069 0.06	< 0.01 ND ND ND ND ND	DP-28186 Test 05
Adra Spain, 2009 (Valverde) Melon	4	0.12	10	1200	6–7	–0 0 1 3 7 14	0.048 0.07 0.078 0.077 0.018 0.059	0.13 0.18 0.21 0.2 0.054 0.17	ND ND ND ND ND ND	DP-28186 Test 07
Adra Spain, 2009 (Galia) Melon	4	0.12	10	1200	6–7	–0 0 1 3 7 14	0.023 0.026 0.049 0.064 0.005 0.019	0.079 0.08 0.14 0.21 0.013 0.053	ND ND ND ND ND ND	DP-28186 Test 08
Adra Spain, 2009 (Cantaloupe) Melon	4	0.12	10	1200		–0 0 1 3 7 14	0.051 0.066 0.096 0.062 0.035 0.024	0.13 0.17 0.24 0.16 0.072 0.056	ND ND ND ND ND < 0.01	DP-28186 Test 09
Mezin S France, 2009 (Charentais)	4	0.1	5	2000	6–8	–0 0 1 3 7 14	ND ND ND ND < 0.01 ND	ND ND ND ND 0.012 ND	ND ND ND ND ND ND	DP-28186 Test 05 (soil dripline)
Adra Spain, 2009 (Valverde)	4	0.1	5	2000	6–7	–0 0 1 3 7 14	ND ND ND ND ND ND	ND ND ND ND ND ND	ND ND ND ND ND ND	DP-28186 Test 07 (soil dripline) May–Jun

MELON Location Country, year (variety)	Application					DAT (days)	Cyantraniliprole residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)		whole fruit	peel	pulp	
Adra Spain, 2009 (Galia)	4	0.1	5	2000	6–7	–0 0 1 3 7 14	ND ND ND ND ND ND	ND ND ND < 0.01 ND ND	ND ND ND ND ND ND	DP-28186 Test 08 (soil dripline) May–Jun
Adra Spain, 2009 (Cantaloupe)	4	0.1	5	2000		–0 0 1 3 7 14	ND ND ND ND ND ND	0.004 ND ND ND ND ND	ND ND ND ND ND ND	DP-28186 Test 09 (soil dripline) October

Fruiting vegetables, other than Cucurbits

Results from supervised trials from Australia, Europe and North America on tomatoes and peppers were provided to the Meeting.

North America

In trials conducted in North America on field tomatoes and peppers (bell and non-bell), two or three foliar applications of 0.15 kg ai/ha cyantraniliprole (OD formulation) were applied at 4–6 day intervals, using 200–500 L/ha, with adjuvant added. Soil treatment plots were also included in three trial sites, where two soil treatments of 0.15 kg ai/ha cyantraniliprole (SC formulation) were applied through drip-line irrigation systems (adjusted to pH 4–5, with no added adjuvant).

Duplicate samples of fruit were stored at –20 °C for up to 8 months before extraction and analysis for cyantraniliprole and six metabolites (within 3 months of extraction) using method DP-15736, with reported LOQs of 0.01 mg/kg. Average concurrent recoveries were 88–92% (cyantraniliprole) and 83–99% (metabolites) in samples spiked with 0.01–1.0 mg/kg and also 4 mg/kg for tomatoes.

Europe

In trials conducted in Europe on protected sweet peppers, chilli peppers and tomatoes, four applications of 10 g ai/hL cyantraniliprole were applied using 1200 L/ha as foliar sprays (OD formulation) or four applications of 5 g ai/hL using 2000 L/ha as soil treatments (SC formulation), applied through the dripline irrigation systems. The retreatment intervals in all trials was 7 days and all foliar treatments included added surfactant.

In outdoor tomatoes, sweet and chilli peppers in Southern Europe, two foliar applications of 0.09 kg ai/ha cyantraniliprole (OD formulation) were applied using 800 L water/ha at 6–7 day intervals with added surfactant.

Samples were stored at -18 C for up to 8 months before extraction and analysis for cyantraniliprole and six metabolites analysis (within 7 days of extraction) using method DP-15736, with reported LOQs of 0.01 mg/kg. Average concurrent recoveries were 75–101% (cyantraniliprole) and 77–101% (metabolites) in samples spiked with 0.01, 0.1, 1.0 and 5.0 (peppers) mg/kg.

Australia

In trials conducted in Australia on outdoor tomatoes and sweet peppers (capsicums), two foliar applications of 0.05, 0.075 or 0.15 kg ai/ha cyantraniliprole (OD formulation) were applied 7 days apart to separate plots using 400 L water/ha with added surfactant.

Samples were stored at or below -18 °C for up to 7 months before extraction and analysis for cyantraniliprole and six metabolites analysis (within 1 day of extraction) using method DP-15736, with reported LOQs of 0.01 mg/kg. Average concurrent recoveries were 80–91% (cyantraniliprole) and 77–94% (metabolites) in samples spiked with 0.01–2.0 mg/kg.

Table 103 Residues in field tomatoes from supervised trials in North America involving two or three foliar applications of cyantraniliprole (OD formulation)

TOMATO Location	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference Comments
Country, year (variety)	no	kg ai/ha	kg ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Bradenton, FL USA, 2008 (FL-47) Tomato/	2	0.15	34	450	5	1	whole fruit	0.05, 0.047	0.048		DP-25643 Trial 1
Fresno, CA USA, 2008 (H8004) Tomato/	2	0.15	54	280	5	1	whole fruit	0.074, 0.1	0.089		DP-25643 Trial 8
Fresno, CA USA, 2008 (H8004) Tomato/	2	0.15	53	280	5	1	whole fruit	0.047, 0.076	0.061		DP-25643 Trial 9
Hickman, CA USA, 2008 (6368) Tomato/	2	0.15	53	280	5	1	whole fruit	0.14, 0.13	0.14		DP-25643 Trial 10
Huron, CA USA, 2008 (Sun 6117) Tomato/	2	0.15	39	390	5	1	whole fruit	0.041, 0.044	0.042		DP-25643 Trial 6
Lemore, CA USA, 2008 (Shady Lady) Tomato/	2	0.15	39	390	5	1	whole fruit	0.13, 0.1	0.12		DP-25643 Trial 4
Oviedo, FL USA, 2008 (Burpee Big Boy) Tomato/	2	0.15	53	280	5	1	whole fruit	0.037, 0.022	0.03		DP-25643 Trial 2
Paso Robles, CA USA, 2008 (Red Cherry) Tomato/	2	0.15	54	280	5	1	whole fruit	0.19, 0.12	0.16		DP-25643 Trial 7
Porterville, CA USA, 2008 (Sun 6117) Tomato/	2	0.15	66 77	230 200		1	whole fruit	0.13, 0.082	0.1		DP-25643 Trial 5
San Ardo, CA USA, 2008 (Shady Lady) Tomato/	2	0.15	55	270	5	0 0 1	whole fruit	0.017, 0.032 0.07, 0.036 0.044, 0.044	0.024 0.053 0.044		DP-25643 Trial 3

TOMATO Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference Comments
	no	kg ai/ha	kg ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Alton, NY USA, 2009 (Polbig F1) Tomato/	3	0.15	53	280	5, 6	1	whole fruit	0.091, 0.072	0.082		DP-25643 Trial 20
Bradenton, FL USA, 2008 (FL-47) Tomato/	3	0.15	34	450	5	1	whole fruit	0.037, 0.045	0.041		DP-25643 Trial 1
Branchton, ON, CAN 2009 (9909) Tomato/	3	0.15	60	250	4, 5	1	whole fruit	0.075, 0.096	0.086		DP-25643 Trial 22
Conklin, MI USA, 2009 (Supersweet 100 VF) Tomato (Cherry)/	3	0.15	50	300	5	1	whole fruit	0.2, 0.14	0.17		DP-25643 Trial 28
Delavan, WI USA, 2009 (Rutgers) Tomato/	3	0.15	75	200	7, 4	1	whole fruit	0.063, 0.078	0.071		DP-25643 Trial 25
Fresno, CA USA, 2008 (H8004) Tomato/	3	0.15	53	290	5	1	whole fruit	0.12, 0.16	0.14		DP-25643 Trial 8
Fresno, CA USA, 2008 (H8004) Tomato/	3	0.15	53	280	5	1	whole fruit	0.055, 0.06	0.058		DP-25643 Trial 9
Hickman, CA USA, 2008 (6368) Tomato/	3	0.15	53	280	5	1	whole fruit	0.16, 0.17	0.16		DP-25643 Trial 10
Huron, CA USA, 2008 (Sun 6117) Tomato/	3	0.15	39	390	5	1	whole fruit	0.08, 0.053	0.067		DP-25643 Trial 6
Lemore, CA USA, 2008 (Shady Lady) Tomato/	3	0.15	41	360	5	1	whole fruit	0.14, 0.15	0.14		DP-25643 Trial 4
Oviedo, FL USA, 2008 (Burpee Big Boy) Tomato/	3	0.15	53	280	5, 4	1	whole fruit	0.052, 0.041	0.046		DP-25643 Trial 2

TOMATO Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference Comments
	no	kg ai/ha	kg ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Paso Robles, CA USA, 2008 (Red Cherry) Tomato/	3	0.15	53	290	5	1	whole fruit	0.28, 0.25	0.26		DP-25643 Trial 7
Richland, IA USA, 2009 (Supersweet) Tomato (Cherry)/	3	0.15	66	220	6, 5	1	whole fruit	0.081, 0.062	0.072		DP-25643 Trial 26
Rockville, IN USA, 2009 (Mountain Spring) Tomato/	3	0.15	48	310	5	1	whole fruit	0.066, 0.05	0.058		DP-25643 Trial 24
San Ardo, CA USA, 2008 (Shady Lady) Tomato/	3	0.15	55	270	5	1	whole fruit	0.076, 0.054	0.065		DP-25643 Trial 3
Seven Springs, NC USA, 2009 (Rutgers) Tomato/	1+ 2	0.15	59 73	250 210	5	1	whole fruit	0.086, 0.066	0.076		DP-25643 Trial 21
Sparta, MI USA, 2009 (Sunoma) Tomato (Roma)/	3	0.15	50	300	5	1	whole fruit	0.099, 0.088	0.093		DP-25643 Trial 29
Verona, WI USA, 2009 (Red Defender) Tomato/	3	0.15	57	260	5	1	whole fruit	0.094, 0.11	0.1		DP-25643 Trial 27
Porterville, CA USA, 2008 (Sun 6117) Tomato/	1+ 2	0.15 0.15	66 77	230 200		1	whole fruit	0.08, 0.074	0.077		DP-25643 Trial 5
Caryle, IL USA, 2009 (Burpee Big Boy) Tomato/	1+ 1+ 1	0.15 0.15 0.15	60 58 44	250 260 340	5	1	whole fruit	0.14, 0.091	0.12		DP-25643 Trial 23
San Ardo, CA USA, 2008 (Shady Lady) Tomato/	2+ 1	0.15 (soil) 0.15	2 54	7700 280	7	1	whole fruit	0.044, 0.052	0.048		DP-25643 Trial 3 2 soil dripline + 1 foliar

Soil (dripline irrigation) treatment rate of 2.7 g ai/100 metres of row

Table 1 Residues in field tomatoes from supervised trials in Europe involving foliar applications of cyantraniliprole (OD formulation)

TOMATO Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Perea Greece, 2009 (Meteor) Tomato/	2	0.09	11	800	7	1	whole fruit	ND			DP-27712 Test 05
Fiorenzuola d'Arda Italy, 2009 (Heinz 690) Tomato/	2	0.09	11	800	7	1	whole fruit	0.079			DP-27712 Test 06
Aguadulce Spain, 2009 (Manitu) Tomato/	2	0.09	11	800	7	1	whole fruit	0.018			DP-27712 Test 07
Les Massonnes S France, 2009 (Perfect Peel) Tomato	2	0.09	11	800	7	–0 0 1 3	whole fruit	0.038 0.071 0.077 0.068			DP-27712 Test 12
Villalba del Alcor Spain, 2009 (Lidia) Tomato/	2	0.09	11	800	6	–0 0 1 3	whole fruit	0.013 < 0.01 0.02 0.01			DP-27712 Test 13

Table 105 Residues in field tomatoes from supervised trials in Australia involving foliar applications of cyantraniliprole (OD formulation)

TOMATO Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Bowen, QLD Australia, 2009 (Danika) Tomato	2	0.05	0.0125	400	7	0 1 7 14 21	whole fruit	0.11 0.09 0.03 ND ND			DP-31520 Site 1
Bowen, QLD Australia, 2009 (Danika) Tomato	2	0.075	0.019	400	7	0 1 7 14 21	whole fruit	0.19 0.16 0.09 0.02 ND			DP-31520 Site 1
Bowen, QLD Australia, 2009 (Pinacle) Tomato	2	0.05	0.0125	400	7	0 1	whole fruit	ND 0.01			DP-31520 Site 2

TOMATO Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Bowen, QLD Australia, 2009 (Pinacle) Tomato	2	0.075	0.019	400	7	0 1	whole fruit	0.01 0.02			DP-31520 Site 2
Bowen, QLD Australia, 2009 (Pinacle) Tomato	2	0.15	0.0375	400	7	0 1	whole fruit	0.01 0.03			DP-31520 Site 2

Table 106 Residues in protected tomatoes from supervised trials in Europe involving soil (drip irrigation) applications of cyantraniliprole (SC formulation)

TOMATO Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Birac Sur Trec S France, 2009 (Carnegie) Tomato	4	0.1	5	2000	7	–0 0 1 3 7	whole fruit	ND ND ND ND ND			DP-28187 Test 09 [SC soil drip]
Mediglia Italy, 2009 (Naram) Tomato	4	0.1	5	2000	7	–0 0 1 3 7	whole fruit	ND ND ND ND ND			DP-28187 Test 05 [SC soil drip]
Puente del Rio Spain, 2009 (HA 04077) Tomato	4	0.1	5	2000	7	–0 0 1 3 7	whole fruit	ND ND ND ND ND			DP-28187 Test 06 [SC soil drip]
Wellerlooi Netherlands, 2009 (Capricia RZ) Tomato	4	0.1	5	2000	7	–0 0 1 3 7	whole fruit	< 0.01 ND 0.02 ND 0.029, ND			DP-28187 Test 07 [SC soil drip]
Wellerlooi Netherlands, 2009 (Capricia RZ) Tomato	4	0.1	5	2000	6–8	–0 0 1 3 7	whole fruit	0.003 ND 0.005 0.007 0.01			DP-28187 Test 10 [SC soil drip]
Nea Magnisia Greece, 2009 (Winner F1) Cherry Tomato	4	0.1	5	2000	7, 8	–0 0 1 3 7	whole fruit	ND ND ND ND ND			DP-28187 Test 08 [SC soil drip]

TOMATO Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Puente del Rio Spain, 2009 (Sirtaki) Cherry Tomato	4	0.1	5	2000	7, 8	–0 0 1 3 7	whole fruit	ND ND ND ND ND			DP-28187 Test 11 [SC soil drip]
La Mojonera Spain, 2009 (Foster) Cherry Tomato	4	0.1	5	2000	7	–0 0 1 3 7	whole fruit	ND ND ND 0.008 ND			DP-28187 Test 12 [SC soil drip]

Table 107 Residues in protected tomatoes from supervised trials in Europe involving foliar applications of cyantraniliprole (OD formulation)

TOMATO Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Birac Sur Trec S France, 2009 (Carnegie) Tomato/ Indoor	4	0.12	10	1200	7	–0 0 1 3 7	whole fruit	0.038 0.058 0.053 0.049 0.053			DP-28187 Test 09
La Mojonera Spain, 2009 (Ikram) Tomato/ Indoor	4	0.12	10	1200	7	1	whole fruit	0.14			DP-28187 Test 04
Mediglia Italy, 2009 (Naram) Tomato/ Indoor	4	0.12	10	1200	7, 6	1	whole fruit	0.072			DP-28187 Test 05
Nea Magnisia Greece, 2009 (Optima) Tomato/ Indoor	4	0.12	10	1200	7	1	whole fruit	0.043			DP-28187 Test 01
Puente del Rio Spain, 2009 (HA 04077) Tomato/ Indoor	4	0.12	10	1200	7	1	whole fruit	0.18			DP-28187 Test 06
Vittoria Italy, 2009 (Arawak) Tomato/ Indoor	4	0.12	10	1200	7	1	whole fruit	0.14			DP-28187 Test 03

TOMATO Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Vittoria Italy, 2009 (Rovente) Tomato/ Indoor	4	0.12	10	1200	7	1	whole fruit	0.18			DP-28187 Test 02
Wellerlooi Netherlands, 2009 (Capricia RZ) Tomato/ Indoor	4	0.12	10	1200	7	1	whole fruit	0.15			DP-28187 Test 07
Wellerlooi Netherlands, 2009 (Capricia RZ) Tomato/ Indoor	4	0.12	10	1200	6–8	–0 0 1 3 7	whole fruit	0.14 0.26 0.19 0.18 0.22			DP-28187 Test 10
Nea Magnisia Greece, 2009 (Winner F1) Cherry Tomato/ Indoor	4	0.12	10	1200	7	1	whole fruit	0.14			DP-28187 Test 08
Puente del Rio Spain, 2009 (Sirtaki) Cherry Tomato/ Indoor	4	0.12	10	1200	7, 8	–0 0 1 3 7	whole fruit	0.095 0.68 0.57 0.62 ND			DP-28187 Test 11
La Mojonera Spain, 2009 (Foster) Cherry Tomato/ Indoor	4	0.12	10	1200	7	–0 0 1 3 7	whole fruit	0.099 0.16 0.42 0.16 0.17			DP-28187 Test 12
Belfeld Netherlands, 2009 (Cabbricia) Cherry Tomato/ Indoor	4	0.12	10	1200	7	–0 0 1 3 7	whole fruit	0.42 0.49 0.59 0.44 0.41			DP-28187 Test 13
Caphan France, 2009 (Amorouso) Cherry Tomato/ Indoor	4	0.1	5	2000	6, 7	–0 0 1 3 7	whole fruit	0.35 0.49 0.37 0.4 0.36			DP-28187 Test 14

Table 108 Residues in sweet peppers from supervised trials in North America involving two or three foliar applications of cyantraniliprole (OD formulation)

PEPPERS, SWEET Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference Comments
	no	kg ai/ha	kg ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Arroyo Grande, CA USA, 2008 (Crusader) Bell Pepper/	2	0.15	70 64	220 240	5	1	whole fruit	0.031, 0.052	0.041		DP-25643 Trial 15
Bradenton, FL USA, 2008 (Camelot) Bell Pepper/	2	0.15	38	390	5	1	whole fruit	0.14, 0.14	0.14		DP-25643 Trial 11
Fresno, CA USA, 2008 (Indra) Bell Pepper/	2	0.15	53	280	5	1	whole fruit	0.19, 0.2	0.2		DP-25643 Trial 14
Raymondville, TX USA, 2008 (Camelot) Bell Pepper/	2	0.15	40	380	5	1	whole fruit	0.17, 0.12	0.15		DP-25643 Trial 12
San Ardo, CA USA, 2008 (Moody) Bell Pepper/	2	0.15	53	290	4	1	whole fruit	0.11, 0.097	0.1		DP-25643 Trial 13
Bradenton, FL USA, 2008 (Camelot) Bell Pepper/	3	0.15	38	390	5	1	whole fruit	0.26, 0.23	0.24		DP-25643 Trial 11
Branchton, ON CAN, 2009 (Permit) Bell Pepper/	3	0.15	73	210	6	1	whole fruit	0.082, 0.068	0.075		DP-25643 Trial 32
Carlyle, IL USA, 2009 (California Wonder) Bell Pepper/	3	0.15	68	230	5	1	whole fruit	0.1, 0.064	0.082		DP-25643 Trial 31
Conklin, MI USA, 2009 (Aristotle X3R) Bell Pepper/	3	0.15	52	290	5	1	whole fruit	0.074, 0.061	0.067		DP-25643 Trial 34
Delavan, WI USA, 2009 (Keystone) Bell Pepper/	3	0.15	76	200	5	1	whole fruit	0.033, 0.03	0.032		DP-25643 Trial 33
Fresno, CA USA, 2008 (Indra) Bell Pepper/	3	0.15	53	280	5	1	whole fruit	0.28, 0.28	0.28		DP-25643 Trial 14

PEPPERS, SWEET Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference Comments
	no	kg ai/ha	kg ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Raymondville, TX USA, 2008 (Camelot) Bell Pepper/	3	0.15	40	380	5	1	whole fruit	0.22, 0.2	0.21		DP-25643 Trial 12
San Ardo, CA USA, 2008 (Moody) Bell Pepper/	3	0.15	53	290	4, 5	1	whole fruit	0.12, 0.17	0.15		DP-25643 Trial 13
Seven Springs, NC USA, 2009 (California Wonder) Bell Pepper/	1+ 2	0.15 0.15	59 73	250 210	5	1	whole fruit	0.066, 0.05	0.058		DP-25643 Trial 30
Verona, WI USA, 2009 (King Arthur) Bell Pepper/	2+ 1	0.15 0.15	42 57	370 260	6, 5	1	whole fruit	0.078, 0.071	0.074		DP-25643 Trial 35
Arroyo Grande, CA USA, 2008 (Crusader) Bell Pepper/	1+ 1+ 1	0.15 0.15 0.14	70 64 58	220 240 240	5	1	whole fruit	0.046, 0.042	0.044		DP-25643 Trial 15
Bradenton, FL USA, 2008 (Camelot) Bell Pepper/	2+ 1	0.15 (soil) 0.15	4 39	3800 380	7	1	whole fruit	0.088, 0.095	0.092		DP-25643 Trial 11 2 soil dripline + 1 foliar

Soil (dripline irrigation) treatment rate was 2.3 g ai/100 metres of row

Table 109 Residues in non-bell peppers from supervised trials in North America involving two or three foliar applications of cyantraniliprole (OD formulation)

PEPPERS Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference Comments
	no	kg ai/ha	kg ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Fresno, CA USA, 2008 (Jalafuego NonBell Pepper/	2	0.15	53	280	5	1	whole fruit	0.39, 0.34	0.36		DP-25643 Trial 18
Paso Robles, CA USA, 2008 (Jalapeno NonBell Pepper/	2	0.15	51	290	5	1	whole fruit	0.19, 0.28	0.24		DP-25643 Trial 19

PEPPERS Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference Comments
	no	kg ai/ha	kg ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Porterville, CA USA, 2008 (Anaheim TMR 23) NonBell Pepper/	2	0.15	39	390	5	1	whole fruit	0.33, 0.41	0.37		DP-25643 Trial 17
Raymondville, TX USA, 2008 (TAM Mild Jalapeno NonBell Pepper/	2	0.15	40	380	5	1	whole fruit	0.22, 0.18	0.2		DP-25643 Trial 16
Branchton, ON CAN, 2009 (Sweet Banana) NonBell Pepper/	3	0.15	72	220	4, 5	1	whole fruit	0.069, 0.071	0.07		DP-25643 Trial 37
Carlyle, IL USA, 2009 (Jalapeno) NonBell Pepper/	3	0.15	57	270	5	1	whole fruit	0.071, 0.066	0.068		DP-25643 Trial 36
Conklin, MI USA, 2009 (New Park) NonBell Pepper/	3	0.15	50	300	5	1	whole fruit	0.098, 0.091	0.095		DP-25643 Trial 40
Delavan, IL USA, 2009 (Poblano Ancho) NonBell Pepper/	3	0.15	75	200	5	1	whole fruit	0.083, 0.074	0.079		DP-25643 Trial 38
Fresno, CA USA, 2008 (Jalafuego NonBell Pepper/	3	0.15	53	280	5	1	whole fruit	0.38, 0.47	0.42		DP-25643 Trial 18
Paso Robles, CA USA, 2008 (Jalapeno NonBell Pepper/	3	0.15	51	290	5	1	whole fruit	0.24, 0.37	0.31		DP-25643 Trial 19
Porterville, CA USA, 2008 (Anaheim TMR 23) NonBell Pepper/	3	0.15	39	390	5, 6	1	whole fruit	0.09, 0.091	0.09		DP-25643 Trial 17
Raymondville, TX USA, 2008 (TAM Mild Jalapeno NonBell Pepper/	3	0.15	40	380	5	1	whole fruit	0.23, 0.28	0.25		DP-25643 Trial 16
Richland, IA USA, 2009 (Early Jalapeno) NonBell Pepper/	3	0.15	70	220	5	1	whole fruit	0.16, 0.25	0.2		DP-25643 Trial 39

PEPPERS Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference Comments
	no	kg ai/ha	kg ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Porterville, CA USA, 2008 (Anaheim TMR 23) NonBell Pepper/	2+1	0.15 (soil) 0.15	0.74 39	20400 380	7	1	whole fruit	0.21, 0.15	0.18		DP-25643 Trial 17 2 soil dripline + 1 foliar

Soil (dripline irrigation) treatment rate was 2.3 g ai/100 metres of row

Table 110 Residues in field peppers from supervised trials in Europe involving foliar applications of cyantraniliprole (OD formulation)

PEPPERS Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Poirino Italy, 2009 (Corno di Cramagnola) Pepper/	2	0.09	11	800	7	1	whole fruit	0.077			DP-27712 Test 01
Los Palacios Spain, 2009 (Negrillo) Pepper/	2	0.09	11	800	7	1	whole fruit	0.096			DP-27712 Test 02
Chalkidona Greece, 2009 (Raiko) Pepper/	2	0.09	11	800	7	1	whole fruit	0.081			DP-27712 Test 03
Clairac S France, 2009 (Alléla) Pepper/	2	0.09	11	800	7	–0 0 1 3	whole fruit	0.062 0.067 0.068 0.059			DP-27712 Test 08
Villalba del Alcor Spain, 2009 (Italico) Pepper/	2	0.09	11	800	6	–0 0 1 3	whole fruit	0.031 0.09 0.066 0.047			DP-27712 Test 09
Contrada Gelso Bianco Italy, 2009 (Pyros) Hot pepper/	2	0.09	11	800	7	1	whole fruit	0.82			DP-27712 Test 04
Perea Greece, 2009 (Magnisia) Hot pepper	2	0.09	11	800	7	–0 0 1 3	whole fruit	0.13 0.35 0.32 0.23			DP-27712 Test 10
Utrera Spain, 2009 (Fire Flame) Hot pepper	2	0.09	11	800	7	–0 0 1 3	whole fruit	0.19 0.61 0.59 0.58			DP-27712 Test 11

PEPPERS Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Contrada Gelso, Bianco, Sicily Italy, 2010 (Not specified) Hot pepper	2	0.09	11	800	7	–0 0 1 3	whole fruit	0.13			DP-27712 Test 14
Aguadulce Spain 2010 (Not specified) Hot pepper	2	0.09	11	800	7	1	whole fruit	0.24			DP-27712 Test 15

Table 111 Residues in field sweet peppers from supervised trials in Australia involving foliar applications of cyantraniliprole (OD formulation)

PEPPER Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Bowen, QLD Australia, 2009 (Warlock) Capsicum	2	0.05	0.0125	400	7	0 1	whole fruit	ND < 0.01			DP-31520 Site 3
Bowen, QLD Australia, 2009 (Warlock) Capsicum	2	0.075	0.019	400	7	0 1	whole fruit	ND 0.02			DP-31520 Site 3
Bowen, QLD Australia, 2009 (Warlock) Capsicum	2	0.15	0.0375	400	7	0 1	whole fruit	0.01 0.02			DP-31520 Site 3

Table 112 Residues in protected sweet and chilli peppers from supervised trials in Europe involving involving soil (drip irrigation) applications of cyantraniliprole (SC formulation)

PEPPERS Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Los Palacios, Andalucia, Spain, 2008 (Palermo) Pepper	2	0.1	5	2000	7	–0 0 1 3 7 14	whole fruit	ND ND ND ND ND ND			DP-23885 Test 03a [soil dripline]

PEPPERS Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Los Palacios, Andalucia, Spain, 2008 (Palermo) Pepper	2	0.1	5	2000	7	–0 0 1	whole fruit	ND ND ND			DP-23885 Test 03b [soil dripline]
NK Belfeld Netherlands, 2008 (Sensation) Sweet Pepper	2	0.1	5	2000	8	–0 0 1 3 7 14	whole fruit	ND ND ND ND ND 0.009			DP-23885 Test 01a [soil dripline]
NK Belfeld Netherlands, 2008 (Sensation) Sweet Pepper	2	0.1	5	2000	8	–0 0 1	whole fruit	ND ND ND			DP-23885 Test 01b [soil dripline]
Berja Spain, 2009 (Melchor) Pepper	4	0.1	5	2000	7	–0 0 1 3 7	whole fruit	0.007 0.005 0.006 0.008 0.008			DP-28188 Test 05 [soil dripline]
El Puente del Rio Spain, 2009 (California Rojo) Pepper	4	0.1	5	2000	7	–0 0 1 3 7	whole fruit	ND ND ND ND ND			DP-28188 Test 11 [soil dripline]
El Puente del Rio Spain, 2009 (Italico) Pepper	4	0.1	5	2000	7	–0 0 1 3 7	whole fruit	0.005 0.005 ND 0.007 0.006			DP-28188 Test 12 [soil dripline]
El Puente del Rio Spain, 2009 (Lamullo) Pepper/	4	0.1	5	2000	7	–0 0 1 3 7	whole fruit	0.004 0.004 0.005 0.004 0.004			DP-28188 Test 04 [soil dripline]
Los Palacios, Andalucia, Spain, 2008 (Palermo) Pepper	4	0.1	5	2000	7	–0 0 1 3 7 14	whole fruit	ND ND ND ND ND ND			DP-23885 Test 03c [soil dripline]
Los Palacios, Andalucia, Spain, 2008 (Palermo) Pepper	4	0.1	5	2000	7	–0 0 1	whole fruit	ND ND ND			DP-23885 Test 03d [soil dripline]

PEPPERS Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
NE Meterik Netherlands, 2009 (Spider) Pepper	4	0.1	5	2000	7	–0 0 1 3 7	whole fruit	ND ND ND 0.003 ND			DP-28118 Test 01 [soil dripline]
Nea Magnisia Greece, 2009 (Raikon) Pepper	4	0.1	5	2000	7	–0 0 1 3 7	whole fruit	0.007 0.006 0.007 0.006 0.011			DP-28118 Test 02 [soil dripline]
NK Belfeld Netherlands, 2008 (Sensation) Sweet Pepper	4	0.1	5	2000	6–8	–0 0 1 3 7 14	whole fruit	0.009 0.007 0.006 0.006 0.006 ND			DP-23885 Test 01c [soil dripline]
NK Belfeld Netherlands, 2008 (Sensation) Sweet Pepper	4	0.1	5	2000	6–8	–0 0 1	whole fruit	ND 0.079 0.004			DP-23885 Test 01d [soil dripline]
NK Belfeld Netherlands, 2009 (Ferrari) Pepper	4	0.1	5	2000	7	–0 0 1 3 7	whole fruit	ND 0.004 ND ND ND			DP-28118 Test 10 [soil dripline]
Pact S France, 2009 (Minerva) Pepper	4	0.1	5	2000	6–8	–0 0 1 3 7	whole fruit	ND ND ND ND ND			DP-28118 Test 09 [soil dripline]
Poirino Italy, 2009 (Sienor) Pepper/	4	0.1	5	2000	7	–0 0 1 3 7	whole fruit	ND ND ND ND ND			DP-28118 Test 03 [soil dripline]
Chalkidona Greece, 2009 (Local "Magnisias") Hot pepper/	4	0.1	5	2000	7	–0 0 1 3 7	whole fruit	ND ND ND 0.004 0.007			DP-28118 Test 06 [soil dripline]
NM Grashoek Netherlands, 2009 (Midal) Hot pepper/	4	0.1	5	2000	6–8	–0 0 1 3 7	whole fruit	0.004 0.007 0.006 0.007 0.007			DP-28118 Test 07 [soil dripline]

PEPPERS Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Contrada Sughero Torto Italy, 2009 (Romitol) Hot pepper/	4	0.1	5	2000	7	–0 0 1 3 7	whole fruit	ND ND ND ND ND			DP-28118 Test 08 [soil dripline]
Pact S France, 2009 (Capel Hot) Hot pepper/	4	0.1	5	2000	7	–0 0 1 3 7	whole fruit	ND ND ND ND ND			DP-28118 Test 13 [soil dripline]
Berja Spain, 2009 (Fire flame) Hot pepper/	4	0.1	5	2000	7	–0 0 1 3 7	whole fruit	ND ND ND ND ND			DP-28118 Test 14 [soil dripline]

Table 113 Residues in protected peppers from supervised trials in Europe involving foliar applications of cyantraniliprole (OD formulation)

PEPPERS Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
K Belfeld Netherlands, 2008 (Sensation) Sweet Pepper	2	0.12	10	1200	8	–0 0 1 3 7 14	whole fruit	0.023 0.081 0.071 0.03 0.032 0.053			DP-23885 Test 01a
Los Palacios, Andalucia, Spain, 2008 (Palermo) Pepper	2	0.12	10	1200	7	–0 0 1 3 7 14	whole fruit	0.24 0.47 0.35 0.41 0.31 0.24			DP-23885 Test 03a
Los Palacios, Andalucia, Spain, 2008 (Palermo) Pepper	2	0.12	10	1200	7	–0 0 1	whole fruit	0.21 0.51 0.31			DP-23885 Test 03b
NK Belfeld Netherlands, 2008 (Sensation) Sweet Pepper	2	0.12	10	1200	6	–0 0 1	whole fruit	0.010 0.057 0.051			DP-23885 Test 01b
Berja Spain, 2009 (Melchor) Pepper	4	0.12	10	1200	7	1	whole fruit	0.076			DP-28118 Test 05

PEPPERS Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
El Puente del Rio Spain, 2009 (California Rojo) Pepper	4	0.12	10	1200	7	–0 0 1 3 7	whole fruit	0.063 0.14 0.13 0.13 0.12			DP-28118 Test 11
El Puente del Rio Spain, 2009 (Italico) Pepper	4	0.12	10	1200	7	–0 0 1 3 7	whole fruit	0.12 0.16 0.15 0.1 0.094			DP-28118 Test 12
El Puente del Rio Spain, 2009 (Lamullo) Pepper	4	0.12	10	1200	7	1	whole fruit	0.096			DP-28118 Test 04
NE Meterik Netherlands, 2009 (Spider) Pepper	4	0.12	10	1250	7	1	whole fruit	0.13			DP-28118 Test 01
Nea Magnisia Greece, 2009 (Raikon) Pepper/	4	0.12	10	1200	7	1	whole fruit	0.14			DP-28118 Test 02
NK Belfeld Netherlands, 2009 (Ferrari) Pepper	4	0.12	10	1200	7	–0 0 1 3 7	whole fruit	0.13 0.13 0.17 0.17 0.11			DP-28118 Test 10
Pact S France, 2009 (Minerva) Pepper	4	0.12	10	1200	6–8	1	whole fruit	0.12 0.2 0.12 0.13 0.16			DP-28118 Test 09
Poirino Italy, 2009 (Sienor) Pepper	4	0.12	10	1200	7	1	whole fruit	0.12			DP-28118 Test 03
Chalkidona Greece, 2009 (Local "Magnisias") Hot pepper	4	0.12	10	1200	7	1	whole fruit	0.12			DP-28118 Test 06
NM Grashoek Netherlands, 2009 (Midal) Hot pepper	4	0.12	10	1200	6–8	1	whole fruit	1			DP-28118 Test 07

PEPPERS Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Contrada Sughero Torto Italy, 2009 (Romitol) Hot pepper	4	0.12	10	1200	7	1	whole fruit	0.86			DP-28118 Test 08
Pact S France, 2009 (Capel Hot) Hot pepper	4	0.12	10	1200	7	–0 0 1 3 7	whole fruit	0.26 0.14 0.34 0.12 0.24			DP-28118 Test 13
Berja Spain, 2009 (Fire flame) Hot pepper	4	0.12	10	1200	7	–0 0 1 3 7	whole fruit	0.27 0.17 0.12 0.16 0.17			DP-28118 Test 14

Leafy vegetables

Results from supervised trials from Australia, Europe and North America on lettuce, spinach, Lamb's lettuce and scarole were provided to the Meeting.

North America

In trials conducted in North America on head and leaf lettuce and spinach, two or three foliar applications of 0.15 kg ai/ha cyantraniliprole (OD formulation) were applied at 5 day intervals, using 200–500 L/ha, with adjuvant added. Soil treatment plots were also included at several trial sites, either as a soil shank (in furrow) treatment of 0.2 kg ai/ha (SC formulation) at planting or two soil treatments of 0.15 kg ai/ha cyantraniliprole (SC formulation) applied through drip-line irrigation systems (adjusted to pH 4–5, with no added adjuvant).

Duplicate samples of spinach and lettuce leaves, lettuce heads (trimmed/washed and also with wrapper leaves) and celery (with tops and also trimmed/washed) were stored at –20 °C for up to 9 months before extraction and analysis for cyantraniliprole and six metabolites (within 3 months of extraction) using method DP-15736, with reported LOQs of 0.01 mg/kg. Average concurrent recoveries were 96–98% (cyantraniliprole) and 80–106% (metabolites) in samples spiked with 0.01–5.0 mg/kg and up to 10 mg/kg in celery and 17 mg/kg in spinach.

In trials conducted in the USA on mustard greens, two or three foliar applications of 0.15 kg ai/ha cyantraniliprole (OD and SE formulations) were applied at 4–6 day intervals, using 200–500 L/ha, with adjuvant added. In several trials, seperate plots were also treated with the equivalent of 0.2 kg ai/ha as a soil injection (soil shank) treatment at planting (without added surfactant).

Duplicate samples were stored at –20 °C for up to 8 months before extraction and analysis for cyantraniliprole and six metabolites (within 5 months of extraction) using method DP-15736, with reported LOQs of 0.01 mg/kg. Average concurrent recoveries were 90–94% (cyantraniliprole) and 80–103% (metabolites) in samples spiked with 0.01 to 25 mg/kg.

Europe

In trials conducted in Europe on protected head lettuce, leaf (open head) lettuce, Lamb's lettuce and scarole, two to three applications of 3.7–10 g ai/hL cyantraniliprole were applied using 750–800 L/ha (0.078–0.096 kg ai/ha) as foliar sprays (OD formulation) or three to four applications of 0.075–

0.1 kg ai/ha using 2000 L/ha as soil treatments (SC formulation), applied through the dripline irrigation systems. The retreatment intervals in all trials was 7 days and no added surfactants were included.

In outdoor head lettuce, leaf (open head) lettuce, Lamb's lettuce and scarole in Southern Europe, three foliar applications of 0.078 kg ai/ha cyantraniliprole (OD formulation) were applied using 800 L water/ha (5 g ai/hL) or three soil treatments (SC formulation) were applied through the dripline irrigation systems at a rate equivalent to 0.1 kg ai/ha. The retreatment intervals in all trials was 7 days and no added surfactants were included.

Samples were stored at –18 °C for up to 7 months before extraction and analysis for cyantraniliprole and six metabolites analysis (within 7 days of extraction) using method DP-15736, with reported LOQs of 0.01 mg/kg. Average concurrent recoveries were 88–102% (cyantraniliprole) and 83–95% (metabolites) in samples spiked with 0.01 and 0.1 mg/kg and also 2 and 10 mg/kg cyantraniliprole and 0.5 mg/kg IN-J9Z38 metabolite in leaf lettuce.

Table 114 Residues in field head lettuce from supervised trials in North America involving soil applications (SC formulation) at planting or through dripline irrigation systems (with foliar applications—OD formulation)

LETTUCE HEAD Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	kg ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Conklin, MI USA, 2009 Head Lettuce Skyline)	1	0.15	0.74	20400		0 3 7	heads	ND, ND ND, ND < 0.01, < 0.01	ND ND < 0.01		DP-25644 Trial 27 [by drip irrigation]
Conklin, MI USA, 2009 Head Lettuce Skyline)	2	0.15	0.74	20400	7	–0 0 3 7	heads	< 0.01, < 0.01 < 0.01, < 0.01 0.01, < 0.01 < 0.01, < 0.01	< 0.01 < 0.01 < 0.01 < 0.01		DP-25644 Trial 27 [by drip irrigation]
Porterville, CA USA, 2008 Head Lettuce/ Vandenberg)	1+ 1+ 1	0.2 0.1 0.15	97 42 62	200 240 240	73 5	1	heads	0.017, 0.017	0.017		DP-25644 Trial 2 1 soil shank treatment + 2 foliar sprays
Conklin, MI USA, 2009 Head Lettuce Skyline)	2+ 1	0.15 0.15	0.74 52	20400 290	7	–0 1	heads	< 0.01, < 0.01 1, 0.81	< 0.01 0.91		DP-25644 Trial 27 [2 by drip irrigation + 1 foliar]

Soil (shank) treatment rate at planting of 1.5 g ai/100 metres of row (Trial 2)

Soil (dripline irrigation) treatment rate of 1.1 g ai/100 metres of row (Trial 27)

Table 115 Residues in field head lettuce from supervised trials in Europe involving soil (drip irrigation) applications of cyantraniliprole (SC formulation)

LETTUCE HEAD Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	metabolites			
									M1	M2	M4	

LETTUCE HEAD Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	metabolites			
									M1	M2	M4	
L'Aldea Spain, 2009 (Maravilla)	3	0.1	5	2000		–0 0 1 3	heads	0.013 0.017 0.013 0.013				DP-27713 Test 05
Lorgies S France, 2009 (Altadis)	3	0.1	5	2010		–0 0 1 3	heads	ND 0.021 0.009 0.006				DP-27713 Test 06
Nea Magnisia Greece, 2009 (Verdunna)	3	0.1	5	2000		7 –0 0 1 3	heads	0.032 0.12 0.12 0.13				DP-27713 Test 07

Table 116 Residues in field head lettuce from supervised trials in North America involving two or three foliar applications of cyantraniliprole (OD formulation unless specified)

LETTUCE HEAD Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	kg ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Bradenton, FL USA, 2008 (Iceberg)	2	0.15	36	420	5	1	heads trimmed	0.68, 0.75 0.21, 0.21	0.71 0.21		DP-25644 Trial 1
Greenfield, CA USA, 2008 (Telluride)	2	0.15	54	280	5	1	heads trimmed	0.023, 0.045 0.009, 0.018	0.034 0.014		DP-25644 Trial 5
Guadalupe, CA USA, 2008 (Durango)	2	0.15	54	280	5	1	heads trimmed	2.1, 2.9 0.046, 0.027	2.5 0.036	M2=0.01	DP-25644 Trial 4
Porterville, CA USA, 2008 (Vandenberg)	2	0.15	60	250	5	1	heads	0.018, 0.013	0.016		DP-25644 Trial 3
Porterville, CA USA, 2008 Head Lettuce/ Vandenberg)	2	0.15	62	240	5	1	heads	0.016, 0.022	0.019		DP-25644 Trial 2
Santa Maria, CA USA, 2008 (Quest)	2	0.15	65	230	5	1	heads	1.3, 1.1	1.2		DP-25644 Trial 6
Alton, NY USA, 2009 (Ithaca MTO)	3	0.15	40	380	5	1	heads	1.8, 1.9	1.8	M1=0.01	DP-25644 Trial 23

LETTUCE HEAD Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	kg ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Alton, NY USA, 2009 (Ithaca MTO)	3	0.15	40	380	5	1	heads	1.2, 1.9	1.5		DP-25644 Trial 23 [100 SE]
Bradenton, FL USA, 2008 (Iceberg)	3	0.15	36	420	5	1	heads trimmed	1.6, 1.7 0.6, 0.6	1.6 0.6	M1=0.02	DP-25644 Trial 1
Conklin, MI USA, 2009 Skyline)	3	0.15	50	300	5	1	heads	1.6, 1.7	1.7		DP-25644 Trial 27
Conklin, MI USA, 2009 Skyline)	3	0.15	50	300	5	1	heads	2.2, 2	2.1	M4=0.01	DP-25644 Trial 27 [100 SE]
Delavan, WI USA, 2009 (Sun Devil)	3	0.15	73	210	5	1	heads	0.2, 0.16	0.18		DP-25644 Trial 26
Delavan, WI USA, 2009 (Sun Devil)	3	0.15	73	210	5	1	heads	0.18, 0.16	0.17		DP-25644 Trial 26 [100 SE]
Fitchburg, WI USA, 2009 (Summertime)	3	0.15	60	250	5	1	heads	0.17, 0.16	0.16		DP-25644 Trial 24
Fitchburg, WI USA, 2009 (Summertime)	3	0.15	60	250	5	1	heads	0.12, 0.15	0.13		DP-25644 Trial 24 [100 SE]
Greenfield, CA USA, 2008 (Telluride)	3	0.15	54	280	5	1	heads trimmed	0.56, 0.95 0.004, ND	0.75 ND		DP-25644 Trial 5
Guadalupe, CA USA, 2008 (Durango)	3	0.15	53	290	5	1	heads trimmed	2.9, 2.6 0.008, 0.012	2.7 0.01	M1=0.01 M2=0.02	DP-25644 Trial 4
Porterville, CA USA, 2008 (Vandenberg)	3	0.15	60	250	5	1	heads	0.021, 0.015	0.018		DP-25644 Trial 3
Porterville, CA USA, 2008 Head Lettuce/ Vandenberg)	3	0.15	62	240	5	1	heads	0.05, 0.12	0.084		DP-25644 Trial 2
Princeton, ON CAN, 2009 (Mighty Joe)	3	0.15	72	210	5	1	heads	0.39, 0.89	0.64		DP-25644 Trial 25
Princeton, ON CAN, 2009 (Mighty Joe)	3	0.15	72	210	5	1	heads	0.53, 0.46	0.5		DP-25644 Trial 25 [100 SE]
Santa Maria, CA USA, 2008 (Quest)	3	0.15	65	230	5	1	heads	1.1, 1.5	1.3	M2=0.01	DP-25644 Trial 6

LETTUCE HEAD Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	kg ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Canby, OR USA, 2009 (Sniper)	1+ 1+ 1	0.15 0.14 0.16	53 52 54	290 270 300	5	1	heads	0.5, 0.56	0.53		DP-25644 Trial 28
Canby, OR USA, 2009 (Sniper)	2+ 1	0.15 0.17	54 53	280 310	5	1	heads	0.85, 0.82	0.83		DP-25644 Trial 28 [100 SE]

Trimmed=washed after outer leaves were removed

M1: Average residues of metabolite IN-J9Z38

M2: Average residues of metabolite IN-MYX98

M4: Average residues of metabolite IN-MLA84

Table 117 Residues in field head lettuce from supervised trials in Europe involving 3 foliar applications of cyantraniliprole (OD formulation)

LETTUCE HEAD Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	metabolites			
									M1	M2	M4	
L'Aldea Spain, 2009 (Maravilla)	3	0.08	9.8	800		–0 0 1 3	heads	0.092 0.64 0.5 0.16				DP-27713 Test 05
Lorgies S France, 2009 (Altadis)	3	0.08	9.8	830		–0 0 1 3	heads	0.062 0.15 0.045 0.037				DP-27713 Test 06
Nea Magnisia Greece, 2009 (Verdunna)	3	0.08	9.8	800		–0 0 1 3	heads	0.37 2.8 2.4 2.3	0.02 0.03 0.02 0.02	 0.01	0.02 0.02 0.02 0.02	DP-27713 Test 07

M1: Average residues of metabolite IN-J9Z38

M2: Average residues of metabolite IN-MYX98

M4: Average residues of metabolite IN-MLA84

Table 118 Residues in protected head lettuce from supervised trials in Europe involving soil (drip irrigation) applications of cyantraniliprole (SC formulation)

LETTUCE HEAD Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	metabolites			
									M1	M2	M4	
Azzano S. Paolo Italy, 2008 (Loira)	2	0.07	3.7	2000	8, 7	–0 0 1 3 7 14	heads	< 0.01, 0.031 < 0.01, < 0.01 ND, 0.01 0.048 0.017 < 0.01				DP-21415 Test 02

LETTUCE HEAD Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	metabolites			
									M1	M2	M4	
Los Palacios Spain, 2008 (Filipu)	2	0.07	3.7	2000	7	–0 0 1 3 7 14	heads	ND, 0.026 < 0.01, 0.019 0.017, 0.013 0.017 0.021 0.013				DP-21415 Test 03
Lucenay France, 2009 (Dedale)	3	0.1	5	2000	7	–0 0 1 3	heads	0.02 0.012 0.006 0.034				DP-28200 Test 10
Utrera Spain, 2009 (Iceberg)	3	0.1	5	2000	7	–0 0 1 3	heads	0.009 0.007 0.009 0.008				DP-28200 Test 09
Azzano S. Paolo Italy, 2008 (Loira)	4	0.07	3.7	2000	7–6	–0 0 1 3 7 14	heads	< 0.01, 0.01 < 0.01, 0.024 < 0.01, ND < 0.01 0.011 0.011				DP-21415 Test 02
Los Palacios Spain, 2008 (Filipu)	4	0.07	3.7	2000	8–6	–0 0 1 3 7 14	heads	0.041, 0.071 0.053, 0.067 0.052, 0.028 0.055 0.049 0.013				DP-21415 Test 03

Table 119 Residues in protected head lettuce from supervised trials in Europe involving foliar applications of cyantraniliprole (SC formulation)

LETTUCE HEAD Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	metabolites		
									M1	M4	
Los Palacios Spain, 2008 (Filipu)	2				7	–0 0 1 3 7 14	heads	1.5, 0.56 2.0, 0.92 2.3, 0.13 1.9 1.4 ND			DP-21415 Test 03
Azzano S. Paolo Italy, 2008 (Loira)	2	0.1	13	750	7, 6	–0 0 1 3 7 14	heads	1.1, 2.9 4.9, 6.1 4.1, 7.4 3.6 3.6 2.6	0.04 0.04, 0.03 0.04, 0.02 0.05 0.05 0.02	0.03 0.02, 0.01 0.03, 0.02 0.02 0.02 0.02	DP-21415 Test 02
Azzano San Paolo Italy, 2009 (Nacre)	3	0.08	9.7	820	6, 8	–0 0 1 3	heads	0.95 3.1 2.9 2.7	 0.01 0.02 0.01	 0.01 0.02 0.01	DP-28200 Test 01

LETTUCE HEAD Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	metabolites		
									M1	M4	
Chalkidona Greece, 2009 (Sonia)	3	0.08	9.8	800	7	–0 0 1 3	heads	2.9 6.4 6.3 5.6	0.02 0.03 0.03 0.02	0.01 0.02 0.02 0.02	DP-28200 Test 02
Lucenay France, 2009 (Dedale)	3	0.08	9.8	780	7	–0 0 1 3	heads	1.4 2 2 3	0.01 0.02 0.02 0.02	 0.01 0.01 0.01	DP-28200 Test 10
Utrera Spain, 2009 (Iceberg)	3	0.08	9.8	810	7	–0 0 1 3	heads	0.006 0.009 0.009 0.006			DP-28200 Test 09

M1: Average residues of metabolite IN-J9Z38

M4: Average residues of metabolite IN-MLA84

Table 120 Residues in field leaf lettuce from supervised trials in North America involving soil applications (SC formulation) at planting or through dripline irrigation systems (with foliar applications-OD formulation)

LETTUCE LEAF Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	kg ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Conklin, MI USA, 2009 (Bergam's Green)	1	0.15	0.74	20400		0 3 7	leaves	ND, ND 0.015, 0.015 0.028, 0.022	ND 0.015 0.025		DP-25644 Trial 33 [drip irrigation]
Conklin, MI USA, 2009 (Bergam's Green)	2	0.15	0.74	20400	7	–0 0 3 7	leaves	0.028, 0.022 0.025, 0.032 0.023, 0.028 0.016, 0.015	0.025 0.028 0.026 0.016		DP-25644 Trial 33 [drip irrigation]
Fresno, CA USA, 2008 (Big Star)	1+ 1+ 1	0.2 0.1 0.15	107 36 54	190 280 280	68 5	1	leaves	1, 1.1	1.1		DP-25644 Trial 12 1 soil shank treatment + 2 foliar sprays
Conklin, MI USA, 2009 (Bergam's Green)	2+ 1	0.15 0.15	0.74 51	20400 290	7 7	–0 1	leaves	0.016, 0.015 1.8, 1.6	0.016 1.7		DP-25644 Trial 33 [2 by drip irrigation + 1 foliar]

Soil (shank) treatment rate of 1.5 g ai/100 metres of row (Trial 12)

Soil (dripline irrigation) treatment rate of 1.1 g ai/100 metres of row (Trial 27)

Table 121 Residues in field leaf (open head) lettuce from supervised trials in Europe involving soil (drip irrigation) applications of cyantraniliprole (SC formulation)

LETTUCE LEAF Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	metabolites			
									M1	M2	M4	
Utrera Spain, 2009 (Filipus)	3	0.1	5	2000		–0 0 1 3	leaves	0.007 0.007 0.01 0.016				DP-27713 Test 01
Mediglia Italy, 2009 (Canasta)	3	0.1	5	2000		–0 0 1 3	leaves	ND 0.006 ND 0.004				DP-27713 Test 02
Lorgies France, 2009 (Kytare)	3	0.1	5	2030		–0 0 1 3	leaves	0.02 0.05 0.016 0.011				DP-27713 Test 03
Chalkidona Greece, 2009 (Simson)	3	0.1	5	2000		–0 0 1 3	leaves	0.004 0.005 0.004 0.009				DP-27713 Test 04

Table 122 Residues in field leaf lettuce from supervised trials in North America involving two or three foliar applications of cyantraniliprole (OD formulation)

LETTUCE LEAF Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	kg ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Bradenton, FL USA, 2008 (Oak Leaf)	2	0.15	36	430	5	1	leaves	1.3, 0.95	1.1		DP-25644 Trial 7
Fresno, CA USA, 2008 (Big Star)	2	0.15	54	280	5	–0 0 1 3 5	leaves	0.28, 0.25 2.9, 3.0 2, 2.2 1.5, 1.2 0.74, 0.59	0.27 3.0 2.1 1.3 0.67	M1=0.01	DP-25644 Trial 12
King City, CA USA, 2008 (”454”)	2	0.15	40	380	5	1	leaves	4.9, 3.8	4.4	M1=0.01 M2=0.02 M4=0.01	DP-25644 Trial 10
King City, CA USA, 2008 (Sunbelt)	2	0.15	65	230	5	1	leaves	0.42, 0.58	0.5		DP-25644 Trial 11
Porterville, CA USA, 2008 (Buttercrunch)	2	0.15	63	240	5	1	leaves	4.6, 3.9	4.2	M1=0.01	DP-25644 Trial 9
Porterville, CA USA, 2008 (Tehema)	2	0.15	410	37	5	1	leaves	3.6, 4.7	4.2	M1=0.02 M2=0.03 M4=0.04	DP-25644 Trial 8

LETTUCE LEAF Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	kg ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Alton, NY USA, 2009 (Harmony)	3	0.15	53	280	5	1	leaves	2.7, 3.1	2.9	M1=0.02 M2=0.01 M4=0.02	DP-25644 Trial 29a
Alton, NY USA, 2009 (Harmony)	3	0.15	53	280	5	1	leaves	3, 3.3	3.2	M1=0.01 M2=0.01 M4=0.01	DP-25644 Trial 29b [100 SE]
Bradenton, FL USA, 2008 (Oak Leaf)	3	0.15	35	430	5	1	leaves	1.1, 1.4	1.2		DP-25644 Trial 7
Conklin, MI USA, 2009 (Bergam's Green)	3	0.15	51	300	5	1	leaves	1.9, 2	1.9	M4=0.02	DP-25644 Trial 33a
Conklin, MI USA, 2009 (Bergam's Green)	3	0.15	51	300	5	1	leaves	2.5, 2.5	2.5		DP-25644 Trial 33b [100 SE]
Corvallis, OR USA, 2009 (Waldmanns Green)	3	0.15	53	280	5	1	leaves	4.2, 3.5	3.9	M1=0.02 M2=0.01 M4=0.03	DP-25644 Trial 34a
Corvallis, OR USA, 2009 (Waldmanns Green)	3	0.15	53	280	5	1	leaves	5.8, 4.8	5.3	M1=0.02 M2=0.02 M4=0.03	DP-25644 Trial 34b [100 SE]
Delavan, WI USA, 2009 (Salad Bowl)	3	0.15	73	210	5	1	leaves	7.4, 6.3	6.8	M1=0.02 M2=0.02 M4=0.02	DP-25644 Trial 32a
Delavan, WI USA, 2009 (Salad Bowl)	3	0.15	75	200	5	1	leaves	7.7, 5.2	6.4	M1=0.02 M2=0.02 M4=0.01	DP-25644 Trial 32b [100 SE]
Fresno, CA USA, 2008 (Big Star)	3	0.15	54	280	5	–0 1	leaves	0.74, 0.59 2.6, 2.1	0.67 2.4		DP-25644 Trial 12
King City, CA USA, 2008 ("454")	3	0.15	40	380	5	1	leaves	3, 3.6	3.3	M1=0.01 M4=0.02	DP-25644 Trial 10
King City, CA USA, 2008 (Sunbelt)	3	0.15	70	220	5	1	leaves	1.4, 0.92	1.1		DP-25644 Trial 11
Porterville, CA USA, 2008 (Buttercrunch)	3	0.15	63	240	5	1	leaves	1.9, 2.2	2.1	M1=0.01	DP-25644 Trial 9
Porterville, CA USA, 2008 (Tehema)	3	0.15	410	37	5	1	leaves	4, 3.9	4.0	M1=0.01 M2=0.02 M4=0.04	DP-25644 Trial 8
Princeton, ON CAN, 2009 (Summer Star)	1+ 1+ 1	0.15 0.15 0.14	76 71 73	200 220 200	5 4	1	leaves	2.2, 2.5	2.4		DP-25644 Trial 31a

LETTUCE LEAF Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	kg ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Princeton, ON CAN, 2009 (Summer Star)	3	0.16 0.15 0.14	76 74 73	200 200 200	5 4	1	leaves	2.3, 2.5	2.4		DP-25644 Trial 31b [100 SE]
Carlyle, IL USA, 2009 (Loose Leaf Oakleaf)	1+ 1+ 1	0.15 0.15 0.15	45 50 72	340 300 200	5	1	leaves	5, 5.8	5.4	M1=0.03 M2=0.02 M4=0.06	DP-25644 Trial 30a
Carlyle, IL USA, 2009 (Loose Leaf Oakleaf)	1+ 1+ 1	0.15 0.15 0.15	46 50 72	330 300 200	5	1	leaves	6.3, 7.4	6.8	M1=0.04 M2=0.02 M4=0.05	DP-25644 Trial 30b [100 SE]

M1: Average residues of metabolite IN-J9Z38

M2: Average residues of metabolite IN-MYX98

M4: Average residues of metabolite IN-MLA84

Table 123 Residues in field leaf (open head) lettuce from supervised trials in Europe involving three foliar applications of cyantraniliprole (OD formulation)

LETTUCE LEAF Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	metabolites			
									M1	M2	M4	
Utrera Spain, 2009 (Filipus)	3	0.08	9.8	800		–0 0 1 3	leaves	0.27 0.49 0.94 0.4				DP-27713 Test 01
Mediglia Italy, 2009 (Canasta)	3	0.08	9.8	800		–0 0 1 3	leaves	0.047 0.69 0.5 0.41			 0.01	DP-27713 Test 02
Lorgies France, 2009 (Kytare)	3	0.08	9.8	810		–0 0 1 3	leaves	0.14 0.42 0.18 0.088			0.01 0.01	DP-27713 Test 03
Chalkidona Greece, 2009 (Simson)	3	0.08	9.8	810		–0 0 1 3	leaves	0.054 0.91 0.81 0.49	 0.01		 0.01	DP-27713 Test 04

M1: Average residues of metabolite IN-J9Z38

M4: Average residues of metabolite IN-MLA84

Table 124 Residues in protected leaf (open head) lettuce from supervised trials in Europe involving soil (drip irrigation) applications of cyantraniliprole (SC formulation)

LETTUCE LEAF Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)		Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	metabolites	

LETTUCE LEAF Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)		Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	metabolites	
Treviolo Italy, 2009 (Gentile)	3	0.1	5	2000	7	–0 0 1 3	leaves	0.01 0.008 0.032 0.083		DP-28820 Test 11
Utrera Spain, 2009 (Filipu)	3	0.1	5	2000	7	–0 0 1 3	leaves	0.011 0.011 0.007 0.18		DP-28200 Test 12
Lleida Spain, 2009 (Inverna)	3	0.1	5	2000	8, 6	–0 0 1 3	leaves	0.012 0.014 0.009 0.009		DP-28200 Test 13

Table 125 Residues in protected leaf (open head) lettuce from supervised trials in Europe involving foliar applications of cyantraniliprole (SC formulation)

LETTUCE LEAF Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	metabolites		
									M1	M4	
Nea Magnisia Greece, 2009 (Bera)	3	0.08	9.8	800	7	–0 0 1 3	leaves	2.5 4.4 4.4 4.9	0.01 0.02 0.02 0.02	0.02 0.02 0.01 0.02	DP-28200 Test 03
Lucenay France, 2009 (Kidam)	3	0.08	9.8	800	7	–0 0 1 3	leaves	1.9 4 4.6 3.6	0.03 0.05 0.07 0.06	0.01 0.02 0.03 0.02	DP-28200 Test 04
Treviolo Italy, 2009 (Gentile)	3	0.08	9.8	800	7	–0 0 1 3	leaves	0.28 2.4 1.6 1.1	 0.01 0.01 	0.01 0.02 0.02 0.01	DP-28200 Test 11
Utrera Spain, 2009 (Filipu)	3	0.08	9.8	810	7	–0 0 1 3	leaves	0.022 1.1 0.49 0.012			DP-28200 Test 12
Lleida Spain, 2009 (Inverna)	3	0.08	9.8	800	8, 6	–0 0 1 3	leaves	0.12 0.8 0.78 0.12			DP-28200 Test 13

M1: Average residues of metabolite IN-J9Z38

M4: Average residues of metabolite IN-MLA84

Table 126 Residues in protected Lamb's lettuce from supervised trials in Europe involving soil (drip irrigation) applications of cyantraniliprole (SC formulation)

LAMB'S LETTUCE Location Country, year	Application					DAT (days)	Matrix	Residues (mg/kg)		Reference & Comments
	no	kg ai/ha	g ai/hL	water	RTI			cyantraniliprole	metabolites	

(variety)				L/ha	(days)				M1		
Treviolo	3	0.1	5	2000	6, 7	–0	leaves	0.025			DP-28200
Italy, 2009						0		0.015			Test 14
(Macholong)						1		0.19	0.01		
						3		0.016			
Azzano San	3	0.1	5	2000	6, 8	–0	leaves	0.055	0.01		DP-28200
Paolo						0		0.064			Test 15
Italy, 2009						1		0.057			
(Tropi)						3		0.078	0.01		
Lleida	3	0.1	5	2000	7, 6	–0	leaves	0.025			DP-28200
Spain, 2009						0		0.099			Test 16
(Verte de						1		0.045			
Cambrai)						3		0.034			

Table 127 Residues in protected Lamb's lettuce from supervised trials in Europe involving foliar applications of cyantraniliprole (SC formulation.

LAMB'S LETTUCE Location	Application					DAT (days)	Matrix	Residues (mg/kg)				Reference & Comments
Country, year (variety)	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	metabolites			
									M1	M2	M4	
Cadreita	3	0.08	9.8	810	8, 6	–0	leaves	2.8	0.11		0.06	DP-28200
Spain, 2009						0		5.3	0.12		0.06	Test 05
(Trophy)						1		4.9	0.12		0.06	
						3		4.9	0.14		0.07	
Dommartin	3	0.08	9.7	790	7	–0	leaves	3	0.02			DP-28200
France, 2010						0		4.4	0.03			Test 06
(Gala)						1		3.7	0.03			
						3		3.7	0.03			
Treviolo	3	0.08	9.7	800	6, 7	–0	leaves	4.7	0.07	0.01	0.02	DP-28200
Italy, 2009						0		3.6	0.08	0.02	0.02	Test 14
(Macholong)						1		6.9	0.09	0.02	0.03	
						3		6.5	0.06	0.02	0.02	
Azzano San	3	0.08	9.7	790	6, 8	–0	leaves	4	0.09	0.01	0.05	DP-28200
Paolo						0		8	0.14		0.07	Test 15
Italy, 2009						1		6.6	0.14	0.01	0.06	
(Tropi)						3		7.4	0.17		0.08	
Lleida	3	0.08	9.8	800	7, 6	–0	leaves	0.37	0.01			DP-28200
Spain, 2009						0		4	0.03			Test 16
(Verte de						1		1.7	0.02			
Cambrai)						3		0.73	0.02			

M1: Average residues of metabolite IN-J9Z38

M2: Average residues of metabolite IN-MYX98

M4: Average residues of metabolite IN-MLA84

Table 128 Residues in spinach from supervised trials in the USA involving soil applications at planting or through dripline irrigation systems (with foliar applications).

SPINACH Location	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
Country, year (variety)	no	kg ai/ha	kg ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	

SPINACH Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	kg ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Conklin, MI USA, 2009 Spinach (Melody)	1	0.15	0.59	25500		0 3 7	leaves	< 0.01, < 0.01 < 0.01, < 0.01 < 0.01, < 0.01	< 0.01 < 0.01 < 0.01		DP-25644 Trial 38 [by drip irrigation]
Conklin, MI USA, 2009 Spinach (Melody)	1+ 1	0.15 0.15	0.59 0.74	25500 20400	7	–0 0 3 7	leaves	< 0.01, < 0.01 < 0.01, < 0.01 0.01, < 0.01 < 0.01, < 0.01	< 0.01 < 0.01 < 0.01 < 0.01		DP-25644 Trial 38 [by drip irrigation]
Terra Bella, CA USA, 2008 Spinach (Shasta)	1+ 1+ 1	0.2 0.1 0.15	97 46 71	210 230 220	51 5	1	leaves	6.7, 6.8	6.7	M1=0.05 M2=0.18	DP-25644 Trial 20 [1 soil shank treatment + 2 foliar sprays]
Conklin, MI USA, 2009 Spinach (Melody)	1+ 1+ 1	0.15 0.15 0.15	0.59 0.74 51	25500 20400 300	7 7	–0 1	leaves	< 0.01, < 0.01 6.7, 6.8	< 0.01 6.8	M1=0.03 M2=0.08	DP-25644 Trial 38 2 [by drip irrigation + 1 foliar]

Soil (shank) treatment rate of 1.5 g ai/100 metres of row (Trial 20)

Soil (dripline irrigation) treatment rate of 1.1 g ai/100 metres of row (Trial 38)

M1: Average residues of metabolite IN-J9Z38

M2: Average residues of metabolite IN-MYX98

Table 129 Residues in spinach from supervised trials in the USA involving two or three foliar applications of cyantraniliprole (OD formulation)

SPINACH Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	kg ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
King City, CA USA, 2008 Spinach/	2	0.15	60	250	6	1	leaves	3.1, 3.9	3.5	M1=0.02 M2=0.31	DP-25644 Trial 22
Raymondville, TX USA, 2008 Spinach (Melody)	2	0.15	40	380	5	1	leaves	14, 13	13.0	M1=0.07 M2=0.29	DP-25644 Trial 19
Santa Maria, CA USA, 2008 Spinach (Mizano)	2	0.15	54	280	5	1	leaves	3.5, 3.2	3.4	M1=0.04 M2=0.07	DP-25644 Trial 21
Terra Bella, CA USA, 2008 Spinach (Shasta)	2	0.15	70	220	5	1	leaves	8.2, 8.9	8.5	M1=0.05 M2=0.2	DP-25644 Trial 20

SPINACH Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	kg ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Alton, NY USA, 2009 Spinach (Lombardia)	3	0.15	40	380	5	1	leaves	5.8, 4	4.9	M1=0.07 M2=0.24 M4=0.02	DP-25644 Trial 35
Conklin, MI USA, 2009 Spinach (Melody)	3	0.15	51	290	6, 4	1	leaves	4.5, 4.7	4.6	M1=0.03 M2=0.26 M4=0.01	DP-25644 Trial 38
Corvallis, OR USA, 2009 Spinach (Tiger Cat)	3	0.15	53	280	5	1	leaves	4, 4.3	4.2	M1=0.03 M2=0.22	DP-25644 Trial 40
Jerome, ID USA, 2009 Spinach (Unipack 151)	3	0.15	76	200	7, 5	1	leaves	5.7, 6	5.8	M1=0.03 M2=0.1	DP-25644 Trial 39
Marengo, IL USA, 2009 Spinach (Bloomsdale Long Standing)	3	0.15	66	230	5	1	leaves	10, 10	10	M1=0.09 M2=0.48 M3=0.01 M4=0.06	DP-25644 Trial 37
Raymondville, TX USA, 2008 Spinach (Melody)	3	0.15	40	380	5	1	leaves	13, 13	13	M1=0.09 M2=0.4 M3=0.01 M4=0.01	DP-25644 Trial 19
Santa Maria, CA USA, 2008 Spinach (Mizano)	3	0.15	54	280	5	1	leaves	4, 4.2	4.1	M1=0.05 M2=0.13	DP-25644 Trial 21
Seven Springs, NC USA, 2009 Spinach (Hybrid 7)	3	0.15	65	230	5, 6	1	leaves	4.9, 4.6	4.7	M1=0.04 M2=0.15 M4=0.01	DP-25644 Trial 36
Terra Bella, CA USA, 2008 Spinach (Shasta)	3	0.15	70	220	5	1	leaves	7.9, 8.4	8.2	M1=0.09 M2=0.33 M4=0.01	DP-25644 Trial 20
King City, CA USA, 2008 Spinach/	2+ 1	0.15 0.15	60 62	250 240	6 5	1	leaves	3.6, 4	3.8	M1=0.02 M2=0.14	DP-25644 Trial 22

M1: Average residues of metabolite IN-J9Z38

M2: Average residues of metabolite IN-MYX98

M3: Average residues of metabolite IN-N7B69

M4: Average residues of metabolite IN-MLA84

Table 130 Residues in mustard greens from supervised trials in the USA involving two or three foliar applications of cyantraniliprole (OD formulation)

MUSTARD GREENS Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
King City, CA, USA, 2008 (Mizuna) Mustard Greens/	2	0.15	60	250	6	1	leaves	4.3, 4.7	4.5	M1=0.01 M2=0.03 M4=0.03 M5=0.015	DP-25641 Trial 11
Raymondville, TX, USA, 2008 (Florida Broadleaf) Mustard Greens/	2	0.15	40	380	5	1	leaves	9.5, 11	10	M1=0.05 M2=0.05 M4=0.06 M5=0.02	DP-25641 Trial 10
Terra Bella, CA, USA, 2008 (Southern Giant Curled) Mustard Greens/	2	0.15	70	220	5	1	leaves	4.2, 5.2	4.7	M1=0.03 M2=0.01 M4=0.05	DP-25641 Trial 12
Alton, NY, USA, 2009 (Pizzo) Mustard Greens/	3	0.15	53	280	4, 6	1	leaves	2.2, 2.5	2.4	M1=0.02 M2=0.01 M4=0.02 M5=0.01	DP-25641 Trial 25
Chula, GA, USA, 2009 (Southern Giant Curled) Mustard Greens/	3	0.15	53	290	5	1	leaves	6.4, 5.6	6.0	M1=0.04 M2=0.02 M4=0.04 M5=0.01	DP-25641 Trial 28
Conklin, MI, USA, 2009 (Green Wave) Mustard Greens/	3	0.15	50	300	5	1	leaves	7.4, 6.8	7.1	M1=0.03 M2=0.03 M4=0.03 M5=0.01	DP-25641 Trial 31
Corvallis, OR, USA, 2009 (Green Wave) Mustard Greens/	3	0.15	54	280	5	1	leaves	6.1, 5.5	5.8	M1=0.02 M2=0.02 M4=0.03 M5=0.01	DP-25641 Trial 32
King City, CA, USA, 2008 (Mizuna) Mustard Greens/	3	0.15	60	250	5, 6	1	leaves	2.9, 4.9	3.9	M1=0.01 M2=0.03 M4=0.03 M5=0.015	DP-25641 Trial 11
Newberry, FL, USA, 2009 (Southern Giant Curled) Mustard Greens/	3	0.15	70	220	5, 6	1	leaves	6.8, 9.1	8.0	M1=0.06 M2=0.02 M4=0.09 M5=0.02	DP-25641 Trial 29
North Rose, NY, USA, 2009 (Savanna) Mustard Greens/	3	0.15	53	290	5	1	leaves	3.6, 3.1	3.4	M1=0.02 M2=0.02 M4=0.07 M5=0.02	DP-25641 Trial 26
Raymondville, TX, USA, 2008 (Florida Broadleaf) Mustard Greens/	3	0.15	40	380	5	1	leaves	14, 13	13	M1=0.07 M2=0.07 M3=0.01 M4=0.12 M5=0.04	DP-25641 Trial 10

MUSTARD GREENS Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Seven Springs, NC, USA, 2009 (Southern Giant Curled) Mustard Greens/	3	0.15	59	260	5	1	leaves	20, 17	19	M1=0.22 M2=0.08 M3=0.02 M4=0.23 M5=0.11	DP-25641 Trial 27
Terra Bella, CA, USA, 2008 (Southern Giant Curled) Mustard Greens/	3	0.15	70	220	5	1	leaves	5.4, 5.7	5.5	M1=0.04 M2=0.02 M4=0.1 M5=0.02	DP-25641 Trial 12
Washington, LA, USA, 2009 (Florida Broadleaf) Mustard Greens/	3	0.15	70	220	5	1	leaves	4.5, 9.9	7.2	M1=0.12 M2=0.05 M4=0.19 M5=0.03	DP-25641 Trial 30
Terra Bella, CA, USA, 2008 (Southern Giant Curled) Mustard Greens/	1+ 1+ 1	0.2 (soil) 0.1 0.15	97 47 70	200 220 220	– 5	1	leaves	3.0, 3.3	3.1	M1=0.02 M2=0.03	DP-25641 Trial 12 soil inject at planting+ 2 foliar sprays

M1: Average residues of metabolite IN-J9Z38

M2: Average residues of metabolite IN-MYX98

M3: Average residues of metabolite IN-N7B69

M4: Average residues of metabolite IN-MLA84

M5: Average residues of metabolite IN-JCZ38

Soil injection (shank) treatment rate was 1.8 g ai/100 metres of row

Table 131 Residues in field scarole from supervised trials in Europe involving soil (drip irrigation) applications of cyantraniliprole (SC formulation)

SCAROLE Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	metabolites			
									M1	M2	M4	
Lorgies S France, 2009 (Sacha)	3	0.1	5	2010		–0 0 1 3	leaves	ND 0.09 0.006 ND				DP-27713 Test 08
Chalkidona Greece, 2009 (Full Heart)	3	0.1	5	2000		–0 0 1 3	leaves	0.017 0.076 0.05 0.036				DP-27713 Test 09
Torrevecchia Pia Italy, 2009 (Samoa)	3	0.1	5	2000		–0 0 1 3	leaves	0.011 ND 0.005 0.036				DP-27713 Test 10

Table 132 Residues in field scarole from supervised trials in Europe involving three foliar applications of cyantraniliprole (OD formulation)

SCAROLE Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	metabolites			
									M1	M2	M4	
Lorgies S France, 2009 (Sacha)	3	0.08	9.8	810		–0 0 1 3	leaves	0.083 0.12 0.056 0.059				DP-27713 Test 08
Chalkidona Greece, 2009 (Full Heart)	3	0.08	9.8	800		–0 0 1 3	leaves	0.21 3.3 4.3 2.4	 0.01 0.01		 0.01	DP-27713 Test 09
Torrevecchia Pia Italy, 2009 (Samoa)	3	0.08	9.8	810		–0 0 1 3	leaves	0.65 1.4 1.2 0.66	 0.01		0.01 0.01 0.02 0.01	DP-27713 Test 10

M1: Average residues of metabolite IN-J9Z38

M4: Average residues of metabolite IN-MLA84

Table 133 Residues in protected scarole from supervised trials in Europe involving soil (drip irrigation) applications of cyantraniliprole (SC formulation)

SCAROLE Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)		Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	metabolites	
Utrera Spain, 2009 (Tosca)	3	0.1	5	2000	7	–0 0 1 3	leaves	0.01 0.02 0.014 0.022		DP-28200 Test 17
Roncoferraro Italy, 2009 (Nuance)	3	0.1	5	2000	6, 7	–0 0 1 3	leaves	0.032 0.1 0.091 0.015		DP-28200 Test 18
Lleida Spain, 2009 (Zigal)	3	0.1	5	2000	7	–0 0 1 3	leaves	0.035 0.011 0.022 0.025		DP-28200 Test 19

Table 134 Residues in protected scarole from supervised trials in Europe involving foliar applications of cyantraniliprole (SC formulation)

SCAROLE Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	metabolites		
									M1	M4	
Dommartin France, 2010 (Natadia)	3	0.08	9.7	820	7	–0 0 1 3	leaves	1.4 3.4 2.7 2.2		0.01 0.02 0.01 0.01	DP-28200 Test 07

SCAROLE Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	metabolites		
									M1	M4	
Los Palacios Spain, 2009 (Seychel)	3	0.08	9.7	820	7	–0	leaves	0.8		0.01	DP-28200 Test 08
								2.2		0.02	
						1		1.3		0.01	
						3		1.8	0.01	0.02	
Utrera Spain, 2009 (Tosca)	3	0.08	9.7	810	7	–0	leaves	1.6			DP-28200 Test 17
						0		4.3	0.01	0.03	
						1		5.3	0.01	0.04	
						3		3.3		0.02	
Roncoferraro Italy, 2009 (Nuance)	3	0.08	9.7	800	6, 7	–0	leaves	1.8		0.02	DP-28200 Test 18
						0		3.4	0.02	0.03	
						1		2.8	0.01	0.02	
						3		2.1		0.02	
Lleida Spain, 2009 (Zigal)	3	0.08	9.8	800	7	–0	leaves	0.1			DP-28200 Test 19
						0		0.91			
						1		1.3			
						3		0.19			

M1: Average residues of metabolite IN-J9Z38

M4: Average residues of metabolite IN-MLA84

Legume vegetables

In trials conducted in Europe on common beans in the field and grown under protection, two applications of 0.09 kg ai/ha cyantraniliprole (OD formulation) were applied 7 days apart, using 800 L spray mix/ha with added surfactants in the protected crops but not in the field crops.

Samples of pods (with seeds) and foliage (leaves and stems) were stored at –18 °C for up to 7 months before extraction and analysis for cyantraniliprole and six metabolites analysis (within 2 days of extraction) using method DP-15736, with reported LOQs of 0.01 mg/kg. Average concurrent recoveries were 90–106% (cyantraniliprole) and 84–93% (metabolites) in samples spiked with 0.01, 0.1, 0.2,0.6 and 1.0 mg/kg and also 8 mg/kg cyantraniliprole in foliage.

Table 135 Residues in field common beans from supervised trials in Europe involving two foliar applications of cyantraniliprole (OD formulation)

COMMON BEAN Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	metabolites			
									M1	M2	M4	
Lleida Spain, 2009 (Festival)	2	0.09	11	820	7	1	foliage	4.9	0.14		0.05	DP-27710 Test 01
							pods	0.22				
Palioura Greece, 2009 (Magirus)	2	0.09	11	800	7	1	foliage	5.5	0.02	0.02		DP-27710 Test 02
							pods	0.16				
Herlies N France, 2009 (Arras)	2	0.09	11	780	7	1	foliage	2.0	0.01			DP-27710 Test 03
							pods	0.19				

COMMON BEAN Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	metabolites			
									M1	M2	M4	
Emilia Romagna Italy, 2009 (Flavio)	2	0.09	11	810	7	–0	foliage	0.31				DP-27710 Test 04
						0		4.9	0.02			
						1		1.7	0.01			
						3		1.8	0.01			
						–0	pods	0.023				
						0		0.27				
						1		0.13				
						3		0.12				
Tayrac S France, 2009 (Banga)	2	0.09	11	800	8	–0	foliage	0.22	0.02		0.01	DP-27710 Test 05
						0		7.1	0.05		0.01	
						1		4.3	0.05		0.01	
						3		1.9	0.03			
						–0	pods	0.062				
						0		0.75	0.01			
						1		0.54	0.01			
						3		0.36	0.01			

Foliage=leaves and stems (without pods)

Pods includes seeds

M1: Residues of metabolite IN-J9Z38

M2: Residues of metabolite IN-MYX98

M3: Residues of metabolite IN-N7B69

Table 136 Residues in protected common beans from supervised trials in Europe involving two foliar applications of cyantraniliprole (OD formulation)

COMMON BEAN Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	metabolites			
									M1	M2	M4	
Chiclana de la Frontera Spain, 2009 (Emerite)	2	0.09	12	790	7	–0	pods	0.052				DP-21413 Test 08
						0		0.14				
						1		0.17				
						3		0.13				
						7		0.1				
						14		0.043				
La Sentiu de Sio Spain, 2008 (Nuria)	2	0.1	12	830	7	–0	pods	0.029				DP-21413 Test 02
						0		0.044				
						1		0.12				
						3		0.099				
						7		0.023				
						14		0.016				
La Sentiu de Sio Spain, 2009 (Kilie)	2	0.1	12	820	7	1	pods	0.42				DP-21413 Test 07
Nea Magnisia Greece, 2009 (Trebona)	2	0.1	12	800	7	1	pods	0.14				DP-21413 Test 06

COMMON BEAN Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)				Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	metabolites			
									M1	M2	M4	
Palefito Greece, 2008 (Trebona)	2	0.1	12	800	7	–0 0 1 3 7 14	pods	0.058 0.19 0.16 0.063 0.067 0.021				DP-21413 Test 04
Roncoferraro Italy, 2008 (Bronco)	2	0.1	12	800	7	–0 0 1 3 7 14	pods	0.13 0.49 0.36 0.19 0.22 0.11				DP-21413 Test 01
Roncoferraro Italy, 2009 (Slankette)	2	0.1	12	820	7	1	pods	0.44	0.02			DP-21413 Test 05
St Chamond S France, 2008 (Hemerite)	2	0.1	12	810	7	–0 0 1 3 7 14	pods	0.27 0.94 0.81 0.81 0.55 0.45	 0.01			DP-21413 Test 03
St Chamond S France, 2009 (Hemerite)	2	0.1	12	810	7	1	pods	0.16				DP-21413 Test 09

M1: Residues of metabolite IN-J9Z38

Tuber vegetables

In trials conducted with cyantraniliprole on potatoes in North America, plots were treated with either a pre-planting seed treatment (FS formulation) followed by one late season foliar spray (OD formulation) or with three late season foliar spray applications (OD formulation). Potato seed was treated with 13.5 g ai cyantraniliprole/100 kg of seed (equivalent to 0.15 kg ai/ha based on 2200 kg seed/ ha) and the foliar application was 0.15 kg ai/ha with added surfactant.

In several trials, additional plots were treated with three late season foliar applications of an SE formulation of cyantraniliprole (with added surfactant) or with a pre-plant in-furrow soil treatment (0.3 kg ai/ha, SC formulation) followed by a single late season foliar spray application (0.15 kg ai/ha, OD formulation).

Samples of tubers were stored at –20 °C for up to 11 months before analysis (within 3 months after extraction) for cyantraniliprole and eight metabolites using method DP-15736, with reported LOQs of 0.01 mg/kg. Average concurrent recoveries were 95% (cyantraniliprole) and 97–102% (metabolites) in samples spiked with 0.01 and 0.1 mg/kg.

Table 137 Residues in potato tubers from supervised trials in North America involving three foliar applications of cyantraniliprole (OD or 100 SE formulations)

POTATO Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Alton, NY USA, 2009 (Superior)	3	0.15	40	370	4, 6	6	tubers	< 0.01, < 0.01	< 0.01		DP-27583 Test 01
Alton, NY USA, 2009 (Superior)	3	0.15	40	370	4, 6	6	tubers	< 0.01, < 0.01	< 0.01		DP-27583 Test 01 [100 SE]
Berwick, NS CAN, 2009 (Goldrush)	3	0.15	36	420	6, 4	8	tubers	0.02, 0.016	0.018		DP-27583 Test 04
Fort Saskatchewan, AB CAN, 2009 (Yukon gold)	3	0.15	50	300	5	7	tubers	ND, < 0.01	ND		DP-27583 Test 21
Gardner, ND USA, 2009 (Gold Rush)	3	0.15				7	tubers	< 0.01, < 0.01	< 0.01		DP-27583 Test 12
Germansville, PA USA, 2009 (Reba)	1+ 2	0.14 0.15	50 50	330 300	5, 4	7	tubers	ND, < 0.01	ND		DP-27583 Test 02
Inglisville, NS CAN, 2009 (Superior)	3	0.15	36	420	6, 5	8	tubers	0.011, 0.01	0.011		DP-27583 Test 05
Jerome, ID USA, 2009 (Norland)	3	0.15	79	190	7	7	tubers	ND, ND	ND		DP-27583 Test 14
Jerome, ID USA, 2009 (Russet Burbank)	3	0.15	80	190	4, 5	7	tubers	ND, ND	ND		DP-27583 Test 18
Merritt, BC CAN, 2009 (Russet Burbank)	3	0.15	30	490	5	6	tubers	0.021, 0.018	0.02		DP-27583 Test 16
Merritt, BC CAN, 2009 (Russet Burbank)	3	0.15	30	490	5	6	tubers	0.021, 0.021	0.021		DP-27583 Test 16 [100 SE]
Merritt, BC CAN, 2009 (Russet Burbank)	3	0.15	63	240	5	7	tubers	< 0.01, < 0.01	< 0.01		DP-27583 Test 17
New Glasgow, PE CAN, 2009 (Gold Rush)	3	0.15	73	200	6	7	tubers	< 0.01, < 0.01	< 0.01		DP-27583 Test 07

POTATO Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
New Glasgow, PE CAN, 2009 (Yukon Gold)	3	0.14 0.15	73 73	200 190	6	7	tubers	ND, ND	ND		DP-27583 Test 06
North Rose, NY USA, 2009 (Superior)	3	0.16	80	190	5	7	tubers	ND, ND	ND		DP-27583 Test 03
Northwood, ND USA, 2009 (Pontiac)	3	0.15	79	190	5, 7	7	tubers	ND, ND	ND		DP-27583 Test 11
Oviedo, FL USA, 2009 Fall application (Red Chieftain)	3	0.15	55	280	4	7	tubers	< 0.01, < 0.01	< 0.01		DP-27583 Test 09
Oviedo, FL USA, 2009 Fall application (Red Chieftain)	3	0.15	54	280	4	7	tubers	< 0.01, < 0.01	< 0.01		DP-27583 Test 09 [100 SE]
Oviedo, FL USA, 2009 Spring application (Red Pontiac)	3	0.15	53	280	4	6	tubers	0.033, 0.034	0.034		DP-27583 Test 09
Oviedo, FL USA, 2009 Spring application (Red Pontiac)	3	0.15	53	280	4	6	tubers	0.035, 0.026	0.03		DP-27583 Test 09 [100 SE]
Payette, ID USA, 2009 (Russet Norkatah)	3	0.15	64	230	5	7	tubers	ND, < 0.01	ND		DP-27583 Test 20
Portage la Prairie, MB CAN, 2009 (Umatilla Russet)	3	0.15	74	200	5, 6	8	tubers	ND, ND	ND		DP-27583 Test 10
Richland, IA USA, 2009 (Kennebec)	3	0.15	77	190	4, 5	7	tubers	ND, ND	ND		DP-27583 Test 13
Sanger, CA USA, 2009 (Red La Soda)	3	0.15	37	400	5	7	tubers	ND, ND	ND		DP-27583 Test 15
Seven Springs, NC USA, 2009 (Red Pontiac)	3	0.15	71	220	6, 4	7	tubers	< 0.01, < 0.01	< 0.01		DP-27583 Test 08

POTATO Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Seven Springs, NC USA, 2009 (Red Pontiac)	3	0.15	70	200	6, 4	7	tubers	< 0.01, < 0.01	< 0.01		DP-27583 Test 08 [100 SE]

Table 138 Residues in potato tubers from supervised trials in North America involving seed treatment (or in-furrow) plus one foliar applications of cyantraniliprole

POTATO Location Country, year (variety)	Application				DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	g ai/ 100 kg seed	kg ai/ha	water L/ha			cyantraniliprole	mean	metabolites	
Alton, NY USA, 2009 (Superior)	1+ 1	13.5	0.15	370	6	tubers	0.037, < 0.01	0.023	M1=0.01	DP-27583 Test 01
Germansville, PA USA, 2009 (Reba)	1+ 1	13.5	0.15	300	7	tubers	0.027, 0.027	0.027	M1=0.01	DP-27583 Test 02
North Rose, NY USA, 2009 (Superior)	1+ 1	13.5	0.15	190	7	tubers	ND, ND	ND		DP-27583 Test 03
Berwick, NS CAN, 2009 (Goldrush)	1+ 1	13.5	0.15	420	8	tubers	0.019, 0.027	0.023		DP-27583 Test 04
Inglisville, NS CAN, 2009 (Superior)	1+ 1	13.5	0.15	420	8	tubers	0.052, 0.052	0.052		DP-27583 Test 05
New Glasgow, PE CAN, 2009 (Yukon Gold)	1+ 1	13.5	0.15	200	7	tubers	< 0.01, 0.011	0.01	M1=0.01	DP-27583 Test 06
New Glasgow, PE CAN, 2009 (Gold Rush)	1+ 1	13.5	0.15	200	7	tubers	0.028, 0.033	0.031	M1=0.03	DP-27583 Test 07
Seven Springs, NC USA, 2009 (Red Pontiac)	1+ 1	13.5	0.15	220	7	tubers	0.11	0.11	M1=0.02	DP-27583 Test 08
Oviedo, FL USA, 2009 Fall (Red Chieftain)	1+ 1	13.5	0.15	280	7	tubers	0.012, 0.011	0.011		DP-27583 Test 09
Portage la Prairie, MB CAN, 2009 (Umatilla Russet)	1+ 1	13.5	0.15	200	8	tubers	< 0.01 (3)	< 0.01		DP-27583 Test 10
Northwood, ND USA, 2009 (Pontiac)	1+ 1	13.5	0.15	370	7	tubers	ND, 0.021, < 0.01	< 0.01		DP-27583 Test 11

POTATO Location Country, year (variety)	Application				DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	g ai/ 100 kg seed	kg ai/ha	water L/ha			cyantraniliprole	mean	metabolites	
Gardner, ND USA, 2009 (Gold Rush)	1+ 1	13.5	0.15	190	7	tubers	0.019, 0.021, 0.019	0.02		DP-27583 Test 12
Richland, IA USA, 2009 (Kennebec)	1+ 1	13.5	0.15	190	7	tubers	< 0.01, < 0.01	< 0.01		DP-27583 Test 13
Jerome, ID USA, 2009 (Norland)	1+ 1	13.5	0.15	190	7	tubers	< 0.01, ND	< 0.01		DP-27583 Test 14
Sanger, CA USA, 2009 (Red La Soda)	1+ 1	13.5	0.15	400	7	tubers	0.014, < 0.01	0.01		DP-27583 Test 15
Merritt, BC CAN, 2009 (Russet Burbank)	1+ 1	13.5	0.15	490	6	tubers	0.035, 0.11	0.072		DP-27583 Test 16
Payette, ID USA, 2009 (Ranger Russet)	1+ 1	13.5	0.15	240	7	tubers	ND, < 0.01	< 0.01		DP-27583 Test 17
Jerome, ID USA, 2009 (Russet Burbank)	1+ 1	13.5	0.15	190	7	tubers	< 0.01, 0.05	0.028		DP-27583 Test 18
Ephrata, WA USA, 2009 (Ranger Russet)	1+ 1	13.5	0.15	190	–0 0 1 5 7	tubers	ND, ND ND, < 0.01 ND, < 0.01 ND, ND ND, ND	ND ND ND ND ND		DP-27583 Test 19
Payette, ID USA, 2009 (Russet Norkatah)	1+ 1	13.5	0.15	230	7	tubers	0.01, < 0.01	< 0.01		DP-27583 Test 20
Fort Saskatchewan, AB CAN, 2009 (Yukon gold)	1+ 1	13.5	0.15	300	7	tubers	0.012, 0.016	0.014		DP-27583 Test 21
Ephrata, WA USA, 2009 (Ranger Russet)	1+ 1	in furrow foliar	0.32 0.15	140 190	7	tubers	ND, ND	ND		DP-27583 Test 19

M1: Average residues of metabolite IN-J9Z38

Note: Second sample from Test site 8 reported high residues (cyantraniliprole 3.1 ppm) attributed to the selection of a treated seed piece

In trials conducted with cyantraniliprole on potatoes in Europe, plots were treated with two foliar applications of cyantraniliprole (OD formulation) with and without added surfactant. Targeted application rates were 0.01 kg ai/ha or 0.08 kg ai/ha, applied about 7 days apart using 450–500 L water/ha.

Samples of tubers were were stored at -18 °C for up to 8 months before analysis (within 2 days after extraction) for cyantraniliprole and six metabolites using method DP-15736, with reported LOQs of 0.01 mg/kg. Average concurrent recoveries were 97% (cyantraniliprole) and 88–101% (metabolites) in samples spiked with 0.01 and 0.1 mg/kg.

Table 140 Residues in potato tubers from supervised trials in Europe involving foliar applications of cyantraniliprole (OD formulation)

POTATO Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Ballencrieff Mains UK North, 2008 (Maris Piper)	2	0.01	2.2	410	7	0 0 7 14	tuber	ND ND ND ND			DP-21414 Test 5a
Ballencrieff Mains UK, 2008 (Maris Piper)	2	0.08	18	460	7	0 0 7 14	tuber	ND ND ND ND			DP-21414 Test 5b (with surfactant)
Castello de Farfanya Spain, 2008 (Kenebec)	2	0.01	2.2	470	7	0 0 7 14	tuber	ND ND ND ND			DP-21414 Test 8a
Castello de Farfanya Spain, 2008 (Kenebec)	2	0.08	18	440	7	0 0 7 14	tuber	ND ND ND ND			DP-21414 Test 8b (with surfactant)
Cervesina Italy, 2008 (Dejsy)	2	0.01	2.2	450	7	14	tuber	ND			DP-21414 Test 6a
Cervesina Italy, 2008 (Dejsy)	2	0.08	18	450	7	14	tuber	ND			DP-21414 Test 6b (with surfactant)
Douai N France, 2008 (Marabella)	2	0.01	2.2	460	7	0 0 7 14	tuber	ND ND ND ND			DP-21414 Test 1a
Douai N France, 2008 (Marabella)	2	0.08	18	450	7	0 0 7 14	tuber	ND ND ND ND			DP-21414 Test 1b (with surfactant)
Galatades Greece, 2008 (Spunta)	2	0.01	2.2	460	7	14	tuber	ND			DP-21414 Test 7a
Galatades, Greece, 2008 (Spunta)	2	0.08	18	470	7	14	tuber	ND			DP-21414 Test 7b (with surfactant)
Herlies N France, 2008 (Bintje)	2	0.01	2.2	450	8	14	tuber	ND			DP-21414 Test 2a
Herlies N France, 2008 (Bintje)	2	0.08	18	470	8	14	tuber	ND			DP-21414 Test 2b (with surfactant)
Le Mas Riller S France, 2008 (Mona Lisa)	2	0.01	2.2	460	7	0 0 7 14	tuber	ND ND ND ND			DP-21414 Test 10a
Le Mas Riller S France, 2008 (Mona Lisa)	2	0.08	18	460	7	0 0 7 14	tuber	ND ND ND ND			DP-21414 Test 10b (with surfactant)

POTATO Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Motterwitz Germany, 2008 (Secura)	2	0.01	2.2	430	7	14	tuber	ND			DP-21414 Test 3a
Motterwitz Germany, 2008 (Secura)	2	0.08	18	470	7	14	tuber	ND			DP-21414 Test 3b (with surfactant)
Prušánky Czech Republic, 2008 (Impala)	2	0.01	2.2	470	8	14	tuber	ND			DP-21414 Test 4a
Prušánky Czech Republic, 2008 (Impala)	2	0.08	18	450	8	14	tuber	ND			DP-21414 Test 4b (with surfactant)
Tora Spain, 2008 (Kenebec)	2	0.01	2.2	470	8	14	tuber	ND			DP-21414 Test 9a
Tora Spain, 2008 (Kenebec)	2	0.08	18	460	8	14	tuber	ND			DP-21414 Test 9b (with surfactant)

Stalk and stem vegetables

In trials conducted in the USA on celery, two or three foliar applications of 0.15 kg ai/ha cyantraniliprole (OD formulation) were applied at 5 day intervals, using 200–500 L/ha, with adjuvant added. Soil treatment plots were also included at several trial sites, either as a soil shank (in furrow) treatment of 0.2 kg ai/ha (SC formulation) at planting or two soil treatments of 0.15 kg ai/ha cyantraniliprole (SC formulation) applied through drip-line irrigation systems (adjusted to pH 4–5, with no added adjuvant).

Duplicate samples of celery (with tops and also trimmed/washed) were stored at –20 °C for up to 9 months before extraction and analysis for cyantraniliprole and six metabolites (within 3 months of extraction) using method DP-15736, with reported LOQs of 0.01 mg/kg. Average concurrent recoveries were 96–98% (cyantraniliprole) and 80–106% (metabolites) in samples spiked with 0.01–10 mg/kg.

Table 140 Residues in celery from supervised trials in North America involving soil applications at planting or through dripline irrigation systems (with foliar applications)

CELERY Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	kg ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Sparta, MI USA, 2009 Celery (Sabroso)	1	0.15	0.42	36000		0 3 7	with tops	ND, ND ND, ND ND, ND	ND ND ND		DP-25644 Trial 45 [by drip irrigation]
Sparta, MI USA, 2009 Celery (Sabroso)	2	0.15	0.42	36000	7	–0 0 3 7	with tops	ND, ND ND, ND ND, < 0.01 ND, ND	ND ND ND ND		DP-25644 Trial 45 [by drip irrigation]

CELERY Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	kg ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Porterville, CA USA, 2008 Celery (Challenger)	1+ 1+ 1	0.2 0.1 0.15	97 41 62	210 240 240	77 5	1	with tops	3, 4.1	3.5	M1=0.02	DP-25644 Trial 14 [1 soil shank treatment + 2 foliar sprays]
Sparta, MI USA, 2009 Celery (Sabroso)	2+ 1	0.15 0.15	0.42 47	36000 320	7 7	–0 1	with tops	ND, ND 1.1, 0.98	ND 1.0		DP-25644 Trial 45 2 [by drip irrigation + 1 foliar]

Soil (shank) treatment rate of 2 g ai/100 metres of row (Trial 14)

Soil (dripline irrigation) treatment rate of 1.5 g ai/100 metres of row (Trial 45)

M1: Average residues of metabolite IN-J9Z38

Table 141 Residues in celery from supervised trials in North America involving two or three foliar applications of cyantraniliprole (OD formulation)

CELERY Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	kg ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Bradenton, FL USA, 2008 Celery (Tall Utah)	2	0.15	36	420	5	1 1	with tops trimmed	5.3, 5.7 4.4, 3.5	5.5 4.0	M1=0.03 M2=0.02 M1=0.02 M2=0.01	DP-25644 Trial 13
Porterville, CA USA, 2008 Celery (Challenger)	2	0.15	62	240	5	1	with tops	4.4, 5.5	5	M1=0.02	DP-25644 Trial 14
Santa Maria, CA USA, 2008 Celery (Conquistador)	2	0.15	54	280	5	1	with tops	1, 0.84	0.92		DP-25644 Trial 15
Fresno, CA USA, 2008 Celery (Command)	2	0.15	54	280	5	1	with tops	2.1, 1.7	1.9	M1 =.02	DP-25644 Trial 16
Guadalupe, CA USA, 2008 Celery (Conquistador)	2	0.15	64	230	5	1	with tops trimmed	1.5, 1.0 0.7, 0.76	1.3 0.73		DP-25644 Trial 17

CELERY Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	kg ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
King City, CA USA, 2008 Celery (G15)	2	0.15	51	290	6	1	with tops trimmed	0.62, 0.73 0.34, 0.46	0.68 0.4		DP-25644 Trial 18
Bradenton, FL USA, 2008 Celery (Tall Utah)	3	0.15	37	430	5	1	with tops trimmed	8.8, 9.5 4.2, 5.4	<u>9.1</u> 4.8	M1=0.04 M2=0.03 M1=0.02 M2=0.01	DP-25644 Trial 13
Branchton, ON CAN, 2009 Celery (Florida 683)	3	0.15	74	200	5, 6	1	with tops	2.4, 2.2	2.3	M1=0.02	DP-25644 Trial 42
Delavan, WI USA, 2009 Celery (XP266)	3	0.15	70	220	5	1	with tops	2.1, 1.9	2.0	M1=0.02	DP-25644 Trial 43
Fitchburg, WI USA, 2009 Celery (Tango)	3	0.15	60	250	5	1	with tops	0.31, 0.24	0.28	M1=0.01	DP-25644 Trial 41
Fresno, CA USA, 2008 Celery (Command)	3	0.15	54	280	5	1	with tops	2.3, 2.6	2.5	M1=0.02	DP-25644 Trial 16
Guadalupe, CA USA, 2008 Celery (Conquistador)	3	0.15	66	230	5	1	with tops trimmed	0.37, 1.7 0.81, 0.97	<u>1.1</u> 0.89		DP-25644 Trial 17
King City, CA USA, 2008 Celery (G15)	2+ 1	0.15 0.16	51 52	290 300	6 5	1	with tops trimmed	0.9, 0.57 0.45, 0.29	<u>0.73</u> 0.37		DP-25644 Trial 18
Porterville, CA USA, 2008 Celery (Challenger)	3	0.15	62	240	5	1	with tops	6.3, 5.1	5.7	M1=0.02	DP-25644 Trial 14
Richland, IA USA, 2009 Celery (Conquistador Pelleted)	3	0.15	74	210	5	1	with tops	4.7, 4.8	4.7	M1=0.04 M2=0.01	DP-25644 Trial 44
Santa Maria, CA USA, 2008 Celery (Conquistador)	3	0.15	54	280	5	1	with tops	1, 1.1	1.0		DP-25644 Trial 15

CELERY Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	kg ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Sparta, MI USA, 2009 Celery (Sabroso)	3	0.15	47	320	5	1	with tops	1.2, 1.2	1.2	M1=0.02	DP-25644 Trial 45

M1: Average residues of metabolite IN-J9Z38

M2: Average residues of metabolite IN-MYX98

Cereals

In three trials on rice, conducted in China, cyantraniliprole (OD formulation) was applied two or three times as foliar sprays, using either 0.1 kg ai/ha or 0.15 kg ai/ha, with a 7 day retreatment interval. No adjuvants were used.

Samples of threshed brown rice, hulls and straw (leaves and stalks) were stored at -20 °C until analysed for cyantraniliprole and the metabolite IN-J9Z38 using an aqueous acetonitrile extraction and LC-MS/MS analysis method based on method DP-15736. The reported LOQs for cyantraniliprole were 0.01 mg/kg in grain and 0.05 mg/kg in hulls and straw, and the LOQs for IN-J9Z38 were 0.02 mg/kg in grain and 0.1 mg/kg in hulls and straw. Average concurrent recoveries were 78-103% (cyantraniliprole) and 79-102% (IN-J9Z38) in samples spiked with 0.01, 0.05, 0.1, 1.0 mg/kg and also 2.0 mg/kg ((IN-J9Z38).

Table 142 Residues in rice grain and hulls from supervised trials in China involving foliar applications of cyantraniliprole (100 FC formulation)

RICE Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	M1	
Hangzhou Zhejiang China 2011 (Xiu Shui 009)	2	0.1			7	7	grain	< 0.01		< 0.02	CL-2010-026 Site 1-11
						14		< 0.01		< 0.02	
						21		< 0.01		< 0.02	
						7	hulls	0.67		0.1	
						14		0.37		< 0.1	
						21		0.43		< 0.1	
						7	straw	< 0.05		0.21	
						14		< 0.05		0.12	
						21		< 0.05		< 0.1	
Hangzhou Zhejiang China, 2010 (Xiu Shui 009)	2	0.1			7	7	grain	< 0.01		< 0.02	CL-2010-026 Site 1-10
						14		< 0.01		< 0.02	
						21		< 0.01		< 0.02	
						7	hulls	3.3		0.28	
						14		4.3		0.35	
						21		2.3		0.2	
						7	straw	0.67		0.29	
						14		1.4		0.28	
						21		1.1		0.2	

RICE Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	M1	
Hunan, Changsha China, 2010 (Yue You 712)	2	0.1			7	7 14 21	grain	< 0.01 < 0.01 < 0.01		< 0.02 < 0.02 < 0.02	CL-2010-026 Site 2-10
						7 14 21	hulls	0.99 1.1 0.92		< 0.1 < 0.1 < 0.1	
						7 14 21	straw	< 0.05 0.083 0.057		< 0.01 < 0.01 < 0.01	
Hunan, Changsha China, 2011 ((Yue You 712)	2	0.1			7	7 14 21	grain	< 0.01 < 0.01 < 0.01		< 0.02 < 0.02 < 0.02	CL-2010-026 Site 2-11
						7 14 21	hulls	1.4 1.4 1.6		< 0.1 < 0.1 < 0.1	
						7 14 21	straw	0.15 0.098 0.18		< 0.1 < 0.1 < 0.1	
Jinan, Shandong China, 2010 (Xin Dao 16)	2	0.1			7	7 14 21	grain	< 0.01 < 0.01 < 0.01		< 0.02 < 0.02 < 0.02	CL-2010-026 Site 3-10
						7 14 21	hulls	0.46 0.38 0.32		< 0.1 < 0.1 < 0.1	
						7 14 21	straw	0.27 0.14 0.4		< 0.1 < 0.1 < 0.1	
Jinan, Shandong China, 2011 (Xin Dao 16)	2	0.1			7	7 14 21	grain	< 0.01 < 0.01 < 0.01		< 0.02 < 0.02 < 0.02	CL-2010-026 Site 3-11
						7 14 21	hulls	0.081 0.092 0.57		< 0.1 < 0.1 < 0.1	
						7 14 21	straw	0.2 0.13 0.075		< 0.1 < 0.1 < 0.1	

RICE Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	M1	
Hangzhou Zhejiang China 2011 (Xiu Shui 009)	2	0.15			7	7 14 21	grain	0.023 0.016 0.01		< 0.02 < 0.02 < 0.02	CL-2010-026 Site 1-11
						7 14 21	hulls	1.7 1.2 0.95		0.21 0.2 0.17	
						7 14 21	straw	0.12 0.063 0.075		0.5 0.46 0.16	
Hangzhou Zhejiang China, 2010 (Xiu Shui 009)	2	0.15			7	7 14 21	grain	0.02 0.013 < 0.01		< 0.02 < 0.02 < 0.02	CL-2010-026 Site 1-10
						7 14 21	hulls	7.5 12 3.4		0.92 0.76 0.41	
						7 14 21	straw	1.1 3.7 1.9		0.46 0.47 0.31	
Hunan, Changsha China, 2010 (Yue You 712)	2	0.15			7	7 14 21	grain	< 0.01 < 0.01 < 0.01		< 0.02 < 0.02 < 0.02	CL-2010-026 Site 2-10
						7 14 21	hulls	1.4 2.2 1.5		< 0.1 < 0.1 < 0.1	
						7 14 21	straw	0.37 0.33 0.17		< 0.1 < 0.1 < 0.1	
Hunan, Changsha China, 2011 ((Yue You 712)	2	0.15			7	7 14 21	grain	0.07 0.029 < 0.01		< 0.02 < 0.02 < 0.02	CL-2010-026 Site 2-11
						7 14 21	hulls	8.2 6.8 2.2		< 0.1 < 0.1 < 0.1	
						7 14 21	straw	0.42 1.1 0.13		< 0.1 < 0.1 < 0.1	

RICE Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	M1	
Jinan, Shandong China, 2010 (Xin Dao 16)	2	0.15			7	7 14 21	grain	< 0.01 < 0.01 < 0.01		< 0.02 < 0.02 < 0.02	CL-2010-026 Site 3-10
						7 14 21	hulls	0.92 0.18 0.14		< 0.1 0.1 < 0.1	
						7 14 21	straw	0.31 0.33 < 0.05		< 0.1 0.1 < 0.1	
Jinan, Shandong China, 2011 (Xin Dao 16)	2	0.15			7	7 14 21	grain	< 0.01 < 0.01 < 0.01		< 0.02 < 0.02 < 0.02	CL-2010-026 Site 3-11
						7 14 21	hulls	< 0.05 < 0.05 < 0.05		< 0.1 < 0.1 < 0.1	
						7 14 21	straw	0.25 0.18 0.079		< 0.1 < 0.1 < 0.1	
Hangzhou Zhejiang China 2011 (Xiu Shui 009)	3	0.1			7	7 14 21	grain	< 0.01 < 0.01 < 0.01		< 0.02 < 0.02 < 0.02	CL-2010-026 Site 1-11
						7 14 21	hulls	1.2 0.71 0.42		0.18 0.16 0.096	
						7 14 21	straw	< 0.05 0.095 < 0.05		0.24 0.24 < 0.1	
Hangzhou Zhejiang China, 2010 (Xiu Shui 009)	3	0.1			7	7 14 21	grain	0.01 < 0.01 < 0.01		< 0.02 < 0.02 < 0.02	CL-2010-026 Site 1-10
						7 14 21	hulls	4.6 3.7 1.7		0.55 0.52 0.37	
						7 14 21	straw	0.78 1.9 1.4		0.34 0.24 0.16	

RICE Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	M1	
Hunan, Changsha China, 2010 (Yue You 712)	3	0.1			7	7 14 21	grain	< 0.01 < 0.01 < 0.01		< 0.02 < 0.02 < 0.02	CL-2010-026 Site 2-10
						7 14 21	hulls	0.87 1.1 1.4		< 0.1 < 0.1 < 0.1	
						7 14 21	straw	0.085 0.058 < 0.05		< 0.1 < 0.1 < 0.1	
Hunan, Changsha China, 2011 ((Yue You 712)	3	0.1			7	7 14 21	grain	0.018 < 0.01 < 0.01		< 0.02 < 0.02 < 0.02	CL-2010-026 Site 2-11
						7 14 21	hulls	4.0 2.5 1.6		< 0.1 0.11 < 0.1	
						7 14 21	straw	0.68 0.4 0.35		< 0.1 < 0.1 < 0.1	
Jinan, Shandong China, 2010 (Xin Dao 16)	3	0.1			7	7 14 21	grain	< 0.01 < 0.01 < 0.01		< 0.02 < 0.02 < 0.02	CL-2010-026 Site 3-10
						7 14 21	hulls	0.55 0.71 0.57		< 0.1 < 0.1 < 0.1	
						7 14 21	straw	0.26 0.44 0.28		< 0.1 0.13 < 0.1	
Jinan, Shandong China, 2011 (Xin Dao 16)	3	0.1			7	7 14 21	grain	< 0.01 < 0.01 < 0.01		< 0.02 < 0.02 < 0.02	CL-2010-026 Site 3-11
						7 14 21	hulls	< 0.05 0.16 0.13		< 0.1 < 0.1 < 0.1	
						7 14 21	straw	0.34 0.2 0.12		0.18 < 0.1 < 0.1	

RICE Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	M1	
Hangzhou Zhejiang China 2011 (Xiu Shui 009)	3	0.15			7	7 14 21	grain	0.041 0.022 0.012		< 0.02 < 0.02 < 0.02	CL-2010-026 Site 1-11
						7 14 21	hulls	2.3 2.4 0.95		0.33 0.3 0.19	
						7 14 21	straw	< 0.05 < 0.05 0.12		0.36 0.59 0.39	
Hangzhou Zhejiang China, 2010 (Xiu Shui 009)	3	0.15			7	7 14 21	grain	0.035 0.019 < 0.01		< 0.02 < 0.02 < 0.02	CL-2010-026 Site 1-10
						7 14 21	hulls	12 11 9.9		1.4 0.6 0.84	
						7 14 21	straw	1.8 4.1 3.0		0.59 0.43 0.51	
Hunan, Changsha China, 2010 (Yue You 712)	3	0.15				7 14 21	grain	< 0.01 < 0.01 < 0.01		< 0.02 < 0.02 < 0.02	CL-2010-026 Site 2-10
						7 14 21	hulls	1.6 1.8 2.1		< 0.1 < 0.1 0.1	
						7 14 21	straw	0.58 0.51 0.36		< 0.1 < 0.1 < 0.1	
Hunan, Changsha China, 2011 ((Yue You 712)	3	0.15				7 14 21	grain	0.039 0.025 0.019		< 0.02 < 0.02 < 0.02	CL-2010-026 Site 2-11
						7 14 21	hulls	6.2 4.1 2.4		< 0.1 < 0.1 < 0.1	
						7 14 21	straw	0.55 0.26 0.33		< 0.1 < 0.1 < 0.1	

RICE Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	M1	
Jinan, Shandong China, 2010 (Xin Dao 16)	3	0.15				7 14 21	grain	< 0.01 < 0.01 < 0.01		< 0.02 < 0.02 < 0.02	CL-2010-026 Site 3-10
						7 14 21	hulls	1.2 0.21 0.66		< 0.1 < 0.1 < 0.1	
						7 14 21	straw	1.3 0.33 < 0.05		0.12 < 0.1 < 0.1	
Jinan, Shandong China, 2011 (Xin Dao 16)	3	0.15				7 14 21	grain	< 0.01 < 0.01 < 0.01		< 0.02 < 0.02 < 0.02	CL-2010-026 Site 3-11
						7 14 21	hulls	< 0.05 < 0.05 < 0.05		< 0.1 < 0.1 < 0.1	
						7 14 21	straw	0.35 0.24 0.18		0.11 < 0.1 < 0.1	

M1: Residues of metabolite IN-J9Z38

Tree nuts

In trials conducted in the USA on almonds and pecans, three foliar (airblast) treatments of 0.15 kg ai/ha cyantraniliprole (OD formulation) were applied at about 7 day intervals using either high volume (1000–3000 L/ha), concentrate (100–800 L/ha) or ultra-low-volume (50 L/ha) sprays, all with adjuvant added. An additional soil (shank) injection side dressing treatment was included in one trial.

Duplicate samples of nutmeat and hulls were stored at –20 °C for up to 6 months before analysis (within 37 days of extraction) for cyantraniliprole and six metabolites using method DP-15736, with reported LOQs of 0.01 mg/kg. Average concurrent recoveries in peel and pulp were 81–93% (cyantraniliprole) and 76–97% (metabolites) in samples spiked with 0.01 and 0.1 mg/kg.

Table 143 Residues in almonds from supervised trials in the USA involving foliar applications of cyantraniliprole (OD or SE formulations)

ALMOND Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
(Turlock, CA USA, 2009) (Butte)	3	0.15	26	580	7, 6	5	nutmeat hulls	0.012, 0.012 4.5, 4.6	0.012 4.6	 M1=0.03 M2=0.01	DP-27446 Trial 01
Kerman, CA USA, 2009 (Non-Pareil)	3	0.15	32	470	7	5	nutmeat hulls	0.009, 0.01 2.0, 1.7	0.009 1.9	 M1=0.01	DP-27446 Trial 02

ALMOND Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Sanger, CA	1+	0.15	23	650	6	5	nutmeat	0.006, 0.007	0.007		DP-27446
USA, 2009	1+		27	600	7						Trial 03
(Neplus)	1			540			hulls	0.78, 0.98	0.88		
Sutter, CA	3	0.15	310	50	7	5	nutmeat	0.024, 0.023	0.023		DP-27446
USA, 2009											Trial 04
(Non-Pareil)							hulls	3.0, 2.8	2.9	M1=0.02	
Sanger, CA	1+	0.15	6	2400	7	5	nutmeat	0.005, 0.005	0.005		DP-27446
USA, 2009	2	0.15	12	1300	8						Trial 05
(Neplus)							hulls	1.2, 1.4	1.3	M1=0.01	
Sanger, CA	1+	0.15	6	2400	7	5	nutmeat	0.008, 0.006	0.007		DP-27446
USA, 2009	2	0.15	12	1300	8						Trial 05
(Neplus)							hulls	2.3, 2.7	2.5	M1=0.01	[100 SE]
Madera, CA	3	0.15	330	50	6, 7	5	nutmeat	0.006, 0.007	0.007		DP-27446
USA, 2009											Trial 06
(Non-Pareil)							hulls	0.88, 0.94	0.91		
Madera, CA	3	0.15	11	1400	6, 7	5	nutmeat	0.016, 0.019	0.018		DP-27446
USA, 2009											Trial 06
(Non-Pareil)							hulls	3.7, 3.5	3.6	M1=0.04	[100 SE]

M1: Average residues of metabolite IN-J9Z38

M2: Average residues of metabolite IN-MYX98

Table 144 Residues in pecans from supervised trials in the USA involving foliar applications of cyantraniliprole (OD or SE formulations) or soil (shank) injection (SC formulation)

PECAN Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Girard, GA	3	0.15	12	1200	7	5	nutmeat	ND, ND	ND		DP-27446
USA, 2009											Trial 07
(Desirables)											
Union Springs,	3	0.15	12	1200	7	5	nutmeat	ND, ND	ND		DP-27446
AL											Trial 08
USA, 2009											
(Stewart)											
Bailey, NC	3	0.15	12	1200	7	4	nutmeat	ND, ND	ND		DP-27446
USA, 2009											Trial 09
(Stuart)											
Alexandria, LA	1+	0.16	24	660	7	5	nutmeat	0.008, 0.010	0.009		DP-27446
USA, 2009	1+	0.16	21	780							Trial 10
(Creek)	1	0.15	23	630							
Eagle Lake, TX	3	0.15	360	40	7, 8	5	nutmeat	0.006, 0.004	0.005		DP-27446
USA, 2009											Trial 11
(Pawnee)											
Pearsall, TX	3	0.15	27	570	7	5	nutmeat	ND, ND	ND		DP-27446
USA, 2009		0.14	24	590							Trial 12
(Wichita		0.15	29	530							

PECAN Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Pearsall, TX USA, 2009 (Wichita)	1	0.46	490	90		57	nutmeat	ND, ND	ND		DP-27446 Trial 12 [200 SC soil injection]

Oilseed crops

In trials conducted with cyantraniliprole on cotton in the USA, plots were treated with three late season foliar sprays (OD formulation) at a rate of 0.15 kg ai/ha, using 200–500 L water/ha, with added surfactant. In one trial, an additional plot was treated with a pre-plant in-furrow soil treatment (0.2 kg ai/ha, SC formulation) followed by two late season foliar spray applications (0.1–0.15 kg ai/ha, OD formulation).

Samples of cotton seed and cotton gin byproducts were stored at –20 °C for up to 8 months before extraction and analysed within 3 months after extraction for cyantraniliprole and eight metabolites using method DP-15736, with reported LOQs of 0.01 mg/kg. Average concurrent recoveries were 75–76% (cyantraniliprole) and 69–120% (metabolites) in samples spiked with 0.01 and 0.1 mg/kg and also 1.0 and 1.6 mg/kg in cotton seed.

Table 145 Residues in cotton from supervised trials in the USA involving three foliar applications of cyantraniliprole (OD formulation)

COTTON SEED Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Cheneyville, LA USA, 2009 (Phytogen 485WRF)	3	0.16	77	200	7, 6	–0 0 5 7	seed	0.28, 0.2 0.63, 0.58 0.2, 0.14 0.17, 0.2	0.24 0.6 0.17 0.18	M1=0.02 M1=0.02 M1=0.02 M1=0.03	DP-27565 Trial 02
East Bernard, TX USA, 2009 (DP0924 B2F)	3	0.15	77	200	8, 6	–0 0 1 5 7	seed	0.27, 0.33 0.94, 0.66 0.63, 0.89 0.56, 0.82 0.26, 0.26	0.3 0.8 0.76 0.69 0.26	M1=0.05 M1=0.05 M1=0.05 M1=0.07 M1=0.06	DP-27565 Trial 05
Edmonson, TX USA, 2009 (DP 924)	3	0.15	96	160	8, 6	7	seed gin trash	0.83, 1.2 4.3, 5.7	0.99 5	 M1=0.02 M2=0.03	DP-27565 Trial 08
Fisk, MO USA, 2009 (DP 164 B2RF)	3	0.15	80	190	8	8	seed	0.023, 0.027	0.025		DP-27565 Trial 03
Hickman, CA USA, 2009 (Pima)	3	0.15	40	370	7	8	seed	0.2, 0.2	0.2		DP-27565 Trial 11
Hinton, OK USA, 2009 (FM1740B2F)	3	0.15	72	200	8, 9	9	seed gin trash	0.18, 0.13 2.6, 2.6	0.16 2.6	 M1=0.03 M2=0.01	DP-27565 Trial 07

COTTON SEED Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Larned, KS USA, 2009 (Delta Pine)	3	0.15	71	210	7	8	seed	0.27, 0.32	0.29	M1=0.01	DP-27565 Trial 06
Levelland, TX USA, 2009 (9063 B2F)	3	0.15	63	230	7	8	seed gin trash	0.11, 0.12 3.5, 3.5	0.12 3.5	 M1=0.07 M2=0.02	DP-27565 Trial 09
Madera, CA USA, 2009 (Acala Riata RR)	3	0.15	64	230	7, 6	7	seed	0.15, 0.12	0.14		DP-27565 Trial 12
Newport, AR USA, 2009 (DP 164 B2RF)	3	0.15	80	190	7	7	seed	0.045, 0.025	0.035		DP-27565 Trial 04
Sanger, CA USA, 2009 (PHY 725 RF Acala)	1+ 2	0.15	54 37	290 400	6 8	7	seed	0.24, 0.21	0.22		DP-27565 Trial 13
Seven Springs, NC USA, 2009 (ST 4554B2RF)	3	0.15	63	240	7	8	seed	0.011, 0.013	0.012		DP-27565 Trial 01
Uvalde, TX USA, 2009 (DP6167 B2RF)	3	0.15	62	230	7	6	seed gin trash	0.1, 0.14 2.8, 2.6	0.12 2.7	 M1=0.07 M2=0.01	DP-27565 Trial 10
Larned, KS USA, 2009 (Delta Pine)	1+ 1+ 1	0.19 0.1 0.15	110 48 72	180 210 210	146 7	8	seed	0.16, 0.14	0.15		DP-27565 Trial 06 [soil inject+ 2 foliar]

M1: Average residues of metabolite IN-J9Z38

M2: Average residues of metabolite IN-MYX98

In trials conducted in Australia on cotton, two foliar applications of 0.06 or 0.12 kg ai/ha cyantraniliprole (SE formulations) were applied at 7 day intervals using 120 L water/ha, with added adjuvant. Several of these trials were reverse decline studies, where different plots were treated at staggered intervals so that the sampling dates co-incided with normal commercial harvest.

Samples were stored at –20 °C for up to 1 month before analysis of forage (leaves, stems etc), seeds and field trash for cyantraniliprole and six metabolites (within 4 days after extraction) using analytical method DP-15736, with reported LOQs of 0.01 mg/kg. Average concurrent recoveries were 95–99% (cyantraniliprole) and 77–101% (metabolites) in samples spiked with 0.01–2.0 mg/kg.

Table 146 Residues in cotton from supervised trials in Australia involving two foliar applications of cyantraniliprole (SE formulation)

COTTONSEED Location	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference Comments
Country, year (variety)	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Boggabilla, NSW	2	0.06	0.05	120	7	7	forage	0.39		M1=0.01	DP-31410
Australia, 2010						14		0.23		M1=0.01	Site 1
(Sicot 71 BRF)						21		0.08			[Reverse
						28		0.03			decline]
						7	seed	ND			
						14		ND			
						21		ND			
						28		ND			
						7	field trash	1.18		M1=0.05	
						14		0.59		M1=0.03	
						21		0.19			
						28		0.11			
Brookstead, QLD	2	0.06	0.05	120	7	14	forage	0.28		M1=0.02	DP-31410
Australia, 2010											Site 3
(Sicot 71 BRF)						14	seed	ND			
						14	field trash	0.74		M1=0.04	
Toobeah, QLD	2	0.06	0.05	120	7	14	forage	0.15		M1=0.01	DP-31410
Australia, 2010											Site 2
(Sicot 71 BRF)						14	seed	ND			
						14	field trash	0.34		M1 =0.02	
Boggabilla, NSW	2	0.12	0.1	120	7	7	forage	0.68		M1=0.03	DP-31410
Australia, 2010						14		0.38		M1=0.01	Site 1
(Sicot 71 BRF)						21		0.12			[Reverse
						28		0.09			decline]
						7	seed	ND			
						14		ND			
						21		ND			
						28		ND			
						7	field trash	1.85		M1=0.08	
						14		1.01		M1=0.04	
						21		0.47		M1=0.02	
						28		0.16			
Brookstead, QLD	2	0.12	0.1	120	7	14	forage	0.43		M1=0.02	DP-31410
Australia, 2010											Site 3
(Sicot 71 BRF)						14	seed	ND			
						14	field trash	1.22		M1=0.04	
Toobeah, QLD	2	0.12	0.1	120	7	14	forage	0.27		M1=0.02	DP-31410
Australia, 2010											Site 2
(Sicot 71 BRF)						14	seed	ND			
						14	field trash	0.76		M1=0.02	

M1: Average residues of metabolite IN-J9Z38

Rape seed

In trials conducted with cyantraniliprole on oil-seed rape (canola) in North America, plots were treated with three late season foliar sprays (OD formulation) at a rate of 0.15 kg ai/ha, using 200–500 L water/ha, with added surfactant. In several trials, seed treated with cyantraniliprole FS formulation) at a rate required to achieve an application rate about 0.08 kg ai/ha planted and the plots were also treated with two late season foliar spray applications (0.15 kg ai/ha, OD formulation).

Seed samples were stored at -20 °C for up to 6.5 months before extraction and analysed within 4 months after extraction for cyantraniliprole and six metabolites using method DP-15736, with reported LOQs of 0.01 mg/kg. Average concurrent recoveries were 92% (cyantraniliprole) and 85–99% (metabolites) in samples spiked with 0.01 and 0.1 mg/kg and also 1.0 mg/kg cyantraniliprole in cotton seed.

Table 147 Residues in oil-seed rape from supervised trials in North America involving foliar applications of cyantraniliprole (OD formulation), with and without the use of cyantraniliprole-treated seed

OILSEED RAPE Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Alvena, SK CAN, 2009 (RR 7145)	3	0.15	75	200		7	seed	0.24, 0.3	0.27	M1=0.02	DP-27582 Test 10
Blaine Lake, SK CAN, 2009 (Dekalb 7145 RR)		0.15	74	200	7	7	seed	0.25, 0.33	0.29	M1=0.01	DP-27582 Trial 16
Brandon, MB CAN, 2009 (Invigor 5030)	3	0.15	60	250	7	7	seed	0.18, 0.16	0.17		DP-27582 Test 09
Carberry, MB CAN, 2009 (D3151)	3	0.15	60	250	7, 6	6	seed	0.054, 0.065	0.059		DP-27582 Test 07
Carrington, ND USA, 2009 (Pioneer D3151)	3	0.15	54	280	7	7	seed	0.017, 0.017	0.017		DP-27582 Test 04
Ephrata, WA USA, 2009 (7145 RR)	3	0.15	73	210	7	7	seed	0.087, 0.08	0.084	M1=0.01	DP-27582 Test 05
Ft. Saskatchewan, AB CAN, 2009 (1818 Roundup Ready)	3	0.15	50	300	7	7	seed	0.13, 0.12	0.12		DP-27582 Trial 12
Ft. Saskatchewan, AB CAN, 2009 (Liberty 1141)	3	0.15	50	300	7, 6	7	seed	0.057, 0.066	0.061		DP-27582 Trial 11
Geneva, MN USA, 2009 (Pioneer 45H21)	3	0.15	79	190	6, 7	8	seed	0.027, 0.016	0.021		DP-27582 Test 02
Jerome, ID USA, 2009 (D3151)	2+ 1	0.15	76 80	200 190	6 8	7	seed	0.29, 0.34	0.32		DP-27582 Test 06
Justice, MB CAN, 2009 (D3151)	3	0.15	60	250	7, 6	6	seed	0.022, 0.023	0.022		DP-27582 Test 08

OILSEED RAPE Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Lamont, AB CAN, 2009 (Invigor 8440)	3	0.15	50	300	6, 7	7	seed	0.14, 0.21	0.18		DP-27582 Trial 13
St. Marcsur Richelieu, QC CAN, 2009 (Pioneer D3150)	3	0.15	50	300	6, 9	1	seed	0.17, 0.16	0.16		DP-27582 Trial 03
Stephens, GA USA, 2009 (Sumner)	1+ 1+ 1	0.15 0.15 0.14	51 62 70	290 240 210	7	7	seed	0.017, 0.022	0.019		DP-27582 Test 01
Wakaw, SK CAN, 2009 (RR 7145)	3	0.15	75	200	7	7	seed	0.066, 0.047	0.057		DP-27582 Trial 17
Waldheim, SK CAN, 2009 (Dekalb 7145 RR)	3	0.15	75	200	7	7	seed	0.57, 0.65	0.61	M1=0.02	DP-27582 Trial 15
Westlock, AB CAN, 2009 (Roundup Ready 1818)	3	0.15	50	300	7, 6	7	seed	0.07, 0.07	0.07	M1=0.01	DP-27582 Trial 14
Carberry, MB CAN, 2009 (D3151)	1+ 1+ 2	0.08+ 0.07 0.15	 29 60	 250 250	 7 6	6	seed	0.029, 0.032	0.031		DP-27582 Test 07 [with treated seed]
Carrington, ND USA, 2009 (Pioneer D3151)	1+ 1+ 2	0.08+ 0.07 0.15	 25 54	 280 280	 7 7	7	seed	0.015, 0.016	0.015		DP-27582 Test 04 [with treated seed]
Jerome, ID USA, 2009 (D3151)	1+ 1+ 1+ 1	0.08+ 0.07 0.15 0.16	 37 74 80	 200 200 190	 6 8	7	seed	0.21, 0.22	0.21		DP-27582 Test 06 [with treated seed]
Justice, MB CAN, 2009 (D3151)	1+ 1+ 2	0.08+ 0.07 0.15	 29 60	 250 250	 7 6	6	seed	0.048, 0.047	0.047		DP-27582 Test 08 [with treated seed]
St. Marcsur Richelieu, QC CAN, 2009 (Pioneer D3150)	1+ 1+ 2	0.08+ 0.07 0.15	 24 50	 310 300	 6 9	1	seed	0.11, 0.13	0.12		DP-27582 Trial 03 [with treated seed]

M1: Average residues of metabolite INJ9Z38

In trials conducted in Australia on oilseed rape, single foliar applications of 0.015 or 0.03 kg ai/ha cyantraniliprole (SE formulations) were applied using about 100 L water/ha, with added adjuvant. One of these trials was a reverse decline study, where different plots were treated at staggered intervals so that the sampling dates co-incided with normal commercial harvest.

Samples, fodder (remaining straw/trash) and processed oil were stored at -20 °C for up to 3 months before extraction for analysis of seeds, trash and oil for cyantraniliprole and six metabolites (within 4 days after extraction) using analytical method DP-15736, with reported LOQs of 0.01 mg/kg. Average concurrent recoveries were 91–102% (cyantraniliprole) and 75–102% (metabolites) in samples spiked with 0.01–2.0 mg/kg.

Table 148 Residues in oilseed rape from supervised trials in Australia involving foliar applications of cyantraniliprole (SE formulation)

OILSEED RAPE Location	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference Comments
Country, year (variety)	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Welshpool, WA Australia, 2009	1	0.015	15	100		7	seed	ND			DP-31412 Site 1 [Reverse decline]
						14		ND			
						21		ND			
						28		ND			
						7	fodder	0.15			
						14		0.06			
						21		0.02			
						28		ND			
						7	oil	ND			
						14		ND			
						21		ND			
						28		ND			
Tranmere, SA Australia, 2009	1	0.015	15	100		14	seed	ND			DP-31412 Site 2
						14	fodder	0.38		M1=0.01	
						14	oil	ND			
Young, NSW Australia, 2009	1	0.015	15	100		14	seed	ND			DP-31412 Site 3
						14	fodder	0.08			
						14	oil	ND			
Tranmere, SA Australia, 2009	1	0.03	30	100		14	seed	< 0.01			DP-31412 Site 2
						14	fodder	0.57		M1=0.03	
						14	oil	ND			
Young, NSW Australia, 2009	1	0.03	30	100		14	seed	ND			DP-31412 Site 3
						14	fodder	0.21		M1=0.01	
						14	oil	ND			

M1: Average residues of metabolite INJ9Z38

Sunflower

In trials conducted with cyantraniliprole on sunflowers in North America, plots were treated with three late season foliar sprays (OD formulation) at a rate of 0.15 kg ai/ha, using 200–500 L water/ha, with added surfactant.

Seed samples were stored at -20 °C for up to 6.5 months before extraction and analysed within 4 months after extraction for cyantraniliprole and six metabolites using method DP-15736,

with reported LOQs of 0.01 mg/kg. Average concurrent recoveries were 92% (cyantraniliprole) and 85–99% (metabolites) in samples spiked with 0.01 and 0.1 mg/kg and also 1.0 mg/kg cyantraniliprole in cotton seed.

Table 149 Residues in sunflower seed from supervised trials in North America involving foliar applications of cyantraniliprole (OD formulation)

SUNFLOWER Location Country, year (variety)	Application					DAT (days)	Matrix	Residues (mg/kg)			Reference & Comments
	no	kg ai/ha	g ai/hL	water L/ha	RTI (days)			cyantraniliprole	mean	metabolites	
Atlantic, IA USA, 2009 (Sunflower/ 8007 Millborn)	3	0.15	80	190	7	7	seed	0.069, 0.065	0.067		DP-27582 Trial 19
Brookdale, MB CAN, 2009 (Sunflower/ 6946)	3	0.15	60	250	7, 6	6	seed	0.36, 0.28	0.32		DP-27582 Trial 25
Carrington, ND USA, 2009 (Sunflower/ Pioneer)	3	0.15	54	280	5, 7	7	seed	0.068, 0.1	0.085		DP-27582 Trial 20
Hinton, OK USA, 2009 (Sunflower/ 8N453DM)	3	0.15	63	240	8, 9	5	seed	0.06, 0.059	0.059		DP-27582 Trial 24
Jamestown, ND USA, 2009 (Sunflower/ IS 8048)	3	0.15	80	190	7	7	seed	0.03, 0.049	0.039		DP-27582 Trial 22
Montpelier, ND USA, 2009 (Sunflower/ IS 8048)	1+ 2	0.16 0.15	83 80	190 190	7	7	seed	0.026, 0.031	0.028		DP-27582 Trial 23
Neepawa, MB CAN, 2009 (Sunflower/ Jaguar)	3	0.15	60	250	7, 6	6	seed	0.092, 0.093	0.092		DP-27582 Trial 26
Stafford, KS USA, 2009 (Sunflower/ Pioneer 63M61)	2+ 1	0.15 0.16	71 75	210	7	7	seed	0.045, 0.082	0.064		DP-27582 Trial 18
Velda, ND USA, 2009 (Sunflower/ 8N835CL)	3	0.15	80	190	8, 7	7	seed	0.14, 0.15	0.14		DP-27582 Trial 21

Coffee

In Brazilian trials on coffee, plots was treated with two soil drench applications of cyantraniliprole (SC formulation), 30 days apart, using from 0.01–0.06 g ai/100 mL/plant to achieve the equivalent of 0.2 kg ai/ha/treatment, followed by two late season foliar applications of 0.175 kg ai/ha cyantraniliprole (OD formulation), 30 days apart, using 400–600 L water/ha with no added adjuvants. In two trials, additional plots received only the two late season foliar sprays.

Samples of beans were dried at room temperature and manually peeled, with the dried beans being stored frozen until analysis for cyantraniliprole and the metabolite IN-J9Z38 using method DP-15736, with reported LOQs of 0.01 mg/kg. Average concurrent recoveries were 93% (cyantraniliprole) and 88% (IN-J9Z38) in samples spiked with 0.01 and 1.0 mg/kg.

Table 150 Residues in coffee beans from supervised trials in Brazil involving soil drench (SC formulation) and foliar applications of cyantraniliprole (OD formulation)

COFFEE Country, year Location (variety)	Application				RTI (days)	DAT, (days)	Matrix	Residues (mg/kg)		Reference & Comments
	no	kg ai/ha	g ai/hL	water (L/ha)				cyantraniliprole	metabolites	
Campinas SP Brazil, 2011	2	0.175	35	500		7 28	beans	< 0.01 <u>< 0.01</u>		BRI- 10/11-008 Test A
Monte Santo de Minas Brazil, 2011	2	0.175	29	600		7 28	beans	0.02 <u>0.02</u>		BRI- 10/11-008 Test F
Cabo Verde Brazil, 2011	2+2	0.2 (soil) 0.175	0.5 35	0.1L/plant 500	30	7 28	beans	0.03 <u>0.01</u>		BRI- 10/11-008 Test C
Campinas SP Brazil, 2011	2+2	0.2 (soil) 0.175	0.5 35	0.1L/plant 500	30	7 14 28 35 45 60	beans	0.02 0.01 < 0.01 < 0.01 < 0.01 ND		BRI- 10/11-008 Test A
Espirito Santo do Pinhal SP Brazil, 2011	2+2	0.2 (soil) 0.175	0.6 35	0.1L/plant 500	30	7 14 28 35	beans	0.01 < 0.01 <u>< 0.01</u> < 0.01		BRI- 10/11-008 Test B
Indianopolis Brazil, 2011	2+2	0.2 (soil) 0.175	0.7 29	0.1L/plant 600	30	7 28 45 60	beans	< 0.01 <u>< 0.01</u> < 0.01 < 0.01		BRI- 10/11-008 Test G
Lohdrina Brazil 2011	2+2	0.2 (soil) 0.175	0.5 44	0.1L/plant 400	30	7 14 28 35	beans	< 0.01 < 0.01 <u>ND</u> ND		BRI- 10/11-008 Test I
Monte Santo de Minas Brazil, 2011	2+2	0.2 (soil) 0.175	0.4 29	0.1L/plant 600	30	7 28	beans	0.01 0.01		BRI- 10/11-008 Test F
Pardinho – SP Brazil, 2011	2+2	0.2 (soil) 0.175	0.6 30	0.1L/plant 580	30	7 28	beans	0.02 <u>< 0.01</u>		BRI- 10/11-008 Test D
Restinga – SP Brazil, 2011	2+2	0.2 (soil) 0.175	0.2 29	0.1L/plant 600	30	7 14 28 35	beans	< 0.01 < 0.01 <u>< 0.01</u> < 0.01		BRI- 10/11-008 Test E

FATE OF RESIDUES IN STORAGE AND PROCESSING

High temperature hydrolysis

In a study reported by Lowrie, 2005 [Ref: DP-16989], the high-temperature hydrolysis of [^{14}C]-cyantraniliprole in buffered solutions of pH 4, 5, and 6 was investigated to simulate representative processing conditions: pasteurization at 90 °C for 20 minutes in pH 4 solution; baking, brewing, or boiling at 100 °C for 60 minutes in pH 5 solution; and sterilization at 120 °C for 20 minutes in pH 6 solution.

Solutions of [CN-^{14}C]-cyantraniliprole and [PC-^{14}C]-cyantraniliprole were incubated at 90, 100, and 120 °C at pH 4, 5, and 6, respectively, using aqueous citrate buffer and samples analyzed

after 20 min. (pH 4 and 6) and 1 h (pH 5) by LSC and HPLC. Identification of parent and transformation products was by co-chromatography and the identifications made were confirmed using LC-MS analysis. The limit of quantification (LOQ) for both labelled forms was 1% AR.

Cyantraniliprole was susceptible to hydrolysis with IN-J9Z38 being the significant hydrolysis product, making up about 4–5% AR under conditions representative of pasteurization (20 minutes at 90 °C and pH 4) and 12-14% AR under sterilisation conditions (20 minutes at 120 °C and pH 6). Under conditions reflecting baking, brewing, or boiling (60 minutes at 100 °C and pH 5) levels of IN-J9Z38 were lower, at 4–6% AR with IN-F6L99 and IN-N5M09 making up a further 5–8% AR. Minor components were detected but represented < 1% AR.

Table 151 Distribution of radioactivity for [CN-^{14}C]-cyantraniliprole in citrate buffer solutions—high temperature hydrolysis (% radioactivity)

Component	pH 4		pH 5		pH 6	
	Control	90 °C	Control	100 °C	Control	120 °C
Cyantraniliprole (parent)	97.54	90.56	96.73	74.54	96.92	93.58
IN-J9Z38	0.69	4.40	1.14	11.51	0.92	6.21
IN-N5M09	ND	1.90	ND	8.41	ND	ND
Others [a]	ND	0.70	0.32	3.39	0.47	1.64
Apparatus wash	0.77	0.84	0.80	0.74	0.74	0.68
Total % recovery	99.00	98.40	98.99	98.59	98.92	102.11

Table 152 Distribution of radioactivity for [PC-^{14}C]-cyantraniliprole in citrate buffer solutions—high temperature hydrolysis (% radioactivity)

Component	pH 4		pH 5		pH 6	
	Control	90 °C	Control	100 °C	Control	120 °C
Cyantraniliprole	97.00	89.23	94.71	75.12	98.43	91.49
IN-J9Z38	0.14	5.36	1.05	13.64	0.36	3.71
IN-F6L99	ND	1.71	ND	5.33	ND	0.12
Other peaks	0.80	1.70	1.33	4.04	1.26	2.14
Total	97.94	97.99	97.09	98.12	100.05	97.46

Potatoes

In three field trials conducted in North America and reported by Thiel, 2010 [Ref: DP-27583], bulk samples of tubers were taken from plots planted with cyantraniliprole-treated seed pieces (13.5 g ai/100 kg seed) and treated with a single foliar application of 0.15 kg ai/ha cyantraniliprole (OD formulation), with added surfactant, 7 days before harvest.

Samples were shipped under ambient conditions to the processing facility where samples were processed to potato flakes, potato waste, peeled potatoes, potato chips, wet peel, culls, fries, cooking water, unpeeled boiled potatoes, and unpeeled microwaved potatoes.

Tubers were washed for 5 minutes and representative samples were batch steam peeled, batch scrubbed for 20 seconds and further processed into flake processing and french fries. The collected peel was hydraulically pressed and blended with the cut trim waste to derive the wet potato waste sample.

A representative sample of steam-peeled potatoes were cut into slabs, batch spray-washed in cold tap water for 30 seconds to remove free starch, precooked at 70–77 °C for 20 minutes in a steam jacketed kettle and cooled to less than 32 °C for 20 minutes. The cooled potato slabs were then steam-cooked at 94–l00 °C for 40 minutes and mixed with an emulsion of pre-weighed food additives. The cooked mash was then hand fed onto a drum dryer to produce a thin sheet of potato flake which was then hammer-milled to give the final potato flakes sample.

An additional representative sample of steam-peeled potatoes were pre-cooked in 54 °C water for 40 minutes and then cut into 3/16" strips using a french fry cutter. After cutting, the strips were spray-washed for 30 seconds to remove free starch, blanched in 79–85 °C water for 5 minutes and then dipped in a solution of 0.5% sodium acid pyrophosphate and 0.5% dextrose for 30 seconds at 71–74 °C. The strips were then air dried (75 °C) to reduce the moisture content of the strips by about 15% and par-fried in vegetable oil for 45–50 seconds at 188–191 °C. After frying, the fried strips were drained to remove excess oil and placed in the freezer for rapid (12 minute) cooling before being labelled and placed back into freezer storage for subsequent analysis.

Potato chips were prepared from the washed potatoes by batch peeling and slicing the peeled potatoes into thin 16mm slices. The sliced potatoes were placed in a tub of hot water to remove free starch, drained and fried at163–191 ° C frying oil for 90 seconds. After draining and salting, samples were labelled and stored frozen for subsequent analysis.

For producing unpeeled boiled potatoes, washed potatoes were quartered and boiled in water until an internal temperature of 88–92 °C was achieved. Once the desired temperature was reached, the unpeeled boiled potato fraction was removed from the water and packaged, labelled and placed into frozen storage.

For producing unpeeled microwaved potatoes, the quartered potatoes were placed into the commercial microwave oven and microwaved in 5 minute increments until an internal temperature of 88–92 °C was achieved. After the desired temperature was reached, the unpeeled microwaved potato fraction was packaged, labelled and placed into frozen storage.

Tubers and processed fractions were stored at –20 °C for up to 9 months before analysis (within 3 months of extraction) for cyantraniliprole and eight metabolites using LC/MS/MS method DP-15736. The reported LOQ in all matrices was 0.01 mg/kg and average concurrent recoveries were 91-95% (cyantraniliprole) and 79–113% (metabolites) in samples spiked with 0.01 and 0.1 mg/kg.

Residues of cyantraniliprole were detected at up to 0.02 mg/kg in fresh tubers, less than 0.01 mg/kg in wet peel and unpeeled microwaved potatoes and were not detectable in all other samples tested. The IN-JZ38 metabolite was also measured at < 0.01 mg/kg in tubers and wet peel, but was not detectable in other matrices. Residues of the other metabolites (IN-JCZ38, IN-K7H19, IN-MLA84, IN-MYX98, IN-N7B69, IN-N5M09, and IN-F6L99) were not detected in tubers or in processed fractions.

Table 153 Residues in potato tubers and processed products from supervised trials in North America involving a seed treatment plus one foliar application of cyantraniliprole

POTATO Study ID	Matrix	Cyantraniliprole	IN-J9Z38	Total	
		mg/kg	mg/kg	mg/kg	PF
DP-27583 Test 10	tubers	< 0.01 (3)	ND	< 0.01	–
	flakes	ND	ND	ND	–
	waste	ND	ND	ND	–
	peeled tubers	ND	ND	ND	–
	chips	ND	ND	ND	–
	wet peel	< 0.01	< 0.01	< 0.02	–
	culls	< 0.01	ND	< 0.01	–
	fries	ND	ND	ND	–
	cooking water	ND	ND	ND	–
	unpeeled, boiled	ND	ND	ND	–
	unpeeled mwaved	< 0.01	ND	< 0.01	–

POTATO Study ID	Matrix	Cyantraniliprole	IN-J9Z38	Total	
		mg/kg	mg/kg	mg/kg	PF
DP–27583	tubers	0.02, < 0.01, ND	ND	< 0.01	–
Test 11	flakes	ND	ND	ND	–
	waste	ND	ND	ND	–
	peeled tubers	ND	ND	ND	–
	chips	ND	ND	ND	–
	wet peel	< 0.01	< 0.01	< 0.02	–
	culls	0.01	ND	< 0.01	–
	fries	ND	ND	ND	–
	cooking water	ND	ND	ND	–
	unpeeled, boiled	ND	ND	ND	–
	unpeeled m'waved	ND	ND	ND	–
DP-27583	tubers	0.02, 0.02, 0.02	< 0.01	0.03	
Test 12	flakes	ND	ND	ND	0.1
	waste	ND	ND	ND	0.1
	peeled tubers	ND	ND	ND	0.1
	chips	ND	ND	ND	0.1
	wet peel	0.02	0.05	0.07	2.3
	culls	0.02	< 0.01	< 0.03	1.0
	fries	ND	ND	ND	0.1
	cooking water	ND	ND	ND	0.1
	unpeeled, boiled	ND	ND	ND	0.1
	unpeeled m'waved	< 0.01	ND	< 0.01	0.33

For calculation purposes, where the residue in the processed commodity was reported as ND (< LOD), a value of 0.003 mg/kg was used and where residues were above the LOD but below the LOQ, a value of 0.01 mg/kg was used. In both cases, the PF was expressed as "less than" (e.g. < 0.01). Where residues of IN-J9Z38 are below the LOQ in the RAC, a value of 0.01 has been used to calculate 'total' residues.

Spinach

The effects of cooking on residues of cyantraniliprole in spinach were investigated by Carringer & Vogl, 2010 [Ref: DP-25644]. Bulk samples of spinach were taken from three field trials in the USA, one day after the last of three foliar applications of 0.15 kg ai/ha cyantraniliprole (OD formulation), applied at 5–6 day intervals with added surfactant.

At each of the three field sites about 400 g spinach leaves were added to 3 L boiling water in a large pot and boiled in the uncovered pot for approximately 15 minutes, stirring as needed to keep the leaves submerged under the water. The cooked spinach was removed and squeezed to remove as much water as possible.

Fresh and cooked leaves and the cooking water were stored at –20 °C for up to 8 months before analysis (within 10 weeks of extraction) for cyantraniliprole and eight metabolites using LC/MS/MS method DP-15736. The reported LOQ in all matrices was 0.01 mg/kg and average concurrent recoveries were 87–98% (cyantraniliprole) and 73–103% (metabolites) in samples spiked with 0.01 and 0.1 mg/kg.

Residues of the IN-J9Z38, IN-MLA84 and IN-N5M09 metabolites increased significantly during the cooking process, with IN-J9Z38 being the predominant residue in cooked spinach. Residues of the other metabolites (IN-MYX98, IN-N7B69, IN-JCZ38 and IN-F6L99) decreased and the metabolite IN-K7H19 was not detected in any samples.

Table 154 Residues in fresh and cooked spinach from supervised trials in North America involving three foliar applications of cyantraniliprole (OD formulation)

SPINACH Study ID	Matrix	Cyantraniliprole	IN-J9Z38	Total		Other metabolites (mg/kg)					
		mg/kg	mg/kg	mg/kg	PF	M2	M3	M4	M5	M6	M7
DP-25644	fresh leaves	4.6	0.07	5.3		0.24	< 0.01	0.02	< 0.01	ND	ND
Trial 35	cooked leaves	0.8	3.5	4.3	0.81	0.01	ND	0.09	ND	0.02	< 0.01
	water	0.03	0.25	0.28	0.05	ND	ND	0.02	ND	ND	< 0.01
DP-25644	fresh leaves	5.3	0.04	5.7		0.15	< 0.01	0.01	ND	< 0.01	ND
Trial 36	cooked leaves	0.86	5.8	6.7	1.3	ND	ND	0.19	ND	0.04	< 0.01
	water	0.24	0.22	0.46	0.08	ND	ND	0.01	ND	ND	< 0.01
DP-25644	fresh leaves	10	0.1	10.1		0.43	0.01	0.05	< 0.01	< 0.01	< 0.01
Trial 37	cooked leaves	0.9	9.2	10.1	1.0	< 0.01	ND	0.35	ND	0.09	0.015
	water	0.18	0.45	0.63	0.06	ND	ND	0.06	ND	< 0.01	0.011

M2: Residues of metabolite IN-MYX98 M3: Residues of metabolite IN-N7B69
M4: Residues of metabolite IN-MLA84 M5: Residues of metabolite IN-JCZ38
M6: Residues of metabolite IN-N5M09 M7: Residues of metabolite IN-F6L99

Tomatoes

Processing studies on tomatoes were reported by Carringer & Vogl, 2009 [Ref: DP-25645]. In three field trials in the USA, involving three foliar applications of 0.3 kg ai/ha cyantraniliprole (OD formulation), applied at 5 day intervals, with added surfactant, bulk samples were taken one day after the last application and shipped under ambient conditions to the processing facility where samples were processed to washed tomatoes, peeled tomatoes, sun-dried tomatoes, canned tomatoes, juice, wet pomace, dried pomace, paste, and puree.

Canned tomatoes were prepared by spray-rinsing the field samples with high pressure warm water (68–74 °C) for 30 seconds and boiling the rinsed tomatoes for about a minute to crack the skin prior to removal and peeled by hand before packing in cans with a teaspoon of salt. The cans were steam heated for about 10 minutes to achieve a temperature of about 80 °C, sealed, cooked in a boiling water bath for about 15 minutes and cooled in cold tap water.

Sun-dried tomatoes were prepared from the rinsed samples by removing the cores and stems, quartering the fruit and spreading them with the peel down on a tray for drying to a moisture content of < 16%.

Washed tomato samples were prepared by batch soaking a representative sample of the field tomatoes in a 5 g/L solution of sodium hydroxide at about 52–60 °C for 3 minutes and then spray-rinsing with high pressure warm water (68–74 °C) for 30 seconds.

Pomace samples were prepared by crushing the washed tomatoes in a hammermill and then rapidly heating the crush to about 79–85 °C for 15–30 seconds (hot break) before separation into pomace and juice. The wet pomace was pressed and the recovered press juice was combined with the fresh juice from the pulper/finisher. A representative sample of the wet pomace was then bin-dried to less than 10% moisture.

Fresh juice was heated to not more than 82 °C in the steam jacketed kettle and an aliquot was vacuum-evaporated to produce puree with the addition of 1% salt and the Brix level adjusted to 12.0–13.0° by the addition of water. The puree was heated to 82–88 °C, canned, sealed and cooked in a boiling water bath for about 15 minutes at 96–100 °C and then cooled under running cold tap water

Fresh juice was also vacuum-evaporated to paste with the addition of 0.5–1.0% salt and the Brix level adjusted to 24.0–33° by the addition of water. The puree was heated to 82–88 °C, canned, sealed and cooked in a boiling water bath for about 15 minutes at 96–100 °C and then cooled under running cold tap water.

An aliquot of the paste was also diluted with hot water, mixed with 0.5%–1.0% salt and the Brix level adjusted to about 4.5–5.5°, to produce juice. This juice was then heated to about 85 °C, canned, sealed and cooked in a boiling water bath for about 15 minutes at 96–100 °C and then cooled in cold tap water.

Processed samples were stored at or below -17 °C for up to 7 months before analysis (within about 2 weeks of extraction) for cyantraniliprole and eight metabolites using LC/MS/MS method DP-15736. The reported LOQ in all matrices was 0.01 mg/kg and average concurrent recoveries were 76–102% (cyantraniliprole) and 70–117% (metabolites) in samples spiked with 0.01 to 2.0 mg/kg.

Cyantraniliprole was the predominant residue in processed tomato products, with significant residues of the IN-J9Z38 metabolite also found in wet and dry pomace. In the wet and dry pomace, paste and sun-dried tomato samples, low levels (up to 0.04 mg/kg) of IN-MYX98, IN-MLA84 or IN-N5M09 were reported. Residues of the other metabolites (IN-N7B69, IN-K7H19 and IN-JCZ38) were not detected in any samples.

Table 155 Residues in fresh and processed tomatoes from supervised trials in North America involving three foliar applications of cyantraniliprole (OD formulation)

TOMATO Study ID	Matrix	Cyantraniliprole	IN-J9Z38	Total		Other metabolites (mg/kg)			
		mg/kg	mg/kg	mg/kg	PF	M2	M4	M6	M7
DP-25645 Trial 1	fresh (field)	0.13	ND	0.13		ND	ND	ND	ND
	washed	0.02	ND	0.02	0.15	ND	ND	ND	ND
	peeled	< 0.01	ND	< 0.01	< 0.08	ND	ND	ND	ND
	sun-dried	0.45	0.03	0.48	3.7	ND	ND	ND	ND
	canned	ND	ND	ND	< 0.02	ND	ND	ND	ND
	juice	< 0.01	< 0.01	< 0.02	< 0.15	ND	ND	ND	ND
	wet pomace	0.08	0.05	0.13	1.0	ND	< 0.01	ND	ND
	dry pomace	0.23	0.18	0.41	3.2	ND	0.01	< 0.01	ND
	paste	0.03	0.05	0.08	0.62	ND	ND	< 0.01	< 0.01
	puree	0.02	0.01	0.03	0.23	ND	ND	ND	ND
DP-25645 Trial 2	fresh (field)	0.2	< 0.01	0.21		ND	ND	ND	ND
	washed	0.05	< 0.01	< 0.06	< 0.29	ND	ND	ND	ND
	peeled	0.02	ND	0.02	0.1	ND	ND	ND	ND
	sun-dried	0.73	0.07	0.8	3.8	ND	< 0.01	< 0.01	< 0.01
	canned	< 0.01	ND	< 0.01	< 0.05	ND	ND	ND	ND
	juice	0.03	0.01	0.04	0.19	ND	ND	ND	ND
	wet pomace	0.29	0.17	0.46	2.2	ND	0.02	< 0.01	ND
	dry pomace	0.5	0.35	0.85	4.0	ND	0.04	0.01	ND
	paste	0.12	0.06	0.18	0.86	ND	ND	< 0.01	< 0.01
	puree	0.07	0.02	0.09	0.43	ND	ND	ND	ND
DP-25645 Trial 3	fresh (field)	0.12	ND	0.12		ND	ND	ND	ND
	washed	0.02	ND	0.02	0.17	ND	ND	ND	ND
	peeled	< 0.01	ND	< 0.01	< 0.08	ND	ND	ND	ND
	sun-dried	0.34	0.02	0.36	3.0	< 0.01	< 0.01	ND	ND
	canned	< 0.01	ND	< 0.01	< 0.08	ND	ND	ND	ND
	juice	0.01	< 0.01	< 0.02	< 0.17	ND	ND	ND	ND
	wet pomace	0.06	0.03	0.09	0.75	ND	ND	ND	ND
	dry pomace	0.12	0.08	0.2	1.7	ND	ND	< 0.01	ND
	paste	0.08	0.04	0.12	1.0	ND	ND	< 0.01	< 0.01
	puree	0.02	0.01	0.03	0.25	ND	ND	ND	ND

M2: Residues of metabolite IN-MYX98 M4: Residues of metabolite IN-MLA84

M6: Residues of metabolite IN-N5M09 M7: Residues of metabolite IN-F6L99

For calculation purposes, where the residue in the processed commodity was reported as ND (< LOD), a value of 0.003 mg/kg was used and where residues were above the LOD but below the LOQ, a value of 0.01 mg/kg was used. In both cases, the PF was expressed as "less than" (e.g. < 0.01). Where residues of IN-J9Z38 are below the LOQ in the RAC, a value of 0.01 has been used to calculate 'total' residues.

Oranges

In three field trials on oranges conducted in North America and reported by Rice, 2010 [Ref: DP-27554], plots were treated 7 days apart with three late season foliar applications of 0.15 kg ai/ha

cyantraniliprole (OD formulation) with added surfactant and bulk samples of fruit were taken 1 day after the last application for processing.

The samples were shipped under ambient conditions to the processing facility where samples were processed to juice, wet pulp, dry pulp, meal, molasses, marmalade, oil, and canned oranges.

Juice was prepared from batch-washing field oranges by scarifying the fruit in a Hobart Peeler for 45 seconds, collecting the oil-water emulsion and passing it through a 180 µm screen to remove flavedo fragments before separating the oil fraction through a cream separator and then an IEC centrifuge. The residual emulsion was then frozen, thawed and centrifuged to collect the remaining oil.

Juice extracted from the scarified oranges was transferred to a pulper/finisher and passed through an approximately 1.2 mm screen to remove the rag and seeds. Peel from the juice extraction process was shredded and combined with scarified flavedo fragments and the residual pulp from the juice extraction to generate wet peel. Lime (about 95% CaO) was mixed with this wet peel and the limed peel was pressed to obtain press liquor which was then vacuum-evaporated to produce molasses.

An aliquot of the wet pulp was also dried to below 10% moisture and hammer-milled to produce the dry pulp sample, with further milling producing the meal sample.

Washed oranges were also peeled, segmented, mixed with a light syrup (Brix 14.0–18.0 °) and canned, with the sealed cans being cooked in a boiling water bath for about 15 minutes at 96°–100 °C and cooled with tap water.

For marmalade, the rinds from washed oranges were chipped in a food processor and cooked for 20 minutes and the chopped pulp (without seeds) was mixed with 20% w/w water and cooked for 45 minutes. The cooked rind and pulp (with about 45 mL lemon juice and 1.5 kg sugar added per kg) was then boiled for 3 minutes, pectin was added and the mixture was boiled for a further 2 minutes. After standing for 1–2 minutes, the finished marmalade was packed in sterilised jars for subsequent analysis.

Fruit and processed fractions were stored at -20 °C for up to 8 months before analysis (within 7 weeks of extraction) for cyantraniliprole and eight metabolites using LC/MS/MS method DP-15736. The reported LOQ in all matrices was 0.01 mg/kg and average concurrent recoveries were 81–96% (cyantraniliprole) and 64–101% (metabolites) in samples spiked with 0.01, 0.1 and 0.2 mg/kg.

The storage stability of frozen samples was also evaluated as part of this study. Whole fruit were cut in half and one-half of each fruit comprising each whole fruit incurred residue sample was pooled, homogenized, and extracted for analysis to provide an initial residue value for incurred residue stability determinations for this high-acid content commodity. The other half of these fruit were homogenized per sample and analyzed ten months later, to complete a ten-month incurred residue storage stability determination for orange fruit. Residues of cyantraniliprole after 10 months storage were 79, 88, and 128% of the inital residues in three composite samples.

Cyantraniliprole was the predominant residue in processed orange products, with residues concentrating in oil. Significant residues of the IN-J9Z38 metabolite were also in oil and other matrices, together with trace levels of the IN-N5M09 metabolite in oil. Residues of the other metabolites (IN-N7B69, IN-MYX98, IN-MLA84, IN-N5M09 IN-K7H19, IN-F6L99 and IN-JCZ38) were not detected in any samples.

Table 156 Residues in fresh and processed oranges from supervised trials in North America involving three foliar applications of cyantraniliprole (OD formulation)

ORANGE Study ID	Matrix	Cyantraniliprole	IN-J9Z38	Total		Other metabolites (mg/kg)
		mg/kg	mg/kg	mg/kg	PF	
DP-27554 Trial 8	oranges	0.13	ND	0.13		
	juice	< 0.01	ND	< 0.01	0.08	
	wet pulp	0.05	< 0.01	0.06	0.46	
	dry pulp	0.11	0.01	0.12	0.92	
	meal	0.1	< 0.01	0.11	0.85	
	molasses	ND	0.09	0.09	0.69	
	marmalade	< 0.01	< 0.01	< 0.02	< 0.15	
	oil	0.81	0.25	1.1	8.5	M6=< 0.01
	canned	ND	ND	ND	< 0.02	
DP-27554 Trial 25	oranges	0.09	ND	0.09		
	juice	ND	ND	ND	< 0.03	
	wet pulp	0.02	ND	0.02	0.22	
	dry pulp	0.02	< 0.01	< 0.03	< 0.33	
	meal	0.02	ND	0.02	0.22	
	molasses	ND	0.03	0.03	0.33	
	marmalade	ND	ND	ND	< 0.03	
	oil	0.21	0.048	0.26	2.9	
	canned	ND	ND	ND	< 0.03	
DP-27554 Trial 26	oranges	0.17	ND	0.17		
	juice	ND	ND	ND	< 0.02	
	wet pulp	0.04	ND	0.04	0.24	
	dry pulp	0.07	0.01	0.08	0.47	
	meal	0.07	0.01	0.08	0.47	
	molasses	ND	0.1	0.1	0.59	
	marmalade	ND	< 0.01	< 0.01	< 0.06	
	oil	1.4	0.08	1.5	8.8	
	canned	< 0.01	ND	< 0.01	< 0.06	

M6: Residues of metabolite IN-N5M09

For calculation purposes, where the residue in the processed commodity was reported as ND (< LOD), a value of 0.003 mg/kg was used and where residues were above the LOD but below the LOQ, a value of 0.01 mg/kg was used. In both cases, the PF was expressed as "less than" (e.g. < 0.01). Where residues of IN-J9Z38 are below the LOQ in the RAC, a value of 0.01 has been used to calculate 'total' residues.

Apples

In three field trials on apples conducted in North America and reported by Thiel, 2010 [Ref: DP-27438], plots were treated 7 days apart with three late season foliar applications of 0.15 kg ai/ha cyantraniliprole (SE formulation) with added surfactant and bulk samples of fruit were harvested 3 day after the last application for processing.

Samples were shipped under ambient conditions to the processing facility where samples were processed to washed apples, puree, canned apples, frozen apples, juice, wet pomace, dry pomace, and sauce.

To produce juice, fresh apples were ground in a hammer-mill and the wet mash was pressed to 2200–3000 psi for at least 5 minutes to extract the juice, leaving the wet pomace. A representative sample of the wet pomace was also dried for 1–4 hours using hot air at 60–88 °C, to achieve a moisture content of 10% or less.

Apple sauce was prepared from peeled, cored and diced apples by heating in a kettle with added water (approximately 25% by weight) at about 70 °C for 45 minutes and mashing to produce puree. Further heating at 82–93 °C for 6–10 hours allowed excess moisture to evaporate, producing

apple sauce. Apple slices were also prepared from peeled, cored and sliced (6–7 mm) fruit by heating to about 70 °C for 20 minutes, resulting in cooked slices in syrup.

Peeled and cored apples were also cut into sliced to 3 mm slices and dehydrated at 65–70 °C for 20–24 hours to achieve a moisture content of about 10%.

Fruit and processed fractions were stored at -20 °C for up to 8 months before analysis (within 11 weeks of extraction) for cyantraniliprole and eight metabolites using LC/MS/MS method DP-15736. The reported LOQ in all matrices was 0.01 mg/kg and average concurrent recoveries were 91–101% (cyantraniliprole) and 81–109% (metabolites) in samples spiked with 0.01, 0.1 and 1.0 mg/kg.

Cyantraniliprole was the predominant residue in processed apple products, with residues concentrating in the dry pomace. Significant residues of the IN-J9Z38 metabolite were present in sauce and to a lesser extent in dry pomace. Residues of IN-N5M09 and IN-F6L99 were also found at levels up to 0.07 mg/kg in sauce. Residues of the other metabolites (IN-N7B69, IN-MYX98, IN-MLA84, IN-K7H19 and IN-JCZ38) were not detected in any samples.

Table 157 Residues in fresh and processed apples from supervised trials in the USA involving foliar applications of cyantraniliprole (SE formulation)

APPLE Study ID	Matrix	Cyantraniliprole	IN-J9Z38	Total		Other metabolites (mg/kg)	
		mg/kg	mg/kg	mg/kg	PF	M6	M7
DP-27438 Trial 3	fruit	0.08	ND	0.08		ND	ND
	washed	0.05	ND	0.05	0.63	ND	ND
	puree	0.08	ND	0.08	1.0	ND	ND
	canned	0.01	ND	0.01	0.13	ND	ND
	frozen	0.12	ND	0.12	1.5	ND	ND
	juice	0.03	ND	0.03	0.38	ND	ND
	wet pomace	0.08	ND	0.08	1.0	ND	ND
	dry pomace	0.3	< 0.01	0.31	3.9	ND	ND
	sauce	0.13	0.06	0.19	2.4	0.014	< 0.01
DP-27438 Trial 10	fruit	0.26	ND	0.26		ND	ND
	washed	0.15	ND	0.15	0.58	ND	ND
	puree	0.23	ND	0.23	0.88	ND	ND
	canned	0.01	ND	0.01	0.04	ND	ND
	frozen	0.25	ND	0.25	0.96	ND	ND
	juice	0.05	ND	0.05	0.19	ND	ND
	wet pomace	0.31	ND	0.31	1.2	ND	ND
	dry pomace	0.68	0.02	0.7	2.7	ND	ND
	sauce	0.21	0.35	0.56	2.2	0.053	0.04
DP-27438 Trial 14	fruit	0.26	ND	0.26		ND	ND
	washed	0.12	ND	0.12	0.46	ND	ND
	puree	0.34	ND	0.34	1.3	ND	ND
	canned	0.03	< 0.01	0.04	0.15	ND	ND
	frozen	0.16	ND	0.16	0.62	ND	ND
	juice	0.08	ND	0.08	0.31	ND	ND
	wet pomace	0.2	ND	0.2	0.77	ND	ND
	dry pomace	0.52	0.01	0.53	2.0	ND	ND
	sauce	0.36	0.35	0.71	2.7	0.07	0.036

M6: Residues of metabolite IN-N5M09 M7: Residues of metabolite IN-F6L99

For calculation purposes, where the residue in the processed commodity was reported as ND (< LOD), a value of 0.003 mg/kg was used and where residues were above the LOD but below the LOQ, a value of 0.01 mg/kg was used. In both cases, the PF was expressed as “less than” (e.g. < 0.01). Where residues of IN-J9Z38 are below the LOQ in the RAC, a value of 0.01 has been used to calculate ‘total’ residues.

Plums

In three field trials on plums conducted in North America and reported by Thiel, 2010 [Ref: DP-27437], plots were treated 7 days apart with three late season foliar applications of 0.15 kg ai/ha cyantraniliprole (SE formulation) with added surfactant and bulk samples of fruit were taken 3 day after the last application for processing.

Samples were shipped under ambient conditions to the processing facility where the plums were stored for up to three days at about 6 °C before processing. The plums were sliced in half, the stones were removed and the pitted plums, on dehydrator trays, were dried at 130°–150 °C for about 14 to 18 hours to produce dried prunes, with a moisture content of 15–18%.

Plums and dried prunes were stored at –20 °C for up to 9 months before analysis (within 15 weeks of extraction) for cyantraniliprole and eight metabolites using LC/MS/MS method DP-15736. The reported LOQ in all matrices was 0.01 mg/kg and average concurrent recoveries were 101–104% (cyantraniliprole) and 92–109% (metabolites) in samples spiked with 0.01, 0.1, 0.2 and 0.5 mg/kg.

Cyantraniliprole was the predominant residue in dried prunes, found a levels up to twice those in plums (without stones). Residues of the IN-J9Z38 metabolite were also found in the dried prunes, with IN-N5M09 and IN-F6L99 also found at levels up to 0.01 mg/kg. Residues of the other metabolites (IN-N7B69, IN-MYX98, IN-MLA84, IN-K7H19 and IN-JCZ38) were not detected in any samples.

Table 158 Residues in plums and dried prunes from supervised trials in North America involving foliar applications of cyantraniliprole (SE formulation)

PLUM Study ID	Matrix	Cyantraniliprole	IN-J9Z38	Total		Other metabolites (mg/kg)	
		mg/kg	mg/kg	mg/kg	PF	M6	M7
DP-27437	plum flesh	0.18	ND	0.18		ND	ND
Trial 14	dried prunes	0.35	0.01	0.36	2.0	ND	ND
DP-27437	plum flesh	0.24	< 0.01	0.25		ND	ND
Trial 16	dried prunes	0.37	0.04	0.41	1.6	< 0.01	< 0.01
DP-27437	plum flesh	0.04	ND	0.04		ND	ND
Trial 20	dried prunes	0.05	ND	0.05	1.3	ND	ND

M6: Residues of metabolite IN-N5M09 M7: Residues of metabolite IN-F6L99

For calculation purposes, where the residue in the processed commodity was reported as ND (< LOD), a value of 0.003 mg/kg was used and where residues were above the LOD but below the LOQ, a value of 0.01 mg/kg was used. In both cases, the PF was expressed as "less than" (e.g. < 0.01). Where residues of IN-J9Z38 are below the LOQ in the RAC, a value of 0.01 has been used to calculate 'total' residues.

Cotton seed

In three field trials on cotton conducted in North America and reported by Thiel, 2010 [Ref: DP-27565], plots were treated 7 days apart with three late season foliar applications of 0.75 kg ai/ha cyantraniliprole (OD formulation) with added surfactant and bulk samples of cotton seed were taken 7 day after the last application for processing.

Ginned cotton seed samples were saw-delinted to remove most of the remaining lint, the hulls were cracked using a roller mill and screened (4–5 mm mesh) to separate out the kernels. After adjusting the kernel moisture content to 12–13.5%, the kernel material was heated to 80–90 °C for 30 minutes, flaked in a roller mill and steam extruded (93–121 °C) to form collets (pellets) which were then dried for 30–40 minutes at 66–82 °C prior to solvent extraction.

Flakes and collets were soaked in hexane at 49–60 °C for 30 minutes (1st cycle) and then 15 minutes each (2nd and 3rd cycles) with the miscella (crude oil and hexane) from each cycle being drained off and combined with the residual hexane from the final extraction (at about 95 °C). The combined miscella was passed through a vacuum evaporator (91–96 °C) to remove the hexane, leaving the raw oil solvent extract. This crude oil was refined by mixing with sodium hydroxide (16 °

Baume) in a water bath, first at 20–24 °C then at 63–67 °C before centrifuging to separate the oil from the soapstock.

Samples of delinted seed were also cold-pressed using a Komet expeller and the crude oil obtained by mechanical pressing was filtered and alkali-refined to produce refined cold-pressed oil.

Samples were shipped under frozen conditions to the processing facility where they were processed to cottonseed raw oil, cottonseed refined oil, cottonseed meal, cottonseed hull, cottonseed raw oil (cold press), cottonseed refined oil (cold press), and cottonseed meal (cold press).

The processing samples were stored at -20 °C for up to 8 months before analysis (within 13 weeks of extraction) for cyantraniliprole and eight metabolites using LC/MS/MS method DP-15736. The reported LOQ in all matrices was 0.01 mg/kg and average concurrent recoveries were 75–114% (cyantraniliprole) and 65–120% (metabolites) in samples spiked with 0.01–1.0 mg/kg.

Cyantraniliprole was the predominant residue in the processed fractions, with residues of the IN-J9Z38 metabolite also present in most oil fractions. Residues of the other metabolites (IN-N7B69, IN-MYX98, IN-MLA84, IN-N5M09, IN-F6L99, IN-K7H19 and IN-JCZ38) were not detected in any samples.

Table 159 Residues in raw and processed cotton seed from supervised trials in the USA involving three foliar applications of cyantraniliprole (OD formulation)

COTTON SEED Study ID	Matrix	Cyantraniliprole	IN-J9Z38	Total		Other metabolites (mg/kg)	
		mg/kg	mg/kg	mg/kg	PF	M6	M7
DP-27565	cottonseed	0.52	< 0.01	0.53		ND	ND
Trial 4	raw oil (solvent extr)	0.02	< 0.01	0.03	0.06	ND	ND
	refined oil (solvent extr)	ND	0.02	0.02	0.04		
	meal (solvent extr)	0.05	ND	0.05	0.09		
	hulls	0.17	< 0.01	0.18	0.34		
	raw oil (cold press)	0.16	ND	0.16	0.3		
	refined oil (cold press)	ND	0.02	0.02	0.04		
	meal (cold press)	0.06	ND	0.06	0.11		
DP-27565	cottonseed	0.71	0.02	0.73		ND	ND
Trial 10	raw oil (solvent extr)	0.017	0.016	0.03	0.04	< 0.01	< 0.01
	refined oil (solvent extr)	ND	0.02	0.02	0.03		
	meal (solvent extr)	0.01	< 0.01	< 0.02	< 0.03		
	hulls	0.25	0.02	0.27	0.37		
DP-27565	cottonseed	1.6	0.01	1.6		ND	ND
Trial 13	raw oil (solvent extr)	0.05	0.07	0.12	0.08	ND	ND
	refined oil (solvent extr)	ND	0.08	0.08	0.05		
	meal (solvent extr)	0.06	0.02	0.08	0.05		
	hulls	0.42	< 0.01	< 0.43	< 0.27		
	raw oil (cold press)	0.34	< 0.01	< 0.34	< 0.21		
	refined oil (cold press)	ND	0.07	0.07	0.04		
	meal (cold press)	0.11	ND	0.11	0.07		

M6: Residues of metabolite IN-N5M09 M7: Residues of metabolite IN-F6L99

For calculation purposes, where the residue in the processed commodity was reported as ND (< LOD), a value of 0.003 mg/kg was used and where residues were above the LOD but below the LOQ, a value of 0.01 mg/kg was used. In both cases, the PF was expressed as “less than” (e.g. < 0.01). Where residues of IN-J9Z38 are below the LOQ in the RAC, a value of 0.01 has been used to calculate ‘total’ residues.

Olives

In three field trials on olives conducted in Europe and reported by Haigh & Cairns, 2011[Ref: DP-27709], plots were treated about 10 days apart with two late season foliar applications of 5 g ai/hL (0.15 kg ai/ha) cyantraniliprole (OD formulation) without added surfactant and bulk samples of olives were taken 14 day after the last application for processing.

Samples were shipped under ambient conditions to the processing facility within 1 day of sampling and were immediately processed to canned olives, raw and refined oil. The fresh whole olives were washed by immersion, drained and placed in a brine solution (220 g salt/2 L/kg olives) and allowed to soak for 30 days at room temperature and stirred daily before being stored for 60 days at 5–10 °C. The olives in brine were then canned (500 g olives with 250 g brine) and sterilised for 10 minutes at 115–120 °C.

For oil production, the fresh olives were hammer-milled and the resulting pulp was mixed and heated for about 30 minutes in a thermo-malaxer with the addition of boiling water and centrifuged to separate the oil/margines/water from the solid cake. The liquid phase was left to settle and the floating oil was decanted, with one portion being filtered to produce raw (virgin) oil and the other portion used to obtain refined oil.

The raw (unfiltered) oil was mixed with 115 g/L soda, heated in an oven at 60–70 °C for at least 30 minutes and the oil decanted from the sediment (soap) and filtered to give refined oil.

The processed samples were stored at –20 °C for up to 5 months before analysis (within 1 week of extraction) for cyantraniliprole and six metabolites using LC/MS/MS method DP-15736. The reported LOQ in all matrices was 0.01 mg/kg and average concurrent recoveries were 84–103% (cyantraniliprole) and 87–107% (metabolites) in samples spiked with 0.01–1.0 mg/kg.

Cyantraniliprole was the predominant residue in the processed fractions, with levels concentrating in oil. Residues of the IN-J9Z38 metabolite were also measured in canned olives and trace residues (< 0.01 mg/kg) of IN-N5M09 and IN-F6L99 were also detected in canned olives and IN-MYX98 and IN-JCZ38 in oil. Residues of the other metabolites (IN-N7B69, IN-MLA84 and IN-K7H19 were not detected in any samples.

Table 160 Residues in processed olives and olive oil from supervised trials in Europe involving foliar applications of cyantraniliprole (SE formulation)

OLIVE Study ID	Matrix	Cyantraniliprole	IN-J9Z38	Total		Other metabolites (mg/kg)			
		mg/kg	mg/kg	mg/kg	PF	M2	M5	M6	M7
DP-27709	whole fruit	0.55	ND	0.55		ND	ND		
Trial 7	canned fruit	0.22	0.12	0.34	0.62	ND	ND	< 0.01	< 0.01
	olive flesh	0.93	< 0.01	0.94	1.7	ND	ND		
	canned flesh	0.34	0.18	0.52	0.95	ND	ND	< 0.01	< 0.01
	raw oil	0.92	< 0.01	0.93	1.7	ND	ND	ND	ND
	refined oil	0.69	< 0.01	0.7	1.3	ND	ND	ND	ND
DP-27709	whole fruit	0.26	ND	0.26		ND	ND		
Trial 9	canned fruit	0.1	0.05	0.15	0.58	ND	ND	ND	ND
	olive flesh	0.45	< 0.01	0.46	1.8	ND	ND		
	canned flesh	0.16	0.09	0.25	0.96	ND	ND	ND	ND
	raw oil	0.31	0.01	0.32	1.2	ND	ND	ND	ND
	refined oil	0.17	0.04	0.21	0.81	ND	ND	ND	ND

OLIVE Study ID	Matrix	Cyantraniliprole	IN-J9Z38	Total		Other metabolites (mg/kg)			
		mg/kg	mg/kg	mg/kg	PF	M2	M5	M6	M7
DP-27709	whole fruit	0.29	< 0.01	0.3		ND	ND		
Trial 10	canned fruit	0.04	0.02	0.06	0.2	ND	ND	ND	ND
	olive flesh	0.45	< 0.01	0.46	1.5	ND	ND	< 0.01	
	canned flesh	0.14	0.07	0.21	0.7	ND	ND	< 0.01	ND
	raw oil	0.16	0.02	0.18	0.6	ND	ND	ND	ND
	refined oil	0.06	0.06	0.12	0.4	< 0.01	< 0.01	ND	ND

M2: Residues of metabolite IN-MYX98 M5: Residues of metabolite IN-JCZ38

M6: Residues of metabolite IN-N5M09 M7: Residues of metabolite IN-F6L99

For calculation purposes, where the residue in the processed commodity was reported as ND (< LOD), a value of 0.003 mg/kg was used and where residues were above the LOD but below the LOQ, a value of 0.01 mg/kg was used. In both cases, the PF was expressed as "less than" (e.g. < 0.01). Where residues of IN-J9Z38 are below the LOQ in the RAC, a value of 0.01 has been used to calculate 'total' residues.

Grapes

In three field trials on wine grapes conducted in Europe and reported by Aitken, 2011 [Ref: DP-27718], plots were treated about 14 days apart with two late season foliar applications of 0.15 kg ai/ha) cyantraniliprole (OD formulation) by either high volume (1500 L/ha) or concentrate (300 L/ha) applications without added surfactant and bulk samples of grapes were taken 14 days after the last application for processing.

Samples were shipped under ambient conditions to the processing facility where samples were processed to stems, must, must deposit, wet pomace, lees, dry pomace, alcoholic fermentation (AF) Wine, malolactic fermentation (MF) Wine, bottled wine, grape juice and raisins.

For wine, grape samples were crushed and stemmed with an electric crusher/stemmer and sub samples taken for storage and analysis. For red wine, potassium metabisulphite (0.06–0.08 g/L) was added to the crushed grapes (must) and dry active yeast (0.1 g/L) added to initiate alcoholic fermentation. For white wine, potassium metabisulphite (0.12 g/L) and pectolytic enzymes were added to the must, which was then allowed to settle for 24 hours, decanted and the yeast was added to initiate alcoholic fermentation. The remaining wet pomace was sub-sampled for subsequent analysis and the wet pomace was also oven-dried at about 60 °C for two days to produce dry pomace.

At the end of the fermentation period, when the wine density had stabilised, the red wine was decanted and the solids were pressed to extract the remaining wine from the wet pomace. The wet pomace and the 'alcoholic fermentation' wine (AF wine) were sub-sampled for subsequent analysis and the wet pomace was also oven-dried at about 60 °C for two days to produce dry pomace. The white wine was separated from the lees and de-acidified by the addition of potassium bicarbonate.

Both the red and white 'AF wine' was then subjected to malolactic fermentation in the absence of air at ambient temperature by the addition of lactic bacteria and on completion, potassium metabisulphite was added. After settling for 4 days, the 'after malolactic fermentation' wine (MF wine) was separated from the lees and the wine was further clarified by the addition of gelatine (0.1 g/L) and potassium metabisulphite (0.04 g/L). After cool storage, the wine was racked and pressure-filtered (cellulose filter plate—2.5 µm pore) under nitrogen and 0.1 g/L potassium metabisulphite added to protect against oxidation.

Grape juice was prepared from the fresh grape samples by removing the stems and manually crushing the berries. After the addition of pectolytic enzymes, the crush was heated to 50 °C, put in jars and sterilised for 2 hours at 45–60 °C before being water-pressed to separate the juice and pomace. The juice was then clarified (5 minutes at about 80 °C), cool stored, racked and pressure-filtered under nitrogen and pasteurised for 1 minute at +85 °C.

Raisins were produced by oven-drying the bunch samples at 60 °C until the weight of the dried grapes was less than 35% of the initial weight and the stems were then removed by hand.

The processed samples were stored at –18 °C for up to 21 months before analysis (within 5 days of extraction) for cyantraniliprole and six metabolites using LC/MS/MS method DP-15736. The reported LOQ in all matrices was 0.01 mg/kg and average concurrent recoveries were 72–111% (cyantraniliprole) and 71–111% (metabolites) in samples spiked with 0.01 and 0.1 mg/kg.

Cyantraniliprole was the predominant residue in the processed fractions, with levels concentrating in the pomace and in raisins. Residues of the IN-J9Z38 metabolite were also measured in dry pomace, lees and juice, with trace levels (< 0.01 mg/kg) also found in wine and raisins. Low levels of IN-N5M09, IN-MYX98 and IN-F6L99 were also detected in pomace and juice. Residues of the other metabolites (IN-N7B69, IN-MLA84, IN-JCZ38 and IN-K7H19 were not detected in any samples.

Table 161 Residues in grapes and processed fractions from supervised trials in Europe involving foliar applications of cyantraniliprole (SE formulation)

GRAPE Study ID	Matrix	Cyantraniliprole	IN-J9Z38	Total		Other metabolites (mg/kg)		
		mg/kg	mg/kg	mg/kg	PF	M2	M6	M7
DP-27718	bunches	0.56	ND	0.56		ND		
Trial 6	grapes	0.16	ND	0.16	0.29	ND		
	stems	0.69	ND	0.69	1.2	< 0.01	ND	ND
red wine	must	0.44	ND	0.44	0.79	ND	ND	ND
	wet pomace	0.77	ND	0.77	1.4	ND	< 0.01	ND
	lees	0.32	0.02	0.34	0.61	ND	ND	ND
	dry pomace	1.4	0.1	1.5	2.7	< 0.01	0.02	ND
	AF wine	0.3	ND	0.3	0.54	ND	ND	ND
	MF wine	0.28	< 0.01	0.29	0.52	ND	ND	ND
	bottled wine	0.27	< 0.01	0.28	0.5	ND	ND	ND
	juice	0.25	0.02	0.27	0.48	ND	< 0.01	< 0.01
	raisins	0.26	< 0.01	0.27	0.48	ND		
DP-27718	bunches	0.21	ND	0.21		ND		
Trial 7	grapes	0.12	ND	0.12	0.57	ND		
	stems	0.48	ND	0.48	2.3	< 0.01		
red wine	must	0.32	ND	0.32	1.5	ND	ND	ND
	wet pomace	0.8	0.02	0.82	3.9	< 0.01	ND	ND
	lees	0.38	0.02	0.4	1.9	ND	ND	ND
	dry pomace	0.73	0.05	0.78	3.7	ND	< 0.01	ND
	AF wine	0.24	ND	0.24	1.1	ND	ND	ND
	MF wine	0.23	< 0.01	0.24	1.1	ND	ND	ND
	bottled wine	0.24	< 0.01	0.25	1.2	ND	ND	ND
	juice	0.1	0.01	0.11	0.52	ND	ND	ND
	raisins	0.1	< 0.01	0.11	0.52	ND		

GRAPE Study ID	Matrix	Cyantraniliprole	IN-J9Z38	Total		Other metabolites (mg/kg)		
		mg/kg	mg/kg	mg/kg	PF	M2	M6	M7
DP-27718	bunches	0.07	ND	0.07		ND		
Trial 8	grapes	0.11	ND	0.11	1.6	ND		
	stems	0.31	< 0.01	0.32	4.6	ND		
white wine	must	0.1	< 0.01	0.11	1.6	ND	ND	ND
	wet pomace	0.18	< 0.01	0.19	2.7	ND	ND	ND
	lees	0.46	ND	0.46	6.6	ND	ND	ND
	dry pomace	0.17	ND	0.17	2.4	ND	ND	ND
	AF wine	0.07	ND	0.07	1.0	ND	ND	ND
	MF wine	0.07	ND	0.07	1.0	ND	ND	ND
	bottled wine	0.07	ND	0.07	1.0	ND	ND	ND
	juice	0.09	< 0.01	0.1	1.4	ND	ND	ND
	raisins	0.15	< 0.01	0.16	2.3	ND		

M2: Residues of metabolite IN-MYX98 M6: Residues of metabolite IN-N5M09

M7: Residues of metabolite IN-F6L99

For calculation purposes, where the residue in the processed commodity was reported as ND (< LOD), a value of 0.003 mg/kg was used and where residues were above the LOD but below the LOQ, a value of 0.01 mg/kg was used. In both cases, the PF was expressed as "less than" (e.g. < 0.01). Where residues of IN-J9Z38 are below the LOQ in the RAC, a value of 0.01 has been used to calculate 'total' residues.

Table 162 Summary of processing factors for cyantraniliprole and cyantraniliprole + IN-J9Z38

RAC	Matrix	Cyantraniliprole + IN-J9Z38 [a]	
		Calculated processing factors	PF median
Potato	tubers		
	flakes	0.1	0.1
	waste	0.1	0.1
	peeled tubers	0.1	< 0.1
	chips	0.1	0.1
	wet peel	2.3	2.3
	culls	<1.0	< 1.0
	fries	0.1	0.1
	cooking water	0.1	0.1
	unpeeled, boiled	0.1	0.1
	unpeeled mwaved	< 0.33	< 0.33
Spinach	fresh leaves		
	cooked leaves	0.81, 1.0 1.3	1.0
	water	0.06, 0.09, 0.06	0.06
Tomato	fresh		
	washed	0.15, < 0.29, 0.17	0.17
	peeled	< 0.08, 0.1, < 0.08	< 0.08
	sun-dried	3.7, 3.8, 3.0	3.7
	canned	< 0.02, < 0.05, < 0.08	< 0.05
	juice	< 0.15, 0.19, < 0.17	< 0.17
	wet pomace	1.0, 2.2, 0.75	1.0
	dry pomace	3.2, 4.0, 1.7	3.2
	paste	0.62, 0.86, 1.0	0.86
	puree	0.23, 0.43, 0.25	0.25
Orange	fruit		
	juice	0.08, < 0.03, < 0.02	< 0.03
	wet pulp	0.46, 0.22, 0.24	0.24
	dry pulp	0.92, < 0.33, 0.47	< 0.33

RAC	Matrix	Cyantraniliprole + IN-J9Z38 [a]	
		Calculated processing factors	PF median
	meal	0.85, 0.22, 0.47	0.47
	molasses	0.69, 0.33, 0.59	0.59
	marmalade	< 0.15, < 0.03, < 0.06	< 0.06
	oil	8.5, 2.9, 8.8	8.5
	canned	< 0.02, < 0.03, < 0.06	< 0.03
Apple	fruit		
	washed	0.63, 0.58, 0.46	0.58
	puree	1.0, 0.88, 1.3	1.0
	canned	0.13, 0.04, 0.15	0.13
	frozen	1.5, 0.96, 0.62	0.96
	juice	0.38, 0.19, 0.31	0.31
	wet pomace	1.0, 1.2, 0.77	1.0
	dry pomace	3.9, 2.7, 2.0	2.7
	sauce	2.4, 2.2, 2.7	2.2
Plum	plum flesh		
	dried prunes	2.0, 1.6, 1.3	1.6
Cottonseed	seed		
	raw oil (solvent extr)	0.06, 0.04, 0.08	0.06
	refined oil (solvent extr)	0.04, 0.03, 0.05	0.04
	meal (solvent extr)	0.09, < 0.03, 0.05	0.05
	hulls	0.34, 0.37, < 0.27	0.34
	raw oil (cold press)	0.3, < 0.21	0.25
	refined oil (cold press)	0.04, 0.04	0.04
	meal (cold press)	0.11, 0.07	0.09
Olive	whole fruit		
	canned fruit	0.62, 0.58, 0.2	0.58
	olive flesh	1.7, 1.8, 1.5	1.7
	canned flesh	0.95, 0.96, 0.7	0.95
	raw oil	1.7, 1.2, 0.6	1.2
	refined oil	1.3, 0.81, 0.4	0.81
Grape	bunches		
	grapes	0.29, 0.57, 1.6	0.57
	stems	1.2, 2.3, 4.6	2.3
	must	0.79, 1.5, 1.6	1.5
	wet pomace	1.4, 3.9, 2.7	2.7
	lees	0.61, 1.9, 6.6	1.9
	dry pomace	2.7, 3.7, 2.4	2.7
	AF wine	0.54, 1.1, 1.0	1.0
	MF wine	0.52, 1.1, 1.0	1.0
	bottled wine	0.5, 1.2, 1.0	1.0
	juice	0.48, 0.52, 1.4	0.52
	raisins	0.48, 0.52, 2.3	0.52

[a] Each value represents a separate study where residues were above the LOQ in the RAC. The factor is the ratio of the combined cyantraniliprole plus IN-J9Z38 metabolite residues in the processed item divided by the residue of cyantraniliprole in the RAC.

RESIDUES IN ANIMAL COMMODITIES

Farm animal feeding studies

Dairy cow

In a dairy cow feeding study reported by Ward & Vance, 2011 [Ref: 27180], cyantraniliprole was administered orally in gelatine capsules to four groups of lactating Holstein cows (three cows/group) twice daily for 28 consecutive days. Dosing was made at target treatment levels of 3, 10, 30, and 100 ppm feed. Based on the average dietary intake and average dosing levels of 0.088, 0.276, 0.82 and 3.15 mg/kg body weight, the actual average dose levels were equivalent to 3.53, 11.7, 35.0, and 112 ppm feed. An additional three cows were dosed at 112 ppm feed to obtain depuration data. Body weights ranged from 457–730 kg and remained relatively constant throughout the study period.

Milk was collected twice daily and samples from afternoon sampling were combined with samples from the next morning. Milk samples from the depuration group were collected during the dosing period and up until sacrifice during the depuration phase. Except for the depuration group, cows were sacrificed within 24 hours after the last dose. One of the animals from the depuration group was sacrificed on Days 4, 10, and 15 after the last dose. Samples of liver, kidney, muscle, and fat were collected from all animals at sacrifice.

Samples were stored at about -80 °C for a maximum of 42 days. Samples were stored at about –80 °C for a maximum of 42 days before analysis, with the liver and kidney samples being extracted on the day of collection. Analysis for cyantraniliprole and seven metabolites was by HPLC/MS/MS method 18844, with an LOQ of 0.01 mg/kg for all analytes in all matrices. Average concurrent recovery rates were 75–103% (cyantraniliprole) and 71–112% (metabolites) in samples spiked with 0.01 and 0.1 mg/kg.

Residues reached a plateau in milk after about 5 days and were concentrated in the cream fraction by a factor of approximately 1.7. Residues in skim milk were approximately 60% of those in raw milk.

Residues in milk were dose-dependant and were predominantly cyantraniliprole and IN-N7B69. In the 3 ppm dose group, the maximum average residue of cyantraniliprole was 0.03 mg/kg and for IN-N7B6, 0.028 mg/kg. In the 10 ppm dose group, residues of cyantraniliprole and IN-N7B69 were 0.11 and 0.074 mg/kg, respectively. In the 30 ppm group, residues of cyantraniliprole, IN-N7B69, IN-J9Z38, and IN-MYX98 were 0.25, 0.17, 0.01, and 0.025 mg/kg, respectively. In the 100 ppm dose group, residues of cyantraniliprole, IN-N7B69, IN-J9Z38, and IN-MYX98 were 0.71, 0.28, 0.034, and 0.085 mg/kg, respectively. Residues of all other metabolites were < LOQ.

Residues in cream were comparable on Days 14 and 21. The predominant residues in cream were cyantraniliprole, IN-N7B69, and IN-J9Z38. In the 3 ppm dose group, analytes present above the LOQ were cyantraniliprole (0.066 mg/kg), IN-N7B69 (0.021 mg/kg) and IN-J9Z38 (0.013 mg/kg).

In the 10 ppm dose group the average residues in cream were: cyantraniliprole, 0.18 mg/kg; IN-N7B69, 0.051 mg/kg; and IN-J9Z38, 0.03 mg/kg. In the 30 ppm dose group the average residues of analytes were: cyantraniliprole, 0.55 mg/kg; IN-N7B69, 0.13 mg/kg; IN-J9Z38, 0.078 mg/kg; IN-MLA84, 0.024 mg/kg; and IN-MYX98, 0.022 mg/kg. In the 100 ppm dose group the average residues were: cyantraniliprole, 1.8 mg/kg; IN-N7B69, 0.22 mg/kg; IN-J9Z38, 0.31 mg/kg; IN-MYX98, 0.073 mg/kg; and IN-MLA84, 0.04 mg/kg. All other residues were < LOQ.

Residues in skim milk were comparable on Days 14 and 21. The predominant residues in cream were cyantraniliprole and IN-N7B69. In the 3 ppm dose group the average residues of analytes present above the LOQ were: cyantraniliprole, 0.017 mg/kg and IN-N7B69, 0.02 mg/kg.

In the 10 ppm dose group the average residues in skim milk were: cyantraniliprole, 0.044 mg/kg and IN-N7B69, 0.052 mg/kg. All other residues were < LOQ. In the 30 ppm dose group the average residues were: cyantraniliprole, 0.14 mg/kg; IN-N7B69, 0.13 mg/kg; and IN-MYX98,

0.021 mg/kg. In the 100 ppm dose group the average residues were: cyantraniliprole, 0.47 mg/kg; IN-N7B69, 0.20 mg/kg; and IN-YX98, 0.059 mg/kg. All other residues were < LOQ.

In tissues, the highest residues were found in liver, followed by kidney, fat, and muscle, with cyantraniliprole being the predominant residue in all tissues and was dose dependent.

In the 3 ppm dose group, average residues of analytes present above the LOQ in liver were cyantraniliprole, 0.054 mg/kg and IN-MLA84, 0.032 mg/kg. In the 10 ppm dose group, the average residues were: cyantraniliprole, 0.15 mg/kg and IN-MLA84, 0.075 mg/kg. In the 30 ppm dose group, the average residues were: cyantraniliprole, 0.46 mg/kg; IN-MLA84, 0.22 mg/kg; and IN-N7B69, 0.042 mg/kg. In the 100 ppm dose group, average residues were: cyantraniliprole, 1.7 mg/kg; IN-MLA84, 0.41 mg/kg; IN-N7B69, 0.075 mg/kg; IN-MYX98, 0.025 mg/kg; IN-J9Z38, 0.015 mg/kg; and IN-HGW87, 0.010 mg/kg. All other residues were < LOQ.

In the 3 ppm dose group, average residues of analytes present above the LOQ in kidney were cyantraniliprole, 0.023 mg/kg and IN-N7B69, 0.012 mg/kg. In the 10 ppm dose group, the average residues were: cyantraniliprole, 0.084 mg/kg; IN-N7B69, 0.031 mg/kg; and IN-MLA84, 0.013 mg/kg. In the 30 ppm dose group, the average residues were: cyantraniliprole, 0.20 mg/kg; IN-N7B69 0.071 mg/kg; IN-MLA84, 0.041 mg/kg; and IN-MYX98, 0.034 mg/kg. In the 100 ppm dose group, average residues were: cyantraniliprole, 0.73 mg/kg; IN-MYX98, 0.14 mg/kg; IN-N7B69, 0.12 mg/kg; IN-MLA84, 0.099 mg/kg; IN-J9Z38, 0.024 mg/kg; and IN-HGW87, 0.011 mg/kg. All other residues were < LOQ.

In the 3 ppm dose group, average residues of analytes present above the LOQ in fat were: cyantraniliprole, 0.014 mg/kg and IN-J9Z38, 0.010 mg/kg. In the 10 ppm dose group, the average residues were: cyantraniliprole, 0.042 mg/kg and IN-J9Z38, 0.023 mg/kg. In the 30 ppm dose group, the average residues were: cyantraniliprole, 0.12 mg/kg; IN-J9Z38, 0.082 mg/kg; and IN-N7B69, 0.01 mg/kg. In the 100 ppm dose group, the average residues were: cyantraniliprole, 0.51 mg/kg; IN-J9Z38, 0.38 mg/kg; and IN-N7B69, 0.02 mg/kg. All other residues were < LOQ.

In the 3 ppm dose group, there were no residues present in muscle above the LOQ. In the 10 ppm dose group, only cyantraniliprole was present above the LOQ (0.026 mg/kg). In the 30 ppm dose group, the average residues were: cyantraniliprole, 0.071 mg/kg and IN-J9Z38, 0.01 mg/kg. In the 100 ppm dose group, the average residues were: cyantraniliprole, 0.28 mg/kg and IN-J9Z38, 0.027 mg/kg. All other residues were < LOQ.

Following cessation of dosing residues declined rapidly. All residues were < LOQ in milk, liver and kidney within 10 days after the last dose, in muscle within 4 days, and in fat within 15 days of the last dose.

Table 163 Average residues of cyantraniliprole and metabolites in milk from cows dosed with cyantraniliprole for 28 days

Matrix	Analyte	Average residues of cyantraniliprole and metabolites (values in milk represent the highest single day value)							
		3 ppm [a]		10 ppm [a]		30 ppm [a]		100 ppm [a]	
		Mean residue (mg/kg)							
Milk	Cyantraniliprole	0.03		0.11		0.25		0.71	
	IN-HGW87	0.0001		0.001		0.004		0.007	
	IN-J9Z38	0.003		0.004		0.010		0.034	
	IN-JCZ38	0.0004		0.0004		0.0008		0.001	
	IN-K7H19	ND		ND		ND		0.001	
	IN-MLA84	0.004		0.005		0.006		0.007	
	IN-MYX98	0.003		0.009		0.025		0.085	
	IN-N7B69	0.028		0.074		0.17		0.28	
Study day		14	21	14	21	14	21	14	21
Cream	Cyantraniliprole	0.072	0.059	0.2	0.15	0.63	0.46	1.9	1.7
	IN-HGW87	0.0002	0.0002	0.0007	0.0007	0.002	0.002	0.008	0.009
	IN-J9Z38	0.014	0.011	0.032	0.027	0.085	0.066	0.37	0.31

Matrix	Analyte	Average residues of cyantraniliprole and metabolites (values in milk represent the highest single day value)							
		3 ppm [a]		10 ppm [a]		30 ppm [a]		100 ppm [a]	
		Mean residue (mg/kg)							
	IN-JCZ38	ND	ND	ND	ND	ND	0.00001	ND	0.00009
	IN-K7H19	ND	ND	ND	ND	ND	ND	ND	0.00001
	IN-MLA84	0.004	0.004	0.009	0.009	0.026	0.021	0.039	0.041
	IN-MYX98	0.002	0.002	0.007	0.006	0.020	0.023	0.066	0.079
	IN-N7B69	0.019	0.022	0.048	0.053	0.12	0.14	0.19	0.25
Skim milk	Cyantraniliprole	0.019	0.014	0.049	0.039	0.15	0.13	0.47	0.47
	IN-HGW87	0.00009	0.00005	0.0004	0.0004	0.001	0.001	0.004	0.003
	IN-J9Z38	ND	0.0002	0.0001	0.0004	0.001	0.002	0.005	0.008
	IN-JCZ38	0.000007	ND	0.00002	ND	0.00003	0.00002	0.030	0.00009
	IN-K7H19	ND	ND	ND	ND	ND	ND	ND	ND
	IN-MLA84	0.0003	0.0002	0.0007	0.0004	0.002	0.001	0.003	0.002
	IN-MYX98	0.002	0.002	0.007	0.006	0.022	0.020	0.066	0.051
	IN-N7B69	0.019	0.020	0.047	0.057	0.12	0.13	0.22	0.18

[a] Actual dose rates were 3.5 ppm, 11.7 ppm, 35 ppm and 112 ppm diet

Table 164 Residues of cyantraniliprole and metabolites in tissues from lactating cows dosed with cyantraniliprole for 28 days

Matrix	Analyte	Residues of cyantraniliprole and metabolites (mg/kg feed) in tissues							
		3 ppm [a]		10 ppm [a]		30 ppm [a]		100 ppm [a]	
		mean	max	mean	max	mean	max	mean	max
Muscle	Cyantraniliprole	0.008	0.011	0.026	0.037	0.071	0.092	0.28	0.33
	IN-HGW87	< 0.0005	< 0.0005	< 0.0005	< 0.0005	< 0.0005	< 0.0005	0.0015	0.0017
	IN-J9Z38	0.001	0.0013	0.002	0.0026	0.010	0.018	0.027	0.043
	IN-JCZ38	ND	ND	ND	ND	< 0.0005	0.0008	0.0008	0.0009
	IN-K7H19	ND	ND	ND	ND	ND	ND	ND	ND
	IN-MLA84	0.0007	0.0009	0.001	0.0014	0.002	0.0029	0.004	0.0045
	IN-MYX98	0.001	0.0012	0.002	0.002	0.003	0.0035	0.01	0.011
	IN-N7B69	0.001	0.0015	0.003	0.0037	0.005	0.0056	0.009	0.0095
Liver	Cyantraniliprole	0.054	0.066	0.15	0.16	0.46	0.6	1.7	2.1
	IN-HGW87	< 0.0005	< 0.0005	0.0006	0.0008	0.002	0.0027	0.01	0.01
	IN-J9Z38	< 0.0005	0.0013	0.002	0.0025	0.006	0.0093	0.015	0.026
	IN-JCZ38	< 0.0005	< 0.005	< 0.0005	0.005	0.001	0.0018	0.008	0.011
	IN-K7H19	ND	ND	ND	ND	< 0.0005	< 0.0005	< 0.0005	< 0.0005
	IN-MLA84	0.032	0.043	0.075	0.099	0.22	0.29	0.41	0.57
	IN-MYX98	0.0008	0.001	0.003	0.0034	0.007	0.0082	0.025	0.026
	IN-N7B69	0.008	0.01	0.021	0.024	0.042	0.046	0.076	0.079
Kidney	Cyantraniliprole	0.023	0.031	0.084	0.14	0.20	0.25	0.73	0.89
	IN-HGW87	< 0.0005	< 0.005	0.0005	0.0006	0.002	0.0028	0.011	0.012
	IN-J9Z38	0.0009	0.0013	0.002	0.003	0.009	0.013	0.024	0.031
	IN-JCZ38	ND	ND	ND	ND	< 0.0005	< 0.005	0.0005	0.0007
	IN-K7H19	ND	ND	ND	ND	ND	ND	ND	ND
	IN-MLA84	0.007	0.011	0.013	0.017	0.041	0.044	0.099	0.13
	IN-MYX98	0.003	0.0037	0.009	0.0094	0.034	0.039	0.14	0.15
	IN-N7B69	0.012	0.015	0.031	0.031	0.071	0.081	0.12	0.15
Fat	Cyantraniliprole	0.014	0.015	0.042	0.066	0.12	0.15	0.51	0.58
	IN-HGW87	ND	ND	< 0.0005	< 0.005	< 0.0005	< 0.005	0.001	0.0014
	IN-J9Z38	0.01	0.012	0.023	0.031	0.082	0.12	0.38	0.45
	IN-JCZ38	ND	ND	ND	ND	< 0.0005	0.0007	ND	ND
	IN-K7H19	ND	ND	ND	ND	ND	ND	ND	ND

Matrix	Analyte	Residues of cyantraniliprole and metabolites (mg/kg feed) in tissues							
		3 ppm [a]		10 ppm [a]		30 ppm [a]		100 ppm [a]	
		mean	max	mean	max	mean	max	mean	max
	IN-MLA84	0.001	0.0012	0.002	0.0022	0.004	0.0051	0.008	0.0087
	IN-MYX98	0.001	0.0012	0.002	0.0021	0.002	0.0024	0.005	0.007
	IN-N7B69	0.002	0.0027	0.006	0.0088	0.01	0.012	0.02	0.024

[a] Actual dose rates were 3.5 ppm, 11.7 ppm, 35 ppm and 112 ppm diet

Table 165 Residue depuration of cyantraniliprole and metabolites in tissues and milk from lactating cows dosed with 100 ppm cyantraniliprole in feed for 28 days

Days after last dose	Analyte	Mean residues of cyantraniliprole and metabolites following withdrawal of dose				
		Milk	Muscle	Liver	Kidney	Fat
4	cyantraniliprole	0.025	0.009	0.063	0.022	0.013
	IN-HGW87	0.0002	0.00007	ND	0.0004	0.00003
	IN-J9Z38	0.002	0.004	0.003	0.011	0.14
	IN-JCZ38	0.0004	ND	0.0002	ND	ND
	IN-K7H19	ND	ND	ND	ND	ND
	IN-MLA84	0.008	0.001	0.17	0.043	0.002
	IN-MYX98	0.004	0.001	0.001	0.004	0.001
	IN-N7B69	0.028	0.001	0.007	0.012	0.002
10	cyantraniliprole	0.002	0.0007	0.005	ND	ND
	IN-HGW87	ND	ND	ND	ND	ND
	IN-J9Z38	ND	0.0006	0.0006	0.001	0.02
	IN-JCZ38	ND	ND	ND	ND	ND
	IN-K7H19	ND	ND	ND	ND	ND
	IN-MLA84	0.00009	ND	0.006	0.0008	0.0005
	IN-MYX98	ND	ND	ND	ND	ND
	IN-N7B69	0.0009	ND	ND	ND	ND
15	cyantraniliprole	0.003	0.0007	0.007	0.004	ND
	IN-HGW87	ND	ND	ND	ND	ND
	IN-J9Z38	ND	ND	0.0005	0.0003	0.004
	IN-JCZ38	ND	ND	ND	ND	ND
	IN-K7H19	ND	ND	ND	ND	ND
	IN-MLA84	0.00009	ND	0.0003	0.0003	ND
	IN-MYX98	ND	ND	0.003	ND	ND
	IN-N7B69	0.002	ND	ND	0.00006	ND

Poultry

In a poultry feeding study reported by Roberts & Ward, 2011 [Ref: DP-27181], cyantraniliprole was administered orally in gelatine capsules to three groups of hens (3 or 4 hens/sub-group with three groups/treatment) daily for 28 consecutive days. Dosing was made at treatment levels of 3, 10, and 30 ppm feed based on the animal diet on a dry weight basis, equivalent to 0.24, 0.86, and 2.34 mg/kg body weight. An additional group (three sub-groups) was dosed at 30 ppm feed (2.37 mg/kg body weight) to obtain depuration data. Body weights ranged from 1.3–1.96 kg and remained relatively constant throughout the study period.

Eggs were collected twice daily and samples from afternoon sampling were combined with samples from the next morning. Eggs from the depuration group were collected during the dosing period and up until sacrifice during the depuration phase. Except for the depuration group, hens were sacrificed within 6 hours after the last dose. Animals from the relevant sub-group from the depuration group were sacrificed on Days 5, 9, and 14 after the last dose. Samples of liver, muscle, and skin with

fat were collected from all animals at sacrifice and were pooled in approximately equal quantities according to sub-group.

Samples were stored at about -80 °C for a maximum of 97 days before analysis, with the liver samples being extracted on the day of collection. Analysis for cyantraniliprole and seven metabolites was by HPLC/MS/MS method 18844, with an LOQ of 0.01 mg/kg for all analytes in all matrices. Average concurrent recovery rates were 95–125% (cyantraniliprole) and 67–126% (metabolites) in samples spiked with 0.01 and 0.1 mg/kg.

Residues in eggs were dose dependant and were predominantly cyantraniliprole and IN-J9Z38. In the 3 ppm dose group, the maximum average residue of cyantraniliprole was 0.08 mg/kg and for IN-J9Z38, 0.04 mg/kg. Residues of IN-MLA84 and IN-MYX98 were also found at 0.016 and 0.014 mg/kg, respectively. In the 10 mg/kg dose group, residues of cyantraniliprole and IN-N7B69 were 0.17 and 0.077 mg/kg, respectively. Residues of IN-MLA84 and IN-MYX98 also exceeded the LOQ (0.038 and 0.035 mg/kg, respectively). In the 30 mg/kg group, residues of cyantraniliprole, IN-J9Z38, IN-MLA84, IN-MYX98, and IN-HGW87 were 0.80, 0.41, 0.12, 0.10, and 0.016 mg/kg, respectively. Residues of all other metabolites were < LOQ.

The predominant residues in egg whites were cyantraniliprole (0.08 mg/kg, 0.17 mg/kg and 0.64 mg/kg) and IN-J9Z38 (0.04 mg/kg, 0.07 mg/kg and 0.27 mg/kg) in the 3 ppm, 10 ppm and 30 ppm dose groups respectively. Other metabolites present were IN-MYX98 and IN-MLA84, at levels less than 0.02 mg/kg (3 ppm dose group), less than 0.04 mg/kg (10 ppm dose group) and less than 0.1 mg/kg in the 10 ppm dose group. All other residues were < LOQ.

The predominant residues in yolks were also cyantraniliprole and IN-J9Z38. In the 3 mg/kg dose group only cyantraniliprole was present above the LOQ (0.015 mg/kg). In the 10 mg/kg dose group the average cyantraniliprole residues were 0.03 mg/kg and were 0.1 mg/kg in the 30 ppm dose group. Residues of IN-J9Z38, IN-MYX98 and IN-MLA84 were less than 0.02 mg/kg in the 10 ppm dose group and less than 0.06 mg/kg in the 30 ppm dose group. All other residues were < LOQ.

In tissues, the highest residues were found in liver with cyantraniliprole, IN-MYX98 and IN-MLA84 being the predominant residues in all tissues. Residues of cyantraniliprole were dose dependant.

In liver, the predominant residue was IN-MYX98, present at average levels of 0.03 mg/kg (3 ppm dose group), 0.07 mg/kg (10 ppm dose group) and 0.19 mg/kg (30 ppm dose group). Average cyantraniliprole residues in these dose groups were 0.017, 0.04 and 0.13 mg/kg respectively and IN-MLA84 was also found at similar levels (0.015, 0.04 and 0.1 mg/kg) in the respective dose groups. Other metabolites found in the 10 ppm and 30 ppm dose groups were IN-HGW87 (0.03 and 0.06 mg/kg respectively) and IN-N7B69 (0.013 and 0.05 mg/kg respectively). All other residues were < LOQ.

In skin with fat, no residues were found above the LOQ, with cyantraniliprole (0.033 mg/kg) and IN-MYX98 (0.015 mg/kg) reported in the 10 mg/kg dose group. In the 30 mg/kg dose group, the average cyantraniliprole residues were 0.08 mg/kg with IN-MYX98, IN-J9Z38 and IN-HGW87 found at levels less than 0.03 mg/kg. All other residues were < LOQ.

In muscle, the only residues found above the LOQ were IN-MYX98, found at 0.012 mg/kg in the 10 ppm dose group and cyantraniliprole (0.025 mg/kg), IN-MYX98 (0.014 mg/kg and IN-HGW87 (0.011 mg/kg) in the 30 ppm dose group. All other residues were < LOQ.

Following cessation of dosing residues rapidly declined, with residues in eggs, muscle and skin with fat all being below the LOQ within 5 days after the last dose. Residues in liver were also below the LOQ after 9 days depuration (first sample taken).

Table 166 Average residues of cyantraniliprole and metabolites in eggs from hems dosed with cyantraniliprole for 28 days

Matrix	Analyte	Average residues of cyantraniliprole and metabolites (mg/kg) (values in eggs represent the highest single day value)					
		3 ppm dose group		10 ppm dose group		30 ppm dose group	
		Mean residue (mg/kg)					
Eggs	cyantraniliprole	0.082		0.17		0.80	
	IN-HGW87	0.0053		0.0069		0.016	
	IN-J9Z38	0.039		0.077		0.41	
	IN-JCZ38	< 0.002		< 0.002		< 0.002	
	IN-K7H19	< 0.002		ND		ND	
	IN-MLA84	0.016		0.038		0.12	
	IN-MYX98	0.014		0.035		0.10	
	IN-N7B69	< 0.002		< 0.002		0.0029	
Study day		14	21	14	21	14	21
Egg whites	cyantraniliprole	0.098	0.059	0.20	0.14	0.68	0.60
	IN-HGW87	< 0.002	0.0020	0.0037	0.0038	0.010	0.0097
	IN-J9Z38	0.045	0.026	0.078	0.066	0.27	0.27
	IN-JCZ38	ND	ND	ND	ND	ND	ND
	IN-K7H19	ND	ND	ND	ND	ND	ND
	IN-MLA84	0.015	0.015	0.034	0.033	0.092	0.093
	IN-MYX98	0.017	0.014	0.037	0.030	0.10	0.089
	IN-N7B69	< 0.002	0.0023	< 0.002	< 0.002	0.0038	0.0036
Egg yolks	cyantraniliprole	0.017	0.012	0.034	0.023	0.090	0.11
	IN-HGW87	< 0.002	< 0.002	0.0040	0.0034	0.0099	0.0081
	IN-J9Z38	0.0081	0.0056	0.018	0.014	0.053	0.062
	IN-JCZ38	ND	ND	ND	ND	ND	ND
	IN-K7H19	ND	ND	ND	ND	ND	ND
	IN-MLA84	0.0064	0.0077	0.017	0.016	0.039	0.046
	IN-MYX98	0.0086	0.0059	0.017	0.012	0.041	0.039
	IN-N7B69	< 0.002	< 0.002	< 0.002	< 0.002	< 0.002	< 0.002

Table 167 Residues of cyantraniliprole and metabolites in poultry tissues

Matrix	Analyte	Residues of cyantraniliprole and metabolites (mg/kg)					
		3 ppm dose group		10 ppm dose group		30 ppm dose group	
		mean	max	mean	max	mean	max
Muscle	cyantraniliprole	0.0034	0.0055	0.0093	0.015	0.025	0.05
	IN-HGW87	< 0.002	< 0.002	0.0052	0.0064	0.011	0.015
	IN-J9Z38	ND	ND	ND	ND	ND	ND
	IN-JCZ38	ND	ND	ND	ND	ND	ND
	IN-K7H19	ND	ND	ND	ND	ND	ND
	IN-MLA84	< 0.002	< 0.002	< 0.002	< 0.002	< 0.002	0.002
	IN-MYX98	0.0041	0.0045	0.012	0.013	0.014	0.022
	IN-N7B69	< 0.002	< 0.002	< 0.002	< 0.002	0.0026	0.0037
Liver	cyantraniliprole	0.017	0.03	0.041	0.064	0.13	0.24
	IN-HGW87	0.0076	0.009	0.028	0.036	0.059	0.083
	IN-J9Z38	< 0.002	< 0.002	< 0.002	< 0.002	< 0.002	0.0034
	IN-JCZ38	0.0030	0.0043	0.0094	0.011	0.0060	0.0074
	IN-K7H19	ND	ND	ND	ND	ND	ND
	IN-MLA84	0.015	0.022	0.043	0.048	0.096	0.099
	IN-MYX98	0.023	0.034	0.068	0.096	0.19	0.32
	IN-N7B69	0.0067	0.012	0.013	0.017	0.045	0.072

Matrix	Analyte	Residues of cyantraniliprole and metabolites (mg/kg)					
		3 ppm dose group		10 ppm dose group		30 ppm dose group	
		mean	max	mean	max	mean	max
Skin with fat	cyantraniliprole	0.0093	0.014	0.033	0.058	0.080	0.16
	IN-HGW87	< 0.002	0.002	0.0078	0.01	0.015	0.021
	IN-J9Z38	0.0029	0.0033	0.0064	0.0079	0.018	0.023
	IN-JCZ38	ND	ND	ND	ND	ND	ND
	IN-K7H19	ND	ND	ND	ND	ND	ND
	IN-MLA84	< 0.002	< 0.002	0.0034	0.0037	0.0080	0.009
	IN-MYX98	0.0037	0.0046	0.015	0.026	0.027	0.049
	IN-N7B69	< 0.002	< 0.002	< 0.002	0.002	0.0030	0.005

Table 168 Residue depuration of cyantraniliprole and metabolites in eggs from hens dosed with 30 ppm cyantraniliprole for 28 days

Analyte	Residues of cyantraniliprole and metabolites in eggs following withdrawal of dose					
	Mean residue (mg/kg)					
	Study day (days post last dose)					
	29 (1)	31 (3)	33 (5)	35 (7)	38 (10)	40 (12)
cyantraniliprole	0.22	0.010	0.0040	< 0.002	< 0.002	< 0.002
IN-HGW87	0.00086	0.0030	< 0.002	< 0.002	ND	ND
IN-J9Z38	0.12	0.0065	0.0024	< 0.002	ND	ND
IN-JCZ38	ND	ND	ND	ND	ND	ND
IN-K7H19	ND	ND	ND	ND	ND	ND
IN-MLA84	0.050	0.0068	< 0.002	ND	ND	ND
IN-MYX98	0.041	0.0079	0.0033	< 0.002	< 0.002	< 0.002
IN-N7B69	< 0.002	< 0.002	ND	ND	ND	ND

Table 169 Residue depuration of cyantraniliprole and metabolites in tissues from hens dosed with 30 ppm cyantraniliprole for 28 days

Study day (days post last dose)	Analyte	Residues of cyantraniliprole and metabolites following withdrawal of dose		
		Mean residue (mg/kg)		
		Muscle	Liver	Skin with fat
33 (5)	cyantraniliprole	< 0.002	-	0.0030
	IN-HGW87	ND	-	ND
	IN-J9Z38	ND	-	ND
	IN-JCZ38	ND	-	ND
	IN-K7H19	ND	-	ND
	IN-MLA84	ND	-	< 0.002
	IN-MYX98	ND	-	< 0.002
	IN-N7B69	ND	-	ND
37 (9)	cyantraniliprole	< 0.002	< 0.002	0.0057
	IN-HGW87	ND	ND	< 0.002
	IN-J9Z38	ND	ND	< 0.002
	IN-JCZ38	ND	ND	ND
	IN-K7H19	ND	ND	ND
	IN-MLA84	ND	< 0.002	< 0.002
	IN-MYX98	ND	ND	< 0.002
	IN-N7B69	ND	ND	ND
42 (14)	cyantraniliprole	< 0.002	< 0.002	0.0044
	IN-HGW87	ND	ND	ND
	IN-J9Z38	ND	ND	< 0.002
	IN-JCZ38	ND	ND	ND

Study day (days post last dose)	Analyte	Residues of cyantraniliprole and metabolites following withdrawal of dose		
		Mean residue (mg/kg)		
		Muscle	Liver	Skin with fat
	IN-K7H19	ND	ND	ND
	IN-MLA84	ND	< 0.002	ND
	IN-MYX98	ND	ND	ND
	IN-N7B69	ND	ND	ND

NATIONAL RESIDUE DEFINITIONS

Table 170 Cyantraniliprole: National residue definitions for MRL-compliance and dietary intake estimation

Country	MRL-compliance	Dietary intake estimation		
	All commodities	Plant commodities (unprocessed)	Plant commodities (processed)	Animal commodities
Canada	cyantraniliprole	cyantraniliprole	cyantraniliprole + IN-J9Z38	
New Zealand	cyantraniliprole	cyantraniliprole	cyantraniliprole	Not established [a]
Global Joint Review (proposed)	cyantraniliprole	cyantraniliprole	cyantraniliprole + IN-J9Z38	cyantraniliprole + IN-J9Z38 + IN-MLA84 +IN-MYX98 + IN-N7B69

[a] A residue definition has not yet been established as the current authorized GAPs are not expected to result in detectable residues in animal products.

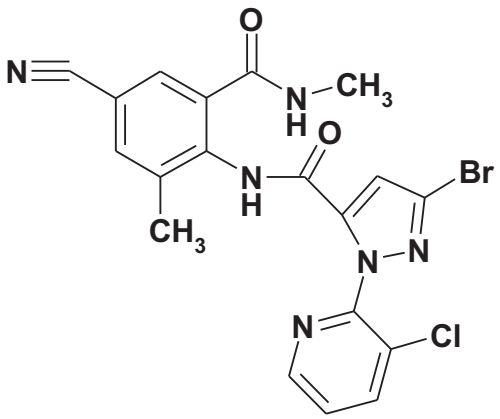
APPRAISAL

Cyantraniliprole is a diamide insecticide with a mode of action (ryanodine receptor activation) similar to that of chlorantraniliprole and flubendiamide with root systemic and translaminar activity against the larval stages of lepidopteran insects; and also on thrips, aphids, and some other chewing and sucking insects.

It was scheduled by the Forty-fourth Session of the CCPR (REP12/PR) as a new compound for consideration by the 2013 JMPR. The manufacturer submitted studies on metabolism, analytical methods, supervised field trials, processing, freezer storage stability, environmental fate in soil and rotational crop residues.

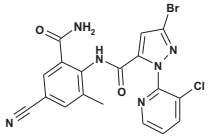
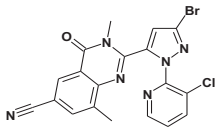
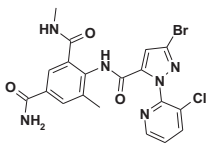
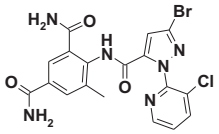
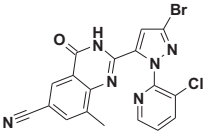
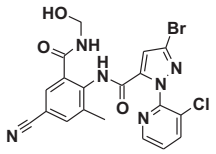
Authorisations exist for the use of cyantraniliprole in Canada, Columbia, Malaysia, New Zealand, Vietnam and in a regional grouping of countries in West Africa (CLISS).

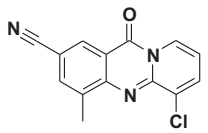
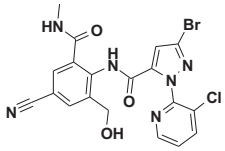
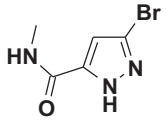
Cyantraniliprole is 3-bromo-1-(3-chloro-2-pyridyl)-4′ -cyano-2′ -methyl-6′ -(methylcarbamoyl) pyrazole -5-carboxanilide. It is relatively insoluble in water (12 mg/L at pH 7, 6 mg/L at pH 9) and hydrolyses under alkaline conditions and at higher temperatures (above 25 °C), the major hydrolysis product being IN-J9Z38. It is not volatile (1.2×10^{-15} Pa at 20 °C), has a log P_{OW} of 1.9, its solubility in organic sovents ranges from < 1 g/L (octanol, xylene) to 5–7 g/L (methanol, dichloromethane, acetone) and is rapidly degraded by photolysis.



Cyantraniliprole (DPX-HGW86)
(MW 473.7)

The following abbreviations are used for the metabolites discussed below:

IN-HGW87		N-[2-(Aminocarbonyl)-4-cyano-6-methylphenyl]-3-bromo-1-(3-chloro-2-pyridinyl)-1*H*-pyrazole-5-carboxamide	
IN-J9Z38		2-[3-Bromo-1-(3-chloro-2-pyridinyl)-1*H*-pyrazol-5-yl]-3,4-dihydro-3,8-dimethyl-4-oxo-6-quinazolinecarbonitrile	MW 455.7
IN-JCZ38		4-[[[3-Bromo-1-(3-chloro-2-pyridinyl)-1*H*-pyrazol-5-yl]carbonyl]amino]-N′3′,5-dimethyl-1,3-benzenedicarboxamide	MW 491.7
IN-K7H19		4-[[[3-Bromo-1-(3-chloro-2-pyridinyl)-1*H*-pyrazol-5-yl]carbonyl]amino]-5-methyl-1,3-benzenedicarboxamide	MW 477.7
IN-MLA84		2-[3-Bromo-1-(3-chloro-2-pyridinyl)-1*H*-pyrazol-5-yl]-1,4-dihydro-8-methyl-4-oxo-6-quinazolinecarbonitrile	MW 441.7
IN-MYX98		3-Bromo-1-(3-chloro-2-pyridinyl)-N-[4-cyano-2[[(hydroxymethyl)amino]carbonyl]-6-methylphenyl]-1*H*-pyrazole-5-carboxamide	MW 489.7

IN-N5M09		6-Chloro-4-methyl-11-oxo-11*H*-pyrido[2,1-*b*]quinazoline-2-carbonitrile	MW 441.6
IN-N7B69		3-Bromo-1-(3-chloro-2-pyridinyl)-N-[4-cyano-2-(hydroxymethyl)-6-[(methylamino)carbonyl]phenyl]-1*H*-pyrazole-5-carboxamide	MW 489.7
IN-F6L99		3-Bromo-N-methyl-1H-pyrazole-5-carboxamide	MW 204

Animal metabolism

The Meeting received information on the metabolism of radiolabelled cyantraniliprole, separately ^{14}C-labelled at the 4-cyano (CN) and the pyrazole carbonyl (PC) groups, in rats, lactating goats and laying hens.

The WHO panel of the 2013 JMPR concluded that in rats, the majority of the dose was excreted within 24 to 48 hours, with about 1–5.5% TRR being recovered in tissues and that tissue elimination half-lives ranged from 2.6 days (fat) to about 6 days in whole blood.

The metabolic pathway was primarily through hydroxylation (to form IN-N7B69 and IN-MYX98), with IN-N7B69 being further metabolized to a glucuronide conjugate. Cyantraniliprole undergoes ring closure to generate IN-J9Z38 which is then in turn hydroxylated to form IN-NBC94, its carboxylic acid, and its glucuronide conjugate. IN-MYX98 is also metabolized to the closed-ring metabolite IN-MLA84, which, like IN-NBC94, is further oxidized to a hydroxylated metabolite, a carboxylic acid, and the glucuronide of the hydroxyl metabolite. Further, the hydroxylated metabolite IN-MYX98 can be N-dealkylated to form IN-HGW87 as well as being hydroxylated a second time to form bis-hydroxy-cyantraniliprole. Cyantraniliprole can also be hydroxylated on the pyridine ring, followed by a ring closure analogous to the conversion of cyantraniliprole to IN-J9Z38.

Lactating goats were orally dosed with [CN-^{14}C]- or [PC-^{14}C]-cyantraniliprole at doses equivalent to approximately 13 ppm in the feed for 7 consecutive days and sacrificed 23 hours after the last dose.

The majority of the administered dose was recovered in excreta (84–88% in faeces, 7% in urine). About 1–2% (0.08–0.15 mg/kg) of the applied radioactivity was retained in milk, with 0.5 mg/kg (0.3% AR) found in liver and < 0.01% AR in other tissues. Solvent extraction was able to retrieve 99% TRR from milk, 90–98% TRR from fat, 61–81% TRR from muscle and 63–79% TRR from kidney. Solvent extractable TRR in the liver were lower (54–60% TRR) but an additional 21–27% TRR were recovered following digestion of the post extracted solids (PES) with protease.

In milk, TRR reached a plateau of 0.09 and 0.18 mg/kg after 2–3 days in the CN and PC label studies respectively. Cyantraniliprole was the major residue component, making up 40–50% (0.03–0.07 mg/kg) of the radiochemical label. Metabolite IN-MYX98 was also present at 15–18% TRR (0.01–0.03 mg/kg). Other metabolites (except IN-N7B69 at 11% TRR in the CN-label study) were found each < 0.01 mg/kg and less than 10% TRR.

In liver, TRR were 0.43–0.5 mg/kg. Cyantraniliprole (0.07–0.14 mg/kg) made up about 17–23% of the TRR and in kidney (TRR 0.14–0.21 mg/kg) with the major residue was also cyantraniliprole, accounting for 13–19% TRR (0.02–0.04 mg/kg).

In muscle, TRR were 0.03–0.04 mg/kg with the significant residue being cyantraniliprole, found at about 30% TRR and 0.01 mg/kg in the CN-label study and 15% TRR, 0.006 mg/kg in the PC-label study. The IN-MYX98 metabolite also accounted for 33% TRR (0.01 mg/kg) in the PC-label study.

TRR in fat were 0.05 and 0.12 mg/kg in the CN-label and PC-label studies respectively, with consistent results in omental, subcutaneous and renal fat. Cyantraniliprole was the major residue, averaging 31–42% TRR (0.01–0.025 mg/kg) with the IN-J9Z38 metabolite also accounted for 24–27% TRR (0.01–0.03 mg/kg).

Laying hens were orally dosed with [CN-^{14}C]- or [PC-^{14}C]-cyantraniliprole at doses equivalent to approximately 11 ppm in the feed for 14 consecutive days and sacrificed 23 hours after the last dose.

The majority of the administered dose was excreted, with 0.4–0.5% (0.20–0.26 mg/kg) remaining in egg whites, 0.07% (0.09 mg/kg) in yolks. Radioactivity in liver (0.14–0.2 mg/kg) accounted for 0.3–0.4% of the applied dose with muscle, abdominal fat and skin with fat each containing ≤ 0.1% AR (< 0.01 mg/kg).

In eggs, %TRR reached a plateau of about 0.1 mg/kg in yolks after 5–7 days. The %TRRs in egg white increased to 0.26–0.56 mg/kg over the first 2 days and decreased to a steady state of about 0.2–0.24 mg/kg after 7 days.

Solvent extraction was able to retrieve 79–99% TRR from eggs, 63–120% TRR from fat, 100% TRR from muscle and 53–72% TRR from skin with fat. Solvent extractable TRR in the liver were lower (17–23% TRR) but an additional 38% TRR were recovered following digestion of the post extracted solids (PES) with protease.

In eggs, cyantraniliprole was the major residue component, making up 33–42% TRR (0.09 mg/kg) in egg whites and 9–10% TRR (< 0.01 mg/kg) in yolks. Metabolite IN-J9Z38 made up about 17–29% TRR (0.03–0.08 mg/kg) in egg whites and 7–13% TRR (0.006–0.011 mg/kg in yolks. IN-MLA84 was also present in egg whites at about 18–19% TRR (0.04–0.05 mg/kg) and in yolks at about 12–17% TRR (0.01–0.015 mg/kg

In liver, TRR were 0.14–0.17 mg/kg. Cyantraniliprole was not found in any samples and while metabolites IN-JCZ38, IN-K5A78, IN-K5A79, IN-K7H19, IN-MLA84, IN-MYX98 and IN-N7B69 were identified, these were each present at <4% TRR (< 0.01 mg/kg).

In muscle, abdominal fat and skin with fat, no further analysis was conducted because of the low TRR (< 0.004 mg/kg) present.

In summary, the metabolism of cyantraniliprole in lactating goats (ruminant) and laying hen was consistent with that in the rat. Cyantraniliprole residues were rapidly eliminated in the excreta (94–100% of the dose) in goats and laying hens, with less than 1% of the total administered dose remaining in tissues and eggs and 1–2% found in milk). Cyantraniliprole was the predominant residue, with IN-N7B69 (milk), IN-MYX98 (muscle and milk), IN-J9Z38 (fat and eggs) and IN-MLA84 (eggs) being the principal metabolites present at more than 10% TRR or > 0.01 mg/kg.

Plant metabolism

The Meeting received plant metabolism studies on cotton, lettuce, tomato and rice seedlings following foliar and soil treatments with [^{14}C]-cyantraniliprole. A 1:1 (μCi/μCi ratio) mixture of [CN-^{14}C]-cyantraniliprole and [PC-^{14}C]-cyantraniliprole was used in the foliar treatments and separate treatments of the two radiolabels were applied as soil treatments.

Cotton

In cotton plants, treated three times with the equivalent of 0.15 kg ai/ha per application as foliar sprays, 7 days apart from 3 weeks after emergence, TRR in leaves immediately after the first treatment were 2.7 and 7.9 mg/kg immediately after the last application and after 13 days had

decreased to 0.43 mg/kg. At harvest (124 days after the last application), TRRs in cotton gin by-products, lint and undelinted seed were 0.13, 0.01, and < 0.01 mg/kg, respectively.

Surface washing removed 56–70% TRR from immature leaves with a further 27–33% extracted into acidified aqueous acetone.

Cyantraniliprole was the major residue in leaves, decreasing from 70% TRR immediately after the first application to 20% TRR seven days later and accounted for 37% TRR (0.19 mg/kg) and 27% TRR (0.12 mg/kg), respectively, 7 and 13 days after the final application.

IN-NXX70, a photodegradate of IN-J9Z38, found predominately in the surface wash, accounted for 22% TRR seven days after the first application but decreased to 1% TRR in leaves sampled 7 days after the third application.

Surface washing of cotton gin by-products removed about 19% TRR (0.03 mg/kg) with a further 65% TRR (0.07 mg/kg) being extracted into acetone or with more aggressive extraction methods. Cyantraniliprole was the predominant residue, accounting for 34% TRR (0.04 mg/kg).

In plants from soil treatments where three applications of 0.15 kg ai/ha cyantraniliprole (SC formulation) were made to wetted soil at 7 day intervals from 7 weeks after emergence, TRRs in leaves from immature plants sampled up to 14 days after the last application were all ≤ 0.005 mg/kg. At maturity, 125 days after the last application, residues in lint and undelinted seed were < 0.001 mg/kg and TRR values in cotton gin by-products were 0.1 mg/kg (CN-label) and 0.02 mg/kg (PC-label).

Cyantraniliprole was the only significant residue in gin by-products, making up 26–47% TRR (0.01–0.03 mg/kg).

Lettuce

In lettuce plants, treated three times with the equivalent of 0.15 kg ai/ha per application as foliar sprays, 7 days apart from 3 weeks after emergence, TRR in leaves immediately after the first treatment was 11 mg/kg, 10 mg/kg immediately after the second application and about 8 mg/kg just after the last application. Seven days after the last application, TRRs had decreased to about 2 mg/kg and were 0.43 mg/kg at maturity, 32 days after the last application. Surface residues decreased from about 91% TRR immediately after the first application and 32 days after the last application surface residues were 13% TRR.

Cyantraniliprole was the major residue in leaves, decreasing from about 98% TRR immediately after the first application to 50% TRR in mature leaves, 32 days after the last application. The IN-J9Z38 metabolite, present in mature leaves at about 23% TRR (0.01 mg/kg) was only metabolite accounting for more than 5% TRR.

In plants from soil treatment where three applications of 0.15 kg ai/ha cyantraniliprole (SC formulation) were made to wetted soil at 7 day intervals from 7 weeks after emergence, TRRs (CN-label) in leaves from immature plants declined from 0.14 mg/kg immediately after the first application to about 0.05 mg/kg (7 days after the 2nd and 3rd applications) and were about 0.01 mg/kg at maturity. TRRs following the PC-label soil treatment were ≤ 0.06 mg/kg in immature leaves and at crop maturity.

Cyantraniliprole was the major radioactive component present in leaves, up to 77–84% TRR in young leaves and 37% TRR (0.004 mg/kg) and 69% TRR (0.04 mg/kg) in mature leaves for the CN-label and PC-labels respectively.

Tomatoes

In tomato plants, treated three times with the equivalent of 0.15 kg ai/ha per application as foliar sprays, 7 days apart from 3 weeks after emergence, TRR in leaves immediately after the first treatment were 2.5 mg/kg, 8.5 mg/kg immediately after the second application and 7.6 mg/kg just after the last application. Seven days after the last application, TRRs had decreased to about

2.2 mg/kg and were 1.3 mg/kg 14 days after the last application. Residues in fruit and leaves at harvest (132 days after the last treatment were < 0.01 mg/kg. The majority of the TRR (66–85%) were found in the surface wash with 15–34% TRR present in the extract.

Cyantraniliprole was the major residue in leaves, decreasing from about 95% TRR immediately after the first application to 61% TRR seven days later and accounted for 64 and 43% TRR respectively, 7 and 14 days after the last application. In leaves taken at maturity, residues in the surface wash were < 0.01 mg/kg and 0.01 mg/kg in the tissue extracts.

Concentrations of the unresolved radioactivity corresponding to both IN-MLA84 and IN-NXX70, (mostly in the surface wash) reached 11.5% TRR 7 days after the first application, decreasing to 4.4% TRR thereafter.

In plants from soil treatments where three applications of 0.15 kg ai/ha cyantraniliprole (SC formulation) were made to wetted soil in pots at 7 day intervals from 7 weeks after emergence, TRRs reached a maximum of 0.03 mg/kg in immature leaves 7 days after the last application and were < 0.01 mg/kg in leaves and 0.001 mg/kg in fruit at harvest (125 days after the last application).

Cyantraniliprole was the major radioactive component in leaves 7–14 days after the last application, ranging from 22–26% TRR (< 0.01 mg/kg).

Rice

Rice seedlings were treated with three foliar applications of 0.15 kg ai/ha at the 3–4 leaf stage and 7 and 14 days later and grown under flooded conditions (pots immersed in about 3 cm water) from 2 days after the initial treatment until 2–3 days before harvest.

TRRs in foliage were 2.1 mg/kg immediately after the first application, decreasing to 0.38 mg/kg after 7 days. Seven and 14 days after the last application, TRRS in leaves were 1.6 mg/kg and 1.2 mg/kg respectively. At harvest, 140 days after the last application, TRRs in straw were 0.45 mg/kg and 0.02 mg/kg in grain. In roots, TRRs increased from 0.24 mg/kg seven days after the first application to 0.68 mg/kg seven days after the last application and were 0.45 mg/kg at harvest. In immature leaves, 7 days after the last application, the surface wash contained about 75% TRR, reducing to 47% TRR in leaves sampled 14 days after the last treatment.

Cyantraniliprole was the major residue in immature leaves, making up 76–81% TRR (about 1.0 mg/kg) in samples taken 7 and 14 days after the last application. IN-J9Z38 was the predominant metabolite found at 0.6% TRR immediately after the first application and increasing to 11% TRR 14 days after the last application.

In straw, cyantraniliprole was the major component, accounting for 24.4% TRR (0.11 mg/kg) and cyantraniliprole was also the predominant residue in grain, accounting for 21% TRR (0.005 mg/kg).

Rice seedlings were also treated with a single soil application of 0.3 kg ai/ha (as surface-applied granules) at the 3–4 leaf stage and the plants were grown under flooded conditions (pots immersed in about 3 cm water) from 2 days after the initial treatment until 2–3 days before harvest, 175 days after treatment.

TRRs in foliage increased from 0.08 mg/kg (7 days after treatment) to 0.15 mg/kg (14 days after treatment) and reached 0.4 mg/kg after 56 days. Residues in roots were about 0.3 mg/kg after 56 days and at harvest (175 days after treatment). At harvest, TRRs were 0.28–0.3 mg/kg in straw and 0.01–0.03 mg/kg in grain.

Cyantraniliprole was the major residue in leaves sampled 56 days after treatment (49–57% TRR and about 0.2 mg/kg). The IN-J9Z38 metabolite was found in these samples at 16–22% TRR (about 0.08 mg/kg).

The major residue in straw was also cyantraniliprole (42–45% TRR, 0.13 mg/kg), with IN-J9Z38 (14–18% TRR) being the only significant metabolite, found at 14–18% TRR and in grain, cyantraniliprole accounted for 46–63% TRR (0.007–0.014 mg/kg).

In summary, cyantraniliprole was the predominant residue in most crop fractions at various sampling points up to crop maturity. Metabolites identified in foliar treated samples (with the exception of the photodegradate IN-NXX70) were also found in samples from plants treated with a soil drench application, indicating that the main metabolic pathways were similar. Overall, total radioactive residues were greater following foliar treatment than following soil application. A similar profile was observed in all studies.

The metabolite IN-J9Z38 was present at levels above 10% TRR only in rice foliage and lettuce (after foliar applications) and also in rice foliage and straw after soil treatment. Where present, residues were significantly lower (10–50%) than the levels of cyantraniliprole.

The metabolism of cyantraniliprole in plants was generally consistent with those in animals, except for the minor plant photodegradation pathway leading to the formation of IN-NXX70 and IN-QKV54).

Environmental fate

The Meeting received information on the environmental fate and behaviour of cyantraniliprole, including aerobic degradation in soil, photolysis on the soil surface, field soil dissipation, hydrolytic stability, soil and water/sediment degradation and confined and field rotational crop studies. Separate treatments of [CN-^{14}C]-cyantraniliprole and [PC-^{14}C]-cyantraniliprole were used in the confined studies.

Hydrolysis

Hydrolysis of cyantraniliprole was pH and temperature dependant. The rate of hydrolysis was significantly higher at high pH and temperature. The half lives at 15 °C decreased from 362 days (pH 4) to 126 days (pH 7) and was about 3 days at pH 9. A similar pattern was observed at the higher temperature of 35 °C, with the respective half-lives being 55 days, 7.5 days and < 1 day at pH 9. Under environmental conditions (pH 7, 25 °C) the half-life for cyantraniliprole was 212 days.

At all pH's, the predominant hydrolysis product was the cyclisation product IN-J9Z38 which accounted for about 28% AR (pH 4), 89% AR (pH 7) and 98% AR in the pH 9 samples.

Photolysis

In aqueous solutions, cyantraniliprole is rapidly degraded by photolysis. Half-lives in natural water and pH 4 sterile buffer exposed to continuous artificial sunlight for 15 days at 25 °C were 4–5 hours, with the formation of IN-NXX69, IN-NXX70, IN-QKV54 and IN-QKV55 as photodegradates. DT_{90s} were less than 16 hours.

In moist (non-sterile) soil (75% field capacity), treated with [^{14}C]-cyantraniliprole at the equivalent of 1 kg ai/ha parent residues decreased to about 1% AR after 30 days in the irradiated samples and to 33% AR in the non-irradiated samples. The IN-J9Z38 metabolite was the predominant residue (up to about 50% AR) with IN-RNU71 and IN-QKV54 also found at about 13–14% AR. Estimated photolysis DT_{50} and DT_{90} values for cyantraniliprole (derived from the difference in the degradation constants (k) for the irradiated and non-irraditaed samples) were 12 and 41 days respectively. Kinetic modeling suggested that nearly 34% of the cyantraniliprole in soil is degraded by photolysis and about 64% through soil degradation pathway.

Aerobic soil metabolism

Two studies were conducted in five soils (one loam soil, two silty clay loams, one silt loam and a sandy loam) with the equivalent of 0.4 kg ai/ha [CN-^{14}C]-cyantraniliprole or [PC-^{14}C]-cyantraniliprole. In these studies, the moist soils were incubated in the dark for up to a year at 20 °C or 22 °C.

Half-lives for cyantraniliprole were 9 days in the loam soil, 21–39 days in the silty clay loams, 44 days in the silty loam and 92 days in the sandy loam.

DT_{50} values for the seven major transformation products (average DT_{50} values in brackets) were: IN-JCZ38 (8 days), IN-K5A79 (64 days), IN-K5A77 (132 days), IN-J9Z38 (139 days), IN-JSE76 (410 days), IN-K5A78 (423 days) and IN-PLT97 (1032 days).

Soil dissipation

Ten field studies were conducted to investigate the degradation and mobility of cyantraniliprole under field conditions. In all of the trials a single application of 0.3 kg ai/ha or 0.45 kg ai/ha was made to bare soil in late spring or early summer and cropped soils were also treated in three of these studies. Soil samples were collected to a maximum depth of 90 cm, immediately prior to application and at pre-determined intervals over an 18 month period.

Cyantraniliprole was rapidly degraded in field soils with half-lives ranging from 17 to 51 days. While laboratory studies suggested that pH had some effect on degradation rates, degradation rate under field conditions does not appear to be pH-dependent. Downward mobility of the parent compound as well as its metabolites was limited, with residues rarely found below 15 cm. Soil metabolites formed in the bare soil treatments at levels greater than 10% of the initial soil concentration were IN-J9Z38 (max 42%), IN-K5A78 (max 17%), IN-JSE76 (max 14%), IN-JCZ38 (max 13%) and IN-K5A77 (max 11%). Lower metabolite levels were observed in soil from the cropped soil treatments, predominantly IN-J9Z38 (max 27%) and IN-JCZ38 (max 13%).

The mean temperature-normalized field DT_{50} value from all studies was 32 days, consistent with the mean laboratory derived value.

Water/sediment dissipation

Under anaerobic conditions, cyantraniliprole degraded in the water phase and also partitioned to the sediment where it was further degraded and incorporated into the sediment organic fraction. The major degradate was IN-J9Z38, present in the total system at up to 23–40% AR (0.09–0.16 ppm) during the first 28 days after treatment and declining to 5–7% AR (0.02–0.03 ppm) at the end of the study period (Day 100).

The aerobic degradation of [^{14}C]-cyantraniliprole was studied in a water/silt-loam system and a water/sand system treated with [PC-^{14}C]-cyantraniliprole at a rate of 0.5 µg ai/g and incubated outdoors for 14 days under natural sunlight at 23 ± 2 °C.

In the water phase, cyantraniliprole residues were 1–2% AR at the end of the 14-day study period and in the sediment phase, after reaching maximum levels of 15–22% AR after 2–3 days, residues declined to 6–9% AR on Day 14.

One significant degradate, IN-J9Z38, was found in the surface water at a maximum of about 15–27% AR after 3–5 days and declining to 4.5% AR (silt-loam system) and 12% AR (sand system) at the end of the 14-day study period. In the sediment extracts residues of IN-J9Z38 increased from about 2% AR at Day 1 to about 42% AR at Day 14.

The calculated half-lives in the water/sediment systems were 3.5–4.4 days for cyantraniliprole and 40 days for the IN-J9Z38 metabolite.

Residues in succeeding crops

In two rotational crop metabolism studies using [PC-^{14}C]-cyantraniliprole or [CN-^{14}C]-cyantraniliprole, wheat, soya bean, lettuce and red beet were planted as rotational crops 25–30 and 120 days after a single bare soil application of 0.3 kg ai/ha in one study and 0.45 kg ai/ha in the second study. In the second study, after 365 days aging, a further planting of wheat was made in the 30-day rotation plots.

In the first rotation crops, total radioactive residues in food items ranged from 0.02–0.06 mg/kg in wheat grain, 0.08–0.11 mg/kg in lettuce, 0.02–0.03 mg/kg in beet roots and 0.04 mg/kg in soya bean seeds. Higher residues were seen in animal feed items; wheat hay and straw (0.97–1.6 mg/kg), soya bean foliage (0.19 mg/kg) and beet foliage (0.11 mg/kg).

The metabolic fates of cyantraniliprole in the three rotational crops were similar. Cyantraniliprole was the predominant residue in wheat straw and hay (41–53% TRR, 0.4–0.85 mg/kg), wheat grain (10–36% TRR, < 0.01–0.02 mg/kg), soya bean foliage (36% TRR, 0.07 mg/kg), red beet roots (21–27% TRR, < 0.01 mg/kg) and lettuce (60–69% TRR, 0.05–0.08 mg/kg). No cyantraniliprole was detected in soya bean seeds and parent residues in beet foliage were 3–4% TRR.

In food commodities, IN-MYX98 was the only significant metabolite present above 10% TRR, being found in 2nd rotation (120 day) lettuce leaves at 16% TRR but < 0.01 mg/kg. In animal feed commodities, IN-J9Z38 was found in wheat hay, forage and straw at up to 13% TRR (0.18 mg/kg) and IN-K7H19 was present in wheat hay and straw at 10–11% TRR (0.04–0.06 mg/kg) in the 365 day rotation crop.

Overall, the metabolism in rotational crops was consistent with metabolism seen in primary crops and in the animal studies.

Rotational crop field studies were conducted in Europe and North America to estimate residue uptake in follow crops. In two European studies, where spinach, lettuce, spring barley, oats, soya bean and radish were planted into bare soil treated with 0.2 kg ai/ha or 0.45 kg ai/ha at plant-back intervals of 14, 30, 120, 270 and 365 days, with the exception of soya bean (seeds and forage) and radish tops, residues of cyantraniliprole and metabolites were not found in succeeding crops. In soya bean forage, residues of cyantraniliprole were 0.02–0.03 mg/kg and in soya bean seed, residues of the IN-N7B69 metabolite were < 0.01 mg/kg.

In five North American studies, bare soil was treated with three applications of cyantraniliprole at about 5 day intervals to achieve a total seasonal application rate of 0.45 kg ai/ha. In three of these studies, four rotational crops (lettuce/spinach, oats, radish and soya bean) were planted 14, 30, 120, and 365 days after the last application. In the other two studies, strawberries, turnip, sugar beet, garden beet, radish, carrot, bean, pea, soya bean, alfalfa, clover, field corn, sweet corn, sorghum, rice, wheat, Bermuda grass, brome grass, clover, bluegrass and peanut were planted 30 days after the last application.

Residues of cyantraniliprole and metabolites in the first rotation crops (30 day plant-back interval were below 0.05 mg/kg in commodities for human consumption (cereal grains, root crops, legumes and pulses, leafy vegetables). Higher residues were reported in animal feed commodities, up to 0.2 mg/kg in forage crops and 0.3 mg/kg in most hays and straws. Highest residues were found in soya bean hay, up to 0.63 mg/kg in one sample.

In the first rotation crops (30 day plant-back interval (PBI)), highest residues of cyantraniliprole were above 0.01 mg/kg in radish roots (0.02 mg/kg), radish and turnip tops (0.02–0.04 mg/kg), legume forages (0.02–0.14 mg/kg), legume hays (0.05–0.63 mg/kg), leafy vegetables (0.03 mg/kg), cereal forages (0.01–0.11 mg/kg), cereal hays and straws (0.07–0.21 mg/kg), forage grasses (0.01–0.09 mg/kg) and grass hays (0.02–0.23 mg/kg).

Metabolites present at more than 0.01 mg/kg were IN-J9Z38 (up to 0.03 mg/kg in cereal and legume hays and straws), IN-JZ38 (in cereal and legumes, up to 0.06 mg/kg hays and 0.03 mg/kg in forages) and IN-MLA84 (in legumes, up to 0.02 mg/kg in forage and 0.07 mg/kg in hays).

Methods of analysis

Several analytical methods have been reported for the analysis of cyantraniliprole and up to eight metabolites in plant and animal commodities. The basic approach employs extraction with acetonitrile/water and analysis by high pressure liquid chromatography with tandem mass spectrometry.

For plant and processed plant commodities, the HPLC-MS/MS method used in most of the supervised residue field trials, was validated for the analysis of cyantraniliprole and its metabolites (IN-N7B69, IN-JCZ38, IN-K7H19, IN-MYX98, IN-MLA84 and IN-J9Z38) in a range of representative matrices. The LOQ is 0.01 mg/kg for each analyte. Adequate extraction efficiencies

were demonstrated in plant matrices using radiolabelled samples from metabolism and confined rotational crop studies.

For animal commodities, the HPLC-MS/MS method was validated for the analysis of cyantraniliprole, IN-HGW87, IN-N7B69, IN-K7H19, IN-JCZ38, IN-MYX98, IN-J9Z38 and IN-MLA84 in livestock tissues, milk and eggs. After extraction with acetonitrile, extracts are partitioned against hexane before SPE clean-up and analysis. The LOQ is 0.01 mg/kg for each analyte. The method was validated by an independent laboratory using kidney, muscle, and milk. The extraction efficiency was successfully demonstrated with samples of liver, muscle, milk, egg white, and egg yolk from livestock metabolism studies.

The DFG S19 multi-residue method with LC-MS/MS analysis was validated for the analysis of cyantraniliprole residues in tomato (representing high water content), orange (high acid content), wheat grain (high starch content) and almond (high oil content), and is suitable as an enforcement method for cyantraniliprole in plant commodities. It was also validated in milk, eggs, meat and liver as being a suitable enforcement method for cyantraniliprole residues in animal commodities.

The US-FDA PAM multi-residue methods were shown to be unsuitable for the detection and enforcement of cyantraniliprole and metabolites (Protocols A through F).

Stability of pesticide residues in stored analytical samples

Freezer storage stability of cyantraniliprole and metabolites IN-F6L99, IN-J9Z38, IN-JCZ38, IN-K7H19, IN-MLA84, IN-MYX98, IN-N5M09 and IN-N7B69 was investigated in five representative commodities: apples (high-water content), grapes (high-acid content), potatoes (high-starch content), dry bean seeds (high-protein content), and peanuts (high-oil content).

Residues were shown to be stable in these representative substrates for at least 24 months in frozen storage, with residues in the stored samples being greater than 80% of the spiked levels except in peanuts (high oil content), where reduced recoveries were observed at all storage intervals for the metabolites IN-JCZ38, IN-K7H19 and IN-N7B69.

Definition of the residue

In animal commodities, the predominant residues identified in the metabolism studies were cyantraniliprole, IN-J9Z38, IN-MLA84, IN-N7B69 and IN-MYX98. Where residues were found in animal tissues in the metabolism studies or the feeding studies, cyantraniliprole was the major or a significant component. Noting that a multi-residue method was available to measure cyantraniliprole in animal commodities, the Meeting agreed that for MRL-compliance, the residue definition for animal commodities should be cyantraniliprole.

The compound cyantraniliprole has a log K_{ow} of 1.9, suggesting that it is not fat soluble, and this is supported by the residue distribution in muscle and fat reported in a cow feeding study, where the residues in fat were generally only about 2-fold higher than in muscle. The Meeting therefore concluded that cyantraniliprole is not fat soluble.

For dietary intake estimation, in addition to the parent compound, metabolites found at significant levels in the animal metabolism studies were IN-N7B69 (milk), IN-MYX98 (muscle and milk), IN-J9Z38 (fat and eggs) and IN-MLA84 (eggs). In the feeding studies, at doses that reflect the expected animal burden, these individual metabolites were also found at levels ranging from about 20% to 100% of the parent concentrations in different matrices but when combined, were generally found at levels close to those of the parent.

IN-N7B69 was found in milk at a level equivalent to parent and at about half the parent concentration in kidney. IN-J9Z38 was present in eggs at about 50% parent concentration. IN-MLA84 was found in cattle liver at about 60% of the parent concentration and in eggs at about 50% parent concentration. IN-MYX98, found in eggs at about 20% parent and in poultry liver at about 135% of parent.

These metabolites also occur in rats, are not considered more toxic than the parent compound, are adequately covered in the derived toxicological reference dose and a validated HPLC-MS/MS method is available to analyse for them.

The Meeting agreed that while not all of these metabolites would occur in all tissues; these four metabolites (IN-N7B69, IN-J9Z38, IN-MLA84 and IN-MYX98) should be included in the residue definition for dietary intake estimation for animal commodities. The Meeting considered that if animal commodities were analysed only for cyantraniliprole, a conservative correction factor of 2 could be applied for the purpose of dietary intake estimation to account for these metabolites.

In plant commodities from treated crops, the metabolism studies indicated that cyantraniliprole was the major residue in rice, lettuce, cotton and tomato. In rotational crops, where residues are present, cyantraniliprole is also the main residue in food commodities. The Meeting noted that a multi-residue method exists to measure parent residues and agreed that for MRL-compliance, the residue definition for plant commodities should be cyantraniliprole.

The only metabolite identified in the plant metabolism studies at more than 10% TRR or greater than 0.01 mg/kg in commodities at harvest was IN-J9Z38, reported in leaves from foliar-treated rice (11% TRR and 0.13 mg/kg), rice straw from rice grown in treated soil (up to 18% TRR and 0.05 mg/kg) and in foliar-treated lettuce where residues of 23% TRR (0.01 mg/kg) were measured in mature leaves from seedling plants treated up to 32 days before sampling. The Meeting noted that in the supervised field trials, residues of IN-J9Z38 were also reported in some trials, but mostly < 0.01 mg/kg and rarely found at levels more than 10% of the cyantraniliprole residue.

In rotational crops, metabolite residues in food commodities did not exceed 0.01 mg/kg, with IN-J9Z38, IN-JZ38 and IN-MLA84 only present in animal feeds (cereal and legume forage, hays and straws) at up to 0.07 mg/kg.

In processed food commodities, metabolites IN-J9Z38 and to a lesser extent IN-N5M09 and IN-F6L99 were formed under conditions of heat and/or hydrolysis. In addition to cyantraniliprole, only IN-J9Z38 was observed at significant levels, being the predominant residue in cooked spinach, cottonseed oil and present at more than 50% of the parent levels in tomato paste, apple sauce and canned olives. IN-N5M09 and IN-F6L99 were only quantifiable in a few processed food commodities (e.g., cooked spinach, apple sauce) and were much lower than the levels of the parent compound and IN-J9Z38.

The main metabolite in some processed commodities (IN-J9Z38) was also observed in the animal metabolism studies and the toxicology of IN-J9Z38 is addressed in the rat studies and covered by the derived reference dose. Sufficient toxicological information is available to confirm that the IN-J9Z38 metabolite is no more toxic than cyantraniliprole and analytical methods are available to measure this metabolite.

The Meeting concluded that for dietary intake risk assessment, the residue definition for plant commodities should be cyantraniliprole but that for processed commodities, the IN-J9Z38 should also be included.

Proposed definition of the residue (for compliance with the MRL, animal and plant commodities*): cyantraniliprole*.

Proposed definition of the residue (for estimation of dietary intake for unprocessed plant commodities): *cyantraniliprole*.

Proposed definition of the residue (for estimation of dietary intake for processed plant commodities): *sum of cyantraniliprole and IN-J9Z38, expressed as cyantraniliprole*.

Proposed definition of the residue (for estimation of dietary intake for animal commodities: *sum of cyantraniliprole, 2-[3-Bromo-1-(3-chloro-2-pyridinyl)-1H-pyrazol-5-yl]-3,4-dihydro-3,8-dimethyl-4-oxo-6-quinazolinecarbonitrile [IN-J9Z38], 2-[3-Bromo-1-(3-chloro-2-pyridinyl)-1H-pyrazol-5-yl]-1,4-dihydro-8-methyl-4-oxo-6-quinazolinecarbonitrile [IN-MLA84], 3-Bromo-1-(3-chloro-2-pyridinyl)-N-[4-cyano-2-(hydroxymethyl)-6-[(methylamino)carbonyl]phenyl]-1H-pyrazole-*

5-carboxamide [IN- N7B69] and 3-Bromo-1-(3-chloro-2-pyridinyl)-N-[4-cyano-2[[(hydroxymethyl)amino]carbonyl]-6-methylphenyl]-1H-pyrazole-5-carboxamide [IN- MYX98], expressed a cyantraniliprole.

The residue is not fat soluble.

Results of supervised residue trials on crops

The Meeting received supervised trial data for foliar and soil applications of cyantraniliprole on a range of fruit and vegetable crops, rice, tree nuts, oilseeds and coffee and for seed treatments (potatoes, oil-seed rape). These trials were conducted mainly in Europe and/or North America.

Where residues have been reported as ND (< LOD) the values have been considered as < LOQ (< 0.01 mg/kg) for the purposes of MRL setting. If a higher residue level was observed at a longer PHI than the GAP, the higher value has been used in MRL setting.

The Meeting noted that GAP has been authorised for the use of cyantraniliprole and that product labels were available from Canada, Columbia, New Zealand Malaysia, Vietnam and from a regional group of countries in West Africa. Supervised trial data were provided for citrus, grapes, olives, pomegranate, beans and sunflower, but no GAP information was available to support maximum residue level estimations for these commodities.

Pome fruits

The critical GAP for cyantraniliprole on pome fruit is in Canada, up to four foliar applications of 0.05–0.15 kg ai/ha applied at least 7 days apart with a PHI of 3 days and with a total of 0.45 kg ai/ha/season.

In trials on apples in North America matching the Canadian GAP (with three applications of 0.15 kg ai/ha, 0.45 kg ai/ha/season), cyantraniliprole residues were: 0.06, 0.07, 0.1, 0.12, 0.13, 0.13, 0.15, 0.15, 0.16, 0.17, 0.18, 0.21, 0.26, 0.26, 0.29 and 0.31 mg/kg (n=16).

In trials on pears in North America matching the Canadian GAP (with three applications of 0.15 kg ai/ha, 0.45 kg ai/ha/season), cyantraniliprole residues were: 0.08, 0.1, 0.12, 0.14, 0.16, 0.23, 0.42, 0.44, 0.56 and 0.58 mg/kg (n=10).

The Meeting noted that the GAP in Canada was for pome fruit and that the medians of the two data sets differed by less than 5-fold and agreed to consider a group maximum residue level. As the Mann-Whitney U-test indicated that the residue populations for apples and pears were not different it was agreed to combine the results to give a data set of: 0.06, 0.07, 0.08, 0.1, 0.1, 0.12, 0.12, 0.13, 0.13, 0.14, 0.15, 0.15, 0.16, 0.16, 0.17, 0.18, 0.21, 0.23, 0.26, 0.26, 0.29, 0.31, 0.42, 0.44, 0.56 and 0.58 mg/kg (n=26) for the pome fruit crop group.

The Meeting estimated an STMR of 0.16 mg/kg and a group maximum residue level of 0.8 mg/kg for cyantraniliprole on pome fruit.

Stone fruits

The critical GAP for cyantraniliprole on stone fruit is in Canada, up to four foliar applications of 0.05–0.15 kg ai/ha with a total of 0.45 kg ai/ha/season, applied at least 7 days apart with a PHI of 3 days.

In trials on cherries in North America matching the Canadian GAP (with three applications of 0.15 kg ai/ha, 0.45 kg ai/ha/season), cyantraniliprole residues in whole fruit were: 0.3, 0.32, 0.7, 0.8, 0.89, 0.9 and 3.4 mg/kg (n=7). In flesh residues were: 0.33, 0.36, 0.89, 0.93, 0.96, 0.98 and 3.8 mg/kg (n=7).

In trials on peaches in North America matching the Canadian GAP (with three applications of 0.15 kg ai/ha, 0.45 kg ai/ha/season), cyantraniliprole residues were: 0.16, 0.18, 0.19, 0.19, 0.23, 0.24, 0.28, 0.34, 0.39, 0.45, 0.51, 0.79 and 0.81 mg/kg. In flesh, residues were: 0.19, 0.19, 0.2, 0.23, 0.25, 0.27, 0.34, 0.35, 0.42, 0.49, 0.56, 0.89 and 0.94 mg/kg (n=13).

In trials on plums in North America matching the Canadian GAP (with three applications of 0.15 kg ai/ha, 0.45 kg ai/ha/season), cyantraniliprole residues were: 0.03, 0.05, 0.06, 0.06, 0.06, 0.07, 0.12, 0.19 and 0.28 mg/kg. In flesh, residues were: 0.03, 0.05, 0.06, 0.06, 0.07, 0.07, 0.13, 0.2 and 0.29 mg/kg (n=9).

The Meeting noted that the GAP in Canada was for stone fruit and that the medians of the data sets for cherries, peaches and plums differed more than 5-fold and agreed not to consider a group maximum residue level for stone fruit.

The Meeting estimated an STMR of 0.93 mg/kg (based on residues in flesh), and based on residues in the whole fruit, estimated a subgroup maximum residue level of 6 mg/kg for cyantraniliprole on cherries.

The Meeting estimated an STMR of 0.34 mg/kg (based on residues in flesh, and based on residues in the whole fruit, estimated a subgroup maximum residue level of 1.5 mg/kg for cyantraniliprole on peaches.

The Meeting estimated an STMR of 0.07 mg/kg (based on residues in flesh) and based on residues in the whole fruit, estimated a subgroup maximum residue level of 0.5 mg/kg for cyantraniliprole on plums.

Bush berries

The critical GAP for cyantraniliprole on bush berries is in Canada, up to four foliar applications of 0.05–0.15 kg ai/ha with a total of 0.45 kg ai/ha/season, applied at least 7 days apart with a PHI of 3 days.

In trials on blueberries in North America matching the Canadian GAP (with three applications of 0.15 kg ai/ha, 0.45 kg ai/ha/season), cyantraniliprole residues were: 0.42, 0.51, 0.52, 0.52, 0.75, 0.8, 1.5, 1.5 and 2.0 mg/kg (n=9).

The Meeting noted that blueberry can be used as a representative crop for bush berries and estimated an STMR of 0.75 mg/kg and a subgroup maximum residue level of 4.0 mg/kg for cyantraniliprole on bush berries.

Bulb vegetables

The critical GAP for cyantraniliprole on bulb vegetables is in Canada, up to four foliar applications of 0.1–0.15 kg ai/ha with a total of 0.45 kg ai/ha/season, applied at least 5 days apart with a PHI of 1 day.

In trials on bulb onions in North America matching the Canadian GAP (with three applications of 0.15 kg ai/ha, 0.45 kg ai/ha/season), cyantraniliprole residues were: < 0.01, < 0.01, < 0.01, < 0.01, 0.01, 0.02, 0.02, 0.02, 0.02 and 0.03 mg/kg (n=10).

The Meeting noted that the GAP in Canada also includes use on garlic and shallot and agreed to extrapolate the data for bulb onions to these commodities.

The Meeting estimated an STMR of 0.02 mg/kg and a maximum residue level of 0.05 mg/kg for cyantraniliprole on onion, bulb, garlic and shallot.

In trials on spring onions (green onions) in North America matching the Canadian GAP (with three applications of 0.15 kg ai/ha, 0.45 kg ai/ha/season), cyantraniliprole residues were: 0.38, 0.63, 1.3, 1.6 and 4.1 mg/kg (n=5).

The Meeting noted that the GAP in Canada also includes use Welsh onion and agreed to extrapolate the data for spring onions to onion, Welsh.

The Meeting estimated an STMR of 1.3 mg/kg and a maximum residue level of 8.0 mg/kg for cyantraniliprole on spring onion and onion, Welsh.

Brassica (cole or cabbage) vegetables

The critical GAP for cyantraniliprole on brassica vegetables is in Canada, up to four foliar applications of 0.025–0.15 kg ai/ha with a total of 0.45 kg ai/ha/season, applied at least 5–7 days apart with a PHI of 1 day.

In trials on broccoli in North America matching the critical Canadian GAP (with three applications of 0.15 kg ai/ha, 0.45 kg ai/ha/season), cyantraniliprole residues were: 0.22, 0.28, 0.51, 0.59, 0.61, 0.69, 0.82 and 1.1 mg/kg (n=8).

In trials on cauliflowers in North America matching the critical Canadian GAP (with three applications of 0.15 kg ai/ha, 0.45 kg ai/ha/season), cyantraniliprole residues in the flower heads were: 0.01 and 0.08 mg/kg (n=2).

In trials on head cabbage in North America matching the critical Canadian GAP (with three applications of 0.15 kg ai/ha, 0.45 kg ai/ha/season), cyantraniliprole residues in cabbages (with wrapper leaves) were: 0.29, 0.32, 0.32, 0.42, 0.47, 0.56, 0.57, 0.65, 0.71, 0.86 and 0.95 mg/kg (n=11).

The Meeting noted that the GAP in Canada was for brassica vegetables and that the medians of the data sets for broccoli and cabbage differed by less than 5-fold (insufficient data on cauliflower) and agreed to consider a group maximum residue level. In deciding on the data set to use for estimating a group maximum residue level, since a Mann-Whitney U-test indicated that the residue populations for broccoli and cabbage were not different, it was agreed to combine the results to give a data set of: 0.01, 0.08, 0.22, 0.28, 0.29, 0.32, 0.32, 0.42, 0.47, 0.51, 0.56, 0.57, 0.59, 0.61, 0.65, 0.69, 0.71, 0.82, 0.86, 0.95 and 1.1 mg/kg (n=21) for brassica vegetables.

The Meeting estimated an STMR of 0.56 mg/kg and a group maximum residue level of 2.0 mg/kg for cyantraniliprole on brassica (cole or cabbage) vegetables. The Meeting also estimated a highest residue of 1.1 mg/kg for calculating animal dietary burdens.

Fruiting vegetables, Cucurbits

The critical GAP for cyantraniliprole on cucurbit vegetables is in Canada, up to four foliar applications of 0.025–0.15 kg ai/ha with a total of 0.45 kg ai/ha/season, applied at least 5–7 days apart with a PHI of 1 day.

In trials on cucumber in North America matching the GAP of Canada (with three applications of 0.15 kg ai/ha, 0.45 kg ai/ha/season), cyantraniliprole residues were: 0.02, 0.02, 0.02, 0.03, 0.04, 0.05, 0.05, 0.07, 0.12 and 0.16 mg/kg (n=10).

In trials on summer squash in North America matching the critical Canadian GAP (with three applications of 0.15 kg ai/ha, 0.45 kg ai/ha/season), cyantraniliprole residues were: 0.01, 0.03, 0.04, 0.05, 0.06, 0.07, 0.08, 0.09 and 0.11 mg/kg (n=9).

In trials on melons in North America matching the critical Canadian GAP (with three applications of 0.15 kg ai/ha, 0.45 kg ai/ha/season), cyantraniliprole residues were: 0.04, 0.05, 0.08, 0.09, 0.09, 0.1, 0.11, 0.15 and 0.17 mg/kg (n=9). In these trials, cyantraniliprole residues in the melon edible portion were all < 0.01 (n=9).

The Meeting noted that the GAP in Canada was for cucurbit vegetables and that the medians of the data sets for cucumber, summer squash and melons differed by less than 5-fold and agreed to consider a group maximum residue level. In deciding on the data set to use for estimating a group maximum residue level, since a Kruskal-Wallis H-test indicated that the residue populations for cucumber, summer squash and melons were not different, it was agreed to combine the results to give a data set of: 0.01, 0.02, 0.02, 0.02, 0.03, 0.03, 0.04, 0.04, 0.04, 0.05, 0.05, 0.05, 0.05, 0.06, 0.07, 0.07, 0.08, 0.08, 0.09, 0.09, 0.09, 0.1, 0.11, 0.11, 0.12, 0.15, 0.16 and 0.17 mg/kg (n=28) for cucurbit vegetables.

The Meeting estimated an STMR of 0.01 mg/kg for cucurbits with an inedible peel (based on the melon data on residues in flesh), an STMR of 0.065 mg/kg (based on the summer squash data) for

cucurbits with an edible peel and a group maximum residue level of 0.3 mg/kg for cyantraniliprole on fruiting vegetables, Cucurbits.

Fruiting vegetables, other than Cucurbits

The critical GAP for cyantraniliprole on fruiting vegetables (except cucurbits) is in Canada, up to four foliar applications of 0.025–0.15 kg ai/ha with a total of 0.45 kg ai/ha/season, applied at least 5–7 days apart with a PHI of 1 day.

In trials on tomatoes in North America matching the critical Canadian GAP (with three applications of 0.15 kg ai/ha, 0.45 kg ai/ha/season), cyantraniliprole residues were: 0.04, 0.05, 0.06, 0.06, 0.07, 0.07, 0.07, 0.07, 0.08, 0.08, 0.08, 0.09, 0.09, 0.1, 0.12, 0.14, 0.14, 0.16, 0.17 and 0.26 mg/kg (n=20).

In trials on sweet peppers in North America matching the critical Canadian GAP (with three applications of 0.15 kg ai/ha, 0.45 kg ai/ha/season), cyantraniliprole residues were: 0.03, 0.04, 0.06, 0.07, 0.07, 0.08, 0.08, 0.15, 0.21, 0.24 and 0.28 mg/kg (n=11).

In trials on chilli peppers (non-bell peppers) in North America matching the critical Canadian GAP (with three applications of 0.15 kg ai/ha, 0.45 kg ai/ha/season), cyantraniliprole residues were: 0.07, 0.07, 0.08, 0.09, 0.1, 0.2, 0.25, 0.31 and 0.42 mg/kg (n=9).

The Meeting noted that the GAP in Canada was for fruiting vegetables (except cucurbits) and that the medians of the data sets for sweet peppers, tomatoes and chilli peppers differed by less than 5-fold and agreed to consider a group maximum residue level. In deciding on the data set to use for estimating a group maximum residue level, since a Kruskal-Wallis H-test indicated that the residue populations for sweet peppers, tomatoes and chili peppers were not different, it was agreed to combine the results to give a data set of: 0.03, 0.04, 0.04, 0.05, 0.06, 0.06, 0.06, 0.07, 0.07, 0.07, 0.07, 0.07, 0.07, 0.07, 0.07, 0.08, 0.08, 0.08, 0.08, 0.08, 0.08, 0.09, 0.09, 0.09, 0.1, 0.1, 0.12, 0.14, 0.14, 0.15, 0.16, 0.17, 0.2, 0.21, 0.24, 0.25, 0.26, 0.28, 0.31 and 0.42 mg/kg (n=40) for the non-cucurbit fruiting vegetables group.

The Meeting estimated an STMR of 0.08 mg/kg and a group maximum residue level of 0.5 mg/kg for cyantraniliprole on fruiting vegetables, other than Cucurbits (excluding sweet corn and mushrooms).

For dried chilli peppers, applying the default processing factor of 7 to the data set for fresh chilli peppers, the Meeting estimated an STMR-P of 0.7 mg/kg and a maximum residue level of 5 mg/kg for cyantraniliprole on dried chilli peppers.

Leafy vegetables (including Brassica leafy vegetables)

The critical GAP for cyantraniliprole on leafy vegetables is in Canada, up to four foliar applications of 0.025–0.15 kg ai/ha with a total of 0.45 kg ai/ha/season, applied at least 5–7 days apart with a PHI of 1 day.

In trials on head lettuce in North America matching the critical Canadian GAP (with three applications of 0.15 kg ai/ha, 0.45 kg ai/ha/season), cyantraniliprole residues were: 0.02, 0.08, 0.16, 0.18, 0.64, 0.75, 0.83, 1.3, 1.6, 1.8, 2.1 and 2.7 mg/kg (n=12).

In trials on leaf lettuce in North America matching the critical Canadian GAP (with three applications of 0.15 kg ai/ha, 0.45 kg ai/ha/season), cyantraniliprole residues were: 1.1, 1.2, 2.1, 2.4, 2.4, 2.5, 3.2, 3.3, 4.0, 5.3, 6.8 and 6.8 mg/kg (n=12).

In trials on spinach in North America matching the critical Canadian GAP (with three applications of 0.15 kg ai/ha, 0.45 kg ai/ha/season), cyantraniliprole residues were: 3.8, 4.1, 4.2, 4.6, 4.7, 4.9, 5.8, 8.2, 10 and 13 mg/kg (n=10).

In trials on mustard greens in North America matching the critical Canadian GAP for vegetable brassicas (with three applications of 0.15 kg ai/ha, 0.45 kg ai/ha/season), cyantraniliprole residues were: 2.4, 3.4, 3.9, 5.5, 5.8, 6.0, 7.1, 7.2, 8.0, 13 and 19 mg/kg (n=11).

The Meeting noted that the GAP in Canada was for leafy vegetables and that the medians of the data sets for leaf lettuce, spinach and mustard greens (but not head lettuce) differed by less than 5-fold and agreed to consider a group maximum residue level for leafy vegetables except head lettuce. In deciding on the data set to use for estimating a group maximum residue level, since a Kruskal-Wallis H-test indicated that the residue populations for leaf lettuce, spinach and mustard greens were not different it was agreed to combine the results to give a data set of: 1.1, 1.2, 2.1, 2.4, 2.4, 2.4, 2.5, 3.2, 3.3, 3.4, 3.8, 3.9, 4.0, 4.1, 4.2, 4.6, 4.7, 4.9, 5.3, 5.5, 5.8, 5.8, 6.0, 6.8, 6.8, 7.1, 7.2, 8.0, 8.2, 10, 13, 13 and 19 mg/kg for leafy vegetables (n=33) except head lettuce and to use the head lettuce data to estimate a maximum residue level for head lettuce.

The Meeting estimated an STMR of 4.7 mg/kg and a group maximum residue level of 20 mg/kg for cyantraniliprole on leafy vegetables (except head lettuce).

The Meeting estimated an STMR of 0.79 mg/kg and a maximum residue level of 5 mg/kg for cyantraniliprole on head lettuce.

Root and tuber vegetables

The critical GAP for cyantraniliprole on root and tuber vegetables is in Canada, up to four foliar applications of 0.05–0.15 kg ai/ha with a total of 0.45 kg ai/ha/season, applied at least 5–14 days apart with a PHI of 7 days.

In trials on potatoes in North America matching the critical Canadian GAP for foliar applications to root and tuber vegetables (with three applications of 0.15 kg ai/ha, 0.45 kg ai/ha/season), cyantraniliprole residues were: < 0.01, < 0.01, < 0.01, < 0.01, < 0.01, < 0.01, < 0.01, < 0.01, < 0.01, < 0.01, < 0.01, < 0.01, < 0.01, < 0.01, 0.01, 0.02, 0.02 and 0.03 mg/kg (n=20).

The Meeting also noted that residues of cyantraniliprole may also arise in potatoes planted as rotational crops, and agreed to consider maximum residue level recommendations for potatoes when discussing rotational crop residues.

Stalk and stem vegetables

The critical GAP for cyantraniliprole on celery is in Canada, up to four foliar applications of 0.025–0.15 kg ai/ha with a total of 0.45 kg ai/ha/season, applied at least 5–7 days apart with a PHI of 1 day.

In trials on celery in North America matching the critical Canadian GAP for leafy vegetables (including celery), with three applications of 0.15 kg ai/ha, 0.45 kg ai/ha/season, cyantraniliprole residues were: 0.28, 0.73, 1.0, 1.1, 1.2, 2.0, 2.3, 2.5, 4.7, 5.7 and 9.1 mg/kg (n=11).

The Meeting estimated an STMR of 2.0 mg/kg and a maximum residue level of 15 mg/kg for cyantraniliprole on celery.

Rice

The critical GAP for cyantraniliprole on rice is in Vietnam, for foliar applications of 0.05–0.1 kg ai/ha with a PHI of 5 days.

Results were available from six trials on rice in China where three foliar applications of cyantraniliprole were applied up to 7 days before harvest.

The Meeting agreed that these data did not match the GAP in Vietnam in that the PHI deviated from GAP by more than 25%.

Tree nuts

The critical GAP for cyantraniliprole on tree nuts is in Canada, up to four foliar applications of 0.05–0.1 kg ai/ha with a total of 0.45 kg ai/ha/season, applied at least 7 days apart with a PHI of 5 days.

In six trials on almonds in the USA, 3 foliar sprays of 0.15 kg ai/ha (0.45 kg ai/ha/season) were applied at 6–8 day intervals up to 5 days before harvest.

In six trials on pecans in the USA, 3 foliar sprays of 0.15 kg ai/ha (0.45 kg ai/ha/season) were applied at 6–8 day intervals up to 5 days before harvest.

The Meeting noted that since both the number of applications and the treatment rates in the trials for almonds and pecans did not match the Canadian GAP, the use of the proportionality approach to estimate maximum residue levels was not appropriate.

Oilseeds

The critical GAP for cyantraniliprole on cotton is in the region of West Africa, up to three foliar applications of 0.05 kg ai/ha with a total of 0.15 kg ai/ha/season, applied at least 14 days apart with a PHI of 7 days.

Results were available from trials conducted in the USA on cotton, where three foliar applications of 0.15 kg ai/ha cyantraniliprole were applied at 6–8 day intervals up to 7–9 days before harvest.

The Meeting noted that the application rates used in the USA trials were higher and the retreatment intervals were shorter than the GAP in West Africa and the Meeting agreed that the concept of proportionality could not be used to recommend a maximum residue level for cyantraniliprole on cotton seed.

The critical GAP for cyantraniliprole on oil seed crops (excluding cotton and peanut) is in Canada, up to 4 foliar applications of 0.025–0.1 kg ai/ha with a total of 0.11 kg ai/ha/season, applied at least 7 days apart with a PHI of 7 days.

Results were available from trials on oilseed rape and on sunflower in North America, where three foliar sprays of 0.15 kg ai/ha (0.45 kg ai/ha/season) were applied at 5–9 day intervals up to 7 days before harvest.

The Meeting noted that for both oilseed rape and sunflower, in addition to the application rate in the field trials differing from the Canadian GAP, the lower seasonal application rate associated with the Canadian GAP supports only a single application of the maximum recommended application rate of 0.1 kg ai/ha, compared to the three applications used in the field trials.

The Meeting concluded that these trials did not match the Canadian GAP.

Seed for beverages and sweets

The critical GAP for cyantraniliprole on coffee is in Columbia, one foliar application of 2.5–3.5 g ai/5 L/100 trees, equivalent to 0.125–0.175 kg ai/ha with a total of 0.3 kg ai/ha/season, with a PHI of 28 days.

In two Brazilian trials matching the Columbian GAP, cyantraniliprole residues were < 0.01 and 0.02 mg/kg.

The Meeting noted that in a further six trials in Brazil involving foliar applications that matched the Columbian GAP but where two soil drenches (0.01–0.06 g ai/100 mL/plant to achieve the equivalent of 0.2 kg ai/ha/treatment) were also applied approximately 90 and approximately 120 days before harvest, cyantraniliprole residues were < 0.01 (5) and 0.01 mg/kg.

The Meeting agreed that since the early season soil drench treatments did not appear to contribute to the final residue in coffee beans, the data from these two sets of results could be combined, giving a data set of: < 0.01, < 0.01, < 0.01, < 0.01, < 0.01, < 0.01, 0.01 and 0.02 mg/kg, to recommend a maximum residue level for cyantraniliprole on coffee beans.

The Meeting estimated an STMR of 0.01 mg/kg and a maximum residue level of 0.03 mg/kg for cyantraniliprole on coffee bean.

Estimation of residues in plant commodities grown as potential succeeding crops

Residues of cyantraniliprole, while not persistent, can be taken up by following crops. In Canada, the GAP includes a maximum seasonal foliar application rate of 0.45 kg ai/ha for most crops except oil seeds where the maximum seasonal rate is 0.11 kg ai/ha. In Columbia, the maximum seasonal rate for onions and Welsh onions is 0.3 kg ai/ha and in West Africa, the total seasonal rate on cotton is 0.15 kg ai/ha.

In Canada, recommended plant-back intervals (PBIs) have been established for crops likely to be grown in rotation with treated crops. In general, for annual crops for human consumption and where MRLs have been established, there is no plant-back interval specified, but for crops likely to be used as animal feed (cereals, grasses, legumes etc) the label recommends a 30-day plant-back interval.

Field rotational crop studies conducted in the USA on a range of representative crops, involving treatment rates equivalent a 0.45 kg ai/ha maximum seasonal rate to bare soil, reported cyantraniliprole residues of less than 0.05 mg/kg in representative food commodities and higher residues in animal feed commodities.

The Meeting agreed that the results of the USA field rotational crop studies, in particular the cyantraniliprole residues reported from the 30 day PBI crops, could be used to estimate residues in follow crops.

Leafy vegetables (including Brassica leafy vegetables)

Highest cyantraniliprole residues in rotational leafy vegetables were < 0.01 mg/kg in spinach and beet tops, 0.02 mg/kg in lettuce and turnip tops and 0.04 mg/kg in radish tops. These levels are adequately covered by the recommendations for leafy vegetables.

Root and tuber vegetables

Cyantraniliprole residues in rotational root and tuber vegetables ranged from < 0.01 to 0.014 mg/kg (n=29, median < 0.01 mg/kg) in beet roots, turnip roots, carrot roots and radish roots.

The Meeting agreed to use the data from 20 field trials on potatoes in North America, treated according to the Canadian GAP for root and tuber vegetables (residues in tubers ranging from < 0.01 to 0.03 mg/kg, STMR 0.01 mg/kg, n=20) and the results of the rotational crop studies on root and tuber vegetables to recommend a group maximum residue level for potatoes (to accommodate residues in follow-crop potatoes that may also be treated with cyantraniliprole).

The Meeting established a maximum residue level of 0.05 mg/kg, a highest residue of 0.044 mg/kg (for estimating animal dietary burdens) and a median residue of 0.02 mg/kg for cyantraniliprole on potato.

The Meeting also agreed to use the rotational crop studies on root and tuber vegetables to recommend a group maximum residue level for the remaining root and tuber vegetables to accommodate residues in these crops grown as follow-crops.

The Meeting established a maximum residue level of 0.05 mg/kg, a highest residue of 0.014 mg/kg and a median residue of 0.01 mg/kg for cyantraniliprole on root and tuber vegetables except potato.

Miscellaneous fodder root crops

The Meeting also agreed to use the results of the rotational crop studies on root and tuber vegetables (residues ranging from < 0.01 to 0.014 mg/kg, median < 0.01 mg/kg, n=29) to recommend maximum residue levels for turnips and fodder beet to accommodate residues in these crops grown as follow-crops.

The Meeting established a maximum residue level of 0.02 mg/kg, a highest residue of 0.014 mg/kg and a median residue of 0.01 mg/kg for cyantraniliprole on fodder beet and turnip fodder.

Legume animal feeds

Cyantraniliprole residues in legume animal feeds (forage) ranged from < 0.01 to 0.14 mg/kg (n=24, median < 0.01 mg/kg) in clover forage, bean forage, pea forage, alfalfa forage and soya bean forage. For the purpose of estimating livestock dietary burdens, the Meeting agreed to combine the data on rotational crop residues in legume animal feeds to estimate residues in legume feed crops grown as follow-crops.

Meeting estimated a median residue of 0.01 mg/kg and a highest residue of 0.14 mg/kg for cyantraniliprole in legume forages (fresh weight).

Cyantraniliprole residues in legume animal feeds (fodders) ranged from < 0.01 to 0.58 mg/kg (n=24, median 0.017 mg/kg) in peanut hay, clover hay, pea hay, bean, alfalfa hay and soya bean hay. The Meeting agreed to combine the data on rotational crop residues in legume fodder crops to recommend a group maximum residue level to accommodate residues in these crops grown as follow-crops.

The Meeting established a median residue of 0.017 mg/kg and a highest residue of 0.58 mg/kg (0.67 mg/kg DM after correction for an average dry matter content of 87%) and recommended a maximum residue level of 0.8 mg/kg (dry weight) for cyantraniliprole in legume animal feeds.

Cereal and grass forages, straws and hays

Cyantraniliprole residues in cereal and grass forage ranged from < 0.01 to 0.053 mg/kg (n=23, median < 0.01 mg/kg) in corn forage, sorghum, Bermuda grass and brome grass forages, oat forage, bluegrass forage and wheat forage. For the purpose of estimating livestock dietary burdens, the Meeting agreed to combine the data on rotational crop residues in cereal and grass forages to estimate residues in cereal and grasses grown as follow-crops.

Meeting established an STMR of 0.01 mg/kg and a highest residue of 0.053 mg/kg for cyantraniliprole in cereal and grass forages (fresh weight).

Cyantraniliprole residues in cereal and grass straws and hays ranged from < 0.01 to 0.14 mg/kg (median < 0.01 mg/kg) in sorghum stover, rice straw, corn stover, brome grass hay, Bermuda grass hay, oat straw, wheat straw, oat hay, wheat hay bluegrass hay. The Meeting agreed to combine the data on rotational crop residues in cereal and grass straws and hays to recommend a group maximum residue level to accommodate residues in these crops grown as follow-crops.

The Meeting established a median residue of 0.01 mg/kg and a highest residue of 0.14 mg/kg (0.16 mg/kg DM after correction for an average dry matter content of 89%) and recommended a maximum residue level of 0.2 mg/kg (dry weight) for cyantraniliprole in straw, fodder (dry) and hay of cereal grains and other grass-like plants.

Miscellaneous fodder leaf crops

The Meeting agreed to use the results of the rotational crop studies on beet, turnip and radish tops (residues ranging from < 0.01 to 0.021 mg/kg (n=22, median < 0.01 mg/kg) to estimate residues in fodder beet tops and the miscellaneous fodder leaf crops listed in the OECD Feedstuffs Table to accommodate residues in these commodities grown as follow-crops.

The Meeting established median residue of 0.01 mg/kg and a highest residue of 0.021 mg/kg for cyantraniliprole on sugar beet tops, fodder beet tops or leaves, kale forage, rape greens and turnip tops (fresh weight).

Fate of residues during processing

The effect of processing on the nature of residues was investigated in buffer solutions under conditions simulating pasteurisation, boiling and sterilisation. Cyantraniliprole was stable under most processing conditions. Hydrolysis to IN-J9Z38 was a significant pathway under sterilisation

conditions (20 minutes at 120 °C and pH 6) making up 12–14% AR. Other degradates present were IN-F6L99 and IN-N5M09 making up a further 5–8% AR.

The fate of cyantraniliprole residues has been examined in a number of studies simulating household and commercial processing of potatoes, spinach, tomatoes, oranges, apples, plums, cottonseed, olives and grapes. Estimated processing factors and STMR-Ps for the commodities considered at this Meeting are summarized below.

Summary of selected processing factors and STMR-P values for cyantraniliprole

RAC	Commodity (RAC: STMR mg/kg [b]	Cyantraniliprole+IN-J9Z38 [a]			
		Processing factors	PF best estimate	RAC STMR (mg/kg)	STMR-P (mg/kg)
Potato	RAC: tubers			0.02	
	flakes	0.1	0.1		0.002
	waste	0.1	0.1		0.002
	peeled tubers	0.1	0.1		0.002
	chips	0.1	0.1		0.002
	wet peel	2.3	2.3		0.046 hi-res 0.1 [c]
	culls	1.0	1.0		0.02
	fries	0.1	0.1		0.002
	unpeeled, boiled	0.1	0.1		0.002
	unpeeled microwaved	< 0.33	< 0.33		0.006
Spinach	RAC: leaves			4.7	
	cooked leaves	0.81, 1.0, 1.2	1.0		4.7
Tomato	RAC: fruit (0.08 mg/kg)				
	washed	0.15, 0.17, < 0.29	0.17		0.014
	peeled	< 0.08, < 0.08, 0.1	< 0.08		0.006
	sun-dried	3.0, 3.7, 3.8	3.7		0.3
	canned	< 0.02, < 0.05, < 0.08	< 0.05		0.004
	juice	< 0.15, < 0.17, 0.19	< 0.17		0.014
	wet pomace	0.75, 1.0, 2.2	1.0		0.08
	dry pomace	1.7, 3.2, 4.0	3.2		0.26
	paste	0.62, 0.86, 1.0	0.86		0.07
	puree	0.23, 0.25, 0.43	0.25		0.02
Apple	RAC: fruit			0.16	
	washed	0.46, 0.58, 0.63	0.58		0.09
	puree	0.88, 1.0, 1.3	1.0		0.16
	canned	0.04, 0.13, 0.15	0.13		0.02
	frozen	0.62, 0.96, 1.5	0.96		0.15
	juice	0.19, 0.31, 0.38	0.31		0.05
	wet pomace	0.77, 1.0, 1.2	1.0		0.16
	dry pomace	2.0, 2.7, 3.9	2.7		0.43
	sauce	2.2, 2.4, 2.7	2.2		0.35
Plum	RAC: flesh			0.34	
	dried prunes	1.3, 1.6, 2.0	1.6		0.54

[a] Each PF value represents a separate study where residues were above the LOQ in the RAC and is the ratio of the combined cyantraniliprole+IN-J9Z38 metabolite residues in the processed item divided by the combined residues of cyantraniliprole+IN-J9Z38 in the RAC.

[b] Residues in the RAC are the sum of cyantraniliprole and IN-J9Z38

[c] Based on the highest residue in the RAC (0.044 mg/kg)

The Meeting noted that in the studies available, cyantraniliprole residues did not concentrate in food commodities during processing except in apple sauce and in dehydrated commodities such as dried prunes and sun-dried tomatoes. Residues also concentrated in dry pomace (apple and tomato).

In three plum processing studies conducted in the USA, cyantraniliprole residues increased (median processing factor of 1.5) when fresh prunes (flesh) were dried to a moisture content of 15–18% (from about 85% in fresh fruit).

The Meeting estimated a maximum residue level for prunes of 0.8 mg/kg based on the maximum residue level estimated for plums of 0.5 mg/kg and a median processing factor of 1.6.

Residues in animal commodities

Farm animal dietary burden

The Meeting estimated the dietary burden of cyantraniliprole in farm animals on the basis of the diets listed in Annex 6 of the 2009 JMPR Report (OECD Feedstuffs Derived from Field Crops). Dietary burden calculations for beef cattle, dairy cattle, broilers and laying poultry are presented in Annex 6 and are summarized below.

Estimated maximum and mean dietary burdens of farm animals

	Animal dietary burden, cyantraniliprole, ppm of dry matter diet							
	US-Canada		EU		Australia		Japan	
	max	mean	max	mean	max	mean	max	mean
Beef cattle	0.41	0.19	1.9 [a]	0.98 [c]	0.68	0.13	0.13	0.004
Dairy cattle	0.44	0.1	1.9 [b]	0.95 [d]	0.67	0.11	0.29	0.02
Poultry-broiler	0.0	0.0	0.05 [e]	0.02 [f]	0.0	0.0	0.0	0.0
Poultry-layer	0.0	0.0	0.37 [g]	0.19 [h]	0.0	0.0	0.0	0.0

[a] Highest maximum beef or dairy cattle dietary burden suitable for MRL estimates for mammalian tissues

[b] Highest maximum dairy cattle dietary burden suitable for MRL estimates for mammalian milk

[c] Highest mean beef or dairy cattle dietary burden suitable for STMR estimates for mammalian tissues.

[d] Highest mean dairy cattle dietary burden suitable for STMR estimates for milk.

[e] Highest maximum poultry dietary burden suitable for MRL estimates for poultry tissues.

[f] Highest mean poultry dietary burden suitable for STMR estimates for poultry tissues.

[g] Highest maximum poultry dietary burden suitable for MRL estimates for poultry eggs.

[h] Highest mean poultry dietary burden suitable for STMR estimates for poultry eggs.

For beef and dairy cattle, the calculated maximum dietary burden suitable for estimating maximum residue levels in mammalian tissues and milk is 1.9 ppm dry weight of feed and the calculated mean dietary burdens, suitable for estimating STMRs in mammalian tissues and in milk are 0.98 ppm and 0.95 ppm dry weight of feed respectively.

For poultry, noting that in some countries, laying hens may also be consumed, the calculated maximum dietary burden suitable for estimating maximum residue levels in poultry tissues and eggs is 0.37 ppm dry weight of feed and the calculated mean dietary burden, suitable for estimating STMRs in poultry tissues and in eggs is 0.19 ppm dry weight of feed.

Farm animal feeding studies

The Meeting received information on the residue levels arising in animal tissues and milk when dairy cows were dosed with cyantraniliprole for 28 days at the equivalent of 3.5, 12, 35 and 112 ppm in the diet. A separate dose group (112 ppm) was used to estimate residue depuration of cyantraniliprole and its major metabolites.

In milk, residues reached a plateau after about 5 days. Average residues of cyantraniliprole were 0.03 mg/kg in the 3.5 ppm dose group and increased to 0.7 mg/kg in the highest dose group (112 ppm). In skim milk, cyantraniliprole residues were about 60% of the whole milk levels (0.016 mg/kg up to 0.47 mg/kg) and 0.066 mg/kg up to 1.8 mg/kg in cream. Residues of IN-N7B69, the predominant metabolite in milk, increased from 0.03 mg/kg to 0.28 mg/kg over the four dose groups while residues of the other metabolites (IN-MLA84, IN-J9Z38 and IN-MYX98) were present at levels at least ten-fold lower than parent.

In muscle, maximum residues of cyantraniliprole increased from 0.01 mg/kg to 0.33 mg/kg in the four dose groups, with IN-J9Z38 being the predominant metabolite, found at more than

0.01 mg/kg only in the two highest dose groups (up to 0.04 mg/kg). Other metabolites were ⩽ 0.01 mg/kg at all dose levels.

In fat, maximum residues of cyantraniliprole increased from 0.015 mg/kg to 0.58 mg/kg in the four dose groups, with IN-J9Z38 being the predominant metabolite, found at 0.012 mg/kg (low dose) up to 0.45 mg/kg (highest dose). Other metabolites were found at lower levels, more than 5-fold lower that parent and only IN-N7B69 was present at more than 0.01 mg/kg, found at 0.02 mg/kg in the highest dose group.

In kidney, maximum residues of cyantraniliprole increased from 0.03 mg/kg to 0.89 mg/kg in the four dose groups, with IN-N7B69 being the predominant metabolite, found at 0.012 mg/kg (low dose) up to 0.15 mg/kg (highest dose). Other metabolites were found at levels more than 5-fold lower that parent, with residues of IN-J9Z38 and IN-MYX98 present above 0.01 mg/kg only in the two higher dose groups.

In liver, maximum residues of cyantraniliprole increased from 0.066 mg/kg to 2.1 mg/kg in the four dose groups, with IN-MLA84 being the predominant metabolite, present at 0.04 mg/kg in the lowest dose group and up to 0.57 mg/kg in the highest dose group. Metabolite IN-N7B69 residues were up to 0.01 mg/kg in the lowest dose group, increasing to 0.08 mg/kg in the highest dose group and other metabolites were all < 0.01 mg/kg except in the highest dose group where levels of < 0.03 mg/kg were found.

Residue depletion was studied in cows dosed orally for 28 days with the equivalent of 112 ppm cyantraniliprole. Parent residues depleted to < 0.01 mg/kg in muscle within 4 days after the last dose, were < 0.01 mg/kg in milk, liver and kidney within 10 days and < 0.01 mg/kg in fat within 15 days. Metabolites were all < 0.01 mg/kg in all matrices after 4 days except IN-J9Z38 (< 0.01 within 10 days in kidney and 15 days in fat), IN-MLA84 (< 0.01 mg/kg within 10 days in liver) and IN-N7B69 (< 0.01 mg/kg within 10 days in kidney).

The Meeting also received information on the residues in tissues and eggs when laying hens were dosed with cyantraniliprole for 28 days at levels equivalent to 3, 10 and 30 ppm in the diet. A separate dose group (30 ppm) was used to estimate residue depuration of cyantraniliprole and its major metabolites.

In eggs, residues reached a plateau after about 3 days. Average residues of cyantraniliprole were 0.08 mg/kg in the 3 ppm dose group and increased to 0.8 mg/kg in the highest dose group (30 ppm). In egg whites, cyantraniliprole was the predominant residue, averaging 0.08 mg/kg in the low dose group up to 0.64 mg/kg in the high dose group. Lower levels of parent were found in egg yolks, averaging 0.015 mg/kg (low dose) up to 0.1 mg/kg (high dose). Residues of IN-J9Z38, the predominant metabolite in eggs, present at levels of about 50% of parent, increased from 0.04 mg/kg to 0.4 mg/kg over the three dose groups while residues of IN-MLA84 and IN-MYX98 were present at levels of 0.015 mg/kg in the low dose group up to 0.12 in the high dose group. In general, residues of these metabolites were about 2-fold higher in egg whites than in the yolks.

In muscle, maximum residues of cyantraniliprole increased from 0.003 mg/kg to 0.05 mg/kg in the three dose groups, with the only metabolites found at more than 0.01 mg/kg being IN-MYX98 and IN-HGW87 (up to 0.02 mg/kg in the highest dose group. Other metabolites were all ⩽ 0.01 mg/kg at all dose levels.

In skin + fat, maximum residues of cyantraniliprole increased from 0.014 mg/kg to 0.16 mg/kg in the three dose groups, with IN-MYX98 being the predominant metabolite, found at 0.005 mg/kg (low dose) up to 0.05 mg/kg (highest dose). Other metabolites were found at lower levels, with IN-J9Z38 and IN-HGW87 present at more than 0.01 mg/kg only in the highest dose group (0.021 mg/kg and 0.023 mg/kg respectively).

In liver, maximum residues of cyantraniliprole increased from 0.03 mg/kg to 0.24 mg/kg in the three dose groups. Metabolite IN-MLA84 was present at levels similar to the parent (0.034 mg/kg in the lowest dose group and up to 0.32 mg/kg in the highest dose group). Maximum IN-MLA84, IN-

HGW87 and IN-N7B69 residues were 0.01–0.02 mg/kg in the lowest dose group and 0.07–0.1 mg/kg in the highest dose group. Other metabolites were all < 0.01 mg/kg in all dose groups.

In the residue depuration dose group (30 ppm), residues depleted to < 0.01 mg/kg in all matrices within 5 days of the last dose (within 9 days in liver, when the first sample was taken).

Animal commodity maximum residue levels

The maximum dietary burden for beef and dairy cattle is 1.9 ppm. The mean dietary burdens are 0.98 ppm (beef cattle) and 0.95 ppm (dairy cattle). Residue levels of cyantraniliprole and the metabolites included in the residue definition in milk and tissues were obtained by extrapolation below the 3.5 ppm feeding level in the dairy cow feeding study.

Cyantraniliprole feeding study	Feed level (ppm) for milk residues	Residues (mg/kg) in milk	Feed level (ppm) for tissue residues	Residues [a] (mg/kg) in			
				Muscle	Liver	Kidney	Fat
MRL beef or dairy cattle							
Feeding study [b]	3.5	0.03	3.5	0.011	0.066	0.031	0.015
Dietary burden and high residue	1.9	0.016	1.9	0.006	0.036	0.017	0.008
STMR beef or dairy cattle							
Feeding study [c]	3.5	0.03	3.5	0.008	0.094	0.042	0.024
Dietary burden and residue estimate	0.95	0.016	0.98	0.002	0.026	0.012	0.007

[a] Residue values used in estimating STMRs are the sum of cyantraniliprole and metabolites IN-N7B69, IN-J9Z38, IN-MLA84 and IN-MYX98, expressed as cyantraniliprole

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

[c] mean residues for tissues and mean residues for milk

Residues of cyantraniliprole expected in cattle milk and tissues for use in estimating maximum residue levels are: 0.008 mg/kg (fat), 0.006 mg/kg (muscle), 0.036 mg/kg (liver) and 0.017 mg/kg (kidney) and the mean residue for milk is 0.016 mg/kg.

The Meeting estimated maximum residue levels of 0.01 mg/kg for cyantraniliprole in meat (from mammals other than marine mammals), 0.05 mg/kg for edible offal (mammalian), 0.01 mg/kg for mammalian fat and 0.02 mg/kg for milks. Estimated STMRs (parent plus metabolites) for dietary intake estimation are 0.002 mg/kg for meat, 0.026 mg/kg for edible offal, 0.007 mg/kg for fat and 0.016 mg/kg for milk.

For poultry, the maximum dietary burden is 0.37 ppm and the mean dietary burden is 0.19 ppm (based on the diet for laying hens). Residue levels of cyantraniliprole and the metabolites included in the residue definition in eggs and tissues were obtained by extrapolation below the 3.0 ppm feeding level in the dairy cow feeding study.

Cyantraniliprole feeding study	Feed level (ppm) for egg residues	Residues (mg/kg) in egg	Feed level (ppm) for tissue residues	Residues [a] (mg/kg) in			
				Muscle	Liver	Skin	Fat
MRL broiler or laying hen							
Feeding study [b]	3.0	0.082	3.0	0.0055	0.03	0.014	
Dietary burden and high residue	0.37	0.01	0.37	0.0007	0.004	0.002	
STMR broiler or laying hen							
Feeding study [c]	3.0	0.082	3.0	0.007	0.062	0.016	
Dietary burden and residue estimate	0.19	0.01	0.19	0.0004	0.004	0.001	

[a] Residue values used in estimating STMRs are the sum of cyantraniliprole and metabolites IN-N7B69, IN-J9Z38, IN-MLA84 and IN-MYX98, expressed as cyantraniliprole

[b] highest residues for tissues and mean residues for egg

[c] mean residues for tissues and mean residues for egg

Residues of cyantraniliprole expected in eggs and poultry tissues for use in estimating maximum residue levels are: 0.002 mg/kg (skin plus fat), 0.0007 mg/kg (muscle), 0.004 mg/kg (liver) and the mean residue for eggs is 0.01 mg/kg.

The Meeting estimated maximum residue levels of 0.01 mg/kg for cyantraniliprole in poultry meat, 0.01 mg/kg for poultry offal, 0.01 mg/kg for poultry fat and 0.015 mg/kg for eggs. Estimated STMRs for dietary intake estimation are 0 mg/kg for poultry fat, 0 mg/kg for poultry meat, 0.004 mg/kg for poultry offal and 0.01 mg/kg for eggs.

RECOMMENDATIONS

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

Definition of the residue (for compliance with the MRL, animal and plant commodities): *cyantraniliprole*.

Definition of the residue (for estimation of dietary intake for unprocessed plant commodities): *cyantraniliprole*.

Definition of the residue (for estimation of dietary intake for processed plant commodities): *sum of cyantraniliprole and 2-[3-Bromo-1-(3-chloro-2-pyridinyl)-1H-pyrazol-5-yl]-3,4-dihydro-3,8-dimethyl-4-oxo-6-quinazolinecarbonitrile*.

Proposed definition of the residue (for estimation of dietary intake for animal commodities: sum of: *cyantraniliprole, 2-[3-Bromo-1-(3-chloro-2-pyridinyl)-1H-pyrazol-5-yl]-3,4-dihydro-3,8-dimethyl-4-oxo-6-quinazolinecarbonitrile, 2-[3-Bromo-1-(3-chloro-2-pyridinyl)-1H-pyrazol-5-yl]-1,4-dihydro-8-methyl-4-oxo-6-quinazolinecarbonitrile, 3-Bromo-1-(3-chloro-2-pyridinyl)-N-[4-cyano-2-(hydroxymethyl)-6-[(methylamino)carbonyl]phenyl]-1H-pyrazole-5-carboxamide and 3-Bromo-1-(3-chloro-2-pyridinyl)-N-[4-cyano-2[[(hydroxymethyl)amino]carbonyl]-6-methylphenyl]-1H-pyrazole-5-carboxamide*.

The residue is not fat soluble.

CCN	Commodity Name	MRL New	STMR or STMR-P	Highest residue [c]
VB 0040	Brassica (cole or cabbage) vegetables	2.0	0.56	1.1
FB 2006	Bush berries	4.0	0.75	
VS 0624	Celery	15	2.0	
FS 0013	Cherries	6.0	0.93	
SB 0716	Coffee beans	0.03	0.01	
DV 0444	Dried chilli peppers	5	0.7	
MO 0105	Edible offal (Mammalian)	0.05	0.026	
PE 0112	Eggs	0.015	0.01	
AM 1050	Fodder beet (follow-crop)	0.02	0.01	0.014
VC 0045	Fruiting vegetables, Cucurbits	0.3	0.065 [a] 0.01 [b]	

CCN	Commodity Name	MRL New	STMR or STMR-P	Highest residue [c]
VO 0050	Fruiting vegetables, other than Cucurbits (except mushrooms & sweetcorn)	0.5	0.08	
VA 0381	Garlic	0.05	0.02	
VL 0053	Leafy vegetables (except lettuce, Head)	20	4.7	
AL 0157	Legume animal feeds (follow-crop)	0.8 (dw)	0.017	0.58
VL 0482	Lettuce, Head	5	0.79	
MM 0069	Mammalian fat	0.01	0.007	
MM 0095	Meat (from mammals other than marine mammals)	0.01	0.002	
ML 0106	Milks	0.02	0.016	
VA 0385	Onion, bulb	0.05	0.02	
VA 0387	Onion, Welsh	8.0	1.3	
FS 2001	Peaches	1.5	0.34	
FS 0014	Plums	0.5	0.07	
FP 0009	Pome fruit	0.8	0.16	
VR 0589	Potato (follow-crop+foliar)	0.05	0.02	0.044
PO 0111	Poultry, Edible offal of	0.01	0.004	
PF 0111	Poultry fat	0.01	0	
PM 0110	Poultry meat	0.01	0	
DF 0014	Prunes	0.8	0.54	
VR 0075	Root and tuber vegetables except potato (follow-crop)	0.05	0.01	0.014
VA 0388	Shallot	0.05	0.02	
VA 0389	Spring onion	8.0	1.3	
AS 0161	Straw, fodder (dry) & hay of cereal grains and other grass-like plants (follow crop)	0.2 (dw)	0.01	0.14
AM 0506	Turnip fodder (fodder-crop)	0.02	0.01	0.014

[a] STMR is for cucurbits with edible peel

[b] STMR is for cucurbits with an inedible peel

[c] Highest residue values for calculating animal dietary burdens

CCN	Commodity Name		STMR or STMR-P	Highest residue [a]
JF 0226	Apple juice		0.05	

CCN	Commodity Name		STMR or STMR-P	Highest residue [a]
	Apple pomace (wet)		0.16	
	Cereal and grass forages (follow crop)		0.01	0.053
AV 1051	Fodder beet leaves or tops		0.01	0.021
	Legume forages (follow-crop)		0.01	0.14
AV 0480	Kale forage		0.01	0.021
	Potato waste (wet)		0.046	0.1
VL 0495	Rape greens		0.01	0.021
	Spinach (cooked)		4.7	
AV 0596	Sugar beet leaves or tops		0.01	0.021
	Tomato (canned)		0.004	
JF 0048	Tomato juice		0.014	
	Tomato paste		0.07	
	Tomato pomace (wet)		0.08	
AV 0506	Turnip leaves or tops		0.01	0.021

[a] Highest residue values for calculating animal dietary burdens

DIETARY RISK ASSESSMENT

Long-term intake

The International Estimated Daily Intake (IEDI) for cyantraniliprole was calculated for the food commodities for which STMRs or HRs were estimated and for which consumption data were available. The results are shown in Annex 3 of the 2013 JMPR Report.

The International Estimated Daily Intakes of cyantraniliprole for the 13 GEMS/Food regional diets, based on estimated STMRs were 1–10% of the maximum ADI of 0.03 mg/kg bw (Annex 3). The Meeting concluded that the long-term intake of residues of cyantraniliprole from uses that have been considered by the JMPR is unlikely to present a public health concern.

Short-term intake

The 2013 JMPR decided that an ARfD was unnecessary. The Meeting therefore concluded that the short-term intake of cyantraniliprole residues is unlikely to present a public health concern.

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CYPROCONAZOLE (239)

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APPRAISAL

Cyproconazole was first evaluated by the 2010 JMPR (T, R), when an ADI of 0–0.02 mg/kg bw and an ARfD of 0.06 mg/kg bw were set, and maximum residue levels were recommended for a variety of crops. Cyproconazole was scheduled at the Forty-fourth Session of the CCPR (2012) for the evaluation of residues in coffee by the 2013 JMPR. The residue definition for cyproconazole in commodities of plant and animal origin is the parent compound.

Methods of analysis

Two methods for the analysis of cyproconazole in coffee beans or roasted coffee were submitted. Residues can be extracted with acetone:water (95:5), the extract cleaned by gel permeation chromatography and the analyte analysed by LC/MS/MS (m/z 292.2 and 70.1), or extracted with methanol and analysed by LC/MS/MS after centrifugation. In both cases, satisfactory recoveries were obtained at 0.01 mg/kg (LOQ). No study was submitted to evaluate the stability of the residues in coffee samples. Studies evaluated by the 2010 JMPR on various fruits, peanuts and wheat had shown that cyproconazole residues are stable for at least 39 months under frozen conditions. The Meeting agreed that this conclusion could be extended to coffee.

Results from supervised residue trials on crops

Cyproconazole is registered in various central and South American countries. The critical GAP is found in Colombia, 3 × 0.06 kg ai/ha and 15 days PHI. A total of nine supervised residue trials were conducted on coffee from 2010 to 2012 in the region.

In two trials conducted in Brazil according to Colombian GAP, residues were 0.02 and 0.03 mg/kg. In two trials conducted in Colombia and one in Guatemala according to Colombian GAP, residues were 0.03 (2) and 0.04 mg/kg. Two declining studies conducted in Colombia according to GAP showed that residues at 10 and 14 days DAT are the same, and can be considered at GAP. Residues are 0.02 and 0.03 mg/kg.

In two other Brazilian trials, a soil drench application was applied in addition to a foliar application at 0.5 kg ai/ha, giving residues of 0.03 and 0.04 mg/kg at 23–37 DAT. Although these trials are not conducted according to GAP, they can be used as supporting information.

The Meeting agreed to combined the residues from trials conducted according to Colombian GAP (n=7) as 0.02 (2), 0.03 (4) and 0.04 mg/kg.

The Meeting estimates a maximum residue level of 0.07 mg/kg, and a STMR of 0.03 mg/kg for cyproconazole in coffee beans.

Fate of residues in processing

In one study conducted in Guatemala, green coffee beans containing 0.094 mg/kg cyproconazole were processed to roasted beans and instant coffee Residues were 0.119 mg/kg in roasted beans and 0.151 mg/kg in instant coffee, resulting in processing factors of 1.3 and 1.6, respectively.

Based on these processing factors, the Meeting recommends a maximum residue level of 0.1 mg/kg and a STMR of 0.039 mg/kg for cyproconazole in roasted coffee beans, and a STMR of 0.048 mg/kg for cyproconazole in instant coffee.

RECOMMENDATIONS

Definition of the residue for compliance with maximum residue levels and estimation of dietary intake in plant commodities: *cyproconazole*.

CCN	Commodity name	Maximum residue level (mg/kg)		STMR (P) mg/kg
		New	Previous	
SB 0716	Coffee beans	0.07		0.03
SM 0716	Coffee beans, roasted	0.1		0.039
	Instant coffee			0.048

DIETARY RISK ASSESSMENT

Long-term intake

The International Estimated Daily Intakes (IEDIs) of cyproconazole calculated for the 13 GEMS/Food Consumption Cluster Diets using STMRs and STMR-Ps estimated by the 2010 Meeting ranged from 0.5 to 2% of the maximum ADI. The impact of coffee on the IEDI is unlikely to affect the previous conclusion that the long-term intake of residues of cyproconazole resulting from the uses considered by the current JMPR is unlikely to present a public health concern.

Short-term intake

The International Estimated Short-Term Intakes (IESTI) of cyproconazole was calculated for coffee using STMR-P estimated by the current Meeting (Annex 4). The ARfD is 0.06 mg/kg and the calculated IESTI was 0% of the ARfD. The Meeting concluded that the short-term intake of residues of cyproconazole, when used in ways that have been considered by the JMPR, is unlikely to present a public health concern.