

PICOXYSTROBIN (258)

First draft prepared by Dr Samuel Margerison, Australian Pesticides and Veterinary Medicines Authority, Canberra, Australia

EXPLANATION

Picoxystrobin is a fungicide belonging to the strobilurin group of chemicals. It is a preventative and curative fungicide with systemic and translaminar movement, acting by inhibition of mitochondrial respiration by blocking electron transfer at the Q_o centre of cytochrome Bc1. It is used for control of a range of fungal diseases, including brown rust, tan spot, powdery mildew, and net blotch in cereals, pulses and oilseeds. At the Forty-third Session of the CCPR (2011), picoxystrobin was scheduled for evaluation as a new compound by the 2012 JMPR.

The Meeting received information on identity and physico-chemical properties, animal and plant metabolism, environmental fate in soil, rotational cropping, analytical methods, storage stability, use patterns, supervised residue trials, animal feeding studies, and the fate of residues in processing.

IDENTITY

Common name: Picoxystrobin

Chemical names

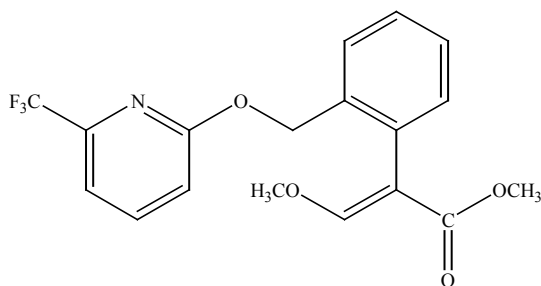
IUPAC: Methyl (E)-3-methoxy-2-[2-(6-trifluoromethyl-2-pyridyloxymethyl)-phenyl]acrylate

CAS: Methyl (E)-(α)-(methoxymethylene)-2-[[[6-(trifluoromethyl)-2-pyridinyl]oxy]methyl]benzeneacetate

CAS number: 117428-22-5

Synonyms: ZA 1963, DPX-YT669

Structural formula:



Molecular formula: C₁₈H₁₆F₃NO₄

Molecular weight: 367.3

SPECIFICATIONS

Specifications for picoxystrobin have not been developed by FAO.

PHYSICAL AND CHEMICAL PROPERTIES

Table 1 Physico-chemical properties of picoxystrobin

Property	Material	Method	Results	Guideline	Reference
Appearance	96.7%	Observation	Cream coloured solid	EPA OPPTS 830.6302,	Husband, 1999, RJ2678B

Property	Material	Method	Results	Guideline	Reference
	99.8%			830.6303	Wollerton and Husband, 1996, RJ2185B
Odour	96.7%	Olfactory	No characteristic odour	EPA OPPTS 830.6304	Husband, 1999, RJ2678B
	99.8%				Wollerton and Husband, 1996, RJ2185B
Melting point	99.66%	Capillary method	74.7 ± 0.2 °C	OECD Guideline 102, EEC Method A.1, EPA OPPTS 830.7200	Anand, 2007, DuPont-21190
	96.7%		71.9-74.3 °C		Husband, 1999, RJ2678B
	99.8%		75.0 °C		Wollerton and Husband, 1996, RJ2185B
Water solubility	99.66%	Shaken flask method with HPLC/UV analysis	3.25 ± 0.17 mg/L (20 °C)	OECD Guideline 105, EEC Method A.6, EPA OPPTS 830.7840	Hosmani, 2007, DuPont-21192
	99.8%	Column elution method with HPLC analysis	3.1 mg/L (20 °C)	CIPAC MT 157.1	Wollerton and Husband, 1996, RJ2185B
Density	96.7%	Pycnometer	1.40 g cm ⁻³ (20 °C)	OECD Guideline 109, EEC Method A.3, EPA OPPTS 830.7300	Husband, 1999, RJ2678B
	99.8%		1.40 g cm ⁻³ (20 °C)		Wollerton and Husband, 1996, RJ2185B
Bulk density	96.7%	Weighing	0.473 g cm ⁻³ (22 °C)	EPA OPPTS 830.7300	Husband, 1999, RJ2678B
Organic solvent solubility (20 °C)	96.7%	Shaken flask method with gravimetric or GC analysis	Xylene: > 200 g/L 1,2-dichloroethane: > 200 g/L Acetone: > 200 g/L Ethyl acetate: > 200 g/L n-Heptane: 4 g/L Methanol: 79 g/L	OECD Guideline 105, EEC Method A.6	Husband, 1999, RJ2678B
pH (1% w/v dispersion in water)	96.7%	pH meter	7.5 (20 °C)	CIPAC MT 75, OPPTS 830.7000	Husband, 1999, RJ2678B
	99.8%		5.6 (20 °C)		Wollerton and Husband, 1996, RJ2185B
Surface tension	96.7%	Torsion balance	71.1 mNm ⁻¹ (20 °C)	OECD Guideline 115, EC Method A.5	Husband, 1999, RJ2678B
Octanol/water partition coefficient	99.66%	Shaken flask method with	3.68 ± 0.01 (20 °C)	OECD Guideline 107,	Manjuntha, 2007, DuPont-21191

Property	Material	Method	Results	Guideline	Reference
(log ₁₀ K _{ow})	99.8%	HPLC/UV analysis	3.6 (20 °C)	EEC Method A.8	Wollerton and Husband, 1996, RJ2185B
Photolysis	Pyridinyl- ¹⁴ C-picoxystrobin and phenylacrylate- ¹⁴ C-picoxystrobin	Irradiation with a xenon lamp for a period equivalent to 30 summer days at 50° latitude, with analysis by TLC, LSC and HPLC	Mean DT ₅₀ = 20.3 days (summer at 50° latitude, 25 °C, pH 7). Major degradation products: IN-QCD12 (isomer) and IN-QGS44	EPA Guideline 161-2 and SETAC-Europe Guideline 10.0	Muller, 1998, RJ2403B
	Pyridinyl- ¹⁴ C-picoxystrobin	Irradiation in sterilised natural pond and pH 7 buffered water with a xenon lamp for a period equivalent to 42 summer days at 40° latitude, with analysis by TLC, LSC and HPLC	DT ₅₀ (pH 7, 25 °C) = 23.9 days DT ₅₀ (natural water, 25 °C) = 68 days DT ₉₀ (pH 7, 25 °C) = 79.5 days DT ₉₀ (natural water, 25 °C) = 226 days. Dark controls showed much slower degradation: DT ₅₀ = 383-1116 days, DT ₉₀ = 1273-3708 days Major degradation product: metabolite 12		Reibach and Freedlander, 2010, DuPont-26619
	Pyridinyl- ¹⁴ C-picoxystrobin	Irradiation in unsterilised natural and ultrapure water with a xenon lamp for a period equivalent to 16-30 summer days at 30° latitude, with analysis by LSC and TLC.	DT ₅₀ (natural water: Old Basing, 23 °C) = 6.1 days DT ₅₀ (natural water: Virginia Water, 23 °C) = 6.7 days DT ₅₀ (ultrapure water, 23 °C) = 15.7 days All results are reported as 30° latitude summer days. Very little degradation took place in the dark control samples.		Hepburn and Joseph, 1996, TMJ 3607B
Hydrolysis	Pyridinyl- ¹⁴ C-picoxystrobin plus unlabelled compound to give a specific activity of 3170 Bq/μg	Incubation in the dark at 25 or 50 °C for up to 32 days, with analysis by TLC, LSC and HPLC	pH 5, 7, 9 (25 °C): no hydrolysis observed pH 4, 7 (50 °C): no hydrolysis observed pH 9 (50 °C): half-life = 15 days. Major degradation products: IN-QDY62 and IN-QFA35.	EPA Guideline 161-1 and Official Journal of the European Commission Legislation (L 383 A: Method C7)	Powell, 1997, RJ2310B
Vapour pressure	99.66%	Gas saturation	0.0034 mPa (20 °C)	OPPTS 830.7950, EEC	Vijayakumar, 2007, DuPont-

Property	Material	Method	Results	Guideline	Reference
		method	0.0069 mPa (25 °C) 0.0491 mPa (40 °C) 0.0975 mPa (45 °C) 0.1647 mPa (50 °C)	Method A.4, OECD Guideline 104	21193
	99.8%		0.0055 mPa (20 °C) 0.014 mPa (25 °C) 1 mPa (50 °C)		Wollerton and Husband, 1996, RJ2185B
Henry's Law constant	99.66%	Calculation	$3.8 \times 10^{-4} \text{ Pam}^3\text{mol}^{-1}$ (20 °C)		Hosmani, 2007, DuPont-21192 and Vijayakumar, 2007, DuPont- 21193
	99.8%		$6 \times 10^{-4} \text{ Pam}^3\text{mol}^{-1}$ (20 °C)		Wollerton and Husband, 1996, RJ2185B
Quantum yield for direct phototransformation	99.8%	Quanta Count actinometer	0.48 (280 ± 10 nm, 20 °C, 50/50 water/acetonitrile)		Wollerton and Husband, 1996, RJ2185B
Calculated environmental half life	99.8%	Calculation	110 days – 1000 years		Wollerton and Husband, 1996, RJ2185B
Calculated environmental lifetime	99.8%		160 days -1500 years		Wollerton and Husband, 1996, RJ2185B

FORMULATIONS

The most commonly used formulation of picoxystrobin is a 250 g/L suspension concentrate.

METABOLISM AND ENVIRONMENTAL FATE

The metabolism of picoxystrobin has been investigated in wheat, soya beans, oilseed rape, rats, laying hens, and lactating goats. The crops selected represent the cereals and pulses/oilseeds groups, the groups for which supervised residue trials have been provided. Extensive investigations on the fate of picoxystrobin residues of soil have also been conducted. Studies were conducted using picoxystrobin labelled with carbon-14 in two locations. Structures are shown in Figure 1.

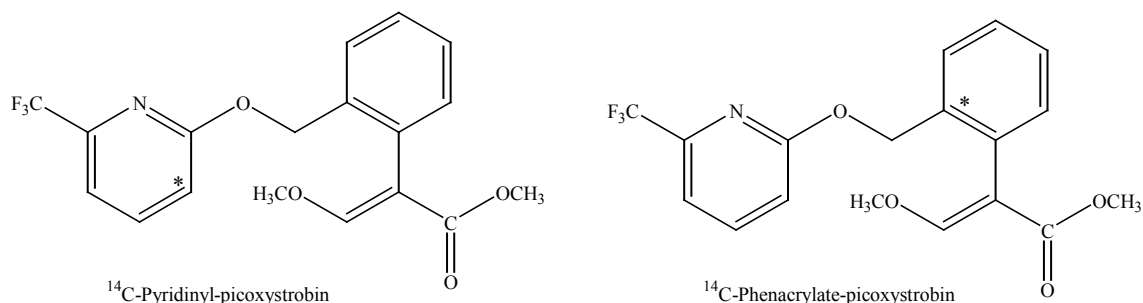
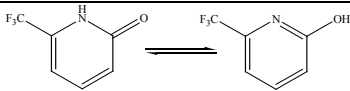
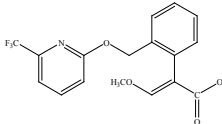
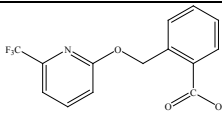
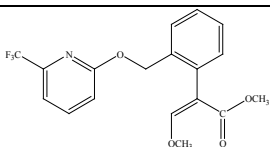
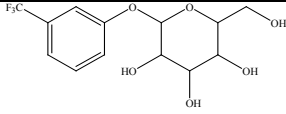
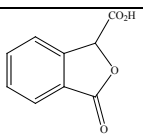
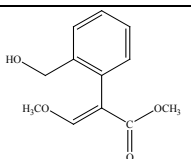
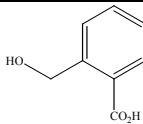


Figure 1 Positions of the ^{14}C labels for picoxystrobin used in metabolism studies

The structures of the metabolites and degradation products of picoxystrobin identified in the plant, animal and environmental metabolism studies are tabulated below.

Table 2 Metabolites and degradation products of picoxystrobin

Code	Chemical name	Structure	Metabolite origin
IN-QDK50, R403814, Metabolite 3	6-(Trifluoromethyl)-1 <i>H</i> -pyridin-2-one		Canola, wheat, hen, soil, rotational crops (wheat, lettuce, carrot)
IN-QDY62, Metabolite 2, R403092	(<i>E</i>)-3-Methoxy-2-[2-(6-trifluoromethyl-2-pyridyloxymethyl)-phenyl]acrylic acid		Canola, wheat, hen, goat, soil, rotational crops (wheat, carrot)
IN-QDY63, Metabolite 8, R408509	2-[2-(6-Trifluoromethyl-2-pyridyloxymethyl) benzoic acid		Canola, wheat, soya bean, goat, soil, rotational crops (carrots)
IN-QCD12, R407782, metabolite 4	Methyl (<i>Z</i>)-3-methoxy-2-[2-(6-trifluoromethyl-2-pyridyloxymethyl)-phenyl]acrylate		Canola, wheat
IN-QGS45, R409465, metabolite 11	2-Glucosyl-6-(trifluoromethyl)pyridine		Canola, wheat, rotational crops (wheat, lettuce, carrot)
IN-H8612, R135305, metabolite 24	1,3-Dihydro-3-oxoisobenzofuran-1-carboxylic acid		Wheat, soya bean, rotational crops (wheat)
IN-QDY60, R233331, metabolite 9	Methyl (<i>E</i>)-3-methoxy-2-(2-hydroxymethylphenyl)acrylate		Wheat, goat
IN-10975, R277643, metabolite 21	2-Hydroxymethylbenzoic acid		Wheat

Code	Chemical name	Structure	Metabolite origin
IN-QGS44, R410101, metabolite 12	Methyl 2-hydroxy-2-[2-(6-trifluoromethyl-2-pyridyloxymethyl)phenyl]acetate		Wheat
IN-QGU66, R407748, metabolite 13	Methyl 2-oxo-2-[2-(6-trifluoromethyl-2-pyridyloxymethyl)phenyl]acetate		Wheat, goat
IN-QGS46, R410639, metabolite 14	2-Hydroxy-2-[2-(6-trifluoromethyl-2-pyridyloxymethyl)phenyl]acetic acid		Wheat, soya bean, goat
IN-QGS46-glucoside, R410639 glucoside	2-Glucosyl-2-[2-(6-trifluoromethyl-2-pyridyloxymethyl)phenyl]acetic acid		Soya bean
IN-QGS46-decarboxy glucoside, R410639-decarboxy glucoside	2-[2-(2-Glucosyl-1-hydroxyethyl)phenylmethoxy]-6-(trifluoromethyl)pyridine		Soya bean
IN-QGU69, R290445, metabolite 32	Methyl 3-hydroxy-2-[2-(6-trifluoromethyl-2-pyridyloxymethyl)phenyl]propionate		Wheat, goat
Hydroxy-IN-QGU69, R290446, metabolite 33	Methyl 3-hydroxy-2-[2-(6-trifluoromethyl-2-pyridyloxymethyl)phenyl]propionate		Wheat
IN-QGU72, R415833, metabolite 20	2-Malonylglucosyl-6-trifluoromethylpyridine		Wheat, rotational crops (wheat, lettuce, carrot)
IN-K2122, R001731, metabolite 15	Phthalic acid		Wheat, soya bean
PAG3, R730529	2-(2-Hydroxymethylphenyl)-2-oxoacetic acid		Wheat

Code	Chemical name	Structure	Metabolite origin
-	2-(2-Formylphenyl)-2-oxoacetic acid		Soya bean
IN-QFA35, R408631, metabolite 7	2-[2-(6-Trifluoromethyl-2-pyridyloxymethyl)phenyl]acetic acid		Projected soya bean intermediate, rotational crops (wheat, carrots), hen, goat
IN-QFA35 glucoside	Glucosyl 2-[2-(6-trifluoromethyl-2-pyridyloxymethyl)phenyl]acetate		Soya bean
IN-QGU73, R414535, metabolite 29	Mixture of isomers, where n = 3, 4 or 6 2-{n-(3-Hydroxy-3-methylglutaryl)glucosyl}-6-trifluoromethylpyridine		Soya bean, rotational crops (carrots)
R290447, metabolite 34	Methyl (E)-3-methoxy-2-[n-hydroxy-2-(6-trifluoromethyl-2-pyridyloxymethyl)-phenyl]acrylate		Goat
R290450, metabolite 37	Methyl (E)-3-hydroxy-2-[n-hydroxy-2-(6-trifluoromethyl-2-pyridyloxymethyl)-phenyl]acrylate		Goat
R290463, metabolite 50	3-Hydroxy-2-[2-(6-trifluoromethyl-2-pyridyloxymethyl)-phenyl]propionic acid		Goat
IN-QCD09, R404843, metabolite 10	Methyl 2-[2-(6-trifluoromethyl-2-pyridyloxymethyl)-phenyl]acetate		Hen, goat
R290449, metabolite 36	2-[n-Hydroxy-2-(6-trifluoromethyl-2-pyridyloxymethyl)-phenyl]acetic acid		Goat

Code	Chemical name	Structure	Metabolite origin
R290461, metabolite 48	Methyl 2,3-dihydroxy-2-[2-(6-trifluoromethyl-2-pyridyloxymethyl)-phenyl]propionate		Soya bean, goat
R290458, metabolite 45	Methyl (<i>E</i>)-3-hydroxy-2-[2-(6-trifluoromethyl-2-pyridyloxymethyl)-phenyl]acrylate		Projected intermediate for wheat and goat
IN-S7529, R206576, metabolite 18	Tetrahydro-2-benzopyran-3-one		Goat
IN-QGY55	Glucosyl (<i>E</i>)-3-methoxy-2-[2-(6-trifluoromethyl-2-pyridyloxymethyl)-phenyl]acrylate (glucosyl-IN-QDY62)		Rotational crops (wheat, lettuce, carrot)
R416021, metabolite 31	(<i>E</i>)-2-Oxo-2-[2-(6-trifluoromethyl-2-pyridyloxymethyl)-phenyl]acetic acid		Soil
R409665, metabolite 30	2-(6-Trifluoromethyl-2-pyridyloxy)acetic acid		Soil, rotational crops (wheat, lettuce, carrot)
PYST2, R290452	6-Trifluoromethyl-2-pyridylsulfuric acid		Rotational crops (wheat)
R290461 malonyl glucose conjugate	Methyl 3-glucosyl-2-malonyl-2-[2-(6-trifluoromethyl-2-pyridyloxymethyl)-phenyl]propionate		Soya bean

Code	Chemical name	Structure	Metabolite origin
R290461 glucosides, R ₁ = H, R ₂ = glucose, or R ₁ = glucose, R ₂ = H	Mixture of glucose conjugates of methyl 2,3-dihydroxy-2-[2-(6-trifluoromethyl-2-pyridyloxymethyl)-phenyl]propionate		Soya bean
Malonyl glucose conjugate of decarboxylate d IN-QGS46			Soya bean

Animal metabolism

The Meeting received studies on the metabolism of picoxystrobin in rats, lactating goats and laying hens.

Rats

The metabolism of [¹⁴C]pyridinyl- and [¹⁴C]phenacrylate-picoxystrobin in rats was investigated and was evaluated by the WHO Panel of the 2012 JMPR. A summary is provided below.

Radiolabelled picoxystrobin administered by oral gavage is rapidly absorbed with peak plasma ¹⁴C levels seen at approximately 2 or 12 hours in rats administered 10 or 100 mg/kg bw respectively. Picoxystrobin is well absorbed with approximately 70% of the radioactivity from an oral dose of 100 mg/kg bw detected in bile and urine. Distribution is extensive, with peak radioactivity levels being detected in liver, pancreas, kidney, and blood plasma. Excretion is predominantly via the bile and thence into faeces and is essentially complete within 120 hours for a dose of 100 mg/kg bw. Excretion in urine was greater in females (approximately 30%) than in males (approximately 20%). Picoxystrobin is extensively metabolised with over 30 identified metabolites. Significant biotransformation reactions include ester hydrolysis, oxidation, O-demethylation and glucuronide conjugation.

Lactating goats

Metabolism of [¹⁴C]pyridinyl- and [¹⁴C]phenacrylate-picoxystrobin was investigated (Webb and Robertson, 1998) in two lactating goats (goats 1 and 2 respectively). The dose was administered orally by capsule twice daily immediately after milking for 7 days, at 10 ppm for goat 1 and 13.5 ppm for goat 2, corresponding to 0.24 and 0.30 mg/kg bw/day. Milk samples were collected twice daily and excreta and cage wash samples collected daily throughout the dosing period as well as a day prior to commencement of dosing. The animals were sacrificed 16 hours after the last doses, and samples of fat (renal, subcutaneous and omental), muscle (forequarter and hindquarter), kidney, liver, gastrointestinal tract and contents, whole blood and plasma, cage washings, bile and remaining urine in the bladder were collected.

Milk samples were centrifuged into cream and skim milk, and skim milk partitioned with ethyl acetate to aid characterisation and identification of residue components. Tissue samples were extracted using a range of solvents (acetonitrile, acetonitrile/water, acetone and hexane). Liver samples were further extracted by weak and strong base hydrolysis and liver, kidney and urine samples were subjected to enzymatic hydrolysis (β -glucuronidase or sulphatase).

The extracts were analysed by thin layer chromatography to quantify the metabolites. Metabolites were identified using reference standards and metabolites isolated from urine. HPLC was used to confirm the specific activity of the test substances and for resolution of the metabolites in urine for identification by LC/MS.

The largest percentage of the administered dose was excreted in urine (46.3% for the pyridinyl label and 49.4% for the phenacrylate label), followed by faeces (35.6% and 27.3% for the pyridinyl and phenacrylate labels respectively). Smaller amounts were found in cage wash and the gastrointestinal tract and contents. Total residue eliminated in milk was 0.20% and 0.06% of the administered dose for the pyridinyl and phenacrylate labels respectively. The total recovery of the administered dose was 85.0% and 81.7% for the pyridinyl and phenacrylate labels respectively.

Table 3 Recovery of the administered dose of radiolabelled picoxystrobin

Sample	¹⁴ C-Pyridinyl label (% dose)	¹⁴ C-Phenacrylate label (%dose)
Urine	46.32	49.41
Faeces	35.61	27.28
GI tract and contents	2.03	2.76
Cage wash	0.67	1.59
Milk	0.20	0.06
Bile	0.03	0.11
Bladder urine at sacrifice	-	0.24
Liver	0.11	0.20
Kidney	0.01	0.02
TOTAL	85.03	81.67

Table 4 Total radioactive residues in milk

Collection interval (hours)	Residue (mg picoxystrobin equivalents/kg)	
	[¹⁴ C]Pyridinyl label	[¹⁴ C]Phenacrylate label
Pre-dose	0	0
8	0.006	0.004
24	0.008	0.006
32	0.011	0.008
48	0.010	0.007
56	0.011	0.008
72	0.009	0.007
80	0.012	0.010
96	0.010	0.006
104	0.011	0.008
120	0.010	0.007
128	0.012	0.008
144	0.010	0.007
152	0.012	0.008
168	0.006	0.006

Residues in milk reached a plateau by day 4, with maximum total radioactive residue of 0.012 and 0.010 mg/kg parent equivalents (mg eq/kg) being observed for goats 1 and 2 respectively.

The total residues found in tissues are tabulated below.

Table 5 Total radioactive residues in tissues of lactating goats

Tissue	Residue (mg picoxystrobin equivalents/kg)	
	[¹⁴ C]Pyridinyl label	[¹⁴ C]Phenacrylate label
Forequarter muscle	0.007	0.009
Hindquarter muscle	0.006	0.010
Renal fat	0.028	0.026
Omental fat	0.034	0.025
Subcutaneous fat	0.033	0.021
Liver	0.12	0.34
Kidney	0.057	0.15
Bile	0.85	5.1
Blood	0.025	0.058
Plasma	0.033	0.073
Gastrointestinal tract and contents	0.26	0.48

Insufficient residues (approximately 0.01 mg eq/kg or less) were found in milk and muscle samples and their extracts to warrant further analysis and residue characterisation and identification.

Components of the residue identified in fat, liver and kidney samples are tabulated below.

Table 6 Picoxystrobin residues identified and characterised in goat liver

Residue component	[¹⁴ C]Pyridinyl label		[¹⁴ C]Phenacrylate label	
	mg/kg equivalents	%TRR	mg/kg equivalents	%TRR
Parent	0.003	2.7	0.003	1.0
IN-QDY62	0.007	6.0	0.017	5.2
IN-QDK50	0.005	4.4	–	–
IN-QFA35	0.004	3.3	0.013	3.9
IN-QDY63	< 0.001	0.2	–	–
IN-QDY60	–	–	0.001	0.3
IN-QCD09	0.001	0.7	0.004	1.1
IN-QGU66	0.001	1.1	0.004	1.1
IN-QGS46	–	–	0.002	0.6
IN-S7529	–	–	< 0.001	0.1
IN-QGU69	0.002	1.5	0.008	2.4
R290447	0.001	0.5	0.007	2.1
R290449	0.001	0.7	0.003	0.9
R290461	0.003	2.3	0.006	1.8
R290463	0.001	1.2	0.010	3.1
Unidentified organosoluble metabolites	0.002	2.1	0.023	7.2

Picoxystrobin

Residue component	¹⁴ C]Pyridinyl label		¹⁴ C]Phenacrylate label	
	mg/kg equivalents	%TRR	mg/kg equivalents	%TRR
Unresolved fractions	0.008	7.0	0.056	17.6
Aqueous fractions (not chromatographed)	0.029	26.3	0.056	17.6
Filter papers	0.004	4.1	0.028	8.8
Precipitates	0.014	12.5	0.024	7.5
Unextracted residues	0.009	7.6	0.007	2.3
Total	0.096	84.2	0.273	84.6

Table 7 Picoxystrobin residues identified and characterised in goat kidney

Residue component	¹⁴ C]Pyridinyl label		¹⁴ C]Phenacrylate label	
	mg/kg equivalents	%TRR	mg/kg equivalents	%TRR
Parent	0.002	3.8	0.004	2.5
IN-QDY62	0.002	3.1	0.004	2.6
IN-QFA35	0.008	15.1	0.020	14.0
IN-QDY63	< 0.001	0.5	0.001	0.9
IN-QDY60	–	–	0.001	0.9
IN-QCD09	0.002	2.9	0.004	3.0
IN-QGU66	0.001	2.0	0.006	3.9
IN-QGU69	0.002	2.9	0.005	3.4
R290447	0.001	1.0	0.001	0.6
R290461	0.001	2.6	0.003	2.0
R290463	0.002	2.8	0.004	3.0
Unidentified organosoluble metabolites	0.003	5.9	0.016	11.2
Unresolved fractions	0.004	7.8	0.005	3.8
Aqueous fractions (not chromatographed)	0.010	18.4	0.026	18.2
Unextracted residues	0.014	25.8	0.026	18.3
Total	0.053	94.6	0.126	88.3

Table 8 Picoxystrobin residues identified and characterised in goat fat

Omental fat				
Component	¹⁴ C]Pyridinyl label		¹⁴ C]Phenacrylate label	
	mg/kg equivalents	%TRR	mg/kg equivalents	%TRR
Parent	0.024	81.0	0.018	76.2
Unidentified organosoluble metabolites	0.002	7.7	0.002	9.5
Unresolved fractions	0.001	3.4	< 0.01	1.0
Organosoluble fractions not	< 0.001	0.1	–	–

Omental fat				
Component	¹⁴ C]Pyridinyl label		¹⁴ C]Phenacrylate label	
	mg/kg equivalents	%TRR	mg/kg equivalents	%TRR
chromatographed				
Unextracted residues	0.002	6.3	0.002	9.9
Total	0.030	98.5	0.023	96.6
Renal fat				
Component	¹⁴ C]Pyridinyl label		¹⁴ C]Phenacrylate label	
	mg/kg equivalents	%TRR	mg/kg equivalents	%TRR
Parent	0.021	71.8	0.018	75.4
Unidentified organosoluble metabolites	0.002	5.4	0.002	7.8
Unresolved fractions	0.001	3.6	< 0.001	1.0
Organosoluble fractions not chromatographed	0.003	12.4	–	–
Unextracted residues	0.002	7.4	0.003	12.3
Total	0.029	100.6	0.023	96.5
Subcutaneous fat				
Component	¹⁴ C]Pyridinyl label		¹⁴ C]Phenacrylate label	
	mg/kg equivalents	%TRR	mg/kg equivalents	%TRR
Parent	0.022	70.0	0.012	54.9
Unidentified organosoluble metabolites	0.002	5.1	0.003	14.3
Unresolved fractions	0.001	2.6	0.001	5.2
Organosoluble fractions not chromatographed	0.005	16.9	–	–
Unextracted residues	0.002	5.2	0.004	19.8
Total	0.032	99.8	0.020	94.2

In fat, picoxystrobin was the major component of the residue, ranging from 54.9% to 81.0% of the TRR. No other components were identified in fat.

Metabolism in liver and kidney was considerably more complex.

In liver, parent compound was found at only 0.003 mg eq/kg for both labels (2.7% and 1.0% of the TRR for the pyridinyl and phenacrylate labels). Fourteen metabolites were identified at low levels. No identified residue component exceeded 10% of the TRR, while only IN-QDY62 and IN-QFA35 exceeded 0.01 mg eq/kg. IN-QDY62 was found at 0.007 mg eq/kg (6.0% TRR) and 0.017 mg eq/kg (5.2% TRR) in the pyridinyl and phenacrylate goats respectively, while IN-QFA35 was found at 0.004 mg eq/kg (3.3% TRR) and 0.013 mg eq/kg (3.9%).

In kidney, parent compound was again a minor component of the residue, being found at 0.002 mg eq/kg (3.8% TRR) and 0.004 mg eq/kg (2.5% TRR) in the pyridinyl and phenacrylate goats respectively. Ten metabolites were identified, as with liver these were at low levels with only one, IN-QFA35, exceeding 10% of the TRR and 0.01 mg eq/kg. IN-QFA35 was found at 0.008 mg eq/kg (15.1% TRR) for the pyridinyl label and 0.020 mg eq/kg (14.0% of TRR) for the phenacrylate label.

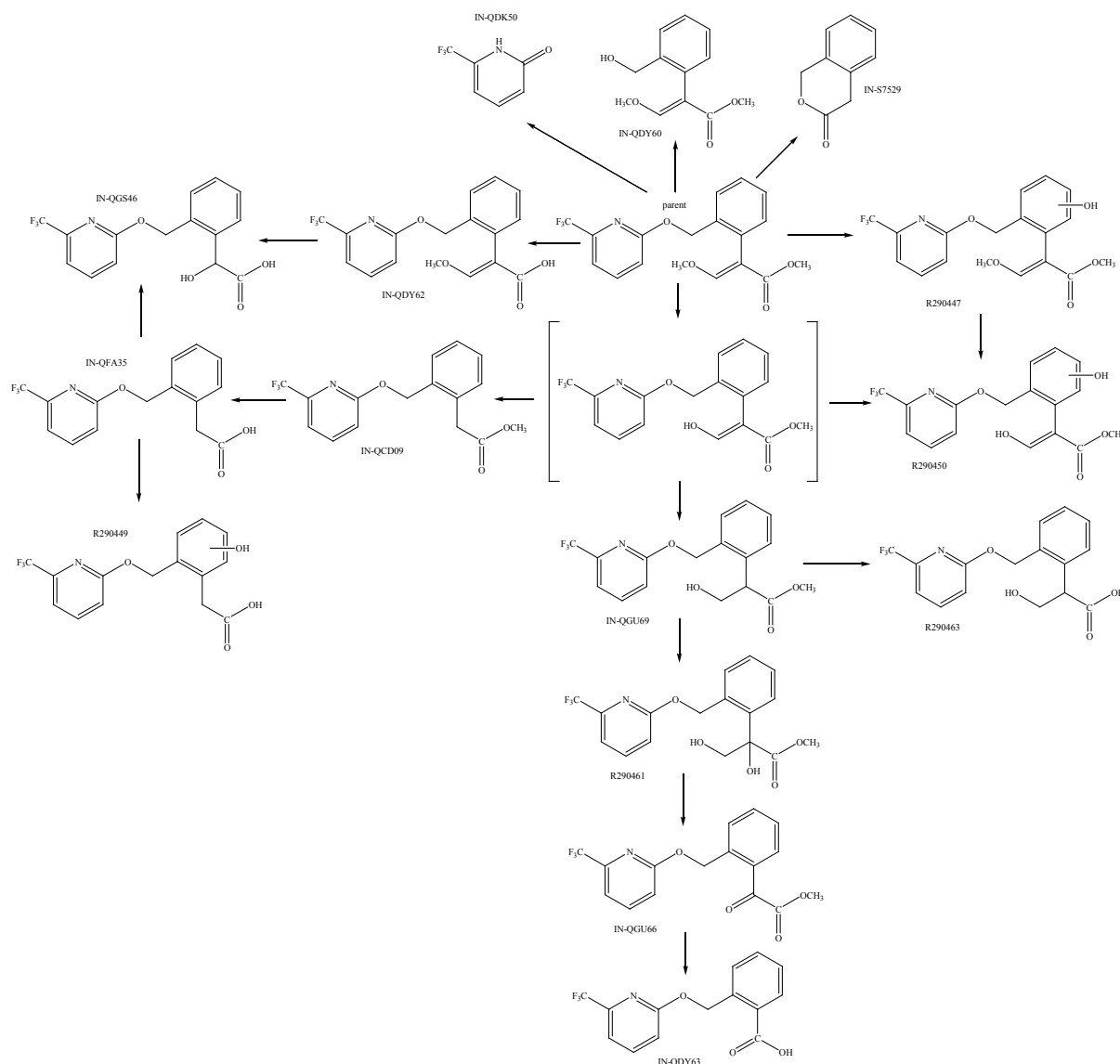


Figure 2 Proposed metabolic pathways for picoxystrobin in lactating goats

Laying hens

Metabolism of [^{14}C]pyridinyl- and [^{14}C]phenacrylate-picoxystrobin was investigated (Robertson *et al.* 1998) in laying hens (three birds per treatment). The hens were dosed orally by capsules administered twice daily for ten days at 11.3 ppm for the [^{14}C]pyridinyl label and 10.9 ppm for the [^{14}C]phenacrylate label, corresponding to 0.95 and 0.88 mg/kg bw/day respectively. Eggs, excreta and cage washings were sampled daily, with cage washings also being collected at sacrifice. The birds were sacrificed 16 hours after the last treatment, and samples of muscle (thigh and breast), abdominal fat, and liver were collected. All samples destined for analysis at the laboratories were stored deep frozen (around $-20\text{ }^{\circ}\text{C}$) between collection and during transport, until extraction and analysis.

Only egg yolk and excreta samples (those collected at 240 hours) were extracted. These were extracted with a range of solvents, including acetonitrile, acetonitrile/water and hexane. Enzymatic hydrolysis (β -glucuronidase) was used to deconjugate metabolites in aqueous excreta extracts. Solvent-solvent partitions (e.g., aqueous/chloroform) were employed in order to further characterise residues as polar or organosoluble, while solid phase extraction (C18 column) was used to effect clean-up of some extracts.

The extracts were analysed by thin layer chromatography to quantify the metabolites, using a range of solvent systems and detection with a bio-imaging analyser. Metabolites were identified using reference standards. HPLC was used to confirm the specific activity of the test substances and for resolution of the metabolites in excreta for identification by LC/MS.

It is noted that egg yolk samples were extracted 10 months after collection, with analysis being completed around 2 months after extraction. Samples were stored deep frozen between collection and analysis. Storage stability data was not provided.

The majority (64.7% and 93.8% for the pyridinyl and phenacrylate labels respectively) of the administered dose was excreted. Around 2% of the total dose was found in cage washings, with much smaller amounts recovered from eggs (maximum 0.10% for the pyridinyl label in egg yolks) and tissues (maximum 0.14% for phenacrylate label liver). The total recovery of the dose from the phenacrylate birds was almost quantitative, at 95.9%, compared with only 67.7% for the pyridinyl label, suggesting a loss of material due to poor recovery of excreta. Full details of the dose recovery are shown in Table 9.

Table 9 Percentage recovery of the administered dose of picoxystrobin (mean of the three birds)

Sample	[¹⁴ C]Pyridinyl label	[¹⁴ C]Phenacrylate label
Excreta	64.7	93.8
Egg yolk	0.10	0.08
Egg white	0.02	0.01
Muscle	0.04	0.05
Fat	0.02	0.01
Liver	0.07	0.14
Cage washings	2.7	1.9
TOTAL	67.7	95.9

Total residues in eggs are tabulated below (Tables 10–11). Residues appeared to reach a plateau around Day 8–10 in both yolks and whites, at 0.015 and 0.006 mg parent equivalents/kg (mg eq/kg) in egg whites for the pyridinyl and phenacrylate labels respectively, and at around 0.21 and 0.19 mg eq/kg in egg yolks for the pyridinyl and phenacrylate labels respectively. Residues were significantly higher (approximately 10–30×) in yolks than in whites for both labels.

Table 10 Total radioactive residues in eggs (range and daily mean of the three birds)

Sample timing (hours)	Total residue (mg parent equivalents/kg)			
	[¹⁴ C]Pyridinyl label		[¹⁴ C]Phenacrylate label	
	Egg whites	Egg yolks	Egg whites	Egg yolks
Pre-dose	0.0000–0.0001 (0)	0	0	0
g	0.0027–0.016 (0.009)	0	0.0005–0.0038 (0.0017)	0.0001–0.0003 (0.0001)
48	0.0083–0.017 (0.012)	0.013–0.023 (0.018)	0.0025–0.0063 (0.0043)	0.0043–0.010 (0.0063)
72	0.0077–0.015 (0.012)	0.040–0.058 (0.048)	0.0029–0.0045 (0.0039)	0.021–0.031 (0.026)
96	0.0093–0.012 (0.0104)	0.0660–0.1031 (0.0842)	0.0042–0.0059 (0.0048)	0.0465–0.0642 (0.0527)
120	0.0093–0.012 (0.010)	0.094–0.15 (0.11)	0.0043–0.0060 (0.0051)	0.067–0.082 (0.074)
144	0.0095–0.015 (0.012)	0.13–0.20 (0.15)	0.0040–0.0089 (0.0058)	0.092–0.13 (0.11)
168	0.0128–0.0129 (0.0129)	0.15–0.23 (0.19)	0.0038–0.0057 (0.0048)	0.12–0.16 (0.14)
192	0.0097–0.013 (0.011)	0.17–0.26 (0.20)	0.0038–0.0065 (0.0048)	0.14–0.18 (0.16)

Sample timing (hours)	Total residue (mg parent equivalents/kg)			
	[¹⁴ C]Pyridinyl label		[¹⁴ C]Phenacrylate label	
	Egg whites	Egg yolks	Egg whites	Egg yolks
216	0.012–0.015 (0.014)	0.18–0.24 (0.20)	0.0060–0.0069 (0.0064)	0.19–0.19 (0.19)
240	0.013–0.018 (0.015)	0.18–0.25 (0.21)	0.0040–0.0061 (0.0051)	0.17–0.21 (0.19)

Levels of residue in tissues reached a maximum of 0.023 mg eq/kg for muscle, 0.070 mg eq/kg for fat and 0.33 mg eq/kg for liver. There was relatively little difference in the residues for the two labels for muscle and fat, while levels in liver for the phenacrylate label were around twice those for the pyridinyl label.

Table 11 Total radioactive residues in tissues (range and mean of the three birds in each dosing group)

Sample	Total residue (mg parent equivalents/kg)	
	[¹⁴ C]Pyridinyl label	[¹⁴ C]Phenacrylate label
Muscle	0.0185–0.0198 (0.0191)	0.0204–0.0232 (0.0219)
Fat	0.0485–0.0591 (0.0537)	0.0277–0.0702 (0.0483)
Liver	0.155–0.192 (0.173)	0.293–0.328 (0.309)

Given the low levels of total residue observed in most samples, only 240 hour egg yolk and excreta were extracted and chromatographed, in order to provide some information on the metabolism of picoxystrobin in hens. Egg yolk was extracted with acetonitrile, acetonitrile/water, and hexane, which together extracted 71% and 32% of the radioactive residue for the pyridinyl and phenacrylate labels. No further attempt was made to characterise the unextracted residue.

Table 12 Identification and characterisation of residues in 240 hour egg yolk

Residue component	[¹⁴ C]Pyridinyl label		[¹⁴ C]Phenacrylate label	
	mg eq/kg	%TRR	mg eq/kg	%TRR
Parent	0.005	2.2	0.003	1.3
IN-QDK50	0.003	1.4	–	–
IN-QFA35	0.005	2.2	0.002	0.9
IN-QCD09	0.001	0.5	–	–
Unidentified organosoluble metabolites ^a	0.084	39.2	0.038	19.6
Unresolved fractions	0.033	15.4	0.020	10.2
Unchromatographed fractions	0.005	2.5	–	–
Unextracted residues	0.062	29.0	0.134	68.5
Total	0.198	92.4	0.197	100.5

^a At least 15 components, maximum individual component 0.026 (12.2% TRR).

The components found in 240 hour egg yolk (parent, and the metabolites IN-QDK50, IN-QFA35 and IN-QCD09) were also found in 240 hour excreta, along with IN-QDY62 (demethylated picoxystrobin), and hydroxyl-IN-QGU69, R290447 and R290461 (phenyl ring or propyl chain hydroxylated compounds). The latter 3 were identified with the aid of LC/MS.

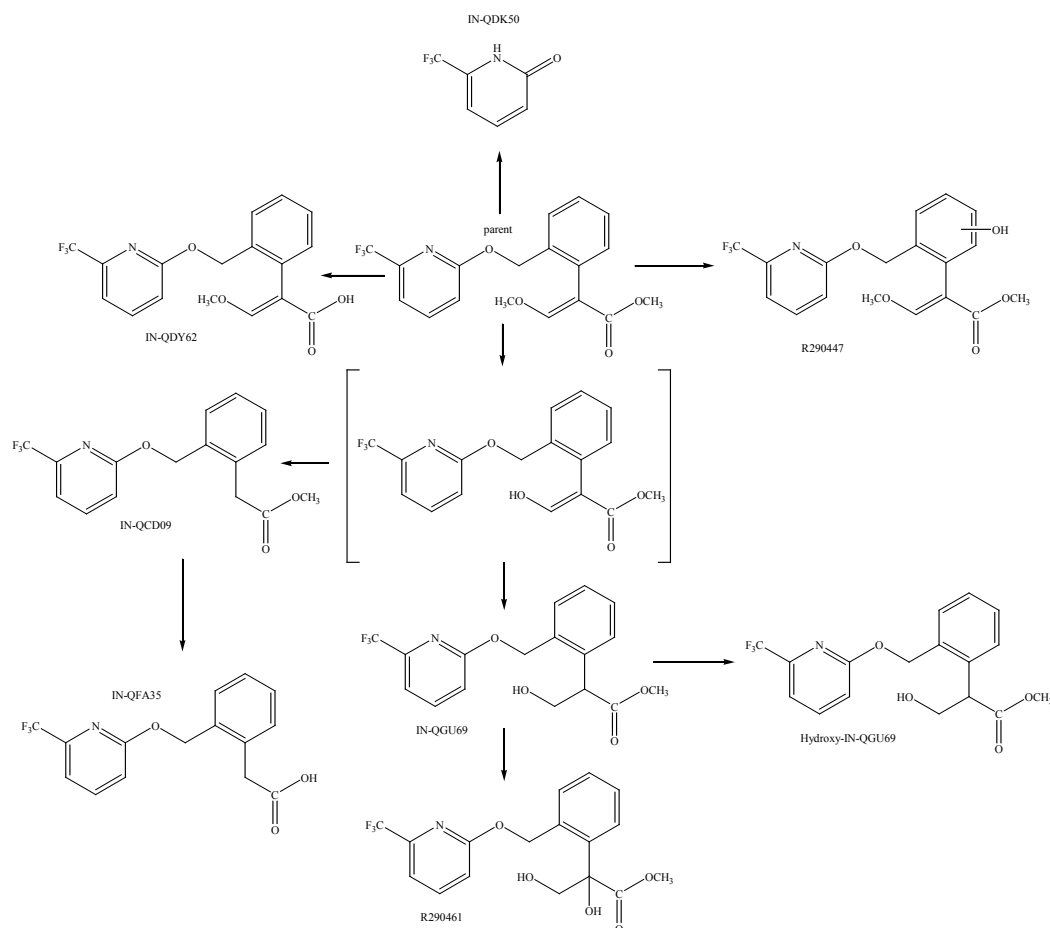


Figure 3 Proposed metabolic pathway for picoxystrobin in laying hens

Summary of animal metabolism

The metabolism of picoxystrobin was similar in lactating goats and laying hens, while rat metabolism was considerably more extensive. Important metabolic pathways were:

Oxidative cleavage of the molecule at the ether bridge to yield 6-(trifluoromethyl)-1*H*-pyridin-2-one and methyl (*E*)-2-(2-hydroxymethylphenyl)3-methoxyacrylate. Only the pyridine moiety metabolite was found in hens, while both metabolites were found in goats. In hens, the significantly lower recovery of radioactivity for the pyridine label experiment compared with the phenacrylate label experiment suggested poor recovery of excreta for the pyridinyl experiment, since amounts recovered in other samples were similar to those for the pyridine label.

Hydrolysis of the methyl ester.

Loss of the methoxy methyl group, with or without subsequent hydroxylation of the carbon side chain, and/or hydrolysis of the methyl ester.

Cleavage of the acrylate side chain at the 2 position to yield phenyl acetate metabolites, with or without subsequent hydrolysis of the methyl ester, and/or hydroxylation at the 2 position.

Hydroxylation of the phenyl ring.

All major metabolites in goats and hens (> 10% and/or > 0.01 mg/kg) were present in rats.

Conjugation of metabolites was not observed in hens or goats; however, glucuronide and sulphate conjugation occurred in rats.

Plant metabolism

Studies were performed on wheat, oilseed rape and soya beans, using [¹⁴C]pyridinyl- and [¹⁴C]phenacrylate-labelled picoxystrobin.

Wheat

In this study (Emburey *et al.*, 1998), field grown winter wheat (Hussar variety) was treated twice by foliar application with either [¹⁴C]pyridinyl-picoxystrobin or [¹⁴C]phenacrylate-picoxystrobin formulated as a suspension concentrate, at Zadok's growth stages 32 and 65–69 (2nd node and mid to late flowering respectively). The total seasonal application rates were 842 and 817 g ai/ha for the pyridinyl and phenacrylate labels respectively (individual application rates ranged from 405 to 437 g ai/ha). Forage was sampled 14 days after the second application, while mature wheat grain and straw were collected at normal harvest, 48 days after the second application. Samples were stored frozen (-20 °C) prior to analysis.

Samples were homogenised, then extracted with a range of solvents (acetonitrile, acetonitrile/water, and water). Solvent-solvent partition was employed for some extracts for further characterisation of radioactivity, and some extracts were cleaned up by solid phase extraction. Grain and straw samples were further subjected to enzymatic hydrolysis and mild base hydrolysis.

Solvent extracts were analysed by TLC and HPLC, with LC/MS/MS being used to confirm the identity of some components.

Table 13 Total radioactive residues in wheat matrices (by direct LSC analysis of the samples)

Sample	Residue (mg/kg parent equivalents)	
	Pyridinyl label	Phenacrylate label
Forage	3.67	6.37
Grain	0.079	0.307
Straw	11.2	12.2

Table 14 Identification of [¹⁴C]pyridinyl-picoxystrobin metabolites in wheat matrices

Residue component	Forage		Grain		Straw	
	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg
Parent compound	49.8	1.96	7.6	0.006	19.9	1.97
IN-QDY62	–	–	–	–	6.1	0.604
IN-QDK50	–	–	–	–	2.0	0.198
IN-QCD12	1.5	0.059	–	–	1.3	0.129
IN-QDY63	1.1	0.043	–	–	4.3	0.426
IN-QGS45	2.9	0.114	–	–	0.2	0.020
IN-QGS44	0.7	0.028	–	–	2.5	0.248
IN-QGU66	1.3	0.051	–	–	1.5	0.149
IN-QGS46	0.4	0.016	–	–	4.6	0.455
IN-QGU72	3.3	0.130	–	–	–	–
IN-QGU69	–	–	–	–	2.3	0.228
Hydroxy-IN-QGU69	–	–	–	–	2.2	0.218
Total identified	61.0	2.401	7.6	0.006	46.9	4.645
Organosoluble unknowns	1.5	0.059 (at least 5)	19.0	0.015 (at least 11)	2.2	0.218 (6 components,

Residue component	Forage		Grain		Straw	
	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg
		components, each ≤ 0.028)		components, each < 0.002)		each < 0.079)
Radioactivity incorporated into natural compounds	–	–	16.3	0.013 (glucose, 0.009, plus others)	–	–
Aqueous soluble unknowns	12.1	0.476 (at least 8 components, each ≤ 0.122)	–	–	11.2	1.11 (12 components, each < 0.347)
Baseline and unassigned components	17.9	0.703	19.1	0.016	18.4	1.818
Unchromatographed aqueous fractions	0.5	0.020	3.8	0.003	4.7	0.465
Unchromatographed organosoluble fractions	–	–	2.2	0.002	–	–
Losses	2.6	0.102	11.1	0.010	12.5	1.24
Total extracted	95.7	3.76161	79.1	0.064	95.9	9.494
Post extraction solids	4.3	0.169	20.9	0.017	4.1	0.406
TOTAL (sum of extracted residue and PES)	100	3.93	100	0.081	100	9.90

Table 15 Identification of [¹⁴C]phenacrylate-picoxystrobin metabolites in wheat matrices

Residue component	Forage		Grain		Straw	
	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg
Parent compound	55.7	3.28	3.5	0.011	21.3	2.35
IN-QDY62	–	–	–	–	4.8	0.528
IN-QCD12	1.0	0.059	–	–	1.3	0.143
IN-QDY63	0.9	0.053	–	–	3.5	0.385
IN-QDY60	–	–	–	–	0.4	0.044
IN-QGS44	0.5	0.029	–	–	2.8	0.308
IN-QGU66	1.3	0.076	–	–	2.0	0.220
IN-QGS46	0.5	0.029	–	–	3.0	0.330
IN-K2122/phthalic acid	1.6	0.094	7.4	0.023	1.8	0.198
IN-10975	0.2	0.012	–	–	1.0	0.110
IN-H8612	1.5	0.088	14.9	0.046	1.8	0.198
PAG 3 ^a	–	–	7.9	0.024	0.8	0.088
IN-QGU69	–	–	–	–	1.4	0.154
Hydroxy-IN-QGU69	–	–	–	–	2.7	0.297
Total identified	63.2	3.72	33.7	0.104	46.8	5.353
Organosoluble	1.0	0.059 (at	16.4	0.051 (9	1.8	0.198 (at

Residue component	Forage		Grain		Straw	
	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg
unknowns		least 4 components, each ≤ 0.041)		components, each, each ≤ 0.012)		least 6 components, each ≤ 0.066)
Radioactivity incorporated into natural compounds	–	–	9.4	0.029 (glucose, 0.013, plus others)	–	–
Aqueous soluble unknowns	13.4	0.788 (at least 10 components, each ≤ 0.194)	–	–	20.8	2.29 (at least 19 components, each ≤ 0.451)
Baseline and unassigned components	17.0	1.0	22.5	0.070	14.8	1.631
Unchromatographed aqueous fractions	–	–	–	–	3.5	0.385
Losses	–	–	10.1	0.031	4.7	0.517
Total extracted	94.5	5.567	92.1	0.285	94.3	10.373
Post extraction solid	5.5	0.323	7.9	0.024	5.7	0.627
TOTAL (sum of extracted residue and PES)	100	5.89	100	0.309	100	11.0

^a PAG 3 = 2-(2-hydroxymethylphenyl)-2-oxoacetic acid.

Sample extraction and profiling was completed within 6 months of harvest, with the exception of the identification of one of the phenacrylate grain metabolites (PAG 3). Retained samples of grain were extracted and subjected to further analyses 5 years after the first study, enabling the identification of PAG 3 as 2-(2-hydroxymethylphenyl)-2-oxoacetic acid (Benner *et al.* 2001), a compound that is also a metabolite in rat urine. No significant changes to the metabolite profile or the amounts of metabolites had occurred during storage, indicating good stability of the residues in wheat grain samples.

The largest individual residue component in most of the matrices was parent compound, ranging from 3.5–7.6% of the TRR in grain, to 49.8–55.7% in forage. A proportion of the radioactivity was incorporated into natural products (glucose and other sugars) in wheat grain (9.4–16.3% of TRR or 0.013–0.029 mg eq/kg, of which glucose comprised 4.2–11.0% of the TRR or 0.009–0.013 mg eq/kg), but not in forage and straw. Few metabolites were identified in grain and these were mostly small molecules. In wheat forage and straw, the metabolic pathways were more evident, with key intermediates as well as terminal metabolites being observed. The major metabolic pathways for picoxystrobin in wheat are:

Oxidative cleavage of the molecule at the ether bridge to yield 6-(trifluoromethyl)-1*H*-pyridin-2-one (IN-QDK50) and methyl (*E*)-2-(2-hydroxymethylphenyl)3-methoxyacrylate (IN-QDY60). The pyridine cleavage product was subsequently conjugated with glucose and malonic acid, while the phenacrylate cleavage product was subject to further oxidation and cleavage giving phthalic acid or 1,3-dihydro-3-oxoisobenzofuran-1-carboxylic acid (IN-H8612)

Loss of the methoxy methyl group followed by reduction of the enol and hydroxylation of the phenyl ring

Hydrolysis of the ester, followed by oxidation and cleavage of the acrylate moiety ultimately yielding the benzoic acid metabolite IN-QDY63 or the phenyl-hydroxy acetic acid metabolite IN-QGS46.

A minor metabolic pathway in wheat is isomerisation about the double bond yielding the *Z* isomer of picoxystrobin (IN-QCD12).

Oilseed rape

Greenhouse-grown oilseed rape plants were treated with two foliar applications of either [¹⁴C]pyridinyl-picoxystrobin or [¹⁴C]phenacrylate-picoxystrobin formulated as a suspension concentrate (Shaffer, 2010). The applications were made 7 days apart, at approximate BBCH growth stages 80 and 85 respectively. Individual and total application rates were 468–483 and 942 g ai/ha, and 403–423 and 827 g ai/ha for the pyridinyl and phenacrylate labels respectively.

Samples of foliage were collected from the treated plots immediately before and 14 days after the second application. All remaining plant material was harvested 21 days after the second application and separated into seed and foliage plus pods. Samples were stored frozen pending homogenisation and analysis.

Homogenised samples were extracted with acetonitrile/water, with seed samples additionally being extracted with dichloromethane. Some samples were additionally subjected to enzymatic and mild base hydrolysis. The extracts were analysed by HPLC, with reference standards being used to identify the metabolites. All analyses were completed within 4 months of harvest.

Table 16 Total radioactive residues in oilseed rape samples

Sample	Sampling interval	TRRs (mg/kg parent equivalents)	
		[¹⁴ C]Pyridinyl label	[¹⁴ C]Phenacrylate label
Foliage	7DAA1	5.93	7.05
Foliage	14DAA2	12.47	11.52
Foliage plus pods	21DAA2	11.80	12.99
Seed	21DAA2	1.66	2.50

7DAA1 = 7 days after application 1, 14 DAA2 = 14 days after application 2, etc.

Table 17 Identification of residues in [¹⁴C]pyridinyl-picoxystrobin treated oilseed rape

Analyte	7DAA1 foliage		14DAA2 foliage		21 DAA2 foliage		21 DAA2 seed	
	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg
Total extracted	97.6	5.79	97.1	12.11	95.3	11.25	92.2	1.53
Picoxystrobin	93.4	5.55	79.5	9.92	70.2	8.29	89.0	1.48
IN-QDY62	–	–	0.2	0.03	0.4	0.05	–	–
IN-QDK50	0.5	0.03	1.8	0.22	2.9	0.34	–	–
IN-QCD12	0.4	0.02	0.7	0.08	0.7	0.08	–	–
IN-QDY63	0.8	0.05	4.6	0.57	7.6	0.90	–	–
IN-QGS45	–	–	–	–	0.2	0.03	–	–
Unknowns ^a	2.5	0.15	10.4	1.30	13.2	1.56	–	–
Enzyme/base extracted residues	–	–	–	–	2.1	0.25	3.2	0.05
Unextracted residue	2.4	0.14	2.9	0.36	2.6	0.30	4.6	0.08
TOTAL		5.93		12.47		11.80		1.66

^a Consists of 2–12 components, ranging from 0.20–4.3% of TRR, or 0.02–0.51 mg/kg.

Table 18 Identification of residues in [¹⁴C]phenacrylate-picoxystrobin treated oilseed rape

Analyte	7DAA1 foliage		14DAA2 foliage		21 DAA2 foliage		21 DAA2 seed	
	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg
Total extracted	98.6	6.95	98.4	11.33	97.4	12.66	96.7	2.42
Picoxystrobin	96.3	6.78	80.7	9.29	71.9	9.35	93.8	2.34
IN-QDY62	–	–	0.6	0.07	0.9	0.11	–	–
IN-QCD12	–	–	0.6	0.07	0.6	0.08	0.6	0.02
IN-QDY63	0.8	0.06	4.9	0.56	7.4	0.96	–	–
Unknowns ^a	1.6	0.11	11.6	1.33	16.6	2.16	0.6	0.01
Enzyme/base extracted residues	–	–	–	–	0.8	0.11	5.6	0.14
Unextracted residue	1.4	0.10	1.6	0.19	1.8	0.23	3.3	0.08
TOTAL		7.05		11.52		12.99		2.50

^a Consists of 1–13 components, ranging from 0.20–4.25% of TRR, or 0.02–0.55 mg/kg.

Metabolism was relatively limited, probably the result of the relatively short times between application and sampling (7–21 days), application at late growth stages (BBCH 80–85) when the plant would be approaching senescence, and the lack of exposure to full sun and rainfall that would be expected to accelerate the degradation in field grown plants. The majority (70.2–96.3% of the TRR) of the residue was present as parent compound. In seed, only one compound other than parent (IN-QCD12, the *Z*-isomer of picoxystrobin) was found, while in forage, small amounts of IN-QDY62 (demethylated picoxystrobin), IN-QCD12, 6-(trifluoromethyl)-1*H*-pyridin-2-one (IN-QDK50) and its glucose conjugate IN-QGS45, and the benzoic acid metabolite IN-QDY63 were also identified. The metabolic pathways in oilseed rape therefore appear to be ester hydrolysis, cleavage at the ether bridge, hydrolysis and oxidation of the acrylate moiety, and isomerisation about the double bond in the acrylic acid moiety.

Soya bean

Foliar applications of either [¹⁴C]pyridinyl-picoxystrobin or [¹⁴C]phenacrylate-picoxystrobin were made to soya beans grown in the field (Close and Brumback, 2006). Two applications were made 14 days apart, the first at around BBCH stage 69 (first pods) and the second at around BBCH stage 73–75 (pod filling). Two treated plots were established for each of the labels, with application rates of 192.8 and 954.4 g ai/ha, and 202.3 and 1038.8 g ai/ha, for the phenacrylate and pyridinyl labels respectively.

Foliage samples were collected 14 days after the second application from all treated plots (simulating a hay harvest). Seed and dry stalk samples were collected at the normal harvest growth stage (61 days after the second application), and dry leaves were collected 42 days after the second application, from the low rate plots only. Samples were stored frozen until homogenisation, extraction and analysis.

Foliage and seed samples were extracted with a number of solvents (acetonitrile/water, hexane, ethyl acetate, methanol, acetonitrile and acidified methanol). Samples containing sufficient unextracted residues were subjected to additional treatments, including solvent and acid reflux, and enzyme hydrolysis. Sample extracts were analysed by TLC, with co-chromatography with reference standards, and MS or NMR being used for metabolite identification. Only low rate forage and seed were fully extracted and profiled, with high rate samples being used to generate material for specific metabolite identifications.

Table 19 Total radioactive residues in soya bean matrices

Sample	Residue (mg/kg parent equivalents)	
	Phenacrylate label	Pyridinyl label
Foliage	1.677	1.795
Seed	0.140	0.074

Table 20 Metabolic profile of picoxystrobin in [¹⁴C]pyridinyl label soya bean samples

Residue component	Forage		Seeds	
	%TRR	mg/kg parent equivalents	%TRR	mg/kg parent equivalents
Parent compound	10.0	0.179	5.9	0.004
IN-QGU73	4.6	0.083	6.8	0.005
IN-QGS46-glucoside	14.4	0.258	6.2	0.005
R290461-glucosides ^a	6.2	0.112	3.5	0.003
	24.4	0.439	7.7	0.006
R290461-malonyl glucose conjugate	10.0	0.180	6.3	0.005
R290461	–	–	4.5	0.003
Unidentified metabolites	17 individual metabolites 0.3–2.4, total 19.1	0.005–0.042, total 0.34	14 individual metabolites 0.6–7.2, total 33.1	< 0.001–0.005, total 0.025
Total identified	69.7	1.250	40.9	0.031
Total extracted	91.2	1.638	82.3	0.061
Post extraction solids	8.8	0.157	17.7	0.013
TRR	100	1.795	100	0.074

^a Two structural isomers of R290461-glucoside were identified.

Table 21 Metabolic profile of picoxystrobin in [¹⁴C]phenacrylate label soya bean samples

Residue component	Forage		Seeds	
	%TRR	mg/kg parent equivalents	%TRR	mg/kg parent equivalents
Parent compound	7.4	0.125	1.5	0.002
IN-H8612	1.7	0.028	2.5	0.003
IN-K2122	1.2	0.020	21.3	0.030
2-Formylphenyl-oxoacetic acid	–	–	25.5	0.036
IN-QGS46-glucoside	8.4	0.140	0.7	< 0.001
R290461-glucosides	4.1	0.068	–	–
	22.3	0.374	3.8	0.005
IN-QFA35-glucoside	6.5	0.109	2.8	0.004
R410639-decarboxy malonyl glucoside	9.9	0.166	0.5	< 0.001

Residue component	Forage		Seeds	
	%TRR	mg/kg parent equivalents	%TRR	mg/kg parent equivalents
IN-QGS46	1.6	0.027	2.0	0.003
IN-QDY63	1.5	0.026	0.6	< 0.001
Unidentified metabolites	15 individual metabolites 0.3–2.8, total 19.4	0.005–0.048, total 0.329	11 individual metabolites 0.7–7.3, total 23.2	< 0.001–0.010, total 0.033
Total identified	64.6	1.083	61.2	0.086
Total extracted	88.8	1.489	91.0	0.127
Post extraction solids	11.2	0.188	9.0	0.013
TRR	100	1.677	100	0.140

The soya bean forage and seed samples were extracted and profiled within 6 months of sampling. Retention samples of forage and soya beans were extracted and profiled again near the end of the study 12–18 months later, showing no significant change in the metabolic profile, evidence that the samples had not deteriorated on storage.

Parent compound was a significant component of the residue in soya bean forage (7.4–10% of the TRR, or 0.125–0.179 mg/kg), while smaller amounts (0.002–0.004 mg/kg or 1.5–5.9% of the TRR) were found in seed. Significant metabolites in soya bean forage included the glucose conjugate of IN-QGS46 (8.4–14.4% of the TRR or 0.140–0.258 mg/kg), glucose conjugates of R290461 (26.4–30.6% of the TRR or 0.442–0.551 mg/kg), the malonyl glucose conjugate of R290461 (10% TRR or 0.180 mg/kg), IN-QFA35 glucoside (6.5% of TRR or 0.109 mg/kg), and R410639 glucoside (9.9% of TRR or R410639). The only metabolites above 0.01 mg/kg or 10% of the TRR in phenacrylate label seed were phthalic acid and 2-formylphenyl-oxoacetic acid. All components in pyridine label seed were < 0.01 mg/kg and < 10% of TRR.

Key metabolic pathways for picoxystrobin in soya beans were:

Oxidative cleavage of the molecule at the ether bridge to yield 6-(trifluoromethyl)-1*H*-pyridin-2-one and methyl (*E*)-2-(2-hydroxymethylphenyl)3-methoxyacrylate. The pyridine cleavage product was subsequently conjugated with glucose and glutaric acid, while the phenacrylate cleavage product was subject to further oxidation and cleavage giving phthalic acid or 1,3-dihydro-3-oxoisobenzofuran-1-carboxylic acid

Loss of the methoxy methyl group followed by reduction of the enol, further hydroxylation of the side chain, and conjugation of the hydroxyl groups with glucose and malonic acid

Hydrolysis of the ester, followed by oxidation and cleavage of the acrylate moiety ultimately yielding the benzoic acid metabolite IN-QDY63 or a phenyl-acetic acid metabolite, with glucose conjugation.

Summary of plant metabolism

The key metabolic pathways in wheat, soya beans and oilseed rape were similar, although the extent of the metabolism differed between crops, with significantly less metabolism occurring in oilseed rape than in wheat or soya beans. This is likely to be the result of application at a late growth stage and the trial being conducted in a greenhouse rather than in the field.

The major metabolic pathways for picoxystrobin in plants were:

Oxidative cleavage of the molecule at the ether bridge to yield 6-(trifluoromethyl)-1*H*-pyridin-2-one and methyl (*E*)-2-(2-hydroxymethylphenyl)3-methoxyacrylate. The pyridine cleavage product was subsequently conjugated with glucose and malonic or glutaric acid, while the

phenacrylate cleavage product was subject to further oxidation and cleavage giving phthalic acid or 1,3-dihydro-3-oxoisobenzofuran-1-carboxylic acid

Loss of the methoxy methyl group followed by reduction of the enol, further hydroxylation of the side chain, and conjugation of the hydroxyl groups with glucose and malonic acid

Hydrolysis of the ester, followed by oxidation and cleavage of the acrylate moiety ultimately yielding the benzoic acid metabolite IN-QDY63 or a phenyl-acetic acid metabolite, with or without glucose conjugation of the hydroxyl or carboxylic acid functionalities.

Hydroxylation of the phenyl ring was also observed in wheat, while small amounts of the *Z*-isomer of picoxystrobin were found in oilseed rape and wheat.

Environmental fate

Data on aerobic soil metabolism, anaerobic soil metabolism, soil surface photolysis, field soil dissipation, hydrolysis, and aqueous photolysis were received. Only those data relevant to the use pattern for picoxystrobin (foliar application to cereal, pulse and oilseed crops) were evaluated.

Aerobic soil metabolism

The metabolism of [^{14}C]pyridinyl- and [^{14}C]phenacrylate-labelled picoxystrobin was studied in four soil types: two sandy loams, a sandy clay loam, and a sand (Harvey and Butters, 1998), with supplementary studies (Muller *et al.*, 1998 and Muller *et al.*, 1999) conducted to further elucidate the structures of volatile metabolites. Soil samples were not sterilised and microbial activity was monitored during the experiment. Radiolabelled picoxystrobin was applied to the soil surface at rates equivalent to field application rates of 23–295 g ai/ha and samples were maintained in the dark under aerobic conditions at 20 °C for up to a year. DT_{50} values for picoxystrobin determined using a first-order multi-compartmental model ranged from 16 to 38 days, while the DT_{90} values ranged from 76–337 days.

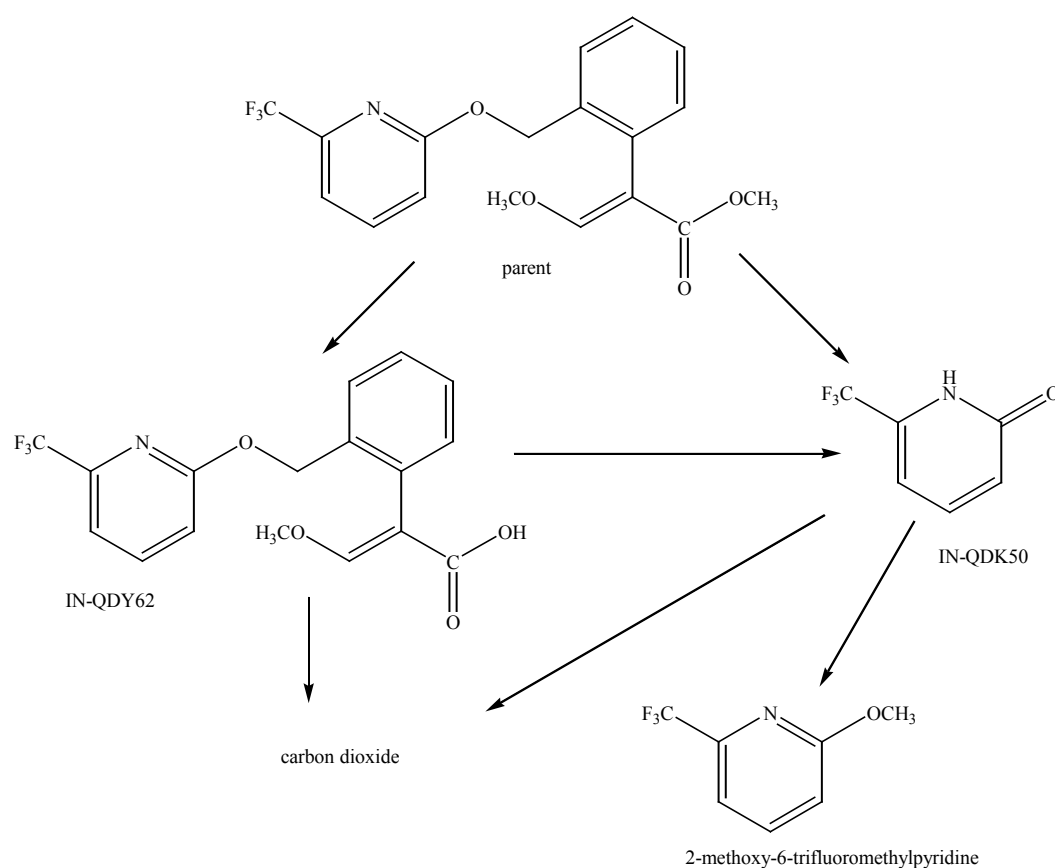


Figure 5 Metabolism of picoxystrobin in aerobic soil in the dark

Major identified degradation products were IN-QDY62 (maximum level of 8–30% of the applied dose, typically around day 14–50), IN-QDK50 (maximum level of 9–15% of the applied dose, typically around day 29–62), and the volatile metabolite 2-methoxy-6-trifluoromethylpyridine (maximum level of 22–31% of applied dose, after 119 days). Mineralisation of picoxystrobin to carbon dioxide was extensive, with 18–33% of the applied radioactivity degrading to CO_2 for the [^{14}C]pyridinyl label and 30–43% for the [^{14}C]phenacrylate label after 119 days incubation in the initial study. For one of the four soil types, incubation was continued to 364 days, with CO_2 comprising 34%

The key parameters (DT₅₀ and DT₉₀ values) for each of the soil dissipation studies are in Table 22).

Table 22 Dissipation rates of picoxystrobin in soil from various field studies

Site/soil type	DT ₅₀ (days)	DT ₉₀ (days)	Model	Reference
Grisolles, Southern France [silty clay loam, pH 7.2, OC 2.0%, 0–10 cm profile]	22.8 (13.5, 32.1)	129 (102, 155)	FOMC	Harradine and Atger, 1998, RJ2520B
Vitray, Southern France [sandy loam, pH 5.9, OC 1.7%, 0–20 cm profile]	22.7 (11.2, 34.2)	286 (184, 387)	FOMC	
Maidenhead, Berkshire, UK [sandy clay loam, pH 6.2, OC 1.6%, 0–10 cm profile]	30.4 (15.3, 45.5)	364 (213, 514)	FOMC	Harradine and Lake, 1998, RJ2555B
Lebien, Sachsen-Anhalt, Germany [sandy clay loam, pH 7.2, OC 1.9%, 0–10 cm profile]	15.6 (6.1, 25.0)	196 (127, 265)	FOMC	Johnson and Chamier, 1998, RJ2492B
St Remy de Provence, southern France [silty clay, pH 8.3, OC 4.0%, 0–20 cm profile]	19	126	FOMC	Nagra and Atger, 1999, RJ2721B
Cessac, southern France [loam, pH 7.5, OC 2.2%, 0–20 cm profile]	35	202	FOMC	
Wangelau, Schleswig-Holstein, Germany [sandy loam, pH 7.2, OC 1.7%, 0–20 cm profile]	9	74	FOMC	Nagra and Chamier, 1998, RJ2722B
Bracknell, Berkshire, UK [sandy clay loam, pH 5.8, OC 3.1%, 0–20 cm profile]	3	42	FOMC	Nagra, Lake and Unsworth, 1998, RJ2735B
Queens County, Prince Edward Island, Canada [sandy loam, pH 6.2, OC 3.6%, 0–5 cm profile]	8.9	96.0	DFOP	Rice, 2010, Study number 25345
Portage la Prairie, Manitoba, Canada [clay loam, pH 7.8, OC 6.6%, 0–5 cm profile]	19.3	437	DFOP	Rice, 2010, Study number 25344
Arkansaw, Wisconsin, USA [sandy loam, pH 6.0, OC 2.7%, 0–5 cm profile]	1.3	66.5	DFOP	Rice, 2010, Study number 26418
Tulare County, California, USA [sandy loam, pH 8.7, OC 0.6%, 0–5 cm profile]	2.6	72.7	DFOP	Shepard, 2010, Study number 24936

FOMC = First Order Multi-Compartmental model

DFOP = Double First Order in Parallel. 95% confidence limits given in brackets for the DT₅₀ and DT₉₀ values for the European studies (where available).

Residues of all metabolites were low, often below the limit of quantification, and less than the level of parent compound. There was no evidence of accumulation of parent or the metabolites. Very few residues were detected in lower soil profiles (below 10 cm in the European studies and below 15 cm in the North American studies), and those that were detected were at or just above the LOQ, and dropped below the LOQ or even the LOD at subsequent sample collection times.

DT₅₀ values in field soil dissipation studies ranged from 1.3 to 35 days, while DT₉₀ values ranged from 42 to 437 days.

Residues in succeeding crops

A confined crop rotation study was conducted to determine the nature of picoxystrobin residues in representative crops planted following a treated crop (Turner *et al.* 1998a).

[¹⁴C]Pyridinyl- or [¹⁴C]phenacrylate-labelled picoxystrobin was applied in a single application as a suspension concentrate formulation directly to the soil surface at a rate of 820–888 g ai/ha. Wheat, lettuce and carrots (representing cereals, leafy and root crops) were sown in the containers at intervals of 30 and 197 days from application. Wheat forage was collected at BBCH growth stage 39, while wheat straw and grain, lettuce heads and carrot roots and leaves were collected at normal harvest maturity. Soil samples were collected from each container for analysis on the day of sowing.

Grain samples were extracted by an enzyme digest. All other plant samples were extracted with solvents including acetonitrile, acetonitrile/water, and water. Some extracts were acid and base hydrolysed to aid in characterising conjugates, while post-extraction samples containing > 0.05 mg parent equivalents/kg (mg eq/kg) of unextracted residues were base hydrolysed. Soil samples were extracted using acetone/HCl, cleaned up and analysed using a method based on method number RAM 291/01 (Mason and French, 1996). Residue components in the plant sample extracts were characterised and quantified using thin layer chromatography with co-chromatography with reference compounds or other identified metabolites, and LC/MS/MS (Tables 23–30).

Table 23 Total radioactive residues (TRRs) in rotational crops grown in soil treated with radiolabelled picoxystrobin (determined as summation of radioactivity in extracts and post-extraction solids)

Sample	TRR (mg/kg parent equivalents)			
	[¹⁴ C]Pyridinyl label		[¹⁴ C]Phenacrylate label	
	30 day PBI	197 day PBI	30 day PBI	197 day PBI
Soil	1.61	1.18	1.35	1.28
Wheat forage	1.02	0.971	0.224	0.357
Wheat grain	0.067	0.034	0.150	0.090
Wheat straw	11.1	4.26	1.64	1.87
Lettuce	0.352	0.186	0.027	0.045
Carrot leaves	1.24	0.746	0.059	0.048
Carrot roots	0.37	0.197	0.031	0.033

PBI = plant-back interval

Table 24 Amount of picoxystrobin and metabolites in treated soil (indoor experiment)

Residue component	[¹⁴ C]Pyridinyl label				[¹⁴ C]Phenacrylate label			
	30 day PBI		197 day PBI		30 day PBI		197 day PBI	
	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg
Picoxystrobin	71.6	1.15	43.0	0.507	68.4	0.923	42.1	0.539
IN-QDY62	2.9	0.047	9.0	0.106	2.9	0.039	12.2	0.156

Residue component	¹⁴ C]Pyridinyl label				¹⁴ C]Phenacrylate label			
	30 day PBI		197 day PBI		30 day PBI		197 day PBI	
	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg
IN-QDK50	–	–	3.1	0.037	–	–	–	–
IN-QDY63	4.7	0.076	5.6	0.066	7.7	0.104	8.4	0.108
R409665	0.6	0.010	3.7	0.044	1.4	0.019	3.8	0.049
R416021	3.3	0.053	4.1	0.048	4.5	0.061	2.9	0.037
Unknowns	0.5 ^a	0.008 ^a	1.9 ^d	0.022 ^d	–	–	6.0 ^e	0.077 ^e
Baseline	0.4	0.006	2.9	0.034	1.0	0.014	0.0	0.0
Unassigned ^b	6.6	0.106	14.8	0.175	9.5	0.128	6.9	0.088
Losses on elution	–	–	2.4	0.028	–	–	2.0	0.026
Post extraction solids	4.0	0.064	7.6	0.090	7.3	0.100	13.1	0.168
Losses on fractionation ^c	5.5	0.089	1.9	0.022	–2.7	0.036	2.6	0.033
TOTAL	100.0	1.61	100.0	1.18	100.0	1.35	100.0	1.28

^a Consists of at least two components, none > 0.3% or 0.005 mg eq/kg.

^b Percentages of identified and discrete unknown components subtracted from the total percentage of recovered residue chromatographed (i.e. areas of streaked radioactivity).

^c Net loss or gain on fractionation.

^d One component.

^e At least four discrete components, none > 1.8% of TRR or 0.023 mg eq/kg.

Table 25 Amount of picoxystrobin and metabolites in wheat forage grown in treated soil (indoor grown)

Residue component	¹⁴ C]Pyridinyl label				¹⁴ C]Phenacrylate label			
	30 day PBI		197 day PBI		30 day PBI		197 day PBI	
	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg
Picoxystrobin	3.1	0.032	2.5	0.024	12.4	0.028	3.4	0.012
IN-QDY62	–	–	0.1	0.001	–	–	–	–
IN-QDK50	1.2	0.012	0.1	0.001	–	–	–	–
IN-QGS45	4.9	0.050	3.4	0.033	–	–	–	–
IN-QGU72	57.2	0.583	44.2	0.429	–	–	–	–
R409665	–	–	9.6	0.093	–	–	40.8	0.146
PAF1 [*]	–	–	–	–	12.5	0.028	–	–
Unknowns	11.3 ^a	0.115 ^a	14.9 ^d	0.145 ^d	34.8 ^e	0.078 ^e	11.0 ^f	0.039 ^f
Baseline	2.4	0.024	5.5	0.053	5.3	0.012	4.1	0.015
Unassigned ^b	15.3	0.156	11.5	0.112	20.4	0.046	28.5	0.102
Unchromatographed fractions	0.7	0.007	1.1	0.011	0.8	0.002	1.5	0.005
Post extraction solids	3.8	0.039	1.9	0.018	11.8	0.026	3.7	0.013
Filter papers	0.2	0.002	3.7	0.036	1.7	0.004	5.9	0.021
Losses on fractionation ^c	0.0	0.0	1.6	0.016	0.3	<0.001	1.1	0.004

Residue component	[¹⁴ C]Pyridinyl label				[¹⁴ C]Phenacrylate label			
	30 day PBI		197 day PBI		30 day PBI		197 day PBI	
	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg
TOTAL	100.1	1.02	100.1	0.972	100.0	0.225	100.0	0.357

*Tentatively identified as a glucose conjugate of IN-QDY62

^a Consists of at least 4 components, none > 4.4% or 0.045 mg eq/kg.

^b Percentages of identified and discrete unknown components subtracted from the total percentage of recovered residue chromatographed (i.e. areas of streaked radioactivity).

^c Net loss or gain on fractionation.

^d At least 5 discrete components, none > 5.8% of TRR or 0.056 mg eq/kg.

^e At least 9 discrete components, none > 7.8% of TRR or 0.017 mg eq/kg.

^f At least 4 discrete components, none > 4.8% of TRR or 0.017 mg eq/kg.

Table 26 Amount of picoxystrobin and metabolites in wheat grain grown in treated soil (indoor grown)

Residue component	[¹⁴ C]Pyridinyl label				[¹⁴ C]Phenacrylate label			
	30 day PBI		197 day PBI		30 day PBI		197 day PBI	
	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg
IN-QDK50	0.9	< 0.001	–	–	–	–	–	–
IN-H8612	–	–	–	–	4.2	0.006	1.7	0.002
R409665	–	–	13.3	0.005	–	–	17.8	0.016
Natural incorporation	36.5 ^a	0.024 ^a	18.4 ^e	0.006 ^e	9.3 ^f	0.014 ^f	15.3 ^h	0.014 ^h
Unknowns	3.2 ^b	0.002 ^b	–	–	30.7 ^g	0.046 ^g	3.7 ⁱ	0.003 ⁱ
Baseline	1.5	0.001	4.1	0.001	6.9	0.010	5.1	0.005
Unassigned ^c	17.9	0.012	26.9	0.009	24.4	0.037	27.7	0.025
Post extraction solid	25.3	0.017	24.8	0.008	16.5	0.025	24.3	0.022
Filter papers	1.3	< 0.001	2.5	< 0.001	1.6	0.002	2.0	0.002
Losses on fractionation ^d	13.2	0.009	10.0	0.003	6.4	0.010	2.5	0.002
TOTAL	99.8	0.067	100.0	0.033	100.0	0.150	100.1	0.091

^a Consists of glucose (16.7% TRR or 0.011 mg eq/kg) and other natural products

^b Consists of at least 3 components, none > 2.0% or 0.001 mg eq/kg

^c Percentages of identified and discrete unknown components subtracted from the total percentage of recovered residue chromatographed (i.e. areas of streaked radioactivity)

^d Net loss or gain on fractionation

^e Consists of glucose (12.2% TRR or 0.004 mg eq/kg) and other natural products

^f Consists of glucose (4.5% TRR or 0.007 mg eq/kg) and other natural products

^g Consists of at least 17 components, none > 11.9% or 0.018 mg eq/kg

^h Consists of glucose (8.8% TRR or 0.008 mg eq/kg) and other natural products.

ⁱ Consists of at least 2 components, none > 1.9% or 0.002 mg eq/kg.

Table 27 Amount of picoxystrobin and metabolites in wheat straw grown in treated soil (indoor grown)

Residue component	¹⁴ C]Pyridinyl label				¹⁴ C]Phenacrylate label			
	30 day PBI		197 day PBI		30 day PBI		197 day PBI	
	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg
Picoxystrobin	1.1	0.122	0.8	0.034	5.1	0.084	0.8	0.015
IN-QDY62	1.3	0.144	0.9	0.038	4.8	0.079	1.7	0.032
IN-QDK50	14.2	1.58	6.7	0.285	–	–	–	–
IN-QGS45	7.5	0.833	4.2	0.179	–	–	–	–
IN-QGU72	21.4	2.38	20.2	0.861	–	–	–	–
IN-QFA35	–	–	–	–	0.7	0.011	0.5	0.009
R409665	0.3	0.033	6.6	0.281	0.6	0.010	9.7	0.181
Unknowns	5.7 ^a	0.633 ^a	16.0 ^d	0.682 ^d	39.8 ^e	0.653 ^e	37.5 ^f	0.701 ^f
PYST2	13.8	1.53	7.3	0.311	–	–	–	–
Baseline	7.3	0.810	3.7	0.158	14.7	0.241	6.8	0.127
Unassigned ^b	20.8	2.31	25.6	1.10	22.7	0.372	31.7	0.593
Unchromatographed fractions	1.3	0.144	1.6	0.068	0.4	0.007	0.3	0.006
Post extraction solids	2.6	0.289	2.6	0.111	3.6	0.059	4.8	0.090
Filter papers	2.0	0.222	3.3	0.141	4.8	0.079	6.8	0.127
Losses on fractionation ^c	0.5	0.056	0.2	0.009	2.9	0.048	–0.6	–0.011
TOTAL	99.8	11.1	99.7	4.26	100.1	1.64	100.0	1.87

^a Consists of at least 3 components, none > 2.5% or 0.278 mg eq/kg

^b Percentages of identified and discrete unknown components subtracted from the total percentage of recovered residue chromatographed (i.e. areas of streaked radioactivity)

^c Net loss or gain on fractionation

^d Consists of at least 4 components, none > 3.1% or 0.132 mg eq/kg

^e Consists of at least 10 components, none > 6.6% or 0.108 mg eq/kg

^f Consists of at least 8 components, none > 7.0% or 0.131 mg eq/kg.

Table 28 Amount of picoxystrobin and metabolites in lettuce grown in treated soil (indoor grown)

Residue component	¹⁴ C]Pyridinyl label				¹⁴ C]Phenacrylate label			
	30 day PBI		197 day PBI		30 day PBI		197 day PBI	
	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg
Picoxystrobin	1.0	0.003	1.1	0.002	4.0	0.001	3.6	0.002
IN-QDY62	–	–	–	–	1.2	< 0.001	–	–
IN-QDK50	2.2	0.008	–	–	–	–	–	–
IN-QGS45	11.4	0.040	8.5	0.016	–	–	–	–
IN-QGU72	51.7	0.182	46.3	0.086	–	–	–	–
R409665	–	–	9.0	0.017	–	–	31.1	0.014
PAF1 [*]	–	–	–	–	38.1	0.010	26.0	0.012
Unknowns	16.0 ^a	0.056 ^a	27.6 ^d	0.051 ^d	25.6 ^e	0.007 ^e	27.5 ^f	0.012 ^f

Residue component	[¹⁴ C]Pyridinyl label				[¹⁴ C]Phenacrylate label			
	30 day PBI		197 day PBI		30 day PBI		197 day PBI	
	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg
Baseline	2.2	0.008	4.0	0.007	1.6	< 0.001	4.5	0.002
Unassigned ^b	11.7	0.041	1.0	0.002	12.1	0.003	2.2	< 0.001
Unchromatographed fractions	1.1	0.004	0.6	0.001	7.2	0.002	0.7	< 0.001
Post extraction solids	2.5	0.009	1.0	0.002	9.0	0.002	5.7	0.003
Filter papers	0.3	0.001	1.0	0.002	0.7	< 0.001	2.8	0.001
Losses on fractionation ^c	0.0	0.0	0.0	0.0	0.5	< 0.001	-4.1	-0.002
TOTAL	100.1	0.352	100.1	0.186	100.0	0.029	100.0	0.046

*Tentatively identified as a glucose conjugate of IN-QDY62

^a Consists of at least 3 components, none > 7.1% or 0.025 mg eq/kg

^b Percentages of identified and discrete unknown components subtracted from the total percentage of recovered residue chromatographed (i.e. areas of streaked radioactivity)

^c Net loss or gain on fractionation

^d Consists of at least 8 components, none > 8.8% or 0.016 mg eq/kg

^e Consists of at least 6 components, none > 10.3% or 0.003 mg eq/kg

^f Consists of at least 8 components, none > 6.3% or 0.003 mg eq/kg.

Table 29 Amount of picoxystrobin and metabolites in carrot leaves grown in treated soil (indoor grown)

Residue component	[¹⁴ C]Pyridinyl label				[¹⁴ C]Phenacrylate label			
	30 day PBI		197 day PBI		30 day PBI		197 day PBI	
	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg
Picoxystrobin	0.9	0.011	1.0	0.007	14.2	0.008	8.6	0.004
IN-QDK50	1.7	0.021	–	–	–	–	–	–
IN-QGS45	5.6	0.069	10.2	0.076	–	–	–	–
IN-QGU72	19.9	0.247	22.9	0.171	–	–	–	–
IN-QGU73	41.3	0.512	43.1	0.322	–	–	–	–
R409665	–	–	–	–	–	–	7.4	0.004
PAF1 [*]	–	–	–	–	17.8	0.011	16.8	0.008
Unknowns	7.7 ^a	0.095 ^a	11.2 ^d	0.084 ^d	19.6 ^e	0.012 ^e	13.1 ^f	0.006 ^f
Baseline	6.4	0.079	1.3	0.010	0.8	< 0.001	3.4	0.002
Unassigned ^b	12.4	0.154	5.0	0.037	1.9	0.001	19.0	0.009
Unchromatographed fractions	0.5	0.006	2.4	0.018	17.3	0.010	4.8	0.002
Post extraction solids	2.6	0.032	2.5	0.019	20.9	0.012	22.5	0.011
Filter papers	1.0	0.012	0.3	0.002	5.0	0.003	4.2	0.002
Losses on fractionation ^c	0.0	0.0	0.0	0.0	2.4	0.001	0.3	< 0.001
TOTAL	100.0	1.24	99.9	0.746	99.9	0.059	100.1	0.049

*Tentatively identified as a glucose conjugate of IN-QDY62

^a Consists of at least 3 components, none > 2.7% or 0.033 mg eq/kg

^b Percentages of identified and discrete unknown components subtracted from the total percentage of recovered residue chromatographed (i.e. areas of streaked radioactivity)

^c Net loss or gain on fractionation

^d Consists of at least 5 components, none > 5.6% or 0.042 mg eq/kg

^e Consists of at least 13 components, none > 3.5% or 0.002 mg eq/kg

^f Consists of at least 3 components, none > 8.7% or 0.004 mg eq/kg.

Table 30 Amount of picoxystrobin and metabolites in carrot roots grown in treated soil (indoor grown)

Residue component	^[14C] Pyridinyl label				^[14C] Phenacrylate label			
	30 day PBI		197 day PBI		30 day PBI		197 day PBI	
	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg
Picoxystrobin	5.5	0.020	10.7	0.021	26.0	0.008	28.8	0.010
IN-QDY62	–	–	–	–	0.5	< 0.001	–	–
IN-QDY63	–	–	–	–	–	–	1.0	< 0.001
IN-QFA35	–	–	–	–	0.5	< 0.001	–	–
IN-QGS45	1.2	0.004	2.6	0.005	–	–	–	–
IN-QGU72	11.1	0.041	5.4	0.011	–	–	–	–
IN-QGU73	28.7	0.106	7.6	0.015	–	–	–	–
R409665	–	–	1.7	0.003			5.9	0.002
PYCR2	14.9	0.055	15.1	0.030	–	–	–	–
PYCR3	24.3	0.090			–	–	–	–
PAF1*	–	–	–	–	15.7	0.005	14.4	0.005
Unknowns	8.2 ^a	0.030 ^a	7.7 ^d	0.015 ^d	3.4 ^e	0.001 ^e	4.5 ^f	0.001 ^f
Baseline	1.4	0.005	1.0	0.002	0.4	< 0.001	2.2	< 0.001
Unassigned ^b	2.7	0.010	20.9	0.041	2.0	< 0.001	6.0	0.002
Unchromatographed fractions	0.7	0.003	4.0	0.008	30.5	0.009	10.5	0.003
Post extraction solids	1.1	0.004	2.0	0.004	15.6	0.005	14.8	0.005
Filter papers	0.2	< 0.001	0.2	< 0.001	3.5	0.001	1.7	< 0.001
Losses on fractionation ^c	0.0	0.0	21.1	0.042	2.0	< 0.001	10.2	0.003
TOTAL	100.0	0.369	100.0	0.198	100.1	0.034	100.0	0.034

*Tentatively identified as a glucose conjugate of IN-QDY62

^a Consists of at least 2 components, none > 6.2% or 0.023 mg eq/kg

^b Percentages of identified and discrete unknown components subtracted from the total percentage of recovered residue chromatographed (i.e. areas of streaked radioactivity)

^c Net loss or gain on fractionation

^d Consists of at least 4 components, none > 4.2% or 0.008 mg eq/kg

^e Consists of at least 5 components, none > 1.6% or 0.001 mg eq/kg

^f Consists of at least 3 components, none > 2.1% or 0.001 mg eq/kg.

The majority of the residue in 30-day interval soil from the confined crop rotation study was parent compound (68.4–71.6% of TRR), dropping to 42.1–43.0% at the 197-day plant back interval.

Other components of the soil residue were the IN-QDK62, IN-QDK50, IN-QDY63, and the oxidised acrylic acid side chain metabolites R409665 and R416021.

The metabolic fate of picoxystrobin was relatively consistent across most of the rotational crop matrices (wheat straw and forage, carrot forage and roots and lettuce). IN-QDK50 and its conjugates IN-QGS45, IN-QGU72, IN-QGU73, PYST2, and PYCR2 and PYCR3 (the latter were not conclusively identified but were shown to be conjugates) were the most significant residue component (totals ranged from 30.7 to 80.2% of the TRR, or 0.061–4.79 mg eq/kg). Levels were highest in wheat straw and lowest in carrot roots. The free metabolite IN-QDK50 was only a small part of the total (0.9–14.2% of TRR, or < 0.001–1.58 mg eq/kg). The IN-QDK50 glutaryl glucosyl conjugate (IN-QGU73) and the compounds characterised as IN-QDK50 conjugates but not conclusively identified (PYCR2 and PYCR3) were only found in the carrot matrices, while the sulphate conjugate of IN-QDK50 (PYST2) was only found in wheat straw. Parent compound was found in all matrices except wheat grain, although mostly at < 10% of the TRR. A lower proportion of the residue was identified for the phenacrylate label than for the pyridinyl label, with generally higher proportions of radioactivity in the post-extraction solids, unknown metabolites and unassigned (streaked) fractions, suggesting perhaps a higher level of bound or naturally incorporated species.

Metabolism in grain differed from the other samples, with a proportion of the picoxystrobin residue incorporated in natural products, particularly glucose, with the total naturally incorporating radioactivity in grain amounting to 9.3–36.5% of the TRR or 0.006–0.024 mg/kg. Other residue components in grain, at a maximum of 0.016 mg eq/kg, were IN-QDK50, the oxidised side chain metabolite R409665, and the benzofuran carboxylic acid IN-H8612.

A rotational crop metabolism study was conducted for [¹⁴C]pyridinyl- and [¹⁴C]phenacrylate-labelled picoxystrobin in field-grown spring wheat, lettuce and carrots planted the season following application (Turner *et al.*, 1998b).

Spring wheat, carrots and lettuce were sown in small plots where the preceding crop was winter wheat that had been treated with two applications 39 days apart at 405–437 g ai/ha, giving a seasonal rate of 842 and 817 g ai/ha for the pyridinyl and phenacrylate labels.

Soil cores were taken at two intervals, just before plot tillage, 22 days after the harvest of the winter wheat crop (and 70 days after the final application), and immediately prior to sowing the rotational crops (304 days after the last application for spring wheat and 308 days after the last application for carrots and lettuce). Wheat forage was harvested at approximately BBCH stage 59 (inflorescence fully emerged), while straw, grain, lettuce and carrot leaves and roots were harvested at normal crop maturity.

Homogenised wheat forage and straw, lettuce and carrot leaf and root samples were extracted with solvents including acetonitrile, acetonitrile/water, and water. Grain samples were not extracted, as the total radioactive residues were < 0.01 mg eq/kg. Soil cores collected pre-tillage were separated into the top 10 cm and the remainder for analysis, while the pre-sowing cores were separated into the top 5 cm and the remainder. Soil samples were extracted using acetone/HCl, cleaned up by solid phase extraction and analysed using a method based on method number RAM 291/01 (Mason and French, 1996).

Residue components in plant sample extracts containing sufficient radioactive residues (> 0.01 mg eq/kg) were characterised and quantified using thin layer chromatography with co-chromatography with reference compounds. Only pyridinyl label wheat forage and straw and carrot foliage, and phenacrylate label wheat forage required chromatographic analysis.

Samples were stored frozen between collection and extraction, and all samples were extracted and analysed within 6 months of harvest.

Table 31 Total radioactive residues (TRRs) in crops grown in the field in rotation with a wheat crop treated with radiolabelled picoxystrobin (determined as summation of radioactivity in extracts and post-extraction solids) ^a

Sample	TRR (mg/kg parent equivalents)	
	[¹⁴ C]Pyridinyl label	[¹⁴ C]Phenacrylate label
Soil (pre-tillage, post-harvest of treated crop)	0.112	0.114
Soil (pre-sowing of rotational crops)	0.141 ^b	0.055 ^b
Wheat forage	0.070	0.018
Wheat grain	0.003	0.009
Wheat straw	0.145	0.023
Lettuce	0.004	< 0.001
Carrot leaves	0.054	0.002
Carrot roots	0.010	0.001

^a Wheat grain TRRs are those determined by direct combustion and LSC of the sample, all others are reported as the sum of the sample extract radioactivity and radioactivity in the post-extraction solids

^b Weighted mean of the results for the top 5 cm and lower sections of the soil core.

Table 32 Amount of picoxystrobin and metabolites in treated plot soil

Residue component	[¹⁴ C]Pyridinyl label				[¹⁴ C]Phenacrylate label			
	Pre-tillage		Pre-sowing		Pre-tillage		Pre-sowing	
	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg
Picoxystrobin	9.8	0.011	8.0	0.011	12.3	0.014	7.5	0.004
IN-QDY62	8.4	0.009	22.4	0.032	13.9	0.016	15.5	0.009
IN-QDK50	16.7	0.019	10.1	0.014	–	–	–	–
IN-QDY63	28.4	0.032	17.0	0.024	27.7	0.032	13.1	0.007
Unknowns	10.0 ^a	0.011 ^a	13.0 ^d	0.018 ^d	9.9 ^e	0.011 ^e	12.7 ^f	0.007 ^f
Baseline	0.5	< 0.001	1.2	0.002	0.7	< 0.001	1.5	0.001
Unassigned ^b	10.2	0.011	6.5	0.009	6.9	0.008	9.6	0.005
Unchromatographed fractions	1.2	0.001	2.3	0.003	3.9	0.004	6.7	0.004
Post extraction solids	11.2	0.013	11.5	0.016	18.3	0.021	23.7	0.013
Losses on fractionation ^c	3.6	0.004	8.1	0.011	6.4	0.007	9.7	0.005
TOTAL	100.0	0.112	100.1	0.140	100.0	0.114	100.0	0.055

^a Consists of at least 2 components, none > 6.2% or 0.007 mg eq/kg

^b Percentages of identified and discrete unknown components subtracted from the total percentage of recovered residue chromatographed (i.e. areas of streaked radioactivity)

^c Net loss or gain on fractionation

^d At least 3 discrete components, none greater than 7.6% or 0.011 mg eq/kg

^e At least 5 discrete components, none greater than 5.3% or 0.006 mg eq/kg

^f At least 3 discrete components, none >6.0% of TRR or 0.003 mg eq/kg.

In the pre-tillage soil samples, > 98% of the residue was found in the top 10 cm of the soil core. In the pre-sowing soil samples, total radioactive residue and individual metabolite levels were

similar in the top 5 cm and the remainder of the soil core. This indicates that the distribution of the picoxystrobin and metabolite residues throughout the core collected prior to sowing was the result of the tillage, rather than the result of leaching.

Results for the chromatographic analyses of wheat forage (both labels), and wheat straw and carrot leaves (pyridinyl label only) are shown in Tables 33-35.

Table 33 Amount of picoxystrobin and metabolites in rotational field grown wheat forage

Residue component	[¹⁴ C]Pyridinyl label		[¹⁴ C]Phenacrylate label	
	%TRR	mg/kg parent equivalents	%TRR	mg/kg parent equivalents
Picoxystrobin	1.3	< 0.001	1.3	< 0.001
IN-QDK50	3.1	0.002	–	–
IN-QGS45	5.4	0.004	–	–
IN-QGU72	31.2	0.022	–	–
Unknowns	20.6 ^a	0.014 ^a	44.3 ^b	0.008 ^b
Baseline	3.8	0.003	0.0	0.000
Unassigned ^c	19.6	0.014	19.9	0.004
Unchromatographed fractions	4.0	0.003	26.7	0.003
Post extraction solids	6.2	0.004	8.0	0.002
Filter papers	1.4	0.001	1.4	0.0003
Losses on fractionation ^d	4.7	0.003	-1.7	-0.0003
TOTAL	100.0	0.070	99.9	0.017

^a Consists of at least 7 components, none > 4.8% or 0.003 mg eq/kg

^b Consists of at least 7 components, none > 11.0% or 0.002 mg eq/kg

^c Percentages of identified and discrete unknown components subtracted from the total percentage of recovered residue chromatographed (i.e. areas of streaked radioactivity)

^d Net loss or gain on fractionation.

Table 34 Amount of picoxystrobin and metabolites in rotational field grown wheat straw

Residue component	[¹⁴ C]Pyridinyl label		[¹⁴ C]Phenacrylate label	
	%TRR	mg/kg parent equivalents	%TRR	mg/kg parent equivalents
IN-QDK50	2.0	0.003	Sample extracted, but no chromatographic analysis performed, as no extract contained greater than 0.01 mg/kg parent equivalents.	
IN-QGS45	36.0	0.052		
IN-QGU72	2.4	0.003		
Unknowns	5.8 ^a	0.008 ^a		
Baseline	1.8	0.003		
Unassigned ^b	10.2	0.015		
Unchromatographed fractions	13.6	0.020		
Post extraction solids	22.3	0.032		
Filter papers	3.29	0.005		
Losses on fractionation ^c	2.60	0.004		

Residue component	[¹⁴ C]Pyridinyl label		[¹⁴ C]Phenacrylate label	
	%TRR	mg/kg parent equivalents	%TRR	mg/kg parent equivalents
TOTAL	100.0	0.142		

^a Consists of at least 2 components, none > 3.8% or 0.006 mg eq/kg

^b Percentages of identified and discrete unknown components subtracted from the total percentage of recovered residue chromatographed (i.e. areas of streaked radioactivity)

^c Net loss or gain on fractionation.

Table 35 Amount of picoxystrobin and metabolites in rotational field grown carrot leaves

Residue component	[¹⁴ C]Pyridinyl label		[¹⁴ C]Phenacrylate label	
	%TRR	mg/kg parent equivalents	%TRR	mg/kg parent equivalents
Picoxystrobin	1.1	< 0.001	Sample extracted, but no chromatographic analysis performed, as no extract contained greater than 0.01 mg/kg parent equivalents.	
IN-QDY62	1.2	< 0.001		
IN-QGS45	6.4	0.003		
IN-QGU72	14.1	0.008		
IN-QGU73	42.6	0.023		
Unknowns	4.3 ^a	0.002 ^a		
Baseline	0.0	0.000		
Unassigned ^b	6.3	0.003		
Unchromatographed fractions	10.6	0.006		
Post extraction solids	3.6	0.002		
Filter papers	0.5	0.003		
Losses on fractionation ^c	9.4	0.005		
TOTAL	100.1	0.054		

^a Consists of at least 1 component.

^b Percentages of identified and discrete unknown components subtracted from the total percentage of recovered residue chromatographed (i.e. areas of streaked radioactivity). ^c Net loss or gain on fractionation.

The identified residue components in soil were parent, IN-QDY62, IN-QDK50, and IN-QDY63, accounting for a total of 36.1–63.3% of the TRR or 0.020–0.081 mg eq/kg. This pattern was similar to that observed in soil for the confined crop rotation study, except that metabolism was more extensive, with a significantly lower proportion of parent being found in field soil.

None of the human food commodities tested in the field rotational crop metabolism study (carrots, lettuce and wheat grain) contained total residues above 0.01 mg eq/kg after application of picoxystrobin to a preceding crop at a seasonal rate of approximately 820 g ai/ha.

In the animal feed commodities tested in the field rotational trial (wheat forage and straw and carrot leaves), the most significant residue components were IN-QDK50 and its glucosyl, malonyl glucosyl and glutaryl glucosyl conjugates (IN-QGS45, IN-QGU72, and IN-QGU73 respectively). The glutaryl glucosyl conjugate was only found in carrot leaves. The total fractions of IN-QDK50 and conjugates in field grown rotational animal feed commodities were 39.7–63.1% of the TRR, or 0.028–0.058 mg eq/kg. The unconjugated metabolite IN-QDK50 comprised only a small part of the totals, at

2.0–3.1% of TRR, or 0.002–0.003 mg eq/kg. Small amounts of parent and IN-QDY62 were found in some commodities.

A further field rotational cropping metabolism study was conducted in winter wheat (Bramley *et al.*, 1998).

Winter wheat was sown in small plots previously treated with [¹⁴C]pyridinyl- or [¹⁴C]phenacrylate-picoxystrobin at 793 and 704 g ai/ha respectively in two applications made to the earlier spring wheat crop grown in the plots. The winter wheat was sown 107 days after the last application of picoxystrobin, and 50 days after harvest of the spring wheat.

Soil cores were sampled 21 days after harvest of the spring wheat, prior to tillage of the soil. Wheat forage was harvested at approximately BBCH stage 67 (late flowering, around 70% of anthers mature), while grain and straw were collected at normal crop maturity.

Homogenised wheat forage and straw samples were extracted with solvents including acetonitrile, acetonitrile/water, and water. Grain samples were not extracted, as the total radioactive residues were < 0.01 mg eq/kg. Soil cores collected pre-tillage were separated into the top 5 cm and the remainder for analysis. Soil samples were extracted using acetone/HCl, cleaned up by solid phase extraction and analysed using a method based on method number RAM 291/01 (Mason and French, 1996).

Residue components in plant sample extracts containing sufficient radioactive residues (> 0.01 mg eq/kg) were characterised and quantified using thin layer chromatography with co-chromatography with reference compounds. Matching of chromatographs with those from the confined crop rotation study was used to aid in component identification. Only pyridinyl label wheat forage and straw for both labels required chromatographic analysis.

Samples were stored frozen between collection and extraction, and all samples were extracted and analysed within 4 months of harvest.

Table 36 Total radioactive residues (TRRs) in rotational winter wheat grown in the field following a spring wheat crop treated with radiolabelled picoxystrobin (determined as summation of radioactivity in extracts and post-extraction solids)

Sample	TRR (mg/kg parent equivalents)	
	[¹⁴ C]Pyridinyl label	[¹⁴ C]Phenacrylate label
Soil (pre-tillage, post-harvest of treated crop): top 5 cm of core	0.146	0.168
Soil (pre-tillage, post-harvest of treated crop): lower core	0.008 ^a	0.042
Wheat forage	0.017	0.011
Wheat grain	0.003 ^a	0.005 ^a
Wheat straw	0.086	0.043

^a Wheat grain and pyridinyl label lower soil TRRs are those determined by direct combustion and LSC of the sample, all others are reported as the sum of the sample extract radioactivity and radioactivity in the post-extraction solids.

Table 37 Amount of picoxystrobin and metabolites in pre-tillage treated plot soil

Residue component	[¹⁴ C]Pyridinyl label				[¹⁴ C]Phenacrylate label			
	Top 5 cm		Lower section		Top 5 cm		Lower section	
	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg
Picoxystrobin	15.5	0.023	Not analysed, as TRR in lower section pyridinyl label soil		14.8	0.025	13.5	0.006
IN-QDY62	23.0	0.034			22.3	0.037	12.8	0.005

Residue component	[¹⁴ C]Pyridinyl label				[¹⁴ C]Phenacrylate label			
	Top 5 cm		Lower section		Top 5 cm		Lower section	
	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg
IN-QDK50	5.0	0.007	was < 0.01 mg/kg.		–	–	–	–
IN-QDY63	16.1	0.024			13.7	0.023	11.2	0.005
Unknowns	3.2 ^a	0.005 ^a			4.8 ^d	0.008 ^d	4.5 ^e	0.002 ^e
Baseline	2.2	0.003			2.3	0.004	2.0	< 0.001
Unassigned ^b	5.6	0.008			2.8	0.005	3	0.002
Post extraction solids	19.8	0.029			38.4	0.065	41.1	0.017
Losses on fractionation ^c	9.6	0.014			0.9	0.002	11.3	0.005
TOTAL	100.0	0.146			100.0	0.168	100.0	0.042

^a Consists of at least 2 components, none > 2.2% or 0.003 mg eq/kg

^b Percentages of identified and discrete unknown components subtracted from the total percentage of recovered residue chromatographed (i.e. areas of streaked radioactivity)

^c Net loss or gain on fractionation

^d At least 3 discrete components, none greater than 2.2% or 0.004 mg eq/kg

^e At least 3 discrete components, none greater than 1.8% or 0.001 mg eq/kg.

Results for the chromatographic analyses of wheat forage (pyridinyl label only), and wheat straw (both labels) are tabulated below.

Table 38 Amount of picoxystrobin and metabolites in rotational field grown winter wheat forage and straw

Residue component	[¹⁴ C]Pyridinyl label				[¹⁴ C]Phenacrylate label	
	Wheat forage		Wheat straw		Wheat straw	
	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg
IN-QDK50	–	–	6.9	0.006	–	–
IN-QGS45	10.4	0.002	3.9	0.003	–	–
IN-QGU72	24.6	0.004	7.8	0.007	–	–
IN-QDY63	–	–	–	–	3.3	0.001
PYST2	–	–	19.1	0.016	–	–
Unknowns	5.4 ^a	< 0.001 ^a	3.9	0.003	17.2 ^b	0.007 ^b
Baseline	ND	ND	3.5	0.003	8.8	0.004
Unassigned ^c	19.7	0.003	10.6	0.009	28.8	0.012
Unchromatographed fractions	28.1	0.005	9.7	0.008	5.4	0.002
Post extraction solids	6.1	0.001	13.5	0.012	25.3	0.011
Filter papers	1.6	< 0.001	1.3	0.001	4.6	0.002
Losses on fractionation ^d	4.0	< 0.001	19.8	0.017	6.6	0.003
TOTAL	99.9	0.017	100.0	0.086	100.0	0.042

^a Consists of at least 2 components, none > 3.0% or 0.001 mg/kg

^b Consists of at least 4 components, none > 4.9% or 0.002 mg eq/kg

^c Percentages of identified and discrete unknown components subtracted from the total percentage of recovered residue chromatographed (i.e. areas of streaked radioactivity)

^d Net loss or gain on fractionation.

The identified residue components in soil were parent, IN-QDY62, IN-QDK50, and IN-QDY63, accounting for a total of 37.5–59.6% of the TRR or 0.016–0.088 mg eq/kg. The soil metabolites were the same as those observed for the other field rotation study and the confined rotation study. The proportions of each residue component differed, with a higher proportion of parent being found in the soil from the confined rotational experiment.

In forage and straw, the most significant residue components were IN-QDK50 and its glucosyl and malonyl glucosyl conjugates (IN-QGS45 and IN-QGU72 respectively). The sulphate conjugate of IN-QDK50, PYST2, was also found in straw. The total fractions of IN-QDK50 and conjugates in field grown rotational wheat straw and forage were 35.0–37.7% of the TRR, or 0.006–0.032 mg eq/kg for the pyridinyl label. The unconjugated metabolite comprised only a small fraction of this total, as a maximum of 0.006 mg eq/kg, or 6.9% of TRR. Only IN-QDY63 was identified in phenacrylate label straw, at 0.001 mg eq/kg or 3.3% of the TRR.

The results of the confined and field crop rotational metabolism studies were consistent, with the majority of the residue in plant matrices comprising the soil and plant metabolite IN-QDK50 and its conjugates with glucose, malonic acid and glutaric acid formed in the plant. The metabolism of picoxystrobin in rotational cropping is summarized in Figure 7.

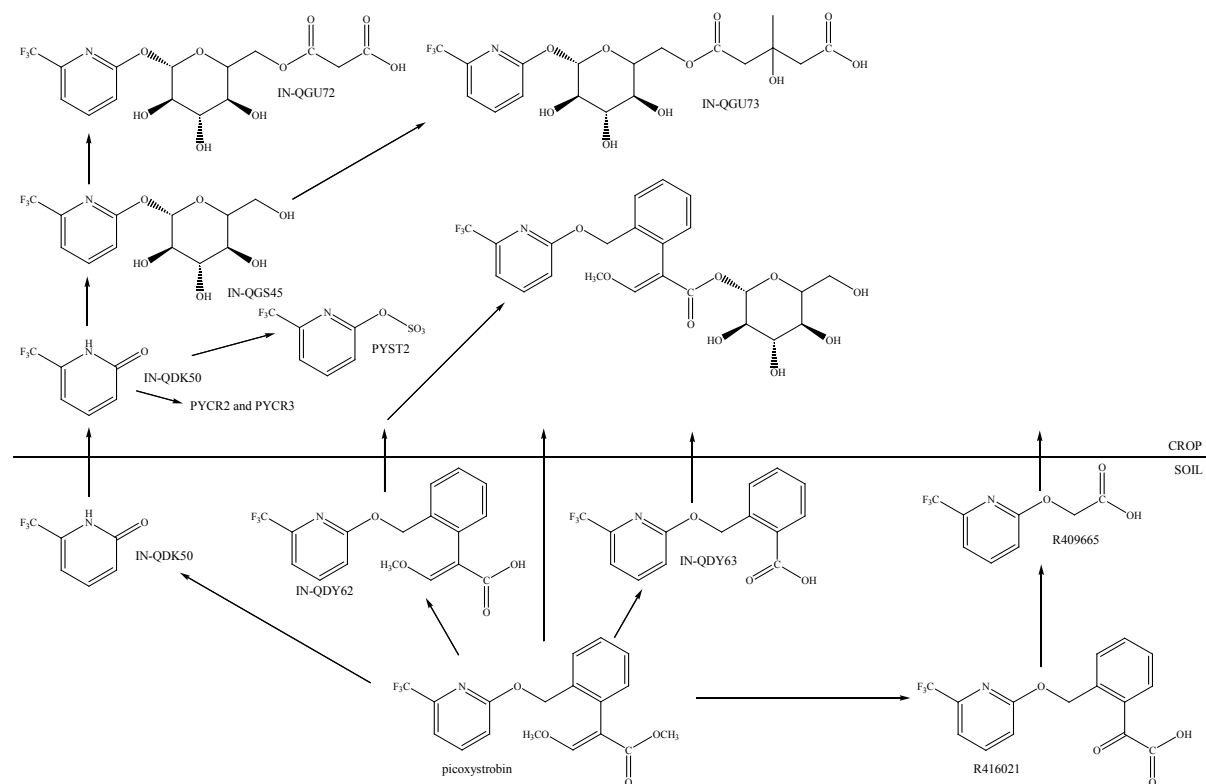


Figure 7 Metabolism of picoxystrobin in rotational crops

METHODS OF RESIDUE ANALYSIS

Methods of residue analysis

Details of analytical methods, including validation data, were supplied for the determination of picoxystrobin and key metabolites in plant and animal matrices, and soil.

*Plant matrices**Method number 24868*

An LC/MS/MS method was developed for the determination of residues of picoxystrobin and the metabolites IN-QDK50, IN-QDY62 and IN-QDY63 in plant matrices (Cabusas and Morgan, 2009 and Chickering and Cabusas, 2009). Samples were extracted using 9:1 v/v acetonitrile/water then extracts were filtered or centrifuged and cleaned up by solid phase extraction (hydrophile-lipophile balance (HLB) cartridges). Samples were analysed by liquid chromatography (conventional or ultra-high performance liquid chromatography; UPLC) with triple quadrupole mass spectrometric detection (LC/MS/MS). Two parent-daughter ion transitions were monitored for each analyte to provide method confirmation (see Table 39 for the transitions used).

Table 39 Recovery data for picoxystrobin and metabolites in plant matrices using method number 24868

Analyte	Matrix	Fortification (mg/kg)	n	Recovery (%) ^a		
				Range	Mean ± RSD	
Picoxystrobin Quantification transition: 368 → 145; Confirmation transition: 368 → 205	Maize stover	0.01	6	92–106	98 ± 5	
		0.10	6	88–101	94 ± 5	
		overall	12	88–106	96 ± 6	
	Maize grain	0.01	5	83–96	90 ± 6	
		0.10	5	79–92	86 ± 7	
		overall	10	79–96	88 ± 6	
	Maize oil	0.01	5	90–117	107 ± 10	
		0.10	5	98–105	101 ± 3	
		overall	10	90–117	104 ± 8	
	Soya bean seed	0.01	5	79–85	82 ± 3	
		0.10	5	84–89	87 ± 2	
		overall	10	79–89	84 ± 4	
	Dried pea	0.01	5	92–100	96 ± 3	
		0.10	5	90–102	95 ± 5	
		overall	10	90–102	96 ± 4	
	Lettuce	0.01	5	92–104	100 ± 5	
		0.10	5	93–106	99 ± 5	
		overall	10	92–106	99 ± 5	
	Orange	0.01	5	86–115	97 ± 12	
		0.10	5	89–115	102 ± 10	
		overall	10	86–115	100 ± 11	
	Oilseed rape plant	0.01	5	91–104	96 ± 6	
		0.10	5	82–94	86 ± 6	
		overall	10	82–104	94 ± 8	
	Oilseed rape seed	0.01	4	90–105	97 ± 6	
		0.10	5	90–99	93 ± 4	
		overall	9	90–105	95 ± 5	
	IN-QDK50	Maize stover	0.01	6	75–110	97 ± 16

Analyte	Matrix	Fortification (mg/kg)	n	Recovery (%) ^a		
				Range	Mean ± RSD	
Quantification transition: 164 → 144; Confirmation transition: 164 → 116		0.10	6	75–103	95 ± 11	
		overall	12	75–110	96 ± 13	
		Maize grain	0.01	5	76–90	86 ± 6
	Maize grain	0.10	5	74–84	79 ± 5	
		overall	10	74–90	82 ± 7	
		Maize oil	0.01	5	89–103	96 ± 6
	Maize oil	0.10	5	86–94	90 ± 4	
		overall	10	86–103	93 ± 6	
		Soya bean seed	0.01	5	73–80	76 ± 4
	Soya bean seed	0.10	5	76–88	82 ± 6	
		overall	10	73–88	79 ± 6	
		Dried pea	0.01	5	92–101	96 ± 4
	Dried pea	0.10	5	83–88	85 ± 3	
		overall	10	83–101	91 ± 7	
		Lettuce	0.01	5	98–105	101 ± 3
	Lettuce	0.10	5	86–97	91 ± 5	
		overall	10	86–105	96 ± 6	
		Orange	0.01	5	98–117	108 ± 7
	Orange	0.10	5	91–96	93 ± 2	
		overall	10	91–117	101 ± 9	
		Oilseed rape plant	0.01	5	73–105	86 ± 14
	Oilseed rape plant	0.10	5	72–90	81 ± 9	
		overall	10	72–105	84 ± 11	
		Oilseed rape seed	0.01	5	77–97	83 ± 10
	Oilseed rape seed	0.10	5	80–95	87 ± 7	
		overall	10	77–97	85 ± 9	
		IN-QDY62	Maize stover	0.01	6	100–112
	Maize stover		0.10	6	83–97	89 ± 6
			overall	12	83–112	97 ± 11
	Quantification transition: 354 → 191; Confirmation transition: 354 → 145	Maize grain	0.01	5	83–115	100 ± 11
Maize grain		0.10	5	88–108	97 ± 8	
		overall	10	83–115	99 ± 10	
Maize oil	0.01	5	97–109	104 ± 5		
	0.10	5	90–102	97 ± 5		
	overall	10	90–109	101 ± 6		
Soya bean seed	0.01	5	85–87	86 ± 1		
	0.10	5	84–91	87 ± 3		
	overall	10	84–91	86 ± 2		
Dried pea	0.01	5	80–97	92 ± 8		

Analyte	Matrix	Fortification (mg/kg)	n	Recovery (%) ^a	
				Range	Mean ± RSD
		0.10	5	82–100	88 ± 9
		overall	10	80–100	90 ± 8
		0.01	5	106–112	108 ± 2
	Lettuce	0.10	5	102–110	107 ± 3
		overall	10	102–111	107 ± 3
		0.01	5	97–128	111 ± 12
	Orange	0.10	5	90–113	98 ± 9
		overall	10	90–128	104 ± 12
		0.01	5	86–96	93 ± 4
Oilseed rape plant	0.10	5	85–96	91 ± 5	
	overall	10	85–96	92 ± 5	
	0.01	5	97–107	102 ± 4	
Oilseed rape seed	0.10	5	94–109	103 ± 6	
	overall	10	94–109	103 ± 5	
	IN-QDY63 Quantification transition: 298 → 164; Confirmation transition: 298 → 135	Maize stover	0.01	6	68–74
0.10			6	77–97	84 ± 9
overall			12	68–97	78 ± 11
Maize grain		0.01	5	90–98	93 ± 4
		0.10	5	82–90	87 ± 3
		overall	10	82–98	90 ± 5
Maize oil		0.01	5	107–112	110 ± 2
		0.10	5	100–105	102 ± 2
		overall	10	100–112	106 ± 4
Soya bean seed	0.01	5	84–88	86 ± 2	
	0.10	5	85–88	86 ± 2	
	overall	10	84–88	86 ± 2	
Dried pea	0.01	5	92–102	98 ± 4	
	0.10	5	94–101	98 ± 3	
	overall	10	92–102	98 ± 3	
Lettuce	0.01	5	96–108	103 ± 6	
	0.10	5	100–105	104 ± 2	
	overall	10	96–108	103 ± 4	
Orange	0.01	5	101–107	104 ± 2	
	0.10	5	89–110	99 ± 7	
	overall	10	89–110	102 ± 6	
Oilseed rape plant	0.01	5	87–92	89 ± 2	
	0.10	5	86–97	93 ± 4	
	overall	10	86–97	91 ± 4	
Oilseed rape seed	0.01	5	84–103	91 ± 8	

Analyte	Matrix	Fortification (mg/kg)	n	Recovery (%) ^a	
				Range	Mean ± RSD
		0.10	5	92–103	97 ± 5
overall	10	84–103	94 ± 7		

^a Recovery values shown are for the quantification transitions.

Method number 24868 was successfully validated, with minor modifications to the method, by an independent laboratory (Nasca, 2010).

Table 40 Independent laboratory recovery data for method number 24868

Analyte	Matrix	Fortification (mg/kg)	n	Recovery (%)	
				Range	Mean ± RSD
Picoxystrobin Quantification transition: 368 → 145; Confirmation transition: 368 → 205	Maize stover	0.01	5	84–97	87 ± 6.3
		0.10	5	77–95	88 ± 8.0
		overall	10	77–97	88 ± 6.8
	Leaf lettuce	0.01	5	96–99	98 ± 1.3
		0.10	5	94–98	96 ± 1.9
		overall	10	94–99	97 ± 1.9
IN-QDK50 Quantification transition: 164 → 144; Confirmation transition: 164 → 116	Maize stover	0.01	5	70–84	76 ± 8.1
		0.10	5	70–79	73 ± 5.3
		overall	10	70–84	74 ± 6.7
	Leaf lettuce	0.01	5	87–113	103 ± 9.8
		0.10	5	90–96	94 ± 2.5
		overall	10	87–113	98 ± 8.8
IN-QDY62 Quantification transition: 354 → 145; Confirmation transition: 354 → 191	Maize stover	0.01	5	78–92	83 ± 7.4
		0.10	5	93–105	98 ± 5.1
		overall	10	78–105	91 ± 11
	Leaf lettuce	0.01	5	77–88	83 ± 4.8
		0.10	5	84–90	87 ± 2.6
		overall	10	77–90	85 ± 4.5
IN-QDY63 Quantification transition: 298 → 135; Confirmation transition: 298 → 164	Maize stover	0.01	5	74–105	89 ± 13
		0.10	5	94–109	101 ± 5.8
		overall	10	74–109	95 ± 11
	Leaf lettuce	0.01	5	83–104	94 ± 8.4
		0.10	5	92–96	94 ± 1.6
		overall	10	83–104	94 ± 5.4

Method number RAM 288/01

An earlier method for determination of residues of picoxystrobin (parent compound only) using GC/MS or LC/MS/MS was validated for cereal grains, straw and forage (Patel, 1996). Samples were

extracted with 9:1 v/v acetonitrile/water, and extracts were cleaned up by C18 and silica solid phase extraction prior to analysis.

Table 41 Recovery of picoxystrobin from cereal matrices (method number RAM 288/01)

Matrix	Fortification level	GC/MS % recoveries (detection ion: m/z = 335; alternative ion: m/z = 303)	LC/MS/MS % recoveries (quantification transition: 368.0 → 145.0)
Cereal straw	0.01	79, 81, 82	82, 88, 88
	0.05	77, 78	78, 78
	0.10	73, 80	81, 86
	0.50	68, 72	74, 78
Cereal grain	0.01	102, 105, 105, 109	98, 101, 103, 104
	0.05	107, 113	89, 91
	0.10	105, 108	96, 99
	0.50	96, 96	91, 93
Cereal forage	0.01	90, 94, 95, 102	87, 87, 93, 95
	0.05	92, 107	84, 87
	0.10	101, 109	78, 92
	0.50	98, 106	86, 101

An independent validation of this method (Kennedy, 1999) in wheat grain gave recoveries of 91–115% (mean = 102%, n = 5, RSD = 10%), and 78–111% (mean = 95%, n = 5, RSD = 14%) at fortifications of 0.01 and 0.20 mg/kg respectively.

Animal matrices

Method number 25997

An LC/MS/MS method was developed for analysis of picoxystrobin in animal matrices (Cabusas, 2010). Samples were extracted with acetonitrile, centrifuged and aliquots diluted for analysis. Two transitions (368.3 → 145.2 and 368.3 → 205.3) were used for quantification (total ion current). The results of the individual transitions were checked for confirmation by ratios. The method was validated in eggs, milk (including whole milk, skim milk and cream), and bovine fat, muscle, liver and kidney (see Table 42 below).

Table 42 Recovery data for picoxystrobin in animal matrices using method number 25997

Matrix	Fortification (mg/kg)	n	Recovery (%) ^a	
			Range	Mean ± RSD
Egg	0.01	5	87–94	90 ± 3.0
	0.10	5	92–96	94 ± 1.8
	overall	10	87–96	92 ± 3.2
Milk	0.01	5	96–110	102 ± 5.7
	0.10	5	83–90	86 ± 3.4
	overall	10	83–110	94 ± 10
Skim milk	0.01	8	81–106	95 ± 10
	0.10	8	78–101	90 ± 10
	overall	16	78–106	93 ± 10

Matrix	Fortification (mg/kg)	n	Recovery (%) ^a	
			Range	Mean ± RSD
Cream	0.01	5	67–109	86 ± 20
	0.10	5	75–109	90 ± 15
	overall	10	67–109	88 ± 17
Bovine muscle	0.01	5	87–96	92 ± 4.0
	0.10	5	97–101	99 ± 1.8
	overall	10	87–101	96 ± 5.1
Bovine kidney	0.01	5	94–99	97 ± 2.5
	0.10	5	97–101	99 ± 1.8
	overall	10	94–101	98 ± 2.3
Bovine liver	0.01	5	86–93	89 ± 3.5
	0.10	5	92–97	93 ± 2.3
	overall	10	86–97	91 ± 3.7
Bovine fat	0.01	5	95–104	100 ± 3.5
	0.10	5	89–97	92 ± 3.2
	overall	10	89–104	96 ± 5.1

^a Recovery values are for the quantification transition.

The method was successfully independently validated with minor modifications (Oden and Whitsel, 2010). The same transitions were monitored as for the original study.

Table 43 Recovery data from independent validation of method number 25997

Matrix	Fortification (mg/kg)	n	Recovery (%)	
			Range	Mean ± RSD
Egg	0.01	5	76–92	84 ± 8.6
	0.10	5	76–88	82 ± 5.7
	overall	10	76–92	83 ± 7.0
Milk	0.01	5	84–108	92 ± 10
	0.10	5	84–94	90 ± 5.5
	overall	10	84–108	91 ± 8.0
Bovine liver	0.01	5	93–110	103 ± 6.3
	0.10	5	92–108	98 ± 7.1
	overall	10	92–110	101 ± 6.7

Method number RAM 304/01

An earlier method for determination of picoxystrobin (parent compound only) residues in animal commodities was developed using GC/MS (Hargreaves, 1998). Samples were extracted by homogenisation with acetonitrile, then centrifuged and aliquots of the extract were subjected to clean up by solid phase extraction (C18 and silica columns) before GC/MS analysis in selected ion monitoring mode (quantification ion: 335; alternative ion: 303). Recovery data are given in Table 44. A method limit of quantification of 0.001 mg/kg was achieved for milk, and 0.01 mg/kg for egg and tissue samples.

Table 44 Recovery data for picoxystrobin in animal matrices using method number RAM 304/01

Matrix	Fortification (mg/kg)	n	Recovery (%)	
			Individual values	Mean \pm RSD
Egg	0.01	4	98, 103, 103, 108	103 \pm 4.0
	0.05	2	95, 96	96
	0.10	2	96, 97	97
	0.20	2	92, 97	95
	overall	10		99 \pm 4.8
Milk	0.001	4	104, 106, 109, 112	108 \pm 3.2
	0.005	2	94, 98	96
	0.01	8	74, 90, 90, 91, 98, 99, 107, 108	95 \pm 12
	0.02	2	95, 96	96
	0.05	2	83, 110	97
	0.10	2	95, 97	96
	0.20	2	77, 107	92
	overall	22		97 \pm 11
Bovine muscle	0.01	4	95, 96, 100, 102	98 \pm 3.4
	0.05	2	91, 100	96
	0.10	2	92, 94	93
	0.20	2	93, 94	94
	overall	10		96 \pm 3.9
Bovine liver	0.01	8	85, 86, 89, 90, 95, 104, 113, 115	97 \pm 12
	0.05	2	105, 119	112
	0.10	2	84, 99	92
	0.20	2	105, 107	106
	overall	14		100 \pm 12
Bovine fat	0.01	4	80, 88, 106, 109	96 \pm 15
	0.05	2	82, 112	97
	0.10	2	68, 79	74
	0.20	2	97, 104	101
	overall	10		93 \pm 16

Table 45 Recovery data obtained by independent laboratory validation of method number RAM 304/01 (Kennedy, 1999)

Matrix	Fortification (mg/kg)	n	Recovery (%)	
			Range	Mean \pm RSD
Egg	0.01	5	81–108	92 \pm 13
	0.05	5	88–108	98 \pm 8.6
	overall	10	81–108	95 \pm 11
Milk	0.001	5	79–103	90 \pm 10

Matrix	Fortification (mg/kg)	n	Recovery (%)	
			Range	Mean ± RSD
	0.02	5	85–97	92 ± 5.3
	overall	10	79–103	91 ± 7.6
	Bovine muscle	0.01	5	73–87
	0.05	5	85–93	89 ± 4.2
	overall	10	73–93	86 ± 6.6
	Bovine liver	0.01	5	65–95
	0.05	5	74–82	78 ± 3.9
	overall	10	65–95	78 ± 13
	Bovine kidney	0.01	5	97–130
	0.05	5	77–90	82 ± 6.4
	overall	10	77–130	95 ± 17

Method number RAM 383/01

A modification of method number RAM 304/01 involving analysis by LC/MS/MS rather than GC/MS was developed partly as a confirmation of the GC/MS method (Hargreaves, 2002). The extraction and clean up procedures were essentially the same as for RAM 304/01. The 368 → 145 transition was used for quantification.

Table 46 Recovery data for LC/MS/MS method number RAM 383/01

Matrix	Fortification (mg/kg)	n	Recovery (%)	
			Range	Mean ± RSD
Egg	0.01	5	81–93	88 ± 5.1
	0.10	5	77–93	85 ± 7.1
	overall	10	77–93	86 ± 6.0
Milk	0.001	5	88–107	99 ± 8.2
	0.01	5	90–103	96 ± 6.5
	overall	10	88–107	97 ± 7.2
Bovine muscle	0.01	5	85–97	90 ± 4.9
	0.10	5	89–97	93 ± 4.1
	overall	10	85–97	92 ± 4.6
Bovine fat	0.01	5	82–92	86 ± 4.6
	0.10	5	84–89	87 ± 2.2
	overall	10	82–92	87 ± 3.4
Bovine liver	0.01	5	71–80	75 ± 5.4
	0.10	5	82–87	83 ± 2.5
	overall	10	71–87	79 ± 6.7
Bovine kidney	0.01	5	92–103	98 ± 4.6
	0.10	5	96–99	98 ± 1.4
	overall	10	92–103	98 ± 3.2

Soil

Method number 24804

An LC/MS/MS method was developed and validated for analysis of picoxystrobin and the metabolites IN-QDK50, IN-QDY62 and IN-QDY63 in four different North American soils (Morgan *et al.*, 2010 and Cabusas, 2010). Samples were extracted by sequential shaking with 75:25 acetone/HCl and neat acetone, followed by centrifuging, combination of the extracts, and clean-up of aliquots of the combined extract by solid phase extraction before analysis. Two transitions were monitored per analyte for the purpose of providing confirmation. The method showed good linearity and a limit of quantification of 0.01 mg/kg. Recoveries are given in Table 47. Acceptable recoveries could also be achieved without the clean-up step if sample extracts were filtered prior to analysis and a sufficiently sensitive LC/MS/MS instrument was used. The recoveries for the Wisconsin and Prince Edward Island sandy loam soil samples were achieved with the clean-up, while those for the Illinois clay loam and Ohio silty clay were determined without clean-up. Matrix effects were monitored by comparing results for pre- and post-extraction fortification, and shown to be minimal.

Table 47 Recovery data for method number 24804 for analysis of picoxystrobin and metabolites in soil

Analyte	Matrix	Fortification (mg/kg)	n	Recovery (%)	
				Range	Mean \pm RSD
Picoxystrobin Quantification transition: 368 \rightarrow 145; Confirmation transition: 368 \rightarrow 205	Sandy loam, Wisconsin, USA	0.01	5	98–104	102 \pm 2.4
		0.40	5	100–113	108 \pm 4.8
		overall	10	98–113	105 \pm 4.6
	Sandy loam, Prince Edward Island, USA	0.01	5	90–100	93 \pm 4.3
		0.40	5	101–109	105 \pm 2.8
		overall	10	90–109	99 \pm 7.1
	Clay loam, Drummer, Illinois, USA	0.01	5	91–95	93 \pm 1.8
		0.10	5	95–100	98 \pm 2.5
		overall	10	91–100	95 \pm 3.2
	Silty clay, Tama, Ohio, USA	0.01	5	89–96	95 \pm 3.3
		0.10	5	90–95	93 \pm 2.1
		overall	10	89–96	94 \pm 2.7
IN-QDK50 Quantification transition: 164 \rightarrow 116; Confirmation transition: 164 \rightarrow 144	Sandy loam, Wisconsin, USA	0.01	5	103–110	107 \pm 3.1
		0.40	5	102–106	104 \pm 1.5
		overall	10	103–110	105 \pm 2.7
	Sandy loam, Prince Edward Island, USA	0.01	5	75–103	90 \pm 14
		0.40	5	103–119	108 \pm 5.9
		overall	10	75–119	99 \pm 13
	Clay loam, Drummer, Illinois, USA	0.01	5	85–109	98 \pm 10
		0.10	5	89–95	91 \pm 2.7
		overall	10	85–109	95 \pm 8.0
	Silty clay, Tama, Ohio, USA	0.01	5	88–103	93 \pm 6.4
		0.10	5	80–85	83 \pm 2.3
		overall	10	80–103	88 \pm 7.8
IN-QDY62	Sandy loam,	0.01	5	99–110	103 \pm 4.0

Analyte	Matrix	Fortification (mg/kg)	n	Recovery (%)	
				Range	Mean \pm RSD
Quantification transition: 354 \rightarrow 131 (SPE clean-up variation), 354 \rightarrow 191 (no clean-up variation); Confirmation transition: 354 \rightarrow 145	Wisconsin, USA	0.40	5	103–109	106 \pm 2.6
		overall	10	99–110	105 \pm 3.4
		Sandy loam, Prince Edward Island, USA	0.01	5	103–120
	0.40		5	96–104	100 \pm 3.5
	overall		10	96–120	103 \pm 6.4
	Clay loam, Drummer, Illinois, USA	0.01	5	91–96	94 \pm 1.9
		0.10	5	96–100	98 \pm 1.7
		overall	10	91–100	96 \pm 2.9
	Silty clay, Tama, Ohio, USA	0.01	5	91–98	96 \pm 3.1
		0.10	5	91–96	93 \pm 2.2
		overall	10	91–98	95 \pm 2.9
	IN-QDY63 Quantification transition: 298 \rightarrow 135; Confirmation transition: 298 \rightarrow 164	Sandy loam, Wisconsin, USA	0.01	5	96–102
0.40			5	103–104	103 \pm 0.5
overall			10	96–104	101 \pm 3.1
Sandy loam, Prince Edward Island, USA		0.01	5	89–96	93 \pm 2.8
		0.40	5	96–98	97 \pm 0.9
		overall	10	89–98	95 \pm 3.0
Clay loam, Drummer, Illinois, USA		0.01	5	89–94	91 \pm 2.4
		0.10	5	104–112	107 \pm 2.9
		overall	10	89–112	99 \pm 9.1
Silty clay, Tama, Ohio, USA		0.01	5	87–99	94 \pm 5.7
		0.10	5	102–106	104 \pm 1.7
		overall	10	87–106	99 \pm 6.7

The method was successfully independently validated without the solid phase extraction clean-up step (Rudroff, 2010), while problems were encountered when validating the method with the clean-up, particularly for metabolite IN-QDK50.

Table 48 Independent recovery data for method number 24804 for analysis of picoxystrobin and metabolites in soil (no SPE clean-up)^a

Analyte	Matrix	Fortification (mg/kg)	n	Recovery (%)	
				Range	Mean \pm RSD
Picoxystrobin	Clay loam, Texas, USA	0.01	5	100–116	108 \pm 5.6
		0.40	5	98–107	103 \pm 3.3
		overall	10	98–116	105 \pm 4.9
IN-QDK50	Clay loam, Texas, USA	0.01	5	90–117	103 \pm 10
		0.40	5	98–107	102 \pm 3.3
		overall	10	90–117	103 \pm 7.3
IN-QDY62	Clay loam, Texas, USA	0.01	5	98–104	101 \pm 2.3
		0.40	5	96–104	99 \pm 3.1

Analyte	Matrix	Fortification (mg/kg)	n	Recovery (%)	
				Range	Mean \pm RSD
		overall	10	96–104	100 \pm 2.7
IN-QDY63	Clay loam, Texas, USA	0.01	5	96–106	100 \pm 3.8
		0.40	5	106–115	110 \pm 3.3
		overall	10	96–115	105 \pm 5.8

^a The same transitions were used for quantification and confirmation as for the no clean-up variation of the original method no. 24804.

Method number RAM 291/01

An earlier GC/MS method was developed and validated for analysis of picoxystrobin and the metabolites IN-QDK50 (ZA1963/03), IN-QDY62 (ZA1963/02) and IN-QDY63 (ZA1963/08) in soil (Mason and French, 1996). The extraction and clean-up methods were essentially the same as those for method number 24804, with the addition of derivatisation of the samples with *N*-methyl-*N*-(*tert*-butyldimethylsilyl) trifluoroacetamide and *tert*-butyldimethylchlorosilane prior to GC/MS analysis. The detection ions were $m/z = 145$, with $m/z = 335$ as an alternative ion for parent, and $m/z = 220$ for the three metabolites.

Table 49 Recovery data for GC/MS method number RAM 291/01 for analysis of picoxystrobin and metabolites in soil

Analyte	Matrix	Fortification (mg/kg)	n	Recovery (%)	
				Range	Mean \pm RSD
Picoxystrobin	Hyde Farm, UK	0.01	5	83–112	96 \pm 11
		0.05	3	97–106	101 \pm 4.5
		0.10	3	95–99	97 \pm 2.1
		0.50	3	107–112	109 \pm 2.7
		1.00	3	88–91	89 \pm 1.9
		overall	17	83–112	98 \pm 8.7
	Poitou-Charentes, France	0.01	5	88–107	93 \pm 8.5
		0.05	3	98–106	101 \pm 4.1
		0.10	3	102–112	106 \pm 5.0
		0.50	3	93–97	95 \pm 2.1
		1.00	3	90–100	94 \pm 5.6
		overall	17	88–112	97 \pm 7.3
	IN-QDK50	Hyde Farm, UK	0.01	5	85–103
0.05			3	101–107	104 \pm 2.9
0.10			3	96–107	101 \pm 5.4
0.50			3	89–104	98 \pm 8.0
1.00			3	87–92	90 \pm 2.9
overall			17	85–107	97 \pm 7.5
Poitou-Charentes, France		0.01	5	110–126	116 \pm 5.2
		0.05	3	105–113	110 \pm 3.8
		0.10	3	73–115	89 \pm 26

Analyte	Matrix	Fortification (mg/kg)	n	Recovery (%)	
				Range	Mean \pm RSD
		0.50	3	90–100	95 \pm 5.3
		1.00	3	90–108	96 \pm 11
		overall	17	73–126	103 \pm 14
IN-QDY62	Hyde Farm, UK	0.01	5	65–85	77 \pm 9.7
		0.05	3	97–99	98 \pm 1.2
		0.10	3	95–98	97 \pm 1.6
		0.50	3	102–106	103 \pm 2.2
		1.00	3	88–94	90 \pm 3.6
		overall	17	65–106	91 \pm 12
	Poitou-Charentes, France	0.01	5	103–113	107 \pm 4.0
		0.05	3	82–87	85 \pm 3.0
		0.10	3	87–104	95 \pm 9.1
		0.50	3	87–90	89 \pm 1.7
		1.00	3	85–94	89 \pm 5.1
		overall	17	82–113	94 \pm 10
IN-QDY63	Hyde Farm, UK	0.01	5	68–80	72 \pm 7.1
		0.05	3	84–87	86 \pm 1.8
		0.10	3	85–92	89 \pm 4.1
		0.50	3	101–106	103 \pm 2.4
		1.00	3	90–91	90 \pm 0.6
		overall	17	68–106	86 \pm 13
	Poitou-Charentes, France	0.01	5	91–120	100 \pm 12
		0.05	3	76–81	79 \pm 3.6
		0.10	3	74–94	82 \pm 13
		0.50	3	90–94	92 \pm 2.3
		1.00	3	89–98	93 \pm 4.9
		overall	17	74–120	91 \pm 12

Regulatory multi-residue methods

The suitability of the US FDA Pesticide Analytical Manual, Volume I (PAM I 3rd edition) protocols for analysis of residues of picoxystrobin and the metabolites IN-QDK50, IN-QDY62 and IN-QDY63 was assessed (Rockwell, 2009). The HPLC method with fluorescence detection (Protocol A) was determined not to be suitable for analysis of picoxystrobin or the metabolites. The gas chromatographic method was suitable for analysis of picoxystrobin (parent only) in non-fatty plant matrices (apple) with the extraction procedures of Protocol D and E and analysis by method DG1, and in fatty plant matrices (soya bean) with the extraction procedure of Protocol F and analysis by method DG1.

Radiovalidation

Radiovalidation studies were not provided to the 2012 JMPR.

Stability of pesticide residues in stored analytical samples*Plant matrices*

Storage stability of picoxystrobin and the metabolites IN-QDK50, IN-QDY62 and IN-QDY63 in a range of crop commodities including high (apples, apple juice, grapes and lettuce), medium (wheat forage and apple pomace) and low (wheat straw and soya bean meal) water content, high protein (dry pea), high starch (potato), and high oil (soya bean seed and refined oil) content was assessed for samples stored frozen (target temperature of -20 °C) for 24 months (Schierhoff, 2012). Homogenised samples were fortified with each analyte at 0.20 mg/kg. Samples were removed from storage and analysed at intervals using an LC/MS/MS method (method number 24868).

Table 50 Stability of picoxystrobin and its metabolites in plant matrices fortified at 0.20 mg/kg and stored at -20 °C

Matrix	Analyte	Storage interval (months)	% remaining	Concurrent recovery (%)	
Wheat forage	Picoxystrobin	0	–	88, 90	
		1	94, 90	88, 103	
		3	82, 81	97, 96	
		6	82, 89	94, 94	
		12	83, 85	91, 86	
		18	68, 76	92, 83	
		24	91, 85	85, 105	
	IN-QDK50	0	–	88, 87	
		1	91, 84	92, 99	
		3	84, 82	93, 86	
		6	85, 92	93, 90	
		12	70, 63	92, 74	
		18	68, 67	92, 80	
		24	92, 84	93, 96	
	IN-QDY62	0	–	81, 85	
		1	94, 92	103, 105	
		3	92, 93	109, 97	
		6	96, 107	112, 110	
		12	97, 99	103, 100	
		18	85, 92	110, 101	
		24	96, 102	103, 113	
	IN-QDY63	0	–	88, 86	
		1	89, 84	98, 104	
		3	81, 82	100, 97	
		6	79, 87	103, 104	
		12	75, 78	97, 93	
		18	78, 83	94, 85	
		24	79, 78	88, 103	
	Wheat straw	Picoxystrobin	0	–	83, 85
			1	81, 85	80, 86

Picoxystrobin

Matrix	Analyte	Storage interval (months)	% remaining	Concurrent recovery (%)	
		3	82, 81	83, 83	
		6	79, 83	76, 78	
		12	92, 84	96, 96	
		18	95, 85	95, 94	
		24	86, 85	90, 88	
	IN-QDK50	0	–	76, 76	
		1	78, 79	76, 75	
		3	81, 82	82, 85	
		6	83, 87	78, 81	
		12	74, 71	75, 80	
		18	81, 73	77, 84	
		24	84, 72	79, 76	
	IN-QDY62	0	–	87, 87	
		1	84, 86	84, 81	
		3	103, 102	105, 106	
		6	105, 107	92, 98	
		12	99, 90	104, 97	
		18	110, 97	111, 105	
		24	92, 87	87, 88	
	IN-QDY63	0	–	89, 92	
		1	79, 86	82, 84	
		3	91, 92	94, 95	
		6	88, 91	84, 88	
		12	94, 85	99, 97	
		18	114, 105	102, 96	
		24	85, 82	83, 81	
	Maize grain	Picoxystrobin	0	–	102, 97
			1	81, 72	86, 87
			3	95, 93	98, 96
			6	93, 89	95, 92
12			96, 93	102, 102	
18			93, 97	103, 106	
24			100, 97	102, 103	
IN-QDK50			0	–	78, 79
		1	75, 66	82, 84	
		3	88, 87	90, 91	
		6	85, 77	79, 83	
		12	81, 78	79, 84	
		18	75	91, 85	
		24	88, 91	95, 82	

Matrix	Analyte	Storage interval (months)	% remaining	Concurrent recovery (%)
	IN-QDY62	0	–	97, 100
		1	75, 79	72, 85
		3	101, 98	105, 103
		6	104, 98	108, 105
		12	102, 98	114, 103
		18	99, 103	108, 116
		24	116, 112	121, 121
	IN-QDY63	0	–	99, 97
		1	71, 68	77, 78
		3	93, 90	100, 100
		6	91, 86	101, 100
		12	91, 86	107, 103
		18	112, 117	108, 114
		24	108, 104	121, 123
Soya bean seed	Picoxystrobin	0	–	90, 88
		1	86, 92	108, 105
		3	80, 78	92, 98
		6	86, 86	102, 98
		12	74, 75	95, 92
		18	70, 67	103, 102
		24	82, 82	97, 101
	IN-QDK50	0	–	87, 85
		1	81, 83	95, 98
		3	72, 69	83, 87
		6	84, 86	89, 84
		12	78, 68	91, 78
		18	59, 60	97, 94
		24	85, 91	86, 99
	IN-QDY62	0	–	91, 99
		1	89, 96	113, 118
		3	86, 91	96, 94
		6	105, 107	114, 110
		12	88, 88	100, 97
		18	85, 86	110, 102
		24	102, 103	106, 99
	IN-QDY63	0	–	92, 94
		1	88, 94	104, 107
		3	86, 87	98, 98
		6	99, 100	113, 108
		12	81, 79	99, 94

Picoxystrobin

Matrix	Analyte	Storage interval (months)	% remaining	Concurrent recovery (%)	
		18	89, 93	98, 95	
		24	92, 92	99, 99	
Soya bean meal	Picoxystrobin	0	–	94, 98	
		1	96, 100	96, 110	
		3	89, 90	94, 94	
		6	90, 86	92, 88	
		12	87, 91	100, 104	
		18	91, 83	103, 107	
		24	92, 90	98, 97	
		IN-QDK50	0	–	79, 84
	1		84, 86	79, 82	
	3		84, 82	90, 88	
	6		88, 86	80, 79	
	12		75, 75	75, 79	
	18		86, 70	81, 92	
	24		86, 80	86, 74	
	IN-QDY62		0	–	91, 101
		1	121, 100	103, 93	
		3	103, 108	117, 108	
		6	99, 107	103, 101	
		12	96, 102	105, 107	
		18	112, 96	118, 123	
		24	103, 104	108, 111	
		IN-QDY63	0	–	98, 100
	1		94, 99	96, 99	
	3		91, 93	103, 96	
	6		90, 89	95, 92	
	12		91, 94	100, 99	
	18		103, 97	97, 100	
	24		78, 81	86, 83	
	Soya bean oil		Picoxystrobin	0	–
		1		105, 112	110, 102
3		100, 103		104, 104	
6		105, 102		101, 100	
12		98, 102		98, 101	
13		111, 120		112, 118	
18		88, 99		103, 111	
24		115, 99		101, 106	
IN-QDK50		0	–	84, 84	
		1	86, 80	93, 8	

Matrix	Analyte	Storage interval (months)	% remaining	Concurrent recovery (%)	
		3	88, 93	96, 98	
		6	98, 99	92, 85	
		12	91	94, 82	
		13	99, 77	93, 90	
		18	72, 86	99, 100	
		24	94, 90	98, 98	
		IN-QDY62	0	–	97, 101
		1	101, 94	99, 96	
		3	108, 113	121, 115	
		6	109, 104	105, 108	
		12	95, 101	107, 105	
		13	111, 98	121, 126	
		18	96, 77	105, 115	
		24	59, 53	92, 99	
		IN-QDY63	0	–	100, 99
			1	98, 94	104, 99
			3	85, 89	112, 107
			6	76, 74	104, 105
			12	61, 65	107, 108
			13	62, 61	117, 123
			18	51, 53	96, 105
			24	13, 4	80, 93
	Potato	Picoxystrobin	0	–	77, 83
			1	105, 85	104, 102
			3	97, 98	103, 101
			6	97, 96	99, 95
			12	91, 89	101, 95
			18	98, 99	108, 109
24			95, 96	118, 111	
IN-QDK50			0	–	90, 93
		1	94, 91	94, 88	
		3	92, 95	93, 102	
		6	97, 91	93, 89	
		12	82, 93	98, 81	
		18	89, 90	107, 101	
		24	93, 102	117, 117	
		IN-QDY62	0	–	103, 97
			1	108, 85	101, 95
			3	99, 101	105, 105
			6	109, 109	105, 102

Picoxystrobin

Matrix	Analyte	Storage interval (months)	% remaining	Concurrent recovery (%)
		12	102, 99	108, 100
		18	103, 107	112, 112
		24	105, 109	122, 116
	IN-QDY63	0	–	94, 101
		1	100, 93	100, 101
		3	100, 103	103, 105
		6	103, 101	103, 99
		12	92, 92	101, 95
		18	96, 100	95, 96
		24	101, 103	117, 113
		Dry pea	Picoxystrobin	0
1	82, 86			94, 98
3	90, 80			96, 100
6	81, 84			93, 90
12	79, 89			102, 103
18	81, 82			107, 102
24	74, 72			105, 96
IN-QDK50	0			–
	1		88, 83	87, 83
	3		83, 77	94, 92
	6		78, 74	84, 82
	12		66, 75	75, 69
	18		84, 75	89, 91
	24		77, 65	74, 66
	IN-QDY62		0	–
1			92, 86	93, 91
3			101, 95	103, 106
6			99, 105	104, 99
12			88, 100	100, 101
18			100, 99	116, 106
24			93, 89	96, 97
IN-QDY63			0	–
	1		91, 92	96, 92
	3		94, 87	99, 101
	6		91, 95	97, 93
	12		83, 93	99, 101
	18		110, 108	110, 104
	24		89, 89	90, 92
	Lettuce	Picoxystrobin	0	–
1			97, 102	103, 99

Matrix	Analyte	Storage interval (months)	% remaining	Concurrent recovery (%)	
		3	91, 92	101, 106	
		6	97, 97	88, 91	
		12	85, 86	90, 101	
		18	82, 83	92, 94	
		24	85, 92	92, 101	
	IN-QDK50	0	–	93, 98	
		1	98, 93	95, 100	
		3	84, 87	94, 95	
		6	92, 93	84, 84	
		12	79, 81	72, 81	
		18	66, 76	78, 76	
		24	69, 80	86, 97	
	IN-QDY62	0	–	99, 95	
		1	99, 94	93, 94	
		3	88, 93	98, 108	
		6	109, 109	91, 99	
		12	103, 105	97, 110	
		18	102, 110	107, 107	
		24	96, 99	95, 103	
	IN-QDY63	0	–	100, 101	
		1	96, 98	97, 98	
		3	88, 92	101, 105	
		6	101, 104	89, 98	
		12	94, 99	94, 109	
		18	89, 95	91, 92	
		24	85, 92	96, 100	
	Apple	Picoxystrobin	0	–	103, 97
			1	84, 122	100, 100
3			91, 95	101, 99	
6			93, 90	99, 101	
12			84, 83	99, 96	
18			85, 81	92, 94	
24			101, 102	108, 115	
IN-QDK50			0	–	105, 106
		1	89, 104	93, 91	
		3	91, 98	99, 94	
		6	100, 92	91, 92	
		12	90, 76	91, 82	
		18	73, 82	85, 76	
		24	105, 104	97, 113	

Picoxystrobin

Matrix	Analyte	Storage interval (months)	% remaining	Concurrent recovery (%)
	IN-QDY62	0	–	112, 112
		1	85, 116	102, 101
		3	96, 96	99, 94
		6	112, 110	110, 107
		12	99, 97	107, 103
		18	105, 103	106, 106
		24	109, 110	112, 120
	IN-QDY63	0	–	108, 109
		1	90, 111	101, 103
		3	98, 100	101, 97
		6	103, 99	105, 106
		12	88, 90	101, 98
		18	96, 95	87, 87
		24	104, 106	109, 114
Apple juice	Picoxystrobin	0	–	99, 98
		1	91, 99	93, 91
		3	85, 95	110, 114
		6	92, 87	90, 91
		12	76, 87	85, 88
		18	96, 72	95, 109
		24	96, 96	105, 105
	IN-QDK50	0	–	90, 92
		1	106, 110	106, 103
		3	90, 91	97, 95
		6	89, 88	92, 86
		12	71, 72	80, 69
		18	77	87, 86
		24	81, 80	86, 94
	IN-QDY62	0	–	87, 95
		1	102, 111	97, 110
		3	100, 92	104, 114
		6	110, 103	99, 98
		12	100, 106	96, 98
		18	104, 83	103, 107
		24	101, 102	105, 108
	IN-QDY63	0	–	94, 94
		1	98, 105	98, 99
		3	95, 101	106, 110
		6	101, 93	98, 94
		12	94, 99	95, 96

Matrix	Analyte	Storage interval (months)	% remaining	Concurrent recovery (%)	
		18	116, 90	94, 97	
		24	98, 99	106, 105	
Apple pomace	Picoxystrobin	0	–	83, 94	
		1	93, 97	97, 95	
		3	93, 96	96, 96	
		6	85, 88	98, 100	
		12	78, 84	98, 94	
		18	78, 76	90, 80	
		24	95, 88	106, 106	
		IN-QDK50	0	–	88, 85
	1		92, 96	92, 87	
	3		95, 99	94, 91	
	6		94, 95	97, 95	
	12		74, 90	100, 92	
	18		86, 82	91, 80	
	24		103, 97	106, 106	
	IN-QDY62		0	–	110, 111
		1	109, 115	113, 111	
		3	119, 122	116, 117	
		6	104, 106	109, 107	
		12	100, 111	122, 113	
		18	98, 96	108, 98	
		24	105, 96	110, 112	
		IN-QDY63	0	–	94, 94
	1		101, 106	101, 102	
	3		103, 108	102, 105	
	6		89, 93	103, 103	
	12		87, 97	104, 103	
	18		102, 101	111, 100	
	24		104, 97	102, 104	
	Grapes		Picoxystrobin	0	–
		1		98, 105	107, 92
3		94, 84		81, 97	
6		91, 93		94, 98	
12		87, 87		92, 94	
18		90, 80		95, 96	
24		92, 98		106, 98	
IN-QDK50		0		–	89, 90
		1	93, 100	83, 95	
		3	88, 85	81, 87	

Matrix	Analyte	Storage interval (months)	% remaining	Concurrent recovery (%)
		6	90, 89	90, 92
		12	70, 79	78, 80
		18	76, 72	83, 86
		24	96, 93	104, 86
	IN-QDY62	0	–	101, 116
		1	103, 101	98, 102
		3	95, 89	81, 89
		6	98, 100	98, 103
		12	101, 100	97, 101
		18	101, 90	107, 105
		24	99, 105	109, 107
		IN-QDY63	0	–
	1		95, 98	96, 96
	3		92, 88	83, 93
	6		95, 98	97, 100
	12		95, 91	96, 99
	18		94, 89	88, 90
	24		93, 101	105, 103

With the exception of the metabolites IN-QDY 62 and IN-QDY63 in soya bean oil, which were stable for 18 and 6 months storage respectively, all analyte/sample combinations were stable (remaining residues in the range 70–120%) for 24 months storage at -20 °C.

Animal matrices

A separate storage stability study was not provided for animal commodities. Storage stability of picoxystrobin residues over the period of sample storage was verified as part of the lactating cattle (Wen, 2009) and laying hen (Wen, 2010) feeding studies, and is considered in detail in the relevant sections of the evaluation below. The stability of residues of picoxystrobin in animal commodity samples over the period of storage in the feeding studies was acceptable.

Soil

Stability of residues of picoxystrobin and the metabolites IN-QDK50, IN-QDY62 and IN-QDY63 in soil samples was studied over two years' storage at -18 °C (Nagra, 1999). Samples of two soil types were fortified with the four analytes at 0.10 mg/kg and stored in a freezer. Samples were withdrawn at intervals and analysed by GC/MS (method number RAM 291/01 and 02).

Table51 Recovery of picoxystrobin and metabolites from soil samples fortified at 0.10 mg/kg and stored at -18 °C

Matrix	Analyte	Storage interval (months)	Residue (mg/kg), normalised for concurrent method recovery
Pickett Piece, Oxfordshire, UK clay loam soil	Picoxystrobin	0	0.09, 0.10
		1	0.10, 0.10
		3	0.10, 0.10
		6	0.10, 0.10

Matrix	Analyte	Storage interval (months)	Residue (mg/kg), normalised for concurrent method recovery	
		12	0.09, 0.10	
		24	0.10, 0.10	
	IN-QDK50	0	0.10, 0.10	
		1	0.10, 0.09	
		3	0.10, 0.09	
		6	0.11, 0.11	
		12	0.12, 0.11	
		24	0.09, 0.09	
		IN-QDY62	0	0.10, 0.10
	1		0.10, 0.10	
	3		0.12, 0.10	
	6		0.10, 0.10	
	12		0.10, 0.10	
	24		0.10, 0.09	
	IN-QDY63		0	0.10, 0.10
		1	0.10, 0.11	
		3	0.10, 0.10	
		6	0.10, 0.10	
		12	0.10, 0.11	
		24	0.11, 0.11	
	Hyde Farm, Berkshire, UK sandy loam soil	Picoxystrobin	0	0.10, 0.10
			1	0.09, 0.09
			3	0.10, 0.09
			6	0.11, 0.10
12			0.10, 0.10	
24			0.10, 0.10	
IN-QDK50		0	0.10, 0.10	
		1	0.09, 0.10	
		3	0.10, 0.09	
		6	0.10, 0.10	
		12	0.11, 0.11	
		24	0.09, 0.09	
IN-QDY62		0	0.10, 0.10	
		1	0.09, 0.10	
		3	0.10, 0.10	
		6	0.09, 0.08	
		12	0.12, 0.11	
		24	0.10, 0.09	
IN-QDY63		0	0.10, 0.10	

Matrix	Analyte	Storage interval (months)	Residue (mg/kg), normalised for concurrent method recovery
		1	0.11, 0.11
		3	0.10, 0.11
		6	0.12, 0.11
		12	0.11, 0.10
		24	0.10, 0.11

Residues of picoxystrobin and the metabolites IN-QDK50, IN-QDY62 and IN-QDY63 were shown to be stable over two years in soil samples stored at -18 °C.

USE PATTERN

Picoxystrobin is registered for use on cereals (barley, oats, rye, triticale and wheat) in a large number of countries in northern and southern Europe, Canada, Argentina, New Zealand, South Africa and Zambia, on oilseed rape (canola) in Canada, the Czech Republic, Slovakia, the UK and Ireland, pulses in Canada, sweet corn in France and Canada, maize in Canada, and soya beans in Canada, Brazil, Bolivia and Argentina for control of various fungal diseases including leaf rust, stripe rust, powdery mildew, net blotch, scald and speckled leaf blotch. The information available to the Meeting on registered use patterns of picoxystrobin is summarized in the table below. All registered formulations are suspension concentrates containing 250 g/L picoxystrobin.

Table 52 Registered uses of picoxystrobin relevant to the evaluation

Crop	Country	Application				PHI (days), or latest growth stage at application
		Method	Rate (g ai/ha, max)	Volume (L/ha)	No. (max)	
Cereal grains						
Barley (brewing)	Argentina	Foliar: aerial application	75	20–30	2	35
		Foliar: ground application	75	150	2	35
Barley	Austria	Foliar	250	200–400	2	35
	Belgium	Foliar	250	200–400	2	Not stated
	Canada	Foliar: ground application	220	110	3	45 (grain) 14 (hay)
		Foliar: aerial application	220	40		
		Foliar: ground application	220	110	1	7 (forage)
	Canada	Foliar: aerial application	220	40		
		Czech Republic	Foliar	250	200–400	2
	Finland	Broadcast	125	100–300	2	35
	France	Foliar	250	80–300	2	42
	Germany	Foliar	250	200–400	2	35
Ireland	Foliar	250	200–300	2	35	
Luxembourg	Foliar	250	200–400	2	Not stated	

Crop	Country	Application				PHI (days), or latest growth stage at application
		Method	Rate (g ai/ha, max)	Volume (L/ha)	No. (max)	
	Norway	Foliar	250	150–167	2	35
	New Zealand	Foliar: aerial application	125	50	2	35 (harvest) 28 (grazing)
		Foliar: ground application	125	200–300	2	
	Portugal	Foliar	250	–	2	42
	Slovakia	Foliar	250	200–400	2	35
	South Africa	Foliar	75	300	2	60
	Sweden	Broadcast	125	150–200	2	35
			250		1	
	UK	Foliar	250	200–300	2	BBCH 71
Barley (spring)	Denmark	Foliar	125	100–200	2	35
	Estonia	Broadcast	125	100–300	2	35
			250		1	
	Hungary	Foliar	250	250–400	2	35
	Latvia	Broadcast	125	100–300	2	35
			250		1	
	Lithuania	Broadcast	125	100–300	2	35
			250		1	
Netherlands	Foliar	250	200–400	2	35	
Poland	Foliar	250	200–400	2	35	
Barley (winter)	Denmark	Foliar	125	100–200	2	35
	Estonia	Broadcast	125	100–300	2	35
			250		1	
	Hungary	Foliar	250	250–400	2	35
	Latvia	Broadcast	125	100–300	2	35
			250		1	
	Lithuania	Broadcast	125	100–300	2	35
			250		1	
Netherlands	Foliar	250	200–400	2	35	
Poland	Foliar	250	200–400	2	35	
Oat	Austria	Foliar	250	200–400	2	35
	Canada	Foliar: ground application	220	110	3	45 (grain) 14 (hay)
		Foliar: aerial application	220	40		
		Foliar: ground application	220	110	1	7 (forage)
Foliar: aerial application		220	40			

Crop	Country	Application				PHI (days), or latest growth stage at application	
		Method	Rate (g ai/ha, max)	Volume (L/ha)	No. (max)		
	Denmark	Foliar	125	100–200	2	35	
	Estonia	Broadcast	125	100–300	2	35	
			250		1		
	Finland	Broadcast	125	100–300	2	35	
	France	Foliar	250	80–300	2	42	
	Hungary	Foliar	250	250–400	2	35	
	Ireland	Foliar	250	200–300	2	35	
	Latvia	Broadcast	125	100–300	2	35	
			250		1		
	Norway	Foliar	250	150–167	2	35	
	Portugal	Foliar	250	–	2	42	
	Sweden	Broadcast	125	150–200	2	35	
			250		1		
	UK	Foliar	250	200–300	2	BBCH 71	
Oat (spring)	Lithuania	Broadcast	125	100–300	2	35	
			250		1		
Oat (winter)	Lithuania	Broadcast	125	100–300	2	35	
			250		1		
Rye	Austria	Foliar	250	200–400	2	35	
	Canada	Foliar: ground application	220	110	3	45 (grain) 14 (hay)	
		Foliar: aerial application	220	40			
		Foliar: ground application	220	110	1		7 (forage)
		Foliar: aerial application	220	40			
	Denmark	Foliar	125	100–200	2	35	
	Finland	Broadcast	125	100–300	2	35	
	France	Foliar	250	80–300	2	42	
	Germany	Foliar	250	200–400	2	35	
	Portugal	Foliar	250	–	2	42	
	Sweden	Broadcast	125	150–200	2	35	
			250		1		
	Rye (spring)	Estonia	Broadcast	125	100–300	2	35
250				1			
Hungary		Foliar	250	250–400	2	35	
Latvia		Broadcast	125	100–300	2	35	
			250		1		

Crop	Country	Application				PHI (days), or latest growth stage at application
		Method	Rate (g ai/ha, max)	Volume (L/ha)	No. (max)	
	Lithuania	Broadcast	125	100–300	2	35
			250		1	
	Norway	Foliar	250	150–167	2	35
Rye (winter)	Estonia	Broadcast	125	100–300	2	35
			250		1	
	Hungary	Foliar	250	250–400	2	35
	Latvia	Broadcast	125	100–300	2	35
			250		1	
	Lithuania	Broadcast	125	100–300	2	35
			250		1	
	Norway	Foliar	250	150–167	2	35
Triticale	Austria	Foliar	250	200–400	2	35
	Canada	Foliar: ground application	220	110	3	45 (grain) 14 (hay)
		Foliar: aerial application	220	40		
		Foliar: ground application	220	110	1	7 (forage)
		Foliar: aerial application	220	40		
	Denmark	Foliar	125	100–200	2	35
	Finland	Broadcast	125	100–300	2	35
	France	Foliar	250	80–300	2	42
	Germany	Foliar	250	200–400	2	35
	New Zealand	Foliar: aerial application	125	50	2	35 (harvest) 28 (grazing)
		Foliar: ground application	125	200–300	2	
	Portugal	Foliar	250	–	2	42
	Triticale (spring)	Hungary	Foliar	250	250–400	2
Triticale (winter)	Estonia	Broadcast	125	100–300	2	35
			250		1	
	Hungary	Foliar	250	250–400	2	35
	Latvia	Broadcast	125	100–300	2	35
			250		1	
	Lithuania	Broadcast	125	100–300	2	35
			250		1	
	Norway	Foliar	250	150–167	2	35
Sweden	Broadcast	125	150–200	2	35	

Picoxystrobin

Crop	Country	Application				PHI (days), or latest growth stage at application
		Method	Rate (g ai/ha, max)	Volume (L/ha)	No. (max)	
			250		1	
Wheat	Argentina	Foliar: aerial application	75	20–30	2	35
		Foliar: ground application	75	150 (min)	2	35
	Austria	Foliar	250	200–400	2	35
	Belgium	Foliar	250	200–400	2	Not stated
	Canada	Foliar: ground application	220	110	3	45 (grain) 14 (hay)
		Foliar: aerial application	220	40		
		Foliar: ground application	220	110	1	7 (forage)
		Foliar: aerial application	220	40		
	Czech Republic	Foliar	250	200–400	2	35
	France	Foliar	250	80–300	2	42
	Germany	Foliar	250	200–400	2	35
	Ireland	Foliar	250	200–300	2	35
	Luxembourg	Foliar	250	200–400	2	Not stated
	New Zealand	Foliar: aerial application	187.5	50	2	35 (harvest) 28 (grazing)
		Foliar: ground application	187.5	200–300	2	
	Portugal	Foliar	250	–	2	42
	Slovakia	Foliar	250	200–400	2	35
	UK	Foliar	250	200–300	2	BBCH 71
	Wheat (spring)	Denmark	Foliar	125	100–200	2
Estonia		Broadcast	125	100–300	2	35
			250		1	
Finland		Broadcast	125	100–300	2	35
Hungary		Foliar	250	250–400	2	35
Ireland		Foliar	250	200–300	2	35
Latvia		Broadcast	125	100–300	2	35
			250		1	
Lithuania		Broadcast	125	100–300	2	35
			250		1	
Netherlands	Foliar	250	200–400	2	35	
Norway	Foliar	250	150–167	2	35	
Sweden	Broadcast	125	150–200	2	35	

Crop	Country	Application				PHI (days), or latest stage application growth at
		Method	Rate (g ai/ha, max)	Volume (L/ha)	No. (max)	
			250		1	
Wheat (winter)	Denmark	Foliar	125	100–200	2	35
	Estonia	Broadcast	125	100–300	2	35
			250		1	
	Finland	Broadcast	125	100–300	2	35
	Hungary	Foliar	250	250–400	2	35
	Ireland	Foliar	250	200–300	2	35
	Latvia	Broadcast	125	100–300	2	35
			250		1	
	Lithuania	Broadcast	125	100–300	2	35
			250		1	
	Netherlands	Foliar	250	200–400	2	35
	Norway	Foliar	250	150–167	2	35
Poland	Foliar	250	200–400	2	35	
Sweden	Broadcast	125	150–200	2	35	
		250		1		
Pulses/oilseeds						
Soya beans	Argentina	Foliar: aerial application	50	20–30	2	30
		Foliar: ground application	50	Min 150	2	30
	Bolivia	Foliar	67.5	250–300	2	21
	Brazil	Foliar: aerial application	62.5	30–40	2	21
		Foliar: ground application	62.5	200	1	21
	Canada	Foliar: ground application	220	110	3	14 (seed) 14 (forage and hay)
		Foliar: aerial application	220	40		
		Foliar: ground application	220	110	1	
		Foliar: aerial application	220	40		
	Oilseeds					
Oilseed rape	Czech Republic	Foliar	250	200–400	2	35
	Ireland	Foliar	250	200–300	1	Do not apply after mid-flowering
	Slovakia	Foliar	250	200–400	2	35
	UK	Foliar	250	200–300	1	BBCH 67

Crop	Country	Application				PHI (days), or latest growth stage at application
		Method	Rate (g ai/ha, max)	Volume (L/ha)	No. (max)	
Pulses						
Legumes, dry	Canada	Foliar: ground application	220	110	2	14 (seed) 0 (vines and hay)
		Foliar: aerial application	220	40		
Corn/maize						
Corn, cob	France	Foliar	250	80–300	2	42
Corn, field	Canada	Foliar: ground application	220	110	3	7 (grain or ear) 0 (forage)
		Foliar: aerial application	220	40		
Corn, pop	Canada	Foliar: ground application	220	110	3	7 (grain or ear) 0 (forage)
		Foliar: aerial application	220	40		
Corn, sweet	Canada	Foliar: ground application	220	110	4	7 (grain or ear) 0 (forage)
		Foliar: aerial application	220	40		

SUPERVISED RESIDUE TRIALS

The Meeting received information on picoxystrobin supervised field residue trials for the following commodities:

Crop group	Commodity	Table
Fruiting vegetables, other than Cucurbits	Sweet corn	53
Pulses	Soya beans	54
	Peas (dry)	55
	Beans (dry)	56
Cereals	Wheat	57
	Barley	58
	Maize	59
Oilseeds	Oilseed rape	60
Animal feeds	Sweet corn forage	61
	Soya bean forage	62
	Soya bean hay	63
	Pea vines	64
	Pea hay	65
	Wheat forage	66
	Wheat hay	67

Crop group	Commodity	Table
	Wheat straw	68
	Barley hay	69
	Barley straw	70
	Maize forage	71
	Maize stover	72

Sweet corn

A series of 11 trials in sweet corn (corn-on-the-cob) was conducted in the USA and Canada in the 2008 growing season in accordance with the Canadian GAP for sweet corn (study number 25881: Shepard 2009). A single treated plot of adequate size (at least 35 m²) was established at each site. Four applications were made at the target rate of 220 g ai/ha, at 7-day intervals, using a backpack sprayer or a tractor-mounted boom sprayer. A spray adjuvant (a non-ionic surfactant) was included in the tank mix for all applications. Duplicates of treated samples of forage and cobs plus kernel with husk removed (ears) were collected, along with a single untreated control sample. Additional decline samples were collected for forage at some sites. Sample sizes were at least 1 kg for forage and for ears, were mostly > 2 kg (with the exception of trial site 06 and the UTC from trial 09).

Samples were analysed for picoxystrobin and the metabolites IN-QDY62, IN-QDY 63 and IN-QDK50 by LC/MS/MS (method number 24868). Acceptable mean concurrent recoveries and precision values were achieved.

Residue data for sweet corn cobs plus kernel with husk removed are presented in Table 53 and data for sweet corn forage are presented in Table 61.

Table 53 Results of residue trials conducted with picoxystrobin (250 g/L SC) in sweet corn in the USA and Canada in 2008 (study number 25881)

Location Trial no., Year (Variety)	Application				Sample	DAT ^b	Residues (mg/kg) ^c			
	No.	Growth stage	g ai/ha ^a	L/ha			Parent	IN-QDY62	IN-QDY63	IN-QDK50
Germansville, PA, USA Trial 01, 2008 (Triple Sweet HYB)	4	Early tassel	222	398	Cobs plus kernel with husk removed	7	<u>ND</u>	ND	ND	ND
		Pollen shed	223	398			(ND, ND)	(ND, ND)	(ND, ND)	(ND, ND)
		R2 blister	220	398						
		Early milk	217	421						
Blackville, SC, USA Trial 02, 2008 (Silver Queen)	4	59	219	177	Cobs plus kernel with husk removed	6	<u>ND</u>	ND	ND	ND
		65	224	179			(ND, ND)	(ND, ND)	(ND, ND)	(ND, ND)
		73	221	179						
		75	220	193						
Oviedo, FL, USA Trial 03, 2008 (Honey Pearl)	4	51	229	281	Cobs plus kernel with husk removed	7	<u>ND</u>	ND	ND	ND
		59	224	281			(ND, ND)	(ND, ND)	(ND, ND)	(ND, ND)
		73	224	281						
		75	226	281						
Branchton, ON,	4	R1	248	200	Cobs plus	7	<u>ND</u>	ND	ND	ND

Location Trial no., Year (Variety)	Application				Sample	DAT ^b	Residues (mg/kg) ^c			
	No.	Growth stage	g ai/ha ^a	L/ha			Parent	IN-QDY62	IN-QDY63	IN-QDK50
Canada Trial 04, 2008 (Ambrosia)		R1 R2 R2	232 213 213	200 200 200	kernel with husk removed		(ND, ND)	(ND, ND)	(ND, ND)	(ND, ND)
Conklin, MI, USA Trial 05, 2008 (Temptation)	4	59 65 71 75	222 223 224 223	204 202 200 201	Cobs plus kernel with husk removed	7	<u>ND</u> (ND, ND)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
Paynesville, MN, USA Trial 06, 2009 (Jubilee)	4	71 72 73 75	216 216 217 215	143 142 143 143	Cobs plus kernel with husk removed	7	<u>ND</u> (ND, ND)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
Richland, IA, USA Trial 07, 2008 (Iochief)	4	R1 R2 R3 R4	224 224 224 213	162 147 161 159	Cobs plus kernel with husk removed	7	<u>ND</u> (ND, ND)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
Taber, AB, Canada Trial 08, 2008 (Northern Supper Sweet)	4	69–74 75–79 83–85 83–85	216 217 222 231	150 152 152 154	Cobs plus kernel with husk removed	9	<u>ND</u> (ND, ND)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
Woodland, CA, USA Trial 09, 2008 (Silver Queen)	4	V15 VT R1 Milk	220 221 222 221	187 187 188 187	Cobs plus kernel with husk removed	7	<u>ND</u> (ND, ND)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
Madras, OR, USA Trial 10, 2008 (Jubilee)	4	63 67 71 75	223 225 221 225	192 194 190 194	Cobs plus kernel with husk removed	7	<u>< 0.01</u> (<u>< 0.01</u> , ND)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
Forest Grove, OR, USA Trial 11, 2008 (Serendipity)	4	Kernel filling Kernels 70% Kernel final size Harvest maturity	212 223 213 217	209 187 189 186	Cobs plus kernel with husk removed	7	<u>ND</u> (ND, ND)	ND (ND, ND)	< 0.01 (ND, < 0.01)	ND (ND, ND)

ND = not detected (< 0.003 mg/kg)

^a Individual application rates shown

^b DAT = Days After Treatment

^c Mean result shown, with individual results for analyses of duplicate samples from the same plot in brackets.

Pulses

Soya bean

A series of 21 trials in soya beans was conducted in the USA and Canada in the 2008 and 2009 growing seasons in accordance with the Canadian GAP for soya beans (study number 24861: Shepard, 2010). A single treated plot was established at each site (except for trial 14, where three plots were established). Plot sizes were sufficient, being at least 60 m². Three applications were made at the target rate of 220 g ai/ha, with the first being timed around BBCH growth stages 61–63, the second around 9–10 weeks later at BBCH stages 81–83, and the third 7 days later. The applications were made using a CO₂-pressurised backpack sprayer or a tractor-mounted boom sprayer. A non-ionic surfactant, or methylated vegetable oil adjuvant was included in all tank mixes. Duplicate treated samples were collected, along with a single untreated control sample. Sample sizes were typically at least 1.0 kg for forage and seed, and 0.5 kg for hay, (except for some of the hay samples for trials 05, 06, and 07, which were only 0.3–0.4 kg).

Samples were analysed for picoxystrobin and the metabolites IN-QDY62, IN-QDY 63 and IN-QDK50 by LC/MS/MS (method number 24868). Acceptable mean concurrent recoveries and precision values were achieved.

Residue data for soya bean seed are presented in Table 54, while data for soya bean forage and hay are presented in Tables 62 and 63 respectively.

Table 54 Results of residue trials conducted with picoxystrobin (250 g/L SC) in soya bean in the USA and Canada in 2008 and 2009 (study number 24861)

Location, Trial No., Year (Variety)	Application				Sample	DAT ^b	Residues (mg/kg) ^c			
	No.	Growth stage	g ai/ha ^a	L/ha			Parent	IN-QDY62	IN-QDY63	IN-QDK50
Blackville, SC, USA Trial 01, 2008 (Asgrow, H7242 RR)	3	63	224	150	Seed	15	≤ 0.01 (< 0.01, < 0.01)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
		95	224	148						
		97	224	146						
Seven Springs, NC, USA, Trial 02, 2008 (DKB-64-51)	3	(R1)61	217	156	Seed	14	≤ 0.01 (< 0.01, < 0.01)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
		(R6)79	219	143						
		(R7)81	216	147						
Cheneyville, LA, USA Trial 03, 2008 (DG 33B52)	3	(R1)61	219	149	Seed	14	≤ 0.01 (< 0.01, < 0.01)	ND (ND, ND)	ND (ND, ND)	< 0.01 (< 0.01, < 0.01)
		98	247	147						
		99	252	131						
Fisk, MO, USA Trial 04, 2008 (Armor 47G7)	3	R1-2/61–65	223	187	Seed	14	≤ 0.01 (< 0.01, < 0.01)	ND (ND, ND)	ND (ND, ND)	< 0.01 (< 0.01, < 0.01)
		81	221	187						
		85	224	187						
Richland, IA, USA Trial 05, 2008 (93M11)	3	(R1)61	213	150	Seed	14	< 0.01 (< 0.01, < 0.01)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
		79	213	142						
		80	224	144						

Picoxystrobin

Location, Trial No., Year (Variety)	Application				Sample	DAT ^b	Residues (mg/kg) ^c			
	No.	Growth stage	g ai/ha ^a	L/ha			Parent	IN-QDY62	IN-QDY63	IN-QDK50
Trial 15, 2008 (Pioneer 93M11)	3	(R1)61	221	141	Seed	14	<u>0.011</u> (0.012, 0.010)	ND	ND	ND
		(R7)81	224	163						
		(R7)81	224	165						
					Process seed	14	0.010	ND	ND	ND
					AGF	14	1.9 c0.018	0.12	0.20	0.048
Branchton, ON, Canada Trial 06, 2008 (Mirra)	3	(R1)61	213	150	Seed	14	<u>0.031</u> (0.024, 0.037)	ND	ND	ND
		81	221	150						
		85-88	229	150						
Paris, ON, Canada Trial 07, 2008 (DK-27-07)	3	(R1)61	224	150	Seed	14	<u>< 0.01</u> (< 0.01, < 0.01)	ND	ND	ND
		85	228	150						
		96-97	224	150						
Paynesville, MN, USA Trial 08, 2009 (AGO0501 Asgrow)	3	(R1)61	214	143	Seed	14	ND (ND, ND)	ND	ND	ND
		73-79	216	142						
		73-79	217	142						
Geneva, MN, USA Trial 09, 2008 (Pioneer 91M80)	3	(R1)61	222	145	Seed	14	ND (ND, ND)	ND	ND	ND
		(R6-7)79-81	221	162						
		(R7)81	220	163						
Lenexa, KS, USA Trial 10, 2008 (395NRR)	3	(R1)61	221	135	Seed	14	<u>< 0.01</u> (< 0.01, < 0.01)	ND	ND	ND
		77	224	138						
		79	221	138						

Location, Trial No., Year (Variety)	Application				Sample	DAT ^b	Residues (mg/kg) ^c			
	No.	Growth stage	g ai/ha ^a	L/ha			Parent	IN-QDY62	IN-QDY63	IN-QDK50
Rochelle, IL, USA Trial 11, 2008 (Pioneer 92M61)	3	(R1)61	224	46	Seed	14	<u>0.039</u> (0.032, 0.045)	ND	ND	ND
		79	224	46						
		81	223	46						
Britton, SD, USA Trial 12, 2008 (Pioneer 90M80 Roundup Ready)	3	(R1)61	224	187	Seed	14	<u>ND</u> (ND, ND)	ND	ND	ND
		(R6-7)79-81	224	187						
		(R7-8)81-89	224	187						
Springfield, NE, USA Trial 13, 2008 (MW GR3631)	3	(R1)61	224	132	Seed	14	<u>< 0.01</u> (< 0.01, < 0.01)	ND	ND	ND
		79	223	134						
		79	224	133						
Carlyle, IL, USA Trial 14, 2008 (NK 37-N4)	3	(R1)61	213	148	Seed	17	<u>0.012</u> (0.011, 0.013)	ND	ND	ND
		(R6-7)79-81	213	183						
		(R7)81	220	126						
					Process seed	17	< 0.01	ND	ND	ND
					AGF	17	3.2 c0.005	0.015	0.098	0.024
LaPlata, MO, USA Trial 16, 2008 (Asgrow AG3802)	3	(R1)61	222	163	Seed	14	<u>0.010</u> (0.010, < 0.01)	ND	ND	ND
		(R7)81	222	190						
		(R7-8)81-89	219	191						

Location, Trial No., Year (Variety)	Application				Sample	DAT ^b	Residues (mg/kg) ^c			
	No.	Growth stage	g ai/ha ^a	L/ha			Parent	IN-QDY62	IN-QDY63	IN-QDK50
Fisk, MO, USA Trial 17, 2009 (54-17 RR/STS)	3	61 81 84	220 221 224	187 187 187	Seed	13	<u>≤ 0.01</u> (<u>< 0.01</u> , <u>< 0.01</u>)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
Dudley, MO, USA Trial 18, 2009 (Jake)	3	61 81 84	221 225 218	187 187 187	Seed	13	<u>0.019</u> (0.015, 0.023)	ND (ND, ND)	< 0.01 (ND, < 0.01)	ND (ND, ND)
Tipton, MO, USA Trial 19, 2009 (48-24 Mor Soy)	3	(R1)61 (R7)81 (R7)81	220 222 224	272 281 281	Seed	14	<u>ND</u> (ND, ND)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
Gardner, KS, USA Trial 20, 2009 (Fontanelle 407NRS)	3	60 81 83	220 217 217	138 138 133	Seed	13	<u>≤ 0.01</u> (<u>< 0.01</u> , <u>< 0.01</u>)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
Springfield, NE, USA Trial 21, 2009 (NC+2A98)	3	60 81 83	213 220 213	129 131 130	Seed	13	<u>0.035</u> (0.036, 0.034)	< 0.01 (ND, < 0.01)	ND (ND, ND)	< 0.01 (ND, < 0.01)

ND = not detected (< 0.003 mg/kg)

^a Individual application rates shown

^b DAT = Days After Treatment

^c Mean result shown, with individual results for analyses of duplicate samples from the same plot in brackets. Residues detected in control samples are indicated with c preceding the reported residue value.

Peas (dry) and beans (dry)

A series of 11 trials each in peas (dry) and beans (dry) was conducted in the USA and Canada in the 2008 growing season in accordance with the Canadian GAP for pulses other than soya beans (study number 24863: Shepard, 2010). A single treated plot was established at each site, except for trials 02 and 10 where two plots were established to aid in generating sufficient material for the decline data points. Plot sizes were sufficient, being at least 45 m². Two applications were made at the target rate of 220 g ai/ha, around 7 days apart and at approximate growth stages of BBCH 75–85, using a CO₂-pressurised backpack sprayer or a quad bike- or tractor-mounted boom sprayer. A spray adjuvant, a non-ionic surfactant, methylated vegetable oil or crop oil, was included in the spray tank at each site. Duplicate treated samples were collected, along with a single untreated control sample. Sample sizes

were typically at least 1.0 kg for vines and seed, and 0.5 kg for hay (except for vine samples from site 07, which were around 0.6–0.75 kg).

Samples were analysed for picoxystrobin and the metabolites IN-QDY62, IN-QDY 63 and IN-QDK50 by LC/MS/MS (method number 24868). Acceptable mean concurrent recoveries and precision values were achieved.

Residue data for peas (dry) and beans (dry) are presented in Tables 55 and 56 respectively, while data for pea vines and hay are presented in Table 64 and 65 respectively.

Table 55 Results of residue trials conducted with picoxystrobin (250 g/L SC) in peas (dry) in the USA and Canada in 2008 (study number 24863)

Location Trial, Year	Application				Sample	DAT ^b	Residues (mg/kg) ^c			
	No.	BBCH stage	g ai/ha ^a	L/ha			Parent	IN-QDY62	IN-QDY63	IN-QDK50
Geneva, MN, USA Trial 01, 2008 (Midas)	2	81	220	165	Seed	14	<u>< 0.01</u>	ND	ND	ND
		85	220	157			(< 0.01, < 0.01)	(ND, ND)	(ND, ND)	(ND, ND)
Parkdale, OR, USA Trial 02, 2008 (Green Arrow)	2	69–73	224	193	Seed	14	<u>0.025</u>	< 0.01	ND	0.037
		79–85	225	190			(0.019, 0.031)	(ND, < 0.01)	(ND, ND)	(0.032, 0.042) c0.020
Payette, ID, USA Trial 03, 2008 (Austrian Winter)	2	74	221	187	Seed	14	<u>0.016</u>	ND	ND	0.013
		79	219	187			(0.012, 0.020)	(ND, ND)	(ND, ND)	(0.011, 0.014)
Jerome, ID, USA Trial 04, 2008 (Pendleton)	2	79	224	186	Seed	14	<u>0.013</u>	ND	ND	0.011
		81	224	183			(0.014, 0.011)	(ND, ND)	(ND, ND)	(0.011, 0.011)
Filer, ID, USA Trial 05, 2008 (Early Resistant Perfection)	2	78	226	168	Seed	14	<u>0.016</u>	ND	ND	0.020
		79	225	168			(0.015, 0.016)	(ND, ND)	(ND, ND)	(0.019, 0.020)
Madras, OR, USA Trial 06, 2008 (K2)	2	79	228	191	Seed	14	<u>ND</u>	ND	ND	ND
		81	221	186			(ND, ND)	(ND, ND)	(ND, ND)	(ND, ND)
Ephrata, WA, Trial 07, 2008	2	81–82	225	188	Seed	14	<u>< 0.01</u>	ND	ND	< 0.01
		88	223	186			(< 0.01, < 0.01,	(ND, (ND, ND)	(ND, ND)	(< 0.01,

Location Trial, Year	Application				Sample	DAT ^b	Residues (mg/kg) ^c			
	No.	BBCH stage	g ai/ha ^a	L/ha			Parent	IN-QDY62	IN-QDY63	IN-QDK50
(Kalamo)							< 0.01)	ND)		< 0.01)
Innisfail, AB, Canada Trial 08, 2008 (SW Cheri)	2	79–81 85–86	223 221	150 151	Seed	14	<u>0.033</u> (0.028, 0.037)	ND (ND, ND)	ND (ND, ND)	< 0.01 (< 0.01, < 0.01)
Rosthern, SK, Canada Trial 09, 2008 (CDC Bronco)	2	75–77 77–82	223 222	204 203	Seed	14	<u>0.010</u> (0.010, 0.010)	ND (ND, ND)	ND (ND, ND)	< 0.01 (< 0.01, < 0.01)
Waldheim, SK, Canada Trial 10, 2008 (Bronco)	2	84–85 87–88	220 217	150 150	Seed	14	<u>< 0.01</u> (< 0.01, < 0.01)	ND (ND, ND)	ND (ND, ND)	< 0.01 (ND, < 0.01)
Fort Saskatchewan, AB, Canada Trial 11, 2008 (Cooper)	2	74 80–81	222 226	180 180	Seed	14	<u>0.012</u> (0.011, 0.013)	ND (ND, ND)	ND (ND, ND)	< 0.01 (< 0.01, < 0.01)

ND = not detected (< 0.003 mg/kg)

^a Individual application rates shown

^b DAT = Days After Treatment

^c Mean result shown, with individual results for analyses of duplicate samples from the same plot in brackets.

Table 56 Results of residue trials conducted with picoxystrobin (250 g/L SC) in beans (dry) in the USA and Canada in 2008 (study number 24863)

Location Trial, Year (Variety)	Application				Sample	DAT ^b	Residues (mg/kg) ^c			
	No.	BBCH stage	g ai/ha ^a	L/ha			Parent	IN-QDY62	IN-QDY63	IN-QDK50
Portage la Prairie, MB, Canada Trial 12, 2008 (Envoy)	2	84 85	215 217	187 187	Seed	14	<u>ND</u> (ND, ND)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
Oakville, MB, Canada Trial 13, 2008 (Envoy)	2	82 85	215 217	187 187	Seed	15	<u>< 0.01</u> (< 0.01, < 0.01)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
Paynesville, MN, USA	2	83	214	143	Seed	14	<u>ND</u> (ND,	ND (ND,	ND	ND (ND,

Location Trial, Year (Variety)	Application				Sample	DAT ^b	Residues (mg/kg) ^c			
	No.	BBCH stage	g ai/ha ^a	L/ha			Parent	IN- QDY62	IN- QDY63	IN- QDK50
Trial 14, 2008 (Black Turtle)		87	216	143			ND)	ND)	(ND, ND)	ND)
Wyoming, IL, USA Trial 15, 2008 (Pinto)	2	R7(81) R7(81)	224 224	159 165	Seed	14	<u>0.038</u> (0.035, 0.040)	< 0.01 (< 0.01, < 0.01)	ND (ND, ND)	ND (ND, ND)
Delavan, WI, USA Trial 16, 2008 (Pinto)	2		221 221	178 178	Seed	14	<u>< 0.01</u> (< 0.01, < 0.01)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
Eldridge, ND, USA Trial 17, 2008 (Navigator)	2	80 85	223 223	187 187	Seed	14	<u>0.011</u> (0.011, < 0.01)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
Taber, AB, Canada Trial 18, 2008 (Black)	2	75–79 77–78	223 222	152 151	Seed	14	<u>0.011</u> (< 0.01, 0.012)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
Larned, KS, USA Trial 19, 2008 (Pinto Field)	2	72 77	224 226	168 168	Seed	14	<u>0.016</u> (0.015, 0.016)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
Jerome, ID, USA Trial 20, 2008 (Othello Pinto)	2	74 78	222 225	192 194	Seed	14	<u>< 0.01</u> (< 0.01, < 0.01)	ND (ND, ND)	ND (ND, ND)	< 0.01 (< 0.01, ND)
Live Oak, CA, USA Trial 21, 2008 (Canario)	2	75 79	220 217	141 141	Seed	14	<u>< 0.01</u> (< 0.01, < 0.01)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
Parkdale, OR, USA Trial 22, 2008 (Blue Lake 91)	2	75 79	221 223	187 191	Seed	14	<u>0.038</u> (0.042, 0.033)	ND (ND, ND)	0.022 (0.025, 0.019)	ND (ND, ND)

ND = not detected (< 0.003 mg/kg)

^a Individual application rates shown

^b DAT = Days After Treatment

^c Mean result shown, with individual results for analyses of duplicate samples from the same plot in brackets.

Cereal grains

Wheat and barley

A series of 26 trials in wheat and 21 trials in barley was conducted in the USA and Canada in the 2008 and 2009 growing seasons in accordance with the Canadian GAP for cereal grains (study number 24860: Thiel 2010). A single treated plot was established at each site. Plot sizes were sufficient, being at least 30 m². Three applications were made at the target rate of 220 g ai/ha, at 7–14 day intervals, using either a CO₂-pressurised backpack sprayer or a tractor- or quad bike-mounted boom sprayer. In all but seven of the trials (trial numbers 07, 09, 12, 15, 28, 30, and 34), a spray adjuvant (a non-ionic surfactant or methylated vegetable oil) was included in the tank mix. Duplicate treated samples were collected, along with a single untreated control sample. Sample sizes were typically at least 1.0 kg for forage and grain, and 0.5 kg for hay and straw (except for one of the treated hay samples and the untreated control hay sample from trial 35, which were only 0.4 kg).

Samples were analysed for picoxystrobin and the metabolites IN-QDY62, IN-QDY 63 and IN-QDK50 by LC/MS/MS (method number 24868). Acceptable mean concurrent recoveries and precision values were achieved.

Residue data for wheat and barley are presented in Tables 57 and 58 respectively, while residue data for wheat forage, wheat hay, wheat straw, barley hay, and barley straw are presented in Tables 66, 67, 68, 69, and 70 respectively.

Table 57 Results of residue trials conducted with picoxystrobin (250 g/L SC) in wheat in the USA and Canada in 2008 and 2009 (study 24860)

Location Trial no., Year (Variety)	Application				Sample	DAT ^b	Residues (mg/kg) ^c			
	No.	BBCH stage	g ai/ha ^a	L/ha			Parent	IN-QDK50	IN-QDY62	IN-QDY63
Seven Springs, NC, USA Trial 01, 2008 (Coker 9478)	3	39 57–58 69–71	217 231 220	135 208 195	Grain	47	<u>< 0.01</u> (<u>< 0.01</u> , < 0.01)	ND (ND, ND)	< 0.01 (ND, < 0.01)	ND (ND, ND)
Fisk, MO, USA Trial 02, 2008 (Coker 9663)	3	39 45–47 69	222 223 222	187 187 187	Grain	35	<u>< 0.01</u> (<u>< 0.01</u> , < 0.01)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
Elm Creek, MB, Canada Trial 03, 2008 (AC Barrie)	3	30–31 32 55	231 230 224	200 200 200	Grain	47	<u>< 0.01</u> (<u>< 0.01</u> , < 0.01)	< 0.01 (ND, < 0.01)	ND (ND, ND)	ND (ND, ND)
Richland, IA, USA Trial 04, 2008	3	30–31 59 65–69	223 213 224	153 178 184	Grain	45	<u>< 0.01</u> (<u>< 0.01</u> , < 0.01)	ND (ND, ND)	< 0.01 (<u>< 0.01</u> , ND)	ND (ND, ND)

Location Trial no., Year (Variety)	Application				Sample	DAT ^b	Residues (mg/kg) ^c			
	No.	BBCH stage	g ai/ha ^a	L/ha			Parent	IN- QDK50	IN- QDY62	IN- QDY63
(Wilcross 07GV6S- 753)										
Lenexa, KS, USA Trial 05, 2008 (Overly)	3	30–31 32–37 59	224 225 224	144 145 144	Grain	45	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
Hinton, OK, USA Trial 06, 2008 (Jagger)	3	39 61 75	222 220 231	125 133 139	Grain	45	< 0.01 (< 0.01, < 0.01)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
Carrington, ND, USA Trial 07, 2008 (Kelby)	3	30–31 45 71	226 228 224	140 140 139	Grain	45	< 0.01 (ND, < 0.01)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
Taber, AB, Canada Trial 08, 2008 (AC Barrie)	3	30 61 71–73	231 230 216	154 154 146	Grain	46	<u>0.022</u> (0.026, 0.018)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
New Rockford, ND, USA Trial 09, 2008 (Kelby)	3	30–31 32 65	221 216 217	141 140 140	Grain	46	< 0.01 (< 0.01, < 0.01)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
Eldridge, ND, USA Trial 10, 2008 (Glynn)	3	30–31 37 59	224 224 224	141 182 172	Grain	45	< 0.01 (< 0.01, < 0.01)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
Dundurn, SK, Canada Trial 11, 2008 (Lillian)	3	31 52–59 69–73	225 222 222	200 200 200	Grain	45	<u>0.019</u> (0.017, 0.020)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
Hanley, SK, Canada Trial 12, 2008 (Lillian)	3	31 51–55 65–69	220 223 224	200 200 200	Grain	45	0.013 (0.016, ND) c0.014	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)

Picoxystrobin

Location Trial no., Year (Variety)	Application				Sample	DAT ^b	Residues (mg/kg) ^c			
	No.	BBCH stage	g ai/ha ^a	L/ha			Parent	IN-QDK50	IN-QDY62	IN-QDY63
Cordell, OK, USA Trial 13, 2008 (Jagger)	3	51 65 83	217 223 222	72 70 82	Grain	40	<u>0.028</u> (0.027, 0.029)	< 0.01 (ND, < 0.01)	ND (ND, ND)	ND (ND, ND)
Levelland, TX, USA Trial 14, 2009 (TAM 105)	3	6–8 in. 10 in. 51–59	230 228 226	140 140 140	Grain	45	<u>0.013</u> (0.016, ND)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
Olton, TX, USA Trial 15, 2008 (Dumas)	3	37 43–51 65–69	224 223 230	157 157 157	Grain	45	<u>ND</u> (ND, ND)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
Larned, KS, USA Trial 16, 2008 (Jagger)	3	30–31 37 61	224 213 224	168 168 168	Grain	44	<u>ND</u> (ND, ND)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
Ephrata, WA, USA Trial 17, 2008 (Dark northern spring)	3	30–31 47–49 57–58	225 226 224	187 189 187	Grain	47	<u>ND</u> (ND, ND)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
Minto, MB, Canada Trial 18, 2008 (Superb)	3	31–32 37–41 57–59	224 226 224	158 162 160	Grain	51	<u>< 0.01</u> (ND, < 0.01)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
Boissevain, MB, Canada Trial 19, 2008 (Strongfield durum)	3	31–32 34–37 41–55	229 228 224	164 163 159	Grain	58	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
Rosthern, SK, Canada Trial 20, 2008 (AC Lillian)	3	31 37–39 59–69	227 224 226	203 199 201	Grain	56	<u>< 0.01</u> (<u>< 0.01</u> , < 0.01)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
Hepburn, SK, Canada Trial 21,	3	31 37–41	223 224	199 199	Grain	54	<u>0.010</u> (0.010,	ND (ND,	ND (ND, ND)	ND (ND,

Location Trial no., Year (Variety)	Application				Sample	DAT ^b	Residues (mg/kg) ^c			
	No.	BBCH stage	g ai/ha ^a	L/ha			Parent	IN-QDK50	IN-QDY62	IN-QDY63
2008 (AC Lillian)		59–69	229	203			< 0.01)	ND)		ND)
Fort Saskatchewan, AB, Canada Trial 22, 2008 (AC Foremost)	3	31 45–54 69	222 224 224	180 180 180	Grain	45	<u>0.010</u> (0.010, 0.010)	< 0.01 (< 0.01, < 0.01)	ND (ND, ND)	ND (ND, ND)
Trial 23, 2008 (AC Foremost)	3	31 45–52 69	222 224 224	180 180 180	Grain	45	0.010 (0.010, < 0.01)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
Alvena, SK, Canada Trial 24, 2008 (Lillian)	3	31 56–59 69–71	223 223 225	200 200 200	Grain	45	<u>0.014</u> (0.016, 0.012)	< 0.01 (< 0.01, ND)	ND (ND, ND)	ND (ND, ND)
Waldheim, SK, Canada Trial 25, 2008 (Lillian)	3	31 55–59 69–71	223 222 224	200 200 200	Grain	45	<u>0.025</u> (0.021, 0.028)	< 0.01 (< 0.01, ND)	ND (ND, ND)	ND (ND, ND)
Northwood, ND, USA Trial 46, 2008 (Kelby)	3	30–31 49 71	214 219 217	184 188 187	Grain	45	<u>ND</u> (ND, ND)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)

^a Individual application rates reported, together with the seasonal rate (underlined)

^b DAT = Days After Treatment

^c Mean result shown, with individual results for analyses of duplicate samples from the same plot in brackets. Residues detected in control samples are indicated with c preceding the reported residue value.

Table 58 Results of residue trials conducted with picoxystrobin (250 g/L SC) in barley in the USA and Canada in 2008 and 2009 (study 24860)

Location Trial no., Year (Variety)	Application				Sample	DAT ^b	Residues (mg/kg) ^c			
	No.	BBCH stage	g ai/ha ^a	L/ha			Parent	IN-QDK50	IN-QDY62	IN-QDY63
Germansville, PA, USA, Trial 26, 2008	3	30–31 39 51	233 230 231	291 288 289	Grain	45	<u>0.047</u> (0.044, 0.049)	ND (ND, ND)	< 0.01 (< 0.01, < 0.01)	ND (ND, ND)

Location Trial no., Year (Variety)	Application				Sample	DAT ^b	Residues (mg/kg) ^c			
	No.	BBCH stage	g ai/ha ^a	L/ha			Parent	IN-QDK50	IN-QDY62	IN-QDY63
(NP)										
Richland, IA, USA Trial 27, 2008 (Robust)	3	30–31 32 59	222 228 219	139 170 159	Grain	45	<u>0.022</u> (0.024, 0.019)	ND (ND, ND)	< 0.01 (< 0.01, < 0.01)	ND (ND, ND)
Delavan, WI, USA Trial 28, 2008 (Kewaunee)	3	30–31 32 55	225 223 224	164 154 161	Grain	46	<u>0.014</u> (0.014, 0.013)	< 0.01 (< 0.01, < 0.01)	< 0.01 (ND, < 0.01)	ND (ND, ND)
Frederick, SD, USA Trial 29, 2008 (Robust)	3	30–31 37 65–71	224 224 224	94 94 94	Grain	45	<u>0.028</u> (0.031, 0.024)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
Carrington, ND, USA Trial 30, 2008 (Tradition)	3	30–31 32 65	221 216 217	139 141 140	Grain	45	<u>0.028</u> (0.027, 0.028)	ND (ND, ND)	ND (ND, ND)	< 0.01 (< 0.01, < 0.01)
Eldridge, ND, USA Trial 31, 2008 (Tradition)	3	30–31 37 59	222 224 221	140 140 140	Grain	45	<u>0.016</u> (0.017, 0.014)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
Velva, ND, USA Trial 32, 2008 (Legacy)	3	30–31 32 47–49	223 224 229	138 139 141	Grain	45	<u>ND</u> (ND, ND)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
Jerome, ID, USA Trial 33, 2008 (Harrington)	3	32 39 71	224 224 230	143 164 161	Grain	45	<u>0.016</u> (0.017, 0.015)	< 0.01 (< 0.01, ND)	ND (ND, ND)	ND (ND, ND)
Live Oak, CA, USA Trial 34, 2008 (UC-937)	3	37–39 49 59	225 224 225	188 187 186	Grain	77	0.012 (0.011, 0.012)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
Madras, OR, USA Trial 35, 2008	3	32 53 83–85	234 233 222	199 192 190	Grain	47	<u>0.087</u> (0.076, 0.098)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.015 (0.015, 0.014)

Location Trial no., Year (Variety)	Application				Sample	DAT ^b	Residues (mg/kg) ^c			
	No.	BBCH stage	g ai/ha ^a	L/ha			Parent	IN-QDK50	IN-QDY62	IN-QDY63
(Bellford)							c0.005			
Minto, MB, Canada Trial 36, 2008 (Conion)	3	31–32 33–37 49–58	220 229 231	157 163 206	Grain	47	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
Boissevain, MB, Canada Trial 37, 2008 (Copelan)	3	31–33 33–37 43–54	224 222 225	160 159 201	Grain	57	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
Rosthern, SK, Canada Trial 38, 2008 (AC Metcalf)	3	31 37 59	230 221 225	205 197 201	Grain	53	<u>0.011</u> (0.011, 0.011)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
Hepburn, SK, Canada Trial 39, 2008 (AC Metcalf)	3	31 39 59	226 220 222	200 196 198	Grain	47	<u>< 0.01</u> (<u>< 0.01</u> , <u>< 0.01</u>)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
Innisfail, AB, Canada Trial 40, 2008 (Metcalf)	3	33–36 39–47 55–59	224 215 224	250 250 250	Grain	58	0.010 (<u>< 0.01</u> , 0.010)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
Fort Saskatchewa n, AB, Canada Trial 41, 2008 (Bold)	3	31 45–52 60–61	228 222 224	180 180 180	Grain	45	<u>0.017</u> (0.020, 0.014)	< 0.01 (<u>< 0.01</u> , <u>< 0.01</u>)	ND (ND, ND)	ND (ND, ND)
Trial 42, 2008 (Bold)	3	31 55–59 59–60	224 220 235	178 180 180	Grain	45	< 0.01 (<u>< 0.01</u> , <u>< 0.01</u>)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
Lamont, AB, Canada Trial 43, 2008 (Bold)	3	31 47–51 72	222 223 223	180 180 180	Grain	45	<u>0.029</u> (0.029, 0.028)	< 0.01 (<u>< 0.01</u> , <u>< 0.01</u>)	ND (ND, ND)	ND (ND, ND)
Alvena, SK, Canada	3	31 56–59	223 223	200 200	Grain	45	<u>0.12</u> (0.15,	< 0.01 (<u>< 0.01</u> ,	< 0.01 (<u>< 0.01</u> ,	0.011 (0.012,

Location Trial no., Year (Variety)	Application				Sample	DAT ^b	Residues (mg/kg) ^c			
	No.	BBCH stage	g ai/ha ^a	L/ha			Parent	IN-QDK50	IN-QDY62	IN-QDY63
Trial 44, 2008 (Legacy)		69–75	223	200			0.082)	< 0.01)	< 0.01)	< 0.01)
Waldheim, SK, Canada Trial 45, 2008 (Legacy)	3	31 55–59 71–73	223 222 217	200 200 200	Grain	45	<u>0.22</u> (0.21, 0.23)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.019 (0.018, 0.019)
Northwood, ND, USA Trial 47, 2008 (Tradition)	3	30–31 32 59	221 216 221	190 186 188	Grain	44	<u>< 0.01</u> (< 0.01, < 0.01)	ND (ND, ND)	ND (ND, ND)	< 0.01 (< 0.01, ND)

^a Individual application rates reported, together with the seasonal rate (underlined)

^b DAT = Days After Treatment

^c Mean result shown, with individual results for analyses of duplicate samples from the same plot in brackets. Residues detected in control samples are indicated with c preceding the reported residue value.

Maize

A series of 15 trials in maize was conducted in the USA and Canada in the 2008 growing season in accordance with the Canadian GAP for maize (study number 24864: Shepard 2009). A single treated plot of sufficient area (at least 45 m²) was established at each site. Three applications were made at the target rate of 220 g ai/ha, with the first being timed around growth stage R1 (silking), the second and third around 6–10 weeks later at stages R5-R6 (between dent and maturity), with about 7 days between applications 2 and 3. Applications were made using a backpack sprayer or a tractor-mounted boom sprayer. A spray adjuvant (non-ionic surfactant, crop oil concentrate or methylated vegetable oil) were included in all tank mixes. Duplicate treated samples were collected at each sampling interval, with single untreated control samples being collected. Sample sizes were at least 1.0 kg for forage and grain and at least 0.5 kg for stover.

Samples were analysed for picoxystrobin and the metabolites IN-QDY62, IN-QDY 63 and IN-QDK50 by LC/MS/MS (method number 24868). Acceptable mean concurrent recoveries and precision values were achieved, with the exception of IN-QDY63 in maize stover and IN-QDK50 in maize grain (RSD = 21% and 23% respectively).

Residue data for maize grain are presented in Table 59, while data for maize forage and stover are presented in Tables 71 and 72 respectively.

Table 59 Results of residue trials conducted with picoxystrobin (250 g/L SC) in maize in the USA and Canada in 2008 (study number 24864)

Location Trial no., Year (variety)	Application				Sample	DAT ^b	Residues (mg/kg) ^c			
	No.	BBCH stage	g ai/ha ^a	L/ha			Parent	IN-QDY62	IN-QDY63	IN-QDK50
Germansville, PA, USA Trial 01,	3	Early R1	226 226	330 433	Grain	7	<u>ND</u> (ND,	ND (ND,	ND (ND,	ND (ND, ND)

Location Trial no., Year (variety)	Application				Sample	DAT ^b	Residues (mg/kg) ^c			
	No.	BBCH stage	g ai/ha ^a	L/ha			Parent	IN-QDY62	IN-QDY63	IN-QDK50
2008 (TA 3892)		89 89	223	428			ND)	ND)	ND)	
Blackville, SC, USA Trial 02, 2008 (OK 69-72)	3	65 89 89	224 224 224	186 181 185	Grain	7	<u>ND</u> (ND, ND)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
Paris, ON, Canada Trial 03, 2008 (DeKalb 50- 20)	3	R1 R5 R5-R6	215 228 217	200 200 200	Grain	7	<u>< 0.01</u> (ND, < 0.01)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
Branchton, ON, Canada Trial 04, 2008 (Pioneer 38A59)	3	R1 R5 R5-R6	213 213 213	200 200 200	Grain	7	<u>0.011</u> (<u>< 0.01</u> , 0.012)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
Richland, IA, USA Trial 05, 2008 (Middle Koop 5513)	3	R1 R6 R6	213 224 224	167 162 165	Grain	6	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
					Process grain	6	<u>0.012</u> (0.010, 0.014)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
					AGF	6	0.15 (0.14, 0.16) c0.008	< 0.01 (<u>< 0.01</u> , < 0.01)	ND (ND, ND)	ND (ND, ND)
Wyoming, IL, USA Trial 06, 2008 (DKC60-18)	3	R1 R6 R6	224 224 224	193 188 186	Grain	7	<u>ND</u> (ND, ND)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
Paynesville, MN, USA Trial 08, 2009 (DKC35)	3	R1 R6 R6	215 217 215	143 142 143	Grain	7	<u>ND</u> (ND, ND)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
Gardner, ND, USA Trial 09, 2008 (2K145)	3	R4 R5 R6	223 221 223	159 159 159	Grain	7	<u>ND</u> (ND, ND)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)

Picoxystrobin

Location Trial no., Year (variety)	Application				Sample	DAT ^b	Residues (mg/kg) ^c			
	No.	BBCH stage	g ai/ha ^a	L/ha			Parent	IN-QDY62	IN-QDY63	IN-QDK50
Lenexa, KS, USA Trial 10, 2008 (08HYBBIO 8REM)	3	R1	220	134	Grain	7	<u>ND</u>	ND	ND	ND
		87	221	135			(ND, ND)	(ND, ND)	(ND, ND)	(ND, ND)
		87	220	137						
Delavan, WI, USA Trial 11, 2008 (DKC51-39)	3	R1	220	196	Grain	7	<u>ND</u>	ND	ND	ND
		R5.5	221	199			(ND, ND)	(ND, ND)	(ND, ND)	(ND, ND)
		R5.75	219	201						
Springfield, NE, USA Trial 12, 2008 (NK N38-04)	3	R1	224	130	Grain	7	<u>ND</u>	ND	ND	ND
		87	224	132			(ND, ND)	(ND, ND)	(ND, ND)	(ND, ND)
		89	220	132						
Tipton, MO, USA Trial 13, 2008 (DeKalb DKC6423)	3	R1	224	262	Grain	7	<u>ND</u>	ND	ND	ND
		R5	224	256			(ND, ND)	(ND, ND)	(ND, ND)	(ND, ND)
		R5	224	259						
Carlyle, IL, USA Trial 14, 2008 (Burrus 616 XLR)	3	R1	225	150	Grain	7	<u>< 0.01</u>	ND	ND	ND
		R6	222	162			(< 0.01, < 0.01)	(ND, ND)	(ND, ND)	(ND, ND)
		R6	216	172						
					Process grain	7	< 0.01 (< 0.01, < 0.01)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
					AGF	7	0.17 (0.18, 0.13) c0.003	0.26 (0.27, 0.25)	< 0.01 (< 0.01, < 0.01)	ND (ND, ND)
La Plata, MO, USA Trial 15, 2008 (LG 2540)	3	R1	221	159	Grain	7	<u>< 0.01</u>	ND	ND	ND
		R6	221	195			(< 0.01, < 0.01)	(ND, ND)	(ND, ND)	(ND, ND)
		R6	223	191						
Hinton, OK, USA Trial 16, 2008 (DKC51-45)	3	75	222	178	Grain	7	<u>< 0.01</u>	< 0.01	ND	ND
		87	224	189			(< 0.01, < 0.01)	(< 0.01, ND)	(ND, ND)	(ND, ND)
		89	219	190						

ND = not detected (< 0.003 mg/kg)

^a Individual application rates shown, together with seasonal rate (underlined)

^b DAT = Days After Treatment

^c Mean result shown, with individual results for analyses of duplicate samples from the same plot in brackets. Residues detected in control samples are indicated with c preceding the reported residue value.

Rape seed

A series of 18 trials in rape seed (canola) was conducted in the USA and Canada in the 2008 growing season in accordance with the Canadian GAP for rape seed (study number 24862: Thiel, 2009). Plot sizes were sufficient, being at least 45 m². Two applications were made at the target rate of 220 g ai/ha, at 7-day intervals, at BBCH growth stages 65–85. A non-ionic surfactant was included in the tank mix for all applications. Seed samples were collected at normal harvest, with duplicate treated samples and a single untreated control sample being collected at each site. Samples were generally at least 1 kg, except for some decline samples which were > 500 grams.

Samples were analysed for picoxystrobin and the metabolites IN-QDY62, IN-QDY 63 and IN-QDK50 by LC/MS/MS (method number 24868). Acceptable mean concurrent recoveries and precision values were achieved.

Table 60 Results of residue trials conducted with picoxystrobin (250 g/L SC) in oilseed rape in the USA and Canada in 2008 (study number 24862)

Location Trial no., Year (Variety)	Application				Sample	DAT ^b	Residues (mg/kg) ^c			
	No.	Growth stage	g ai/ha ^a	L/ha			Parent	IN-QDY62	IN-QDY63	IN-QDK50
Montezuma, GA, USA Trial 01, 2008 (Flint)	2	Podfill Podfill	225 224	218 193	Seed	21	< 0.01 (< 0.01, < 0.01)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
Conklin, MI, USA Trial 02, 2008 (Dekalb DKL72-55)	2	79 80	223 222	204 203	Seed	19	0.018 (0.015, 0.021)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
Perley, MN, USA Trial 04, 2008 (Patriot)	2	69 76	222 233	140 140	Seed	22	0.016 (0.013, 0.018)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
Sykeston, ND, USA Trial 05, 2008 (45H26)	2	62 65	220 219	187 187	Seed	21	0.043 (0.045, 0.040)	0.010 (0.010, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.014 (0.016, 0.011)
Taber, AB, Canada Trial 06, 2008 (75-45RR)	2	78–80 80–82	214 234	213 220	Seed	20	< 0.01 (0.004, 0.005)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
Jerome, ID,	2	79	225	199	Pod and	–0	0.044	ND	ND	0.027

Location Trial no., Year (Variety)	Application				Sample	DAT ^b	Residues (mg/kg) ^c			
	No.	Growth stage	g ai/ha ^a	L/ha			Parent	IN-QDY62	IN-QDY63	IN-QDK50
USA Trial 07, 2008 (Phoenix)		82	224	188	seed		(0.052, 0.036)	(ND, ND)	(ND, ND)	(0.028, 0.026)
						+0	4.5 (4.9, 4.1)	ND (ND, ND)	< 0.01 (< 0.01, ND)	0.028 (0.026, 0.030)
						7	0.90 (0.80, 1.0)	ND (ND, ND)	0.032 (0.025, 0.039)	0.062 (0.065, 0.058)
						14	0.31 (0.27, 0.34)	ND (ND, ND)	0.019 (0.020, 0.017)	0.062 (0.054, 0.069)
					Seed	21	< 0.01 (< 0.01, < 0.01)	ND (ND, ND)	ND (ND, ND)	< 0.01 (ND, < 0.01)
					28	< 0.01 (< 0.01, < 0.01)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)	
Madras, OR, USA Trial 08, 2008 (Cracker Jack)	2	79	229	192	Seed	21	0.021 (0.024, 0.018)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
		83	232	196						
Ephrata, WA, USA Trial 09, 2008 (71-45RR)	2	65-69 72-74	226 226	188 190	Seed	21	0.011 (0.011, 0.011)	ND (ND, ND)	ND (ND, ND)	< 0.01 (< 0.01, < 0.01)
Minto, MB, Canada Trial 10, 2008 (5030)	2	69-75	222	159	Pod and seed	-0	0.016 (0.016, 0.015)	ND (ND, ND)	ND (ND, ND)	0.011 (0.012, 0.010)
		79	226	162		+0	3.5 (3.3, 3.6)	ND (ND, ND)	ND (ND, ND)	0.014 (0.014, 0.014)
						7	0.088 (0.087, 0.089)	ND (ND, ND)	ND (ND, ND)	0.019 (0.019, 0.018)
						15	0.044 (0.044, 0.044)	ND (ND, ND)	ND (ND, ND)	0.017 (0.016, 0.017)
					Seed	21	0.013 (0.012, 0.014)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
						28	0.012	ND	ND	ND

Location Trial no., Year (Variety)	Application				Sample	DAT ^b	Residues (mg/kg) ^c			
	No.	Growth stage	g ai/ha ^a	L/ha			Parent	IN-QDY62	IN-QDY63	IN-QDK50
							(0.012, 0.011)	(ND, ND)	(ND, ND)	(ND, ND)
Rosthern, SK, Canada Trial 11, 2008 (SP Banner)	2	69–75 74–77	227 232	202 207	Seed	21	0.039 (0.041, 0.036)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
Hepburn, SK, Canada Trial 12, 2008 (46A76)	2	69–74 73–77	228 231	203 206	Seed	21	0.023 (0.021, 0.025)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
Innisfail, AB, Canada Trial 13, 2008 (33-95)	2	69–75 79–80	220 217	250 250	Seed	21	0.032 (0.031, 0.032)	ND (ND, ND)	ND (ND, ND)	< 0.01 (< 0.01, < 0.01)
Innisfail, AB, Canada Trial 14, 2008 (7145)	2	81–83 83–85	234 222	300 300	Seed	21	0.045 (0.045, 0.045)	ND (ND, ND)	ND (ND, ND)	< 0.01 (ND, < 0.01)
Alvena, SK, Canada Trial 15, 2008 (Pioneer 45H72)	2	75–79 80–81	222 223	150 150	Seed	21	0.043 (0.041, 0.044)	ND (ND, ND)	ND (ND, ND)	< 0.01 (< 0.01, < 0.01)
Waldheim, SK, Canada Trial 16, 2008 (Pioneer 45H72)	2	80 81–82	225 228	150 150	Seed	21	0.047 (0.035, 0.059)	ND (ND, ND)	ND (ND, ND)	< 0.01 (ND, < 0.01)
Lamont, AB, Canada Trial 17, 2008 (45H72)	2	72 78	224 224	180 180	Seed	21	0.022 (0.024, 0.019)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
Fort Saskatchewan, AB, Canada Trial 18, 2008	2	66 71–72	224 223	250 250	Seed	26	0.031 (0.029, 0.033)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)

Location Trial no., Year (Variety)	Application				Sample	DAT ^b	Residues (mg/kg) ^c			
	No.	Growth stage	g ai/ha ^a	L/ha			Parent	IN-QDY62	IN-QDY63	IN-QDK50
(45H73)										
Trial 19, 2008 (Pioneer 45H72)	2	69 70	222 224	250 250	Seed	28	0.014 (0.014, 0.013)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)

ND = not detected (< 0.003 mg/kg)

^a Individual application rates shown, together with seasonal rate (underlined)

^b DAT = Days After Treatment

^c Mean result shown, with individual results for analyses of duplicate samples from the same plot in brackets.

Forage and fodder

Table 61 Results of residue trials conducted with picoxystrobin (250 g/L SC) in sweet corn forage in the USA and Canada in 2008 (study number 25881)

Location, Trial no., Year (Variety)	Application				Sample [%water]	DA T ^b	Residues (mg/kg) ^c				
	No.	Growth stage	g ai/ha ^a	L/ha			Parent		IN-QDY62	IN-QDY63	IN-QDK50
							FW ^d	DW ^e			
Germansville, PA, USA Trial 01, 2008 (Triple Sweet HYB)	4	Early tassel	222	398	Forage [83]	7	0.80 (0.63, 0.96)	4.7 (3.7, 5.6)	0.016 (0.016, 0.015)	< 0.01 (< 0.01, < 0.01)	0.31 (0.28, 0.34)
		Pollen shed	220	398							
		R2 blister	217	421							
		Early milk									
Blackville, SC, USA Trial 02, 2008 (Silver Queen)	4	59	219	177	Forage [80]	6	0.32 (0.29, 0.35)	1.7 (1.5, 1.8)	0.024 (0.022, 0.026)	< 0.01 (< 0.01, < 0.01)	0.083 (0.083, 0.082)
		65	224	179							
		73	221	179							
		75	220	193							
Oviedo, FL, USA Trial 03, 2008 (Honey 'n' Pearl)	4	51	229	281	Forage [85]	7	0.53 (0.68, 0.37) c0.019	3.5 (4.5, 2.5)	0.046 (0.051, 0.040)	< 0.01 (< 0.01, < 0.01)	0.17 (0.16, 0.18)
		59	224	281							
		73	224	281							
		75	226	281							
Branchton, ON, Canada Trial 04, 2008 (Ambrosia)	4	R1	248	200	Forage [82]	-0	0.20 (0.21, 0.19)	1.2 (1.2, 1.1)	< 0.01 (< 0.01, < 0.01)	ND (ND, ND)	0.069 (0.079, 0.059)
		R1	232	200							
		R2	213	200		+0	1.5 (1.4, 1.6)	8.4 (7.8, 8.9)	0.013 (0.010, 0.015)	ND (ND, ND)	0.076 (0.080, 0.071)
		R2	213	200							
					1	0.65 (0.63, 0.67)	3.6 (3.5, 3.7)	0.017 (0.013, 0.021)	< 0.01 (< 0.01, < 0.01)	0.077 (0.071, 0.083)	

Location, Trial no., Year (Variety)	Application				Sample [%water]	DA T ^b	Residues (mg/kg) ^c				
	No.	Growth stage	g ai/ha ^a	L/ha			Parent		IN-QDY62	IN-QDY63	IN-QDK50
							FW ^d	DW ^e			
						4	0.25 (0.24, 0.25)	1.4 (1.3, 1.4)	0.010 (< 0.01 , 0.010)	< 0.01 (< 0.01 , < 0.01)	0.081 (0.078, 0.083)
						7	0.19 (0.20, 0.18)	1.1 (1.1, 1.0)	0.012 (0.011, 0.013)	< 0.01 (< 0.01 , < 0.01)	0.080 (0.082, 0.077)
Conklin, MI, USA Trial 05, 2008 (Temptation)	4	59	222	204	Forage [84]	-0	0.68 (0.41, 0.95)	4.3 (2.6, 5.9)	0.014 (0.006, 0.021)	0.011 (< 0.01 , 0.012)	0.061 (0.046, 0.076)
		65	223	202			+0	2.5 (2.1, 2.9)	16 (13, 18)	0.019 (0.017, 0.021)	0.010 (< 0.01 , 0.010)
		71	224	200		1		2.6 (3.0, 2.2)	17 (19, 14)	0.020 (0.023, 0.016)	0.013 (0.014, 0.011)
		75	223	201		4	2.0 (1.5, 2.4)	12 (9.4, 15)	0.023 (0.020, 0.025)	0.018 (0.016, 0.019)	0.077 (0.075, 0.079)
						7	1.5 (1.5, 1.5)	9.4 (9.4, 9.4)	0.023 (0.024, 0.021)	0.021 (0.021, 0.021)	0.089 (0.091, 0.087)
									7	ND (ND, ND)	ND (ND, ND)
Paynesville, MN, USA Trial 06, 2009 (Jubilee)	4	71	216	143	Forage [78]	7	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)	
		72	216	142							
		73	217	143							
		75	215	143							
Richland, IA, USA Trial 07, 2008 (Iochief)	4	R1	224	162	Forage [82]	7	0.24 (0.26, 0.22)	1.3 (1.4, 1.2)	0.074 (0.080, 0.068)	0.014 (0.015, 0.013)	0.078 (0.086, 0.070)
		R2	224	147							
		R3	224	161							
		R4	213	159							
Taber, AB, Canada Trial 08, 2008 (Northern Supper Sweet)	4	69-74	216	150	Forage [82]	9	0.89 (0.96, 0.81)	4.9 (5.3, 4.5)	0.038 (0.039, 0.037)	< 0.01 (< 0.01 , < 0.01)	0.090 (0.11, 0.070)
		75-79	217	152							
		83-85	222	152							
		83-85	231	154							
Woodland, CA, USA Trial 09, 2008 (Silver Queen)	4	V15	220	187	Forage [84]	7	1.3 (0.87, 1.8)	8.2 (5.4, 11)	ND (ND, ND)	0.018 (0.014, 0.022)	0.10 (0.081, 0.12)
		VT	221	187							
		R1	222	188							
		Milk	221	187							
Madras, OR, USA Trial 10, 2008 (Jubilee)	4	63	223	192	Forage [80]	7	2.2 (2.2, 2.2)	11 (11, 11)	< 0.01 (ND, < 0.01)	0.035 (0.034, 0.035)	0.12 (0.12, 0.11)
		67	225	194							
		71	221	190							
		75	225	194							

Location, Trial no., Year (Variety)	Application				Sample [%water]	DAT ^b	Residues (mg/kg) ^c					
	No.	Growth stage	g ai/ha ^a	L/ha			Parent		IN-QDY62	IN-QDY63	IN-QDK50	
							FW ^d	DW ^e				
Forest Grove, OR, USA Trial 11, 2008 (Serendipity)	4	Kernel filling	212	209	Forage [82]	7	0.12	0.74	< 0.01	< 0.01	0.020	
			223	187			(0.16, 0.086)	(0.89, 0.48)	(< 0.01, < 0.01)	(< 0.01, ND)	(0.022, 0.017)	
		Kernels 70%	213	189								
			217	186								
		Kernel final size										
		Harvest maturity										

ND = not detected (< 0.003 mg/kg)

^a Individual application rates shown

^b DAT = Days After Treatment

^c Mean result shown, with individual results for analyses of duplicate samples from the same plot in brackets

^d Fresh weight

^e Dry weight. Residues detected in control samples are indicated with c preceding the reported residue value.

Table 62 Results of residue trials conducted with picoxystrobin (250 g/L SC) in soya bean forage in the USA and Canada in 2008 and 2009 (study number 24861)

Location, Trial no., Year (Variety)	Application				Sample [water %]	DAT ^a	Residues (mg/kg) ^b				
	No.	Growth stage	g ai/ha	L/ha			Parent		IN-QDY62	IN-QDY63	IN-QDK50
							FW ^c	DW ^d			
Blackville, SC, USA Trial 01, 2008 (Asgrow, H7242 RR)	1	63	224	150	Forage [79]	14	0.19	<u>0.88</u>	ND	< 0.01	0.055
							(0.19, 0.18)	(0.90, 0.86)	(ND, ND)	(< 0.01, < 0.01)	(0.057, 0.052)
Seven Springs, NC, USA Trial 02, 2008 (DKB-64-51)	1	61	217	140	Forage [78]	14	0.13	<u>0.57</u>	< 0.01	0.010	0.037
							(0.13, 0.12)	(0.59, 0.55)	(< 0.01, < 0.01)	(0.010, 0.010)	(0.039, 0.035)
Cheneyville, LA, USA Trial 03, 2008 (DG 33B52)	1	61	219	149	Forage [76]	14	0.19	<u>0.80</u>	< 0.01	0.012	0.040
							(0.15, 0.23)	(0.63, 0.96)	(< 0.01, < 0.01)	(0.012, 0.012)	(0.040, 0.039)
Fisk, MO, USA Trial 04, 2008 (Armor 47G7)	1	61-65	223	119	Forage [76]	14	0.34	<u>1.4</u>	0.010	0.011	0.080
							(0.31, 0.37)	(1.3, 1.5)	(< 0.01, 0.010)	(0.010, 0.011)	(0.078, 0.081)
Richland, IA, USA Trial 05, 2008 (93M11)	1	61	213	150	Forage [83]	0	13	77	ND	ND	0.022
							(14, 12)	(71, 82)	(ND, ND)	(ND, ND)	(0.022, 0.021)
						3	5.2	31	< 0.01	0.064	0.064
							(5.2, 5.3)	(31,)	(< 0.01,)	(0.067,)	(0.067,)

Location, Trial Year (Variety)	Application				Sample [water %]	DAT a	Residues (mg/kg) ^b				
	No.	Growth stage	g ai/ha	L/ha			Parent		IN- QDY62	IN- QDY63	IN- QDK50
							FW ^c	DW ^d			
							c0.003	31)	ND)	0.06)	0.06)
						7	0.79 (0.65, 0.92)	4.6 (3.8, 5.4)	ND (ND, ND)	0.011 (0.010, 0.012)	0.052 (0.049, 0.055)
						10	0.36 (0.35, 0.36)	2.1 (2.1, 2.1)	ND (ND, ND)	< 0.01 (< 0.01, < 0.01)	0.031 (0.034, 0.027)
						14	0.20 (0.23, 0.17)	1.2 (1.4, 1.0)	ND (ND, ND)	ND (ND, ND)	0.031 (0.037, 0.025)
Trial 15, 2008 (Pioneer 93M11)	1	61	221	141	Forage [80]	14	0.30 (0.25, 0.35)	<u>1.6</u> (1.3, 1.8)	< 0.01 (< 0.01, < 0.01)	< 0.01 (ND, < 0.01)	0.040 (0.034, 0.046)
Branchton, ON, Canada Trial 06, 2008 (Mirra)	1	61	213	150	Forage [84]	0	20 (21, 19)	125 (130, 120)	ND (ND, ND)	ND (ND, ND)	0.013 (0.013, 0.013)
						3	0.97 (1.0, 0.94)	6.1 (6.3, 5.9)	< 0.01 (ND, < 0.01)	< 0.01(0.0 09, 0.009)	0.024 (0.027, 0.021)
						7	0.33 (0.24, 0.42)	2.1 (1.5, 2.6)	ND (ND, ND)	< 0.01 (< 0.01, < 0.01)	0.021 (0.016, 0.026)
						10	0.26 (0.20, 0.31)	1.6 (1.3, 1.9)	ND (ND, ND)	< 0.01 (< 0.01, < 0.01)	0.026 (0.023, 0.028)
						14	0.15 (0.12, 0.17)	<u>0.93</u> (0.75, 1.1)	ND (ND, ND)	< 0.01 (ND, < 0.01)	0.021 (0.020, 0.022)
Paris, ON, Canada Trial 07, 2008 (DK-27-07)	1	61	224	150	Forage [83]	14	0.50 (0.51, 0.48)	<u>2.9</u> (3.0, 2.8)	ND (ND, ND)	ND (ND, ND)	0.047 (0.048, 0.046)
Paynesville, MN, USA Trial 08, 2009 (AGO0501 Asgrow)	1	61	214	143	Forage [76]	14	<u>ND</u> (ND, ND)		ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
Geneva, MN, USA Trial 09, 2008 (Pioneer 91M80)	1	61	222	145	Forage [86]	13	0.27 (0.28, 0.26)	<u>2.0</u> (2.0, 1.9)	ND (ND, ND)	ND (ND, ND)	0.047 (0.057, 0.037)

Picoxystrobin

Location, Trial Year (Variety)	Application				Sample [water %]	DAT ^a	Residues (mg/kg) ^b				
	No.	Growth stage	g ai/ha	L/ha			Parent		IN- QDY62	IN- QDY63	IN- QDK50
							FW ^c	DW ^d			
Lenexa, KS, USA Trial 10, 2008 (395NRR)	1	61	221	135	Forage [77]	14	0.43 (0.40, 0.46)	<u>1.9</u> (1.7, 2.0)	ND (ND, ND)	0.015 (0.015, 0.014)	0.054 (0.053, 0.055)
Rochelle, IL, USA Trial 11, 2008 (Pioneer 92M61)	1	61	224	46	Forage [84]	14	0.34 (0.34, 0.33)	<u>2.1</u> (2.1, 2.1)	ND (ND, ND)	< 0.01 (< 0.01, < 0.01)	0.047 (0.047, 0.047)
Britton, SD, USA Trial 12, 2008 (Pioneer 90M80 Roundup Ready)	1	61	224	187	Forage [78]	14	0.13 (0.12, 0.13)	<u>0.57</u> (0.55, 0.59)	ND (ND, ND)	ND (ND, ND)	0.025 (0.025, 0.025)
Springfield, NE, USA Trial 13, 2008 (MW GR3631)	1	61	224	132	Forage [82]	14	0.37 (0.38, 0.35)	<u>2.0</u> (2.1, 1.9)	ND (ND, ND)	< 0.01 (< 0.01, < 0.01)	0.11 (0.10, 0.12)
Carlyle, IL, USA Trial 14, 2008 (NK 37-N4)	1	61	213	148	Forage [81]	14	0.31 (0.35, 0.26)	<u>1.6</u> (1.8, 1.4)	< 0.01 (< 0.01, < 0.01)	0.011 (0.012, 0.010)	0.095 (0.098, 0.091)
LaPlata, MO, USA Trial 16, 2008 (Asgrow AG3802)	1	61	222	163	Forage [79]	14	0.052 (0.060, 0.044)	<u>0.25</u> (0.29, 0.21)	ND (ND, ND)	ND (ND, ND)	0.019 (0.018, 0.020)
Fisk, MO, USA Trial 17, 2009 (54-17 RR/STS)	1	61	220	187	Forage [81]	15	0.16 (0.16, 0.16)	<u>0.84</u> (0.84, 0.84)	< 0.01 (< 0.01, < 0.01)	0.011 (0.011, 0.010)	0.081 (0.079, 0.083)
Dudley, MO, USA Trial 18, 2009 (Jake)	1	61	221	187	Forage [78]	14	0.10 (0.11, 0.093)	<u>0.46</u> (0.50, 0.42)	ND (ND, ND)	< 0.01 (< 0.01, < 0.01)	0.027 (0.027, 0.027)
Tipton, MO, USA Trial 19, 2009 (48-24 Mor Soy)	1	61	220	272	Forage [82]	21	0.11 (0.075, 0.14)	0.60 (0.42, 0.78)	ND (ND, ND)	< 0.01 (< 0.01, < 0.01)	0.064 (0.043, 0.084)
Gardner, KS,	1	60	220	138	Forage	14	0.76	<u>3.5</u>	< 0.01	< 0.01	0.060

Location, Trial no., Year (Variety)	Application				Sample [water %]	DAT ^a	Residues (mg/kg) ^b				
	No.	Growth stage	g ai/ha	L/ha			Parent		IN-QDY62	IN-QDY63	IN-QDK50
							FW ^c	DW ^d			
USA Trial 20, 2009 (Fontanelle 407NRS)					[78]		(0.72, 0.80)	(3.3, 3.6)	(< 0.01, < 0.01)	(< 0.01, < 0.01)	(0.062, 0.058)
Springfield, NE, USA Trial 21, 2009 (NC+2A98)	1	60	213	129	Forage [82]	14	0.29 (0.26, 0.32)	<u>1.6</u> (1.4, 1.8)	< 0.01 (< 0.01, ND)	< 0.01 (< 0.01, < 0.01)	0.069 (0.058, 0.079)

ND = not detected (< 0.003 mg/kg)

^a DAT = Days After Treatment

^b Mean result shown, with individual results for analyses of duplicate samples from the same plot in brackets

^c Fresh weight

^d Dry weight. Residues detected in control samples are indicated with c preceding the reported residue value.

Table 63 Results of residue trials conducted with picoxystrobin (250 g/L SC) in soya bean hay in the USA and Canada in 2008 and 2009 (study number 24861)

Location, Trial no., Year (Variety)	Application				Sample [water %]	DAT ^a	Residues (mg/kg) ^b				
	No.	Growth stage	g ai/ha	L/ha			Parent		IN-QDY62	IN-QDY63	IN-QDK50
							FW ^c	DW ^d			
Blackville, SC, USA Trial 01, 2008 (Asgrow, H7242 RR)	1	63	224	150	Hay [51]	14 + 6	0.25 (0.22, 0.28)	<u>0.51</u> (0.45, 0.57)	0.15 (0.13, 0.16)	0.072 (0.061, 0.083) c0.005	0.015 (0.014, 0.016)
Seven Springs, NC, USA Trial 02, 2008 (DKB-64-51)	1	61	217	140	Hay [39]	14 + 2	0.30 (0.31, 0.29)	<u>0.50</u> (0.51, 0.48)	0.017 (0.013, 0.02)	0.036 (0.035, 0.036)	0.078 (0.075, 0.080)
Cheneyville, LA, USA Trial 03, 2008 (DG 33B52)	1	61	219	149	Hay [23]	14 + 4	0.40 (0.46, 0.33)	<u>0.52</u> (0.60, 0.43)	0.028 (0.028, 0.027)	0.057 (0.054, 0.059)	0.077 (0.077, 0.077)
Fisk, MO, USA Trial 04, 2008 (Armor 47G7)	1	61-65	223	119	Hay [31]	14 + 10	0.85 (0.92, 0.78)	<u>1.2</u> (1.3, 1.1)	0.45 (0.43, 0.47)	0.18 (0.17, 0.19)	0.12 (0.11, 0.12)

Picoxystrobin

Location, Trial no., Year (Variety)	Application				Sample [water %]	DAT a	Residues (mg/kg) ^b				
	No.	Grow th stage	g ai/ha	L/ha			Parent		IN- QDY62	IN- QDY63	IN- QDK50
							FW ^c	DW ^d			
Richland, IA, USA Trial 05, 2008 (93M11)	1	61	213	150	Hay [17]	0 + 5	58 (60, 56)	70 (72, 67)	0.034 (0.033, 0.034)	0.83 (0.87, 0.78)	0.079 (0.077, 0.081)
						3 + 5	23 (21, 24) c0.00 6	27 (25, 29)	0.054 (0.052, 0.056)	0.81 (0.87, 0.75)	0.098 (0.10, 0.096)
						7 + 5	3.1 (2.9, 3.3)	3.8 (3.5, 4.0)	0.026 (0.022, 0.030)	0.10 (0.082, 0.12)	0.12 (0.11, 0.12)
						10 + 3	1.8 (1.8, 1.7)	2.1 (2.2, 2.0)	0.015 (0.015, 0.014)	0.041 (0.040, 0.041)	0.12 (0.12, 0.11)
						14 + 3	0.80 (0.73, 0.87)	0.94 (0.88, 1.0)	0.010 (<u><0.01</u> , 0.010)	0.019 (0.018, 0.019)	0.085 (0.083, 0.086)
Trial 15, 2008 (Pioneer 93M11)	1	61	221	141	Hay [17]	14 + 5	1.3 (1.4, 1.2) c0.00 3	<u>1.6</u> (1.7, 1.4)	0.076 (0.065, 0.087)	0.026 (0.025, 0.026)	0.084 (0.085, 0.082)
Branchton, ON, Canada Trial 06, 2008 (Mirra)	1	61	213	150	Hay [27]	0 + 14	59 (51, 66)	80 (70, 90)	0.086 (0.075, 0.097)	0.47 (0.42, 0.52)	0.048 (0.043, 0.052)
						3 + 11	3.3 (3.6, 2.9) c0.00 7	4.5 (4.9, 4.0)	0.16 (0.10, 0.21)	0.13 (0.14, 0.12) c0.004	0.042 (0.031, 0.052)
						7 + 7	1.4 (1.2, 1.6)	1.9 (1.6, 2.2)	0.024 (0.025, 0.022)	0.040 (0.037, 0.043)	0.039 (0.035, 0.042)
						10 + 14	1.3 (1.4, 1.1)	1.7 (1.9, 1.5)	0.035 (0.037, 0.032)	0.049 (0.056, 0.041) c0.005	0.035 (0.038, 0.031)
						14 + 10	0.54 (0.63, 0.44)	<u>0.73</u> (0.86, 0.60)	0.015 (0.014, 0.016)	0.025 (0.023, 0.027)	0.034 (0.031, 0.036)
Paris, ON, Canada Trial 07, 2008	1	61	224	150	Hay [31]	14 + 17	1.6 (1.6, 1.6)	<u>2.3</u> (2.3, 2.3)	0.16 (0.17, 0.15)	0.12 (0.11, 0.12)	0.053 (0.054, 0.052)

Location, Trial no., Year (Variety)	Application				Sample [water %]	DAT ^a	Residues (mg/kg) ^b				
	No.	Grow th stage	g ai/ha	L/ha			Parent		IN- QDY62	IN- QDY63	IN- QDK50
							FW ^c	DW ^d			
(DK-27-07)											
Paynesville, MN, USA Trial 08, 2009 (AGO0501 Asgrow)	1	61	214	143	Hay [22]	14 + 3	ND (ND, ND)		ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
Geneva, MN, USA Trial 09, 2008 (Pioneer 91M80)	1	61	222	145	Hay [47]	13 + 3	1.1 (1.1, 1.1)	<u>2.1</u> (2.1, 2.1)	0.010 (ND, 0.020)	0.020 (0.020, 0.019)	0.095 (0.097, 0.093)
Lenexa, KS, USA Trial 10, 2008 (395NRR)	1	61	221	135	Hay [24]	14 + 3	1.3 (1.1, 1.5)	<u>1.7</u> (1.4, 2.0)	< 0.01 (< 0.01, < 0.01)	0.048 (0.043, 0.053)	0.084 (0.080, 0.087)
Rochelle, IL, USA Trial 11, 2008 (Pioneer 92M61)	1	61	224	46	Hay [32]	14 + 2	1.1 (1.2, 0.90) c0.00 3	<u>1.6</u> (1.8, 1.3)	0.014 (0.012, 0.015)	0.020 (0.022, 0.018)	0.10 (0.12, 0.088)
Britton, SD, USA Trial 12, 2008 (Pioneer 90M80 Roundup Ready)	1	61	224	187	Hay [28]	14 + 5	0.43 (0.50, 0.35)	<u>0.59</u> (0.69, 0.49)	0.019 (0.021, 0.016)	0.020 (0.023, 0.017)	0.034 (0.032, 0.035)
Springfield, NE, USA Trial 13, 2008 (MW GR3631)	1	61	224	132	Hay [29]	14 + 5	1.3 (1.3, 1.3)	<u>1.8</u> (1.8, 1.8)	0.013 (0.012, 0.014)	0.051 (0.050, 0.052)	0.24 (0.23, 0.24)
Carlyle, IL, USA Trial 14, 2008 (NK 37-N4)	1	61	213	148	Hay [53]	14 + 4	0.80 (0.81, 0.79)	<u>1.7</u> (1.7, 1.7)	0.025 (0.027, 0.023)	0.042 (0.040, 0.043)	0.13 (0.12, 0.13)
LaPlata, MO, USA Trial 16, 2008	1	61	222	163	Hay [17]	14 + 5	0.11 (0.098, , 0.13)	<u>0.14</u> (0.12, 0.16)	0.021 (0.020, 0.022)	0.014 (0.011, 0.016)	0.034 (0.030, 0.038)

Location, Trial no., Year (Variety)	Application				Sample [water %]	DAT ^a	Residues (mg/kg) ^b					
	No.	Grow th stage	g ai/ha	L/ha			Parent		IN- QDY62	IN- QDY63	IN- QDK50	
							FW ^c	DW ^d				
(Asgrow AG3802)												
Fisk, MO, USA Trial 17, 2009 (54-17 RR/STS)	1	61	220	187	Hay [20]	15 + 8	0.66 (0.82, 0.49)	<u>0.81</u> (1.0, 0.61)	0.033 (0.036, 0.030)	0.075 (0.084, 0.066)	0.12 (0.12, 0.11)	
Dudley, MO, USA Trial 18, 2009 (Jake)	1	61	221	187	Hay [20]	14 + 9	0.31 (0.30, 0.32)	<u>0.39</u> (0.38, 0.40)	0.012 (0.013, 0.011)	0.028 (0.027, 0.029)	0.038 (0.040, 0.035)	
Tipton, MO, USA Trial 19, 2009 (48-24 Mor Soy)	1	61	220	272	Hay [46]	21 + 3	0.22 (0.25, 0.19)	0.41 (0.46, 0.35)	< 0.01 (0.008, 0.005)	0.016 (0.018, 0.013)	0.044 (0.049, 0.039)	
Gardner, KS, USA Trial 20, 2009 (Fontanelle 407NRS)	1	60	220	138	Hay [29]	14 + 3	1.9 (1.9, 1.9)	<u>2.7</u> (2.7, 2.7)	0.034 (0.036, 0.031)	0.035 (0.037, 0.032)	0.098 (0.095, 0.10)	
Springfield, NE, USA Trial 21, 2009 (NC+2A98)	1	60	213	129	Hay [44]	14 + 3	1.1 (1.2, 0.98)	<u>2.0</u> (2.1, 1.8)	0.015 (0.016, 0.013)	0.032 (0.034, 0.029)	0.10 (0.11, 0.097)	

ND = not detected (< 0.003 mg/kg)

^a DAT = Days After Treatment. The first number reported is the interval between application and harvest, the second is the field drying interval (between harvest and sampling)

^b Mean result shown, with individual results for analyses of duplicate samples from the same plot in brackets

^c Fresh weight

^d Dry weight. Residues detected in control samples are indicated with c preceding the reported residue value.

Table 64 Results of residue trials conducted with picoxystrobin (250 g/L SC) in pea vines in the USA and Canada in 2008 (study number 24863)

Location Trial no., Year (variety)	Application				Sample [water %]	DAT ^b	Residues (mg/kg) ^c				
	No.	BBCH stage	g ai/ha ^a	L/h a			Parent		IN- QDY62	IN- QDY63	IN- QDK50
							FW ^d	DW ^e			
Parkdale, OR, USA	2	65	229	183	Vines	-0	0.42 (0.48,	3.3 (3.7,	ND (ND,	< 0.01 (< 0.01,	0.13 (0.15,

Location Trial no., Year (variety)	Application				Sample [water %]	DAT ^b	Residues (mg/kg) ^c				
	No.	BBCH stage	g ai/ha ^a	L/h a			Parent		IN- QDY62	IN- QDY63	IN- QDK50
							FW ^d	DW ^e			
Trial 02, 2008 (Green Arrow)		71	226	183	[87]		0.36)	2.8)	ND)	< 0.01)	0.11)
	+0						7.2 (7.2, 7.2)	<u>55</u> (55, 55)	< 0.01 (ND, < 0.01)	< 0.01 (< 0.01, < 0.01)	0.15 (0.16, 0.14)
	3						3.9 (3.6, 4.1)	30 (28, 32)	ND (ND, ND)	0.014 (0.011, 0.016)	0.26 (0.26, 0.26)
	7						0.61 (0.66, 0.56)	4.7 (5.1, 4.3)	ND (ND, ND)	0.011 (0.011, < 0.01)	0.18 (0.18, 0.17)
	10						0.28 (0.29, 0.26)	2.1 (2.2, 2.0)	ND (ND, ND)	< 0.01 (< 0.01, < 0.01)	0.16 (0.17, 0.14)
	14						0.17 (0.18, 0.16)	1.3 (1.4, 1.2)	ND (ND, ND)	ND (ND, ND)	0.13 (0.14, 0.11)
Payette, ID, USA Trial 03, 2008 (Austrian Winter)	2	74	221	187	Vines	0	9.4	<u>35</u>	0.044	0.026	0.34
		79	219	187	[73]		(11, 7.7)	(41, 29)	(0.042, 0.046)	(0.026, 0.025)	(0.32, 0.35)
Jerome, ID, USA Trial 04, 2008 (Pendleton)	2	79	224	186	Vines	0	4.8	<u>19</u>	< 0.01	0.016	0.073
		81	224	183	[75]		(5.2, 4.3)	(21, 17)	(< 0.01, < 0.01)	(0.015, 0.016)	(0.073, 0.072)
Madras, OR, USA Trial 06, 2008 (K2)	2	79	228	191	Vines	0	3.4	<u>14</u>	< 0.01	< 0.01	0.072
		81	221	186	[75]		(4.0, 2.7)	(16, 11)	(< 0.01, < 0.01)	(0.006, 0.004)	(0.076, 0.067)
Ephrata, WA, 2008 Trial 07, 2008 (Kalamo)	2	81–82	225	188	Vines	0	8.0	<u>9.5</u>	0.032	0.033	0.049
		88	223	186	[16]		(8.4, 7.5)	(10, 8.9)	(0.022, 0.042)	(0.034, 0.032)	(0.042, 0.055)
Waldheim, SK, Canada Trial 10, 2008 (Bronco)	2	71–74	219	150	Vines	–0	0.69	4.3	< 0.01	< 0.01	0.087
		74–75	220	150	[84]		(0.64, 0.74)	(4.0, 4.6)	(< 0.01, < 0.01)	(< 0.01, < 0.01)	(0.082, 0.092)
		+0						3.5 (3.7, 3.3)	<u>22</u> (23, 21)	< 0.01 (< 0.01, ND)	< 0.01 (< 0.01, < 0.01)
						3	3.0	19	< 0.01	0.013	0.13

Location Trial no., Year (variety)	Application				Sample [water %]	DAT ^b	Residues (mg/kg) ^c				
	No.	BBCH stage	g ai/ha ^a	L/h a			Parent		IN- QDY62	IN- QDY63	IN- QDK50
							FW ^d	DW ^e			
							(3.0, 3.0)	(19, 19)	(< 0.01, < 0.01)	(0.012, 0.013)	(0.13, 0.13)
						7	2.0 (2.0), 1.9)	13 (13, 12)	0.012 (0.013, < 0.01)	0.016 (0.015, 0.017)	0.14 (0.15, 0.13)
						10	2.0 (1.9, 2.1)	13 (12, 13)	0.011 (< 0.01, 0.011)	0.016 (0.014, 0.017)	0.18 (0.17, 0.19)
						14	1.4 (1.5, 1.3)	8.8 (9.4, 8.1)	< 0.01 (< 0.01, < 0.01)	0.016 (0.015, 0.016)	0.16 (0.15, 0.17)

ND = not detected (< 0.003 mg/kg)

^a Individual application rates shown

^b DAT = Days After Treatment

^c Mean result shown, with individual results for analyses of duplicate samples from the same plot in brackets

^d Fresh weight

^e Dry weight.

Table 65 Results of residue trials conducted with picoxystrobin (250 g/L SC) in pea hay in the USA and Canada in 2008 (study number 24863)

Location Trial no., Year (variety)	Application				Sample [water %]	DAT ^b	Residues (mg/kg) ^c				
	No.	BBCH stage	g ai/ha ^a	L/ha			Parent		IN- QDY62	IN- QDY63	IN- QDK50
							FW ^d	DW ^e			
Parkdale, OR, USA Trial 02, 2008 (Green Arrow)	2	65	229	183	Hay [64]	-0 + 3	0.90	2.5	ND	0.20	0.090
		71	226	183			(0.83, 0.96)	(2.3, 2.7)	(ND, ND)	(0.18, 0.21)	(0.089, 0.091)
						+0 + 3	23 (28, 18) c0.005	<u>6</u> (78, 50)	0.017 (0.016, 0.017)	0.19 (0.20, 0.17)	0.24 (0.27, 0.20)
						3 + 4	7.0 (6.2, 7.8)	20 (17, 22)	0.018 (0.017, 0.019)	0.055 (0.048, 0.062)	0.23 (0.21, 0.24)
						7 + 3	0.77 (0.91, 0.63)	2.2 (2.5, 1.8)	ND (ND, ND)	0.017 (0.022, 0.012)	0.20 (0.21, 0.19)
						10 + 4	1.5 (1.5, 1.5)	4.2 (4.2, 4.2)	0.024 (0.034, 0.013)	0.039 (0.034, 0.043)	0.33 (0.37, 0.28)

Location Trial no., Year (variety)	Application				Sample [water %]	DAT ^b	Residues (mg/kg) ^c				
	No.	BBCH stage	g ai/ha ^a	L/ha			Parent		IN- QDY62	IN- QDY63	IN- QDK50
							FW ^d	DW ^e			
						14 + 4	0.54 (0.58, 0.50)	1.5 (1.6, 1.4)	< 0.01 (ND, < 0.01)	0.021 (0.019, 0.022)	0.25 (0.26, 0.24)
Payette, ID, USA Trial 03, 2008 (Austrian Winter)	2	74 79	221 219	187 187	Hay [17]	0 + 4	12 (13, 10) c0.007	<u>14</u> (16, 12)	0.13 (0.15, 0.11)	0.18 (0.18, 0.17)	0.89 (0.88, 0.89)
Jerome, ID, USA Trial 04, 2008 (Pendleton)	2	79 81	224 224	186 183	Hay [14]	0 + 11	9.2 (11, 7.3)	<u>11</u> (13, 8.5)	0.011 (0.011, 0.011)	0.18 (0.20, 0.15)	0.20 (0.19, 0.21)
Madras, OR, USA Trial 06, 2008 (K2)	2	79 81	228 221	191 186	Hay [19]	0 + 6	3.4 (3.1, 3.6) c0.007	<u>4.1</u> (3.8, 4.4)	0.021 (0.018, 0.024)	0.086 (0.083, 0.088)	0.17 (0.16, 0.17)
Ephrata, WA, 2008 Trial 07, 2008 (Kalamo)	2	81–82 88	225 223	188 186	Hay [11]	0 + 2	6.3 (6.5, 6.1)	<u>7.1</u> (7.3, 6.9)	0.034 (0.026, 0.041)	0.060 (0.062, 0.058)	0.062 (0.066, 0.057)
Waldheim, SK, Canada Trial 10, 2008 (Bronco)	2	71–74 74–75	219 220	150 150	Hay [46]	–0 + 7	1.9 (2.0, 1.8)	3.5 (3.7, 3.3)	0.015 (< 0.01, 0.019)	0.017 (0.017, 0.017)	0.10 (0.098, 0.11)
						+0 + 7	9.3 (9.6, 9.0)	<u>18</u> (18, 17)	0.019 (0.018, 0.019)	0.038 (0.041, 0.035)	0.18 (0.19, 0.16)
						3 + 6	7.7 (7.9, 7.5)	15 (15, 14)	< 0.01 (< 0.01, < 0.01)	0.023 (0.024, 0.021)	0.12 (0.12, 0.11)
						7 + 6	5.0 (5.6), 4.3)	9.0 (10, 8.0)	0.035 (0.028, 0.041)	0.028, 0.020	0.16 (0.16, 0.15)
						10 + 4	4.2 (4.3, 4.1)	7.8 (8.0, 7.6)	0.015 (0.011, 0.018)	0.027 (0.025, 0.028)	0.16 (0.16, 0.15)
						14 + 6	3.6 (3.5,	6.7 (6.5,	0.028 (0.017,	0.048 (0.042,	0.18 (0.17,

Location Trial no., Year (variety)	Application				Sample [water %]	DAT ^b	Residues (mg/kg) ^c				
	No.	BBCH stage	g ai/ha ^a	L/ha			Parent		IN- QDY62	IN- QDY63	IN- QDK50
							FW ^d	DW ^e			
							3.7) c0.003	6.9)	0.038)	0.054)	0.18)

ND = not detected (< 0.003 mg/kg)

^a Individual application rates shown

^b DAT = Days After Treatment. The first number reported is the interval between application and harvest, the second is the field drying interval (between harvest and sampling)

^c Mean result shown, with individual results for analyses of duplicate samples from the same plot in brackets

^d Fresh weight

^e Dry weight. Residues detected in control samples are indicated with c preceding the reported residue value.

Table 66 Results of residue trials conducted with picoxystrobin (250 g/L SC) in wheat forage in the USA and Canada in 2008 and 2009 (study number 24860)

Location Trial no., Year (Variety)	Application				Sample [water %]	DA T ^b	Residues (mg/kg) ^c				
	No.	Growth stage	g ai/ha ^a	L/ha			Parent		IN- QDK50	IN- QDY62	IN- QDY63
							FW ^d	DW ^e			
Seven Springs, NC, USA Trial 01, 2008 (Coker 9478)	1	39	217	135	Forage [75.35]	7	0.93 (0.92, 0.93)	<u>3.8</u> (3.7, 3.8)	0.010 (0.010, < 0.01)	ND (ND, ND)	< 0.01 (< 0.01, < 0.01)
Fisk, MO, USA Trial 02, 2008 (Coker 9663)	1	39	222	187	Forage [79.02]	7	2.3 (2.4, 2.2)	<u>11</u> (11, 10)	0.035 (0.037, 0.032)	ND (ND, ND)	0.016 (0.018, 0.013)
Elm Creek, MB, Canada Trial 03, 2008 (AC Barrie)	1	30-31	231	200	Forage [83.05]	7	0.32 (0.33, 0.31)	<u>1.9</u> (1.9, 1.8)	0.035 (0.033, 0.037)	ND (ND, ND)	< 0.01 (ND, < 0.01)
Richland, IA, USA Trial 04, 2008 (Wilcross 07GV6S- 753)	1	30-31	223	153	Forage [84.14]	7	1.0 (0.91, 1.1)	<u>6.3</u> (5.7, 6.9)	0.031 (0.028, 0.033)	ND (ND, ND)	< 0.01 (< 0.01, < 0.01)
Lenexa, KS, USA Trial 05, 2008	1	30-31	224	144	Forage [80.88]	7	0.68 (0.68, 0.68)	<u>3.6</u> (3.6, 3.6)	0.011 (0.011, < 0.01)	ND (ND, ND)	0.011 (0.011, < 0.01)

Location Trial no., Year (Variety)	Application				Sample [water %]	DA T ^b	Residues (mg/kg) ^c				
	No.	Growth stage	g ai/ha ^a	L/ha			Parent		IN- QDK50	IN- QDY62	IN- QDY63
							FW ^d	DW ^e			
(Overly)											
Hinton, OK, USA Trial 06, 2008 (Jagger)	1	39	222	125	Forage [66.72]	7	1.3 (1.3, 1.3)	<u>3.9</u> (3.9, 3.9)	0.011 (0.011, 0.010)	ND (ND, ND)	< 0.01 (< 0.01, < 0.01)
Carrington, ND, USA Trial 07, 2008 (Kelby)	1	30–31	226	140	Forage [85.87]	-0	ND (ND, ND)		ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
						+0	16 (16, 15)	110 (110, 110)	< 0.01 (< 0.01, < 0.01)	ND (ND, ND)	ND (ND, ND)
						3	2.2 (2.2, 2.1)	16 (16, 15)	0.025 (0.024, 0.026)	ND (ND, ND)	0.013 (0.012, 0.013)
						7	0.65 (0.67, 0.62)	<u>4.6</u> (4.8, 4.4)	0.010 (< 0.01, 0.010)	ND (ND, ND)	ND (ND, ND)
						10	0.29 (0.24 0.33)	2.1 (1.7, 2.4)	< 0.01 (< 0.01, < 0.01)	ND (ND, ND)	ND (ND, ND)
Taber, AB, Canada Trial 08, 2008 (AC Barrie)	1	30	231	154	Forage [77.31]	9	0.36 (0.41, 0.30)	<u>1.6</u> (1.8, 1.3)	0.010 (0.010, 0.010)	ND (ND, ND)	ND (ND, ND)
New Rockford, ND, USA Trial 09, 2008 (Kelby)	1	30–31	221	141	Forage [84.73]	7	0.17 (0.17, 0.16)	<u>1.1</u> (1.1, 1.1)	< 0.01 (< 0.01, < 0.01)	(ND, ND)	ND (ND, ND)
Eldridge, ND, USA Trial 10, 2008 (Glynn)	1	30–31	224	141	Forage [85.63]	7	4.5 (4.4, 4.5)	<u>31</u> (31, 31)	0.030 (0.028, 0.032)	ND (ND, ND)	< 0.01 (< 0.01, < 0.01)
Dundurn, SK, Canada Trial 11, 2008 (Lillian)	1	31	225	200	Forage [77.96]	7	0.38 (0.39, 0.36)	<u>1.7</u> (1.8, 1.6)	0.017 (0.018, 0.016)	ND (ND, ND)	ND (ND, ND)

Location Trial no., Year (Variety)	Application				Sample [water %]	DA T ^b	Residues (mg/kg) ^c				
	No.	Growth stage	g ai/ha ^a	L/ha			Parent		IN- QDK50	IN- QDY62	IN- QDY63
							FW ^d	DW ^e			
Hanley, SK, Canada Trial 12, 2008 (Lillian)	1	31	220	200	Forage [81.64]	7	0.40 (0.40, 0.39)	<u>2.2</u> (2.2, 2.1)	0.013 (0.012, 0.014)	ND (ND, ND)	< 0.01 (< 0.01, ND)
Cordell, OK, USA Trial 13, 2008 (Jagger)	1	51	217	72	Forage [68.61]	6	3.0 (3.4, 2.6)	<u>9.7</u> (11, 8.3)	0.021 (0.024, 0.018)	ND (ND, ND)	0.011 (0.011, < 0.01)
Levelland, TX, USA Trial 14, 2009 (TAM 105)	1	6–8 in.	230	140	Forage [69.57]	8	3.5 (3.6, 3.3)	<u>12</u> (12, 11)	0.029 (0.028, 0.030)	ND (ND, ND)	0.038 (0.037, 0.039)
Olton, TX, USA Trial 15, 2008 (Dumas)	1	37	224	157	Forage [72.99]	7	2.3 (2.1, 2.4)	<u>8.9</u> (7.8, 10)	0.018 (0.016, 0.019)	ND (ND, ND)	0.046 (0.045, 0.046)
Larned, KS, USA Trial 16, 2008 (Jagger)	1	30–31	224	168	Forage [79.58]	7	2.3 (2.3, 2.2)	<u>11</u> (11, 11)	0.017 (0.017, 0.017)	ND (ND, ND)	0.011 (0.011, 0.011)
Ephrata, WA, USA Trial 17, 2008 (Dark northern spring)	1	30–31	225	187	Forage [79.10]	7	0.48 (0.49, 0.46)	<u>2.3</u> (2.3, 2.2)	0.010 (0.010, 0.010)	ND (ND, ND)	0.017 (0.017, 0.017)
Minto, MB, Canada Trial 18, 2008 (Superb)	1	31–32	224	158	Forage [85.25]	–0	ND (ND, ND)		ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
						+0	17 (17, 17)	120 (120, 120)	< 0.01 (< 0.01, < 0.01)	ND (ND, ND)	ND (ND, ND)
						3	2.6 (2.2, 3.0)	18 (15, 20)	0.029 (0.028, 0.029)	ND (ND, ND)	< 0.01 (< 0.01, < 0.01)
						7	0.67 (0.61,	<u>4.5</u> (4.1,	0.015 (0.014,	ND (ND,	ND (ND,

Location Trial no., Year (Variety)	Application				Sample [water %]	DA T ^b	Residues (mg/kg) ^c				
	No.	Growth stage	g ai/ha ^a	L/ha			Parent		IN- QDK50	IN- QDY62	IN- QDY63
							FW ^d	DW ^e			
							0.73)	4.9)	0.015)	ND)	ND)
Boissevain, MB, Canada Trial 19, 2008 (Strongfield (durum))	1	31–32	229	164	Forage [81.20]	7	1.4 (1.4, 1.4)	<u>7.4</u> (7.4, 7.4)	0.016 (0.014, 0.017)	ND (ND, ND)	0.011 (< 0.01, 0.011)
Rosthern, SK, Canada Trial 20, 2008 (AC Lillian)	1	31	227	203	Forage [85.66]	7	0.51 (0.50, 0.52)	<u>3.6</u> (3.5, 3.6)	0.012 (0.010, 0.013)	ND (ND, ND)	ND (ND, ND)
Hepburn, SK, Canada Trial 21, 2008 (AC Lillian)	1	31	223	199	Forage [82.45]	7	0.65 (0.64, 0.66)	<u>3.7</u> (3.6, 3.8)	0.013 (0.013, 0.012)	ND (ND, ND)	ND (ND, ND)
Fort Saskatchew an, AB, Canada Trial 22, 2008 (AC Foremost)	1	31	222	180	Forage [81.53]	7	1.3 (1.3, 1.3)	<u>7.0</u> (7.0, 7.0)	0.013 (0.013, 0.012)	ND (ND, ND)	0.012 (0.012, 0.012)
Trial 23, 2008 (AC Foremost)	1	31	222	180	Forage [79.94]	8	0.70 (0.70, 0.70)	3.5 (3.5, 3.5)	0.012 (0.012, 0.011)	ND (ND, ND)	0.010 (0.010, 0.010)
Alvena, SK, Canada Trial 24, 2008 (Lillian)	1	31	223	200	Forage [77.11]	7	1.5 (1.3, 1.6)	<u>6.4</u> (5.7, 7.0)	0.023 (0.023, 0.023)	ND (ND, ND)	< 0.01 (< 0.01, < 0.01)
Waldheim, SK, Canada Trial 25, 2008 (Lillian)	1	31	223	200	Forage [78.22]	7	1.0 (1.1, 0.99) c0.005	<u>4.8</u> (5.1, 4.5)	0.020 (0.021, 0.018)	ND (ND, ND)	< 0.01 (< 0.01, < 0.01)
Northwood, ND, USA Trial 46, 2008 (Kelby)	1	30–31	214	184	Forage [81.31]	9	0.23 (1.2), 0.26 (1.4)	<u>1.3</u> (1.2, 1.4)	< 0.01 (< 0.01, < 0.01)	ND (ND, ND)	ND (ND, ND)

ND = not detected (< 0.003 mg/kg)

^a Individual application rates shown

^b DAT = Days After Treatment

^c Mean result shown, with individual results for analyses of duplicate samples from the same plot in brackets

^d Fresh weight

^e Dry weight. Residues detected in control samples are indicated with c preceding the reported residue value.

Table 67 Results of residue trials conducted with picoxystrobin (250 g/L SC) in wheat hay in the USA and Canada in 2008 and 2009 (study number 24860)

Location Trial no., Year (Variety)	Application				Sample [water %]	DAT ^b	Residues (mg/kg) ^c				
	No.	Growth stage	g ai/ha ^a	L/ha			Parent		IN-QDK50	IN-QDY62	IN-QDY63
							FW ^d	DW ^e			
Seven Springs, NC, USA Trial 01, 2008 (Coker 9478)	3	39 57–58 69–71	217 231 220	135 208 195	Hay [38.77]	14 + 2	0.37 (0.40, 0.34)	<u>0.61</u> (0.65, 0.56)	0.054 (0.053, 0.054)	0.033 (0.035, 0.030)	0.012 (0.012, 0.011)
Fisk, MO, USA Trial 02, 2008 (Coker 9663)	3	39 45–47 69	222 223 222	187 187 187	Hay [13.65]	14 + 8	0.70 (0.69, 0.70)	<u>0.81</u> (0.80, 0.81)	0.081 (0.082, 0.080)	0.016 (0.015, 0.018)	0.013 (0.013, 0.013)
Elm Creek, MB, Canada Trial 03, 2008 (AC Barrie)	3	30–31 32 55	231 230 224	200 200 200	Hay [41.91]	14 + 2	0.52 (0.55, 0.49)	<u>0.90</u> (0.95, 0.84)	0.075 (0.083, 0.067)	0.078 (0.090, 0.065)	0.023 (0.024, 0.021) c0.008
Richland, IA, USA Trial 04, 2008 (Wilcross 07GV6S-753)	3	30–31 59 65–69	223 213 224	153 178 184	Hay [22.60]	14 + 6	0.39 (0.42, 0.35)	<u>0.51</u> (0.55, 0.46)	0.082 (0.075, 0.088)	0.13 (0.15, 0.11)	0.085 (0.091, 0.078)
Lenexa, KS, USA Trial 05, 2008 (Overly)	3	30–31 32–37 59	224 225 224	144 145 144	Hay [32.13]	14 + 4	0.28 (0.28, 0.28)	<u>0.41</u> (0.41, 0.41)	0.083 (0.079, 0.086) c0.079	0.026 (0.023, 0.028)	0.011 (0.011, 0.011)
Hinton, OK, USA Trial 06, 2008 (Jagger)	3	39 61 75	222 220 231	125 133 139	Hay [31.89]	15 + 1	0.46 (0.35, 0.57)	<u>0.68</u> (0.51, 0.84)	0.040 (0.041, 0.038)	< 0.01 (< 0.01, < 0.01)	0.011 (< 0.01, 0.012)
Carrington, ND, USA Trial 07, 2008 (Kelby)	3	30–31 45 71	226 228 224	140 140 139	Hay [41.45]	-0 + 7	1.0 (1.1, 0.99)	1.8 (1.9, 1.7)	0.027 (0.027, 0.026)	< 0.01 (< 0.01, ND)	0.022 (0.022, 0.022)

Location Trial no., Year (Variety)	Application				Sample [water %]	DAT ^b	Residues (mg/kg) ^c				
	No.	Growth stage	g ai/ha ^a	L/ha			Parent		IN- QDK50	IN- QDY62	IN- QDY63
							FW ^d	DW ^e			
						+0	12 (12, 11)	20 (21, 19)	0.039 (0.041, 0.037)	< 0.01 (< 0.01, ND)	0.039 (0.038, 0.039) c0.004
						3 + 8	6.6 (6.0, 7.2)	11 (10, 12)	0.036 (0.038, 0.034)	< 0.01 (< 0.01, < 0.01)	0.019 (0.020, 0.018)
						7 + 4	5.4 (6.4, 4.3)	9.2 (11, 7.4)	0.050 (0.052, 0.048)	0.012 (0.012, 0.012)	0.028 (0.030, 0.025)
						14 + 4	0.98 (0.86, 1.1)	<u>1.7</u> (1.5, 1.9)	0.11 (0.11, 0.10)	0.025 (0.028, 0.021)	0.016 (0.016, 0.016)
Taber, AB, Canada Trial 08, 2008 (AC Barrie)	3	30 61 71–73	231 230 216	154 154 146	Hay [43.94]	14 + 1	2.2 (2.3, 2.1)	<u>4.0</u> (4.1, 3.8)	0.015 (0.057, 0.057)	0.015 (0.015, 0.015)	0.016 (0.013, 0.018)
New Rockford, ND, USA Trial 09, 2008 (Kelby)	3 (7, 14)	30–31 32 65	221 216 217	141 140 140	Hay [31.23]	14 + 6	0.76 (0.76, 0.75)	<u>1.1</u> (1.1, 1.1)	0.046 (0.047, 0.044)	0.024 (0.024, 0.023)	0.022 (0.022, 0.021)
Eldridge, ND, USA Trial 10, 2008 (Glynn)	3	30–31 37 59	224 224 224	141 182 172	Hay [27.99]	16 + 5	0.14 (0.14, 0.14)	<u>0.19</u> (0.19, 0.19)	0.23 (0.23, 0.23)	0.046 (0.043, 0.049)	0.013 (0.012, 0.013)
Dundurn, SK, Canada Trial 11, 2008 (Lillian)	3	31 52–59 69–73	225 222 222	200 200 200	Hay [12.43]	14 + 13	2.1 (2.2, 1.9)	<u>2.4</u> (2.5, 2.2)	0.082 (0.077, 0.087)	0.013 (0.012, 0.013)	0.049 (0.045, 0.052)
Hanley, SK, Canada Trial 12, 2008 (Lillian)	3	31 51–55 65–69	220 223 224 <u>667</u>	200 200 200	Hay [14.13]	14 + 13	1.6 (1.5, 1.6)	<u>1.8</u> (1.7, 1.9)	0.13 (0.11, 0.14)	0.061 (0.042, 0.079)	0.062 (0.050, 0.074)
Cordell, OK, USA Trial 13, 2008 (Jagger)	3	51 65 83	217 223 222	72 70 82	Hay [9.29]	17 + 0	2.5 (2.8, 2.2)	<u>2.8</u> (3.1, 2.4)	0.13 (0.13, 0.12)	0.052 (0.055, 0.049)	0.085 (0.092, 0.078)

Location Trial no., Year (Variety)	Application				Sample [water %]	DAT ^b	Residues (mg/kg) ^c				
	No.	Growth stage	g ai/ha ^a	L/ha			Parent		IN- QDK50	IN- QDY62	IN- QDY63
							FW ^d	DW ^e			
Levelland, TX, USA Trial 14, 2009 (TAM 105)	3	6–8 in. 10 in. 51–59	230 228 226	140 140 140	Hay [18.21]	16 + 6	2.8 (3.1, 2.4)	<u>3.4</u> (3.8, 2.9)	0.20 (0.15, 0.25)	0.097 (0.043, 0.15)	0.080 (0.087, 0.073) c0.014
Olton, TX, USA Trial 15, 2008 (Dumas)	3	37 43–51 65–69	224 223 230	157 157 157	Hay [28.39]	14 + 3	0.76 (0.81, 0.70)	<u>1.0</u> (1.1, 0.98)	0.073 (0.074, 0.071)	ND (ND, ND)	0.061 (0.063, 0.058) c0.012
Larned, KS, USA Trial 16, 2008 (Jagger)	3	30–31 37 61	224 213 224 <u>661</u>	168 168 168	Hay [37.82]	14 + 1	0.30 (0.29, 0.30)	<u>0.48</u> (0.47, 0.48)	0.051 (0.051, 0.050)	0.029 (0.025, 0.032)	0.011 (0.010, 0.012) c0.005
Ephrata, WA, USA Trial 17, 2008 (Dark northern spring)	3	30–31 47–49 57–58	225 226 224	187 189 187	Hay [14.13]	14 + 12	0.21 (0.21, 0.21)	<u>0.24</u> (0.24, 0.24)	0.063 (0.063, 0.063)	ND (ND, ND)	0.015 (0.014, 0.015) c0.005
Minto, MB, Canada Trial 18, 2008 (Superb)	3	31–32 37–41 57–59	224 226 224	158 162 160	Hay [21.85]	-0 + 10	3.8 (3.6, 3.9)	4.8 (4.6, 5.0)	0.047 (0.042, 0.051)	0.033 (0.033, 0.032)	0.042 (0.043, 0.040)
						+0 + 10	27 (27, 26) c0.003	34 (35, 33)	0.075 (0.077, 0.073)	0.14 (0.13, 0.15)	0.065 (0.068, 0.062) c0.004
						3 + 7	15 (15, 14)	19 (19, 18)	0.073 (0.075, 0.071)	0.090 (0.094, 0.086)	0.036 (0.039, 0.033)
						7 + 9	3.5 (3.3, 3.7) c0.003	4.5 (4.2, 4.7)	0.044 (0.045, 0.043)	0.029 (0.032, 0.026)	0.022 (0.027, 0.017) c0.005
						14 + 9	2.0 (1.9, 2.0)	<u>2.5</u> (2.4, 2.6)	0.067 (0.065, 0.068)	0.023 (0.026, 0.020)	0.021 (0.021, 0.020)
Boissevain, MB, Canada Trial 19,	3	31–32 34–37 41–55	229 228 224	164 163 159	Hay [31.16]	14 + 7	0.96 (0.91, 1.0)	<u>1.4</u> (1.3, 1.5)	0.049 (0.045, 0.052)	0.019 (0.015, 0.022)	ND (ND, ND)

Location Trial no., Year (Variety)	Application				Sample [water %]	DAT ^b	Residues (mg/kg) ^c				
	No.	Growth stage	g ai/ha ^a	L/ha			Parent		IN- QDK50	IN- QDY62	IN- QDY63
							FW ^d	DW ^e			
2008 (Strongfield (durum))											
Rosthern, SK, Canada Trial 20, 2008 (AC Lillian)	3	31 37–39 59–69	227 224 226	203 199 201	Hay [19.30]	14 + 12	0.86 (0.81, 0.91) c0.004	<u>1.1</u> (1.0, 1.1)	0.16 (0.12, 0.19)	0.092 (0.10, 0.083)	0.019 (0.021, 0.016)
Hepburn, SK, Canada Trial 21, 2008 (AC Lillian)	3	31 37–41 59–69	223 224 229	199 199 203	Hay [19.31]	14 + 11	0.58 (0.67, 0.49)	<u>0.72</u> (0.83, 0.61)	0.078 (0.083, 0.073)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)
Fort Saskatchewan , AB, Canada Trial 22, 2008 (AC Foremost)	3	31 45–54 69	222 224 224	180 180 180	Hay [17.22]	14 + 20	0.64 (0.65, 0.63)	<u>0.78</u> (0.79, 0.76)	0.12 (0.11, 0.12)	0.19 (0.19, 0.19)	0.033 (0.033, 0.032)
Trial 23, 2008 (AC Foremost)	3	31 45–52 69	222 224 224	180 180 180	Hay [17.77]	14 + 20	0.43 (0.50, 0.36)	0.53 (0.61, 0.44)	0.048 (0.050, 0.045)	0.13 (0.14, 0.12)	0.027 (0.028, 0.026)
Alvena, SK, Canada Trial 24, 2008 (Lillian)	3	31 56–59 69–71	223 223 225	200 200 200	Hay [11.32]	14 + 13	1.3 (1.4, 1.2)	<u>1.5</u> (1.6, 1.4)	0.091 (0.094, 0.087)	0.16 (0.14, 0.17)	0.036 (0.036, 0.036)
Waldheim, SK, Canada Trial 25, 2008 (Lillian)	3	31 55–59 69–71	223 222 224	200 200 200	Hay [13.96]	14 + 13	3.1 (2.7, 3.4)	<u>3.6</u> (3.1, 4.0)	0.079 (0.064, 0.093)	0.058 (0.045, 0.070)	0.042 (0.032, 0.051)
Northwood, ND, USA Trial 46, 2008 (Kelby)	3	30–31 49 71	214 219 217	184 188 187	Hay [23.68]	14 + 7	0.14 (0.15, 0.12)	<u>0.18</u> (0.20, 0.16)	0.058 (0.064, 0.051)	0.14 (0.14, 0.13)	0.022 (0.022, 0.022)

ND = not detected (< 0.003 mg/kg)

^a Individual application rates shown

^b DAT = Days After Treatment. The first number reported is the interval between application and harvest, the second is the field drying interval (between harvest and sampling)

^c Mean result shown, with individual results for analyses of duplicate samples from the same plot in brackets

^d Fresh weight^e Dry weight. Residues detected in control samples are indicated with c preceding the reported residue value.

Table 68 Results of residue trials conducted with picoxystrobin (250 g/L SC) in wheat straw in the USA and Canada in 2008 and 2009 (study number 24860)

Location Trial no., Year (Variety)	Application				Sample [water %]	DA T ^b	Residues (mg/kg) ^c				
	No.	Growth stage	g ai/ha a	L/ha			Parent		IN- QDK50	IN- QDY62	IN- QDY63
							FW ^d	DW ^e			
Seven Springs, NC, USA Trial 01, 2008 (Coker 9478)	3	39 57–58 69–71	217 231 220	135 208 195	Straw [11.84]	47	0.087 (0.093, 0.081)	<u>0.10</u> (0.11, 0.092)	0.033 (0.035, 0.031)	0.093 (0.090, 0.095)	0.031 (0.033, 0.029)
Fisk, MO, USA Trial 02, 2008 (Coker 9663)	3	39 45–47 69	222 223 222	187 187 187	Straw [12.73]	35	0.25 (0.26, 0.24)	<u>0.29</u> (0.30, 0.28)	0.18 (0.17, 0.18)	0.16 (0.17, 0.14)	0.040 (0.041, 0.038)
Elm Creek, MB, Canada Trial 03, 2008 (AC Barrie)	3	30–31 32 55	231 230 224	200 200 200	Straw [38.10]	47	0.021 (0.026, 0.015)	<u>0.033</u> (0.04 2, 0.024)	0.055 (0.062, 0.047)	0.032 (0.039, 0.024)	< 0.01 (< 0.01, < 0.01)
Richland, IA, USA Trial 04, 2008 (Wilcross 07GV6S-753)	3	30–31 59 65–69	223 213 224	153 178 184	Straw [20.42]	45	0.018 (0.015, 0.020)	<u>0.022</u> (0.01 9, 0.025)	0.023 (0.034, 0.012)	0.066 (0.066, 0.066)	0.012 (0.011, 0.012)
Lenexa, KS, USA Trial 05, 2008 (Overly)	3	30–31 32–37 59	224 225 224	144 145 144	Straw [21.40]	45	0.013 (0.013, 0.013)	<u>0.016</u> (0.01 6, 0.016)	0.056 (0.056, 0.055)	0.062 (0.063, 0.061)	0.026 (0.026, 0.025)
Hinton, OK, USA Trial 06, 2008 (Jagger)	3	39 61 75	222 220 231	125 133 139	Straw [11.17]	45	0.29 (0.27, 0.30)	<u>0.32</u> (0.30, 0.34)	0.032 (0.031, 0.033)	0.12 (0.13, 0.11)	0.028 (0.027, 0.029)
Carrington, ND, USA Trial 07, 2008 (Kelby)	3	30–31 45 71	226 228 224	140 140 139	Straw [13.40]	45	1.5 (1.6, 1.4)	<u>1.7</u> (1.8, 1.6)	0.052 (0.052, 0.052)	0.21 (0.20, 0.22)	0.049 (0.050, 0.047)
Taber, AB, Canada Trial 08, 2008 (AC Barrie)	3	30 61 71–73	231 230 216	154 154 146	Straw [25.60]	45	0.46 (0.57, 0.34)	<u>0.62</u> (0.77, 0.46)	0.039 (0.041, 0.037)	0.042 (0.060, 0.024)	0.014 (0.019, 0.009)
New Rockford, ND, USA	3 (7, 14)	30–31 32	221 216	141 140	Straw [19.96]	46	0.12 (0.12, 0.12)	<u>0.15</u> (0.15, 0.15)	0.012 (0.013, 0.011)	0.10 (0.11, 0.099)	0.020 (0.020, 0.019)

Location Trial no., Year (Variety)	Application				Sample [water %]	DA T ^b	Residues (mg/kg) ^c				
	No.	Growth stage	g ai/ha a	L/ha			Parent		IN- QDK50	IN- QDY62	IN- QDY63
							FW ^d	DW ^e			
Trial 09, 2008 (Kelby)		65	217	140							
Eldridge, ND, USA Trial 10, 2008 (Glynn)	3	30–31 37 59	224 224 224	141 182 172	Straw [26.23]	45	0.017 0.012, 0.021)	<u>0.022</u> (0.01 6, 0.028)	0.15 (0.15, 0.15)	0.031 (0.027, 0.035)	0.013 (0.012, 0.013)
Dundurn, SK, Canada Trial 11, 2008 (Lillian)	3	31 52–59 69–73	225 222 222	200 200 200	Straw [13.19]	45	0.42 (0.47, 0.37)	<u>0.49</u> (0.54, 0.43)	0.083 (0.078, 0.087)	0.032 (0.034, 0.029)	0.023 (0.026, 0.019)
Hanley, SK, Canada Trial 12, 2008 (Lillian)	3	31 51–55 65–69	220 223 224	200 200 200	Straw [15.58]	45	0.42 (0.31, 0.52)	<u>0.50</u> (0.37, 0.62)	0.071 (0.084, 0.058)	0.049 (0.038, 0.060)	0.025 (0.020, 0.029)
Cordell, OK, USA Trial 13, 2008 (Jagger)	3	51 65 83	217 223 222	72 70 82	Straw [9.75]	40	1.1 (1.0, 1.1)	<u>1.2</u> (1.1, 1.2)	0.091 (0.083, 0.099)	0.12 (0.12, 0.11)	0.069 (0.065, 0.072)
Levelland, TX, USA Trial 14, 2009 (TAM 105)	3	6–8 in. 10 in. 51–59	230 228 226	140 140 140	Straw [9.80]	45	1.0 (0.95, 1.1)	<u>1.2</u> (1.1, 1.2)	0.13 (0.15, 0.11)	0.082 (0.080, 0.083)	0.069 (0.065, 0.072) c0.003
Olton, TX, USA Trial 15, 2008 (Dumas)	3	37 43–51 65–69	224 223 230	157 157 157	Straw [25.24]	45	0.21 (0.26, 0.15)	<u>0.28</u> (0.35, 0.20)	0.18 (0.19, 0.17)	0.048 (0.056, 0.039)	0.032 (0.037, 0.027)
Larned, KS, USA Trial 16, 2008 (Jagger)	3	30–31 37 61	224 213 224	168 168 168	Straw [9.28]	44	0.072 (0.070, 0.073)	<u>0.079</u> (0.07 7, 0.080)	0.15 (0.15, 0.15)	0.12 (0.12, 0.11)	0.034 (0.034, 0.033)
Ephrata, WA, USA Trial 17, 2008 (Dark northern spring)	3	30–31 47–49 57–58	225 226 224	187 189 187	Straw [34.50]	47	0.019 (0.020, 0.018)	<u>0.029</u> (0.03 1, 0.027)	0.64 (0.59, 0.69)	0.010 (0.009, 0.011)	< 0.01 (< 0.01, ND)
Minto, MB, Canada Trial 18, 2008 (Superb)	3	31–32 37–41 57–59	224 226 224	158 162 160	Straw [20.71]	51	< 0.01 (ND, < 0.01)	<u>≤ 0.0 1</u> (ND, < 0.0 1)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
Boissevain, MB, Canada	3	31–32	229	164	Straw	58	0.012 (0.011,	0.017 (0.01	0.037 (0.034,	< 0.01 (0.006,	ND (ND,

Location Trial no., Year (Variety)	Application				Sample [water %]	DA T ^b	Residues (mg/kg) ^c				
	No.	Growth stage	g ai/ha ^a	L/ha			Parent		IN- QDK50	IN- QDY62	IN- QDY63
							FW ^d	DW ^e			
Trial 19, 2008 (Strongfield durum)		34–37 41–55	228 224	163 159	[32.07]		0.012)	6, 0.018)	0.039)	0.008)	ND)
Rosthern, SK, Canada Trial 20, 2008 (AC Lillian)	3	31 37–39 59–69	227 224 226	203 199 201	Straw [28.22]	56	0.080 (0.077, 0.082 c0.003	<u>0.11</u> (0.11, 0.11)	0.039 (0.040, 0.037)	0.025 (0.025, 0.025)	0.016 (0.016, 0.016) c0.007
Hepburn, SK, Canada Trial 21, 2008 (AC Lillian)	3	31 37–41 59–69	223 224 229	199 199 203	Straw [32.27]	54	0.068 (0.062, 0.073)	<u>0.10</u> (0.09 2, 0.11)	0.044 (0.046, 0.041)	0.010 (< 0.01, 0.010)	< 0.01 (< 0.01, < 0.01)
Fort Saskatchewan , AB, Canada Trial 22, 2008 (AC Foremost)	3	31 45–54 69	222 224 224	180 180 180	Straw [14.79]	45	0.23 (0.26, 0.19)	0.25 (0.30, 20)	0.11 (0.11, 0.10)	0.12 (0.15, 0.092)	0.031 (0.039, 0.023)
Trial 23, 2008 (AC Foremost)	3	31 45–52 69	222 224 224 <u>670</u>	180 180 180	Straw [15.49]	45	0.30 (0.34, 0.26)	<u>0.36</u> (0.40, 0.31)	0.075 (0.085, 0.065)	0.089 (0.10, 0.078)	0.038 (0.046, 0.029)
Alvena, SK, Canada Trial 24, 2008 (Lillian)	3	31 56–59 69–71	223 223 225	200 200 200	Straw [28.42]	45	0.37 (0.39, 0.35)	<u>0.52</u> (0.54, 0.49)	0.079 (0.076, 0.081)	0.048 (0.054, 0.042)	0.020 (0.020, 0.019)
Waldheim, SK, Canada Trial 25, 2008 (Lillian)	3	31 55–59 69–71	223 222 224	200 200 200	Straw [20.81]	45	0.67 (0.85, 0.48)	<u>0.86</u> (1.1, 0.61)	0.052 (0.049, 0.055)	0.043 (0.053, 0.032)	0.023 (0.028, 0.018)
Northwood, ND, USA Trial 46, 2008 (Kelby)	3	30–31 49 71	214 219 217	184 188 187	Straw [14.11]	45	0.037 (0.037, 0.037)	<u>0.043</u> (0.04 3, 0.043)	0.043 (0.045, 0.041)	0.074 (0.075, 0.072)	0.023 (0.023, 0.023)

ND = not detected (< 0.003 mg/kg). ^a Individual application rates shown. ^b DAT = Days After Treatment. ^c Mean result shown, with individual results for analyses of duplicate samples from the same plot in brackets. ^d Fresh weight. ^e Dry weight. Residues detected in control samples are indicated with c preceding the reported residue value.

Table 69 Results of residue trials conducted with picoxystrobin (250 g/L SC) in barley hay in the USA and Canada in 2008 and 2009 (study number 24860)

Location Trial no., Year (Variety)	Application				Sample [water %]	DAT ^b	Residues (mg/kg) ^c				
	No.	Growth stage	g ai/ha ^a	L/ha			Parent		IN- QDK50	IN- QDY62	IN- QDY63
							FW ^d	DW ^e			
Germansville, PA, USA Trial 26, 2008 (NP)	3	30–31 39 51	233 230 231	291 288 289	Hay [22.40]	14 + 3	0.61 (0.61, 0.60)	<u>0.78</u> (0.79, 0.77)	0.23 (0.21, 0.24)	0.28 (0.26, 0.29)	0.077 (0.073, 0.080)
Richland, IA, USA Trial 27, 2008 (Robust)	3	30–31 32 59	222 228 219	139 170 159	Hay [36.56]	14 + 2	0.21 (0.22, 0.20)	<u>0.34</u> (0.35, 0.32)	0.13 (0.14, 0.12)	0.031 (0.031, 0.030)	0.011 (0.011, 0.011)
Delavan, WI, USA Trial 28, 2008 (Kewaunee)	3	30–31 32 55	225 223 224	164 154 161	Hay [37.82]	14 + 4	0.20 (0.23, 0.16)	<u>0.32</u> (0.37, 0.26)	0.13 (0.16, 0.098)	0.011 (0.014, 0.008)	< 0.01 (0.006, 0.005)
Frederick, SD, USA Trial 29, 2008 (Robust)	3	30–31 37 65–71	224 224 224	94 94 94	Hay [32.59]	14 + 3	1.2 (1.2, 1.1)	<u>1.7</u> (1.8, 1.6)	0.19 (0.19, 0.18)	0.073 (0.072, 0.073)	0.045 (0.047, 0.042)
Carrington, ND, USA Trial 30, 2008 (Tradition)	3	30–31 32 65	221 216 217	139 141 140	Hay [33.60]	14 + 3	1.6 (1.6, 1.5)	<u>2.4</u> (2.4, 2.3)	0.062 (0.065, 0.058)	0.010 (0.012, 0.007)	0.016 (0.018, 0.013)
Eldridge, ND, USA Trial 31, 2008 (Tradition)	3	30–31 37 59	222 224 221	140 140 140	Hay [18.68]	16 + 5	0.16 (0.16, 0.16)	<u>0.20</u> (0.20, 0.20)	0.15 (0.16, 0.14)	0.017 (0.019, 0.014)	< 0.01 (0.005, 0.004)
Velva, ND, USA Trial 32, 2008 (Legacy)	3	30–31 32 47–49	223 224 229	138 139 141	Hay [37.47]	14 + 2	1.0 (1.1, 0.92)	<u>1.7</u> (1.8, 1.5)	0.082 (0.079, 0.084)	< 0.01 (0.004, 0.004)	0.012 (0.013, 0.011)
Jerome, ID, USA Trial 33, 2008 (Harrington)	3	32 39 71	224 224 230	143 164 161	Hay [13.03]	14 + 9	0.33 (0.31, 0.35)	<u>0.38</u> (0.35, 0.40)	0.074 (0.070, 0.077)	ND (ND, ND)	0.017 (0.016, 0.018)
Live Oak, CA, USA Trial 34,	3	37–39 49	225 224	188 187	Hay [33.47]	14 + 5	3.7 (3.0,	<u>5.5</u> (4.5,	0.12 (0.10,	< 0.01 (< 0.01,	0.071 (0.060,

Location Trial no., Year (Variety)	Application				Sample [water %]	DAT ^b	Residues (mg/kg) ^c				
	No.	Growth stage	g ai/ha ^a	L/ha			Parent		IN- QDK50	IN- QDY62	IN- QDY63
							FW ^d	DW ^e			
2008 (UC-937)		59	225	186			4.3)	6.5)	0.13)	< 0.01)	0.082)
Madras, OR, USA Trial 35, 2008 (Bellford)	3	32 53 83-85	234 233 222	199 192 190	Hay [14.78]	14 + 6	0.75 (0.61, 0.88)	<u>0.86</u> (0.72, 1.0)	0.044 (0.044, 0.044)	0.034 (0.033, 0.035)	0.070 (0.062, 0.078)
Minto, MB, Canada Trial 36, 2008 (Conion)	3	31-32 33-37 49-58	220 229 231	157 163 206	Hay [27.72]	11 + 7	0.90 (0.91, 0.88)	<u>1.3</u> (1.3, 1.2)	0.12 (0.11, 0.12)	0.013 (0.011, 0.014)	0.013 (0.013, 0.013)
Boissevain, MB, Canada Trial 37, 2008 (Copelan)	3	31-33 33-37 43-54	224 222 225	160 159 201	Hay [8.19]	14 + 10	2.1 (2.0, 2.2) c0.00 8	<u>2.3</u> (2.2, 2.4)	0.20 (0.21, 0.18)	0.10 (0.10, 0.10)	0.065 (0.064, 0.066)
Rosthern, SK, Canada Trial 38, 2008 (AC Metcalf)	3	31 37 59	230 221 225	205 197 201	Hay [15.31]	14 + 11	0.56 (0.58, 0.53)	<u>0.66</u> (0.68, 0.63)	0.15 (0.15, 0.14)	0.025 (0.026, 0.023)	0.012 (0.012, 0.012)
Hepburn, SK, Canada Trial 39, 2008 (AC Metcalf)	3	31 39 59	226 220 222	200 196 198	Hay [16.33]	14 + 18	0.33 (0.31, 0.34)	<u>0.39</u> (0.37, 0.41)	0.079 (0.075, 0.082)	< 0.01 (< 0.01, < 0.01)	0.012 (0.011, 0.012)
Innisfail, AB, Canada Trial 40, 2008 (Metcalf)	3	33-36 39-47 55-59	224 215 224	250 250 250	Hay [32.72]	9 + 6	1.8 (1.7, 1.8)	2.6 (2.5, 2.7)	0.069 (0.063, 0.075)	0.039 (0.040, 0.037)	0.033 (0.033, 0.032)
Fort Saskatchew an, AB, Canada Trial 41, 2008 (Bold)	3	31 45-52 60-61	228 222 224	180 180 180	Hay [15.95]	14 + 26	0.46 (0.41, 0.51)	<u>0.55</u> (0.49, 0.61)	0.13 (0.10, 0.15)	0.24 (0.21, 0.26)	0.053 (0.049, 0.056)
Trial 42, 2008 (Bold)	3	31 55-59 59-60	224 220 235	178 180 180	Hay [13.79]	14 + 26	0.28 (0.28, 0.27)	0.32 (0.32, 0.31)	0.058 (0.056, 0.060)	0.19 (0.18, 0.20)	0.031 (0.031, 0.031)
Lamont,	3	31	222	180	Hay	13 +	0.37	<u>0.46</u>	0.11	0.17	0.050

Location Trial no., Year (Variety)	Application				Sample [water %]	DAT ^b	Residues (mg/kg) ^c				
	No.	Growth stage	g ai/ha ^a	L/ha			Parent		IN- QDK50	IN- QDY62	IN- QDY63
							FW ^d	DW ^e			
AB, Canada Trial 43, 2008 (Bold)		47–51 72	223 223	180 180	[20.71]	31	(0.39, 0.34)	(0.49, 0.43)	(0.12, 0.092)	(0.18, 0.16)	(0.051, 0.049)
Alvena, SK, Canada Trial 44, 2008 (Legacy)	3	31 56–59 69–75	223 223 223	200 200 200	Hay [12.79]	14 + 13	1.2 (0.90, 1.5)	<u>1.4</u> (1.0, 1.7)	0.22 (0.17, 0.26)	0.33 (0.19, 0.47)	0.064 (0.035, 0.092)
Waldheim, SK, Canada Trial 45, 2008 (Legacy)	3	31 55–59 71–73	223 222 217	200 200 200	Hay [13.12]	14 + 13	3.1 (2.8, 3.3)	<u>3.5</u> (3.2, 3.8)	0.10 (0.091, 0.11)	0.10 (0.055, 0.14)	0.076 (0.072, 0.080)
Northwood, ND, USA Trial 47, 2008 (Tradition)	3	30–31 32 59	221 216 221	190 186 188	Hay [27.76]	14 + 5	0.63 (0.68, 0.57)	<u>0.77</u> (0.94, 0.79)	0.15 (0.15, 0.14)	0.015 (0.014, 0.015)	< 0.01 (< 0.01, < 0.01)

ND = not detected (< 0.003 mg/kg)

^a Individual application rates shown

^b DAT = Days After Treatment. The first number reported is the interval between application and harvest, the second is the field drying interval (between harvest and sampling)

^c Mean result shown, with individual results for analyses of duplicate samples from the same plot in brackets

^d Fresh weight

^e Dry weight. Residues detected in control samples are indicated with c preceding the reported residue value.

Table 70 Results of residue trials conducted with picoxystrobin (250 g/L SC) in barley straw in the USA and Canada in 2008 and 2009 (study number 24860)

Location Trial no., Year (Variety)	Application				Sample [water %]	DAT ^b	Residues (mg/kg) ^c				
	No.	Growth stage	g ai/ha ^a	L/ha			Parent		IN- QDK50	IN- QDY62	IN- QDY63
							FW ^d	DW ^e			
Germansville, PA, USA Trial 26, 2008 (NP)	3	30–31 39 51	233 230 231	291 288 289	Straw [18.97]	45	0.18 (0.19, 0.16)	<u>0.22</u> (0.23, 0.20)	0.098 (0.11, 0.085)	0.080 (0.077, 0.083)	0.082 (0.080, 0.083)
Richland, IA, USA Trial 27, 2008 (Robust)	3	30–31 32 59	222 228 219	139 170 159	Straw [28.72]	45	0.035 (0.041, 0.028)	<u>0.049</u> (0.058, 0.039)	0.060 (0.063, 0.056)	0.031 (0.031, 0.030)	0.011 (0.012, 0.009)

Location Trial no., Year (Variety)	Application				Sample [water %]	DAT ^b	Residues (mg/kg) ^c				
	No.	Growth stage	g ai/ha ^a	L/ha			Parent		IN- QDK50	IN- QDY62	IN- QDY63
							FW ^d	DW ^e			
Delavan, WI, USA Trial 28, 2008 (Kewaunee)	3	30-31 32 55	225 223 224	164 154 161	Straw [35.28]	46	0.033 (0.024, 0.041)	<u>0.050</u> (0.037, 0.063)	0.16 (0.14, 0.17)	0.021 (0.017, 0.025)	0.011 (0.009, 0.013)
Frederick, SD, USA Trial 29, 2008 (Robust)	3	30-31 37 65-71	224 224 224	94 94 94	Straw [15.25]	45	0.11 (0.10, 0.11)	<u>0.13</u> (0.12, 0.13)	0.058 (0.051, 0.065)	0.038 (0.028, 0.048)	0.014 (0.008, 0.019)
Carrington, ND, USA Trial 30, 2008 (Tradition)	3	30-31 32 65	221 216 217	139 141 140	Straw [18.90]	45	0.34 (0.36, 0.31)	<u>0.41</u> (0.44, 0.38)	0.041 (0.040, 0.041)	0.054 (0.059, 0.048)	0.037 (0.039, 0.034)
Eldridge, ND, USA Trial 31, 2008 (Tradition)	3	30-31 37 59	222 224 221	140 140 140	Straw [60.81]	45	0.032 (0.031, 0.033)	<u>0.082</u> (0.079, 0.084)	0.095 (0.079, 0.11)	0.014 (0.012, 0.015)	< 0.01 (0.005, 0.005)
Velva, ND, USA Trial 32, 2008 (Legacy)	3	30-31 32 47-49	223 224 229	138 139 141	Straw [17.27]	45	0.33 (0.31, 0.35)	<u>0.40</u> (0.37, 0.42)	0.061 (0.051, 0.070)	0.025 (0.022, 0.028)	0.012 (0.011, 0.012)
Jerome, ID, USA Trial 33, 2008 (Harrington)	3	32 39 71	224 224 230	143 164 161	Straw [11.52]	45	0.059 (0.064, 0.054)	<u>0.066</u> (0.072, 0.061)	0.18 (0.17, 0.19)	0.023 (0.024, 0.022)	0.018 (0.019, 0.017)
Live Oak, CA, USA Trial 34, 2008 (UC-937)	3	37-39 49 59	225 224 225	188 187 186	Straw [20.80]	77	0.13 (0.12, 0.14)	0.17 (0.15, 0.18)	0.068 (0.067, 0.068)	0.027 (0.026, 0.027)	0.060 (0.053, 0.066)
Madras, OR, USA Trial 35, 2008 (Bellford)	3	32 53 83-85	234 233 222	199 192 190	Straw [13.81]	47	0.69 (0.68, 0.70) c0.082	<u>0.80</u> (0.79, 0.81)	0.025 (0.026, 0.024) c0.004	0.014 (0.015, 0.013)	0.059 (0.058, 0.060) c0.009
Minto, MB, Canada Trial 36, 2008 (Conion)	3	31-32 33-37 49-58	220 229 231	157 163 206	Straw [60.48]	47	0.027 (0.026, 0.028)	<u>0.069</u> (0.066, 0.071)	0.046 (0.044, 0.048)	0.027 (0.025, 0.029)	0.013 (0.012, 0.014)

Location Trial no., Year (Variety)	Application				Sample [water %]	DAT ^b	Residues (mg/kg) ^c				
	No.	Growth stage	g ai/ha ^a	L/ha			Parent		IN- QDK50	IN- QDY62	IN- QDY63
							FW ^d	DW ^e			
Boissevain, MB, Canada Trial 37, 2008 (Copelan)	3	31–33 33–37 43–54	224 222 225	160 159 201	Straw [34.58]	57	0.050 (0.059, 0.040)	0.076 (0.090, 0.061)	0.062 (0.068, 0.056)	0.018 (0.020, 0.016)	< 0.01 (< 0.01, < 0.01)
Rosthern, SK, Canada Trial 38, 2008 (AC Metcalf)	3	31 37 59	230 221 225	205 197 201	Straw [30.30]	53	0.16 (0.16, 0.15) c0.005	<u>0.23</u> (0.23, 0.22)	0.060 (0.058, 0.061)	0.033 (0.031, 0.035)	0.016 (0.014, 0.017)
Hepburn, SK, Canada Trial 39, 2008 (AC Metcalf)	3	31 39 59	226 220 222	200 196 198	Straw [25.63]	47	0.18 (0.18, 0.18)	<u>0.24</u> (0.24, 0.24)	0.096 (0.097, 0.094)	0.031 (0.030, 0.031)	0.011 (0.011, 0.011)
Innisfail, AB, Canada Trial 40, 2008 (Metcalf)	3	33–36 39–47 55–59	224 215 224	250 250 250	Straw [17.32]	58	0.20 (0.21, 0.18)	0.25 (0.25, 0.25)	0.034 (0.035, 0.033)	0.089 (0.094, 0.083)	0.032 (0.032, 0.031)
Fort Saskatchewan , AB, Canada Trial 41, 2008 (Bold)	3	31 45–52 60–61	228 222 224	180 180 180	Straw [35.80]	45	0.15 (0.16, 0.14)	0.24 (0.25, 0.22)	0.064 (0.067, 0.060)	0.063 (0.065, 0.060)	0.024 (0.027, 0.020)
Trial 42, 2008 (Bold)	3	31 55–59 59–60	224 220 235	178 180 180	Straw [32.60]	45	0.19 (0.16, 0.21)	<u>0.28</u> (0.24, 0.31)	0.071 (0.074, 0.067)	0.056 (0.051, 0.061)	0.030 (0.027, 0.032)
Lamont, AB, Canada Trial 43, 2008 (Bold)	3	31 47–51 72	222 223 223	180 180 180	Straw [27.06]	45	0.26 (0.27, 0.24)	<u>0.35</u> (0.37, 0.33)	0.13 (0.13, 0.12)	0.066 (0.066, 0.066)	0.029 (0.030, 0.027)
Alvena, SK, Canada Trial 44, 2008 (Legacy)	3	31 56–59 69–75	223 223 223	200 200 200	Straw [45.67]	45	0.18 (0.18, ND) c0.27	0.33 (0.33, ND)	0.010 (0.010, ND)	0.037 (0.037, ND)	0.014 (0.014, ND)
Waldheim, SK, Canada Trial 45, 2008	3	31 55–59 71–73	223 222 217	200 200 200	Straw [36.28]	45	0.74 (0.88, 0.60)	<u>1.2</u> (1.4, 0.94)	0.057 (0.059, 0.055)	0.073 (0.083, 0.063)	0.048 (0.051, 0.044)

Location Trial no., Year (Variety)	Application				Sample [water %]	DAT ^b	Residues (mg/kg) ^c				
	No.	Growth stage	g ai/ha ^a	L/ha			Parent		IN-QDK50	IN-QDY62	IN-QDY63
							FW ^d	DW ^e			
(Legacy)											
Northwood, ND, USA Trial 47, 2008 (Tradition)	3	30-31 32 59	221 216 221	190 186 188	Straw [24.39]	44	0.066 (0.072, 0.060)	<u>0.087</u> (0.095, 0.079)	0.081 (0.091, 0.070)	0.018 (0.019, 0.016)	< 0.01 (< 0.01, < 0.01)

ND = not detected (< 0.003 mg/kg)

^a Individual application rates shown

^b DAT = Days After Treatment

^c Mean result shown, with individual results for analyses of duplicate samples from the same plot in brackets

^d Fresh weight

^e Dry weight. Residues detected in control samples are indicated with c preceding the reported residue value.

Table 71 Results of residue trials conducted with picoxystrobin (250 g/L SC) in maize forage in the USA and Canada in 2008 (study number 24864)

Location Trial no., Year (Variety)	Application				Sample [water %]	DAT ^b	Residues (mg/kg) ^c				
	No.	Growth stage	g ai/ha ^a	L/ha			Parent		IN-QDY62	IN-QDY63	IN-QDK50
							FW ^d	DW ^e			
Germansville, PA, USA Trial 01, 2008 (TA 3892)	3	Early R 89 89	226 226 223	330 433 428	Forage [48]	0	6.4 (6.2, 6.6)	<u>13</u> (12, 13)	0.076 (0.052, 0.099)	0.041 (0.041, 0.041)	0.040 (0.037, 0.043)
Blackville, SC, USA Trial 02, 2008 (OK 69-72)	3	65 89 89	224 224 224	186 181 185	Forage [33]	0	2.4 (2.7, 2.0) c0.003	<u>3.5</u> (4.0, 3.0)	0.25 (0.31, 0.18)	0.030 (0.033, 0.026)	< 0.01 (< 0.01, < 0.01)
Paris, ON, Canada Trial 03, 2008 (DeKalb 50-20)	3	R1 R5 R5-R6	215 228 217	200 200 200	Forage [45]	0	4.7 (5.0, 4.3) c0.004	<u>8.5</u> (9.1, 7.8)	0.13 (0.13, 0.12)	0.015 (0.016, 0.014)	0.010 (0.011, < 0.01)
Branchton, ON, Canada Trial 04, 2008 (Pioneer 38A59)	3	R1 R5 R5-R6	213 213 213	200 200 200	Forage [46]	-0 +0	0.020 (0.017, 0.023) 2.5 (2.4, 2.6)	0.037 (0.031, 0.043) <u>4.6</u> (4.4, 4.8)	< 0.01 (< 0.01, ND) < 0.01 (< 0.01, < 0.01)	ND (ND, ND) ND (ND, ND)	ND (ND, ND) ND (ND, ND)

Location Trial no., Year (Variety)	Application				Sample [water %]	DAT b	Residues (mg/kg) ^c				
	No.	Growth stage	g ai/ha ^a	L/ha			Parent		IN- QDY62	IN- QDY63	IN- QDK50
							FW ^d	DW ^e			
						1	1.1 (1.0, 1.1)	2.0 (1.9, 2.0)	0.024 (0.022, 0.025)	ND (ND, ND)	ND (ND, ND)
						3	0.84 (0.84, 0.83)	1.6 (1.6, 1.5)	0.035 (0.040, 0.029)	< 0.01 (< 0.01, < 0.01)	ND (ND, ND)
						6	0.83 (0.77, 0.88)	1.5 (1.4, 1.6)	0.054 (0.044, 0.064)	< 0.01 (< 0.01, < 0.01)	ND (ND, ND)
Richland, IA, USA Trial 05, 2008 (Middle Koop 5513)	3	R1 R6 R6	213 224 224	167 162 165	Forage [38]	0	3.1 (3.4, 2.7) c0.005	<u>5.0</u> (5.5, 4.4)	0.072 (0.076, 0.067)	0.020 (0.022, 0.017)	< 0.01 (< 0.01, < 0.01)
Wyoming, IL, USA Trial 06, 2008 (DKC60-18)	3	R1 R6 R6	224 224 224	193 188 186	Forage [38]	-0	0.016 (0.019, 0.013)	0.026 (0.031, 0.021)	0.011 (0.011, 0.010)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)
						+0	3.9 (4.6, 3.1)	<u>6.2</u> (7.4, 5.0)	0.019 (0.021, 0.016)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, ND)
						1	3.3 (3.6, 3.0)	5.3 (5.8, 4.8)	0.032 (0.030, 0.034)	< 0.01 (< 0.01, < 0.01)	< 0.01 (< 0.01, < 0.01)
						3	3.3 (3.1, 3.5)	5.3 (5.0, 5.6)	0.081 (0.065, 0.097)	0.012 (0.010, 0.013)	< 0.01 (< 0.01, < 0.01)
						7	3.3 (3.9, 2.7)	4.7 (5.5, 3.8)	0.096 (0.082, 0.11)	0.030 (0.028, 0.031)	0.016 (0.022, 0.01)
Paynesville, MN, USA Trial 08, 2009 (DKC35)	3	R1 R6 R6	215 217 215	143 142 143	Forage [41]	0	8.1 (6.1, 10)	<u>14</u> (10, 17)	ND (ND, ND)	ND (ND, ND)	< 0.01 (< 0.01, < 0.01)
Gardner, ND, USA Trial 09, 2008 (2K145)	3	R4 R5 R6	223 221 223	159 159 159	Forage [66]	0	2.7 (3.4, 2.0)	<u>8.0</u> (10, 6.0)	0.053 (0.061, 0.045)	< 0.01 (< 0.01, < 0.01)	ND (ND, ND)
Lenexa, KS, USA Trial 10,	3	R1 87	220 221	134 135	Forage [57]	0	4.8 (4.5,	<u>9.7</u> (10, 9.4)	0.24 (0.23,	0.093 (0.096,	0.035 (0.033,

Picoxystrobin

Location Trial no., Year (Variety)	Application				Sample [water %]	DAT b	Residues (mg/kg) ^c				
	No.	Growth stage	g ai/ha ^a	L/ha			Parent		IN- QDY62	IN- QDY63	IN- QDK50
							FW ^d	DW ^e			
2008 (08HYBBIO 8REM)		87	220	137			5.0)		0.25)	0.089)	0.037)
Delavan, WI, USA Trial 11, 2008 (DKC51-39)	3	R1 R5.5 R5.75	220 221 219	196 199 201	Forage [47]	0	3.0 (3.7, 2.3) c0.005	<u>5.7</u> (7.0, 4.3)	0.12 (0.14, 0.092)	0.028 (0.035, 0.021)	0.011 (0.011, < 0.01)
Springfield, NE, USA Trial 12, 2008 (NK N38- 04)	3	R1 87 89	224 224 220	130 132 132	Forage [50]	0	3.4 (2.9, 3.8)	<u>6.7</u> (5.8, 7.6)	0.036 (0.031, 0.041)	0.012 (0.011, 0.012)	0.023 (0.022, 0.023)
Tipton, MO, USA Trial 13, 2008 (DeKalb DKC6423)	3	R1 R5 R5	224 224 224	262 256 259	Forage [63]	0	2.6 (2.7, 2.5) c0.004	<u>7.1</u> (7.3, 6.8)	0.043 (0.049, 0.037)	0.010 (0.010, < 0.01)	0.025 (0.022, 0.028)
Carlyle, IL, USA Trial 14, 2008 (Burrus 616 XLR)	3	R1 R6 R6	225 222 216	150 162 172	Forage [50]	0	5.4 (4.8, 6.0)	<u>11</u> (9.6, 12)	0.12 (0.093, 0.14)	0.023 (0.02, 0.027)	0.036 (0.035, 0.036)
La Plata, MO, USA Trial 15, 2009 (LG 2540)	3	R1 R6 R6	221 221 223	159 195 191	Forage [52]	0	5.7 (6.1, 5.3)	<u>12</u> (13, 11)	0.20 (0.18, 0.21)	0.033 (0.034, 0.032)	0.012 (0.013, < 0.01)
Hinton, OK, USA Trial 16, 2009 (DKC51-45)	3	75 87 89	222 224 219	178 189 190	Forage [52]	0	3.0 (2.7, 3.3)	<u>6.3</u> (5.6, 6.9)	0.021 (0.020, 0.022)	< 0.01 (< 0.01, < 0.01)	0.014 (< 0.01, 0.018)

ND = not detected (< 0.003 mg/kg)

^a Individual application rates shown

^b DAT = Days After Treatment

^c Mean result shown, with individual results for analyses of duplicate samples from the same plot in brackets

^d Fresh weight

^e Dry weight. Residues detected in control samples are indicated with c preceding the reported residue value.

Table 72 Results of residue trials conducted with picoxystrobin (250 g/L SC) in maize stover in the USA and Canada in 2008 (study number 24864)

Location Trial no., Year (Variety)	Application				Sample [water %]	DAT ^b	Residues (mg/kg) ^c					
	No.	Growth stage	g ai/ha ^a	L/ha			Parent		IN-QDY62	IN-QDY63	IN-QDK50	
							FW ^d	DW ^e				
Germansville, PA, USA Trial 01, 2008 (TA 3892)	3	Early R` 89 89	226	330	Stover [70]	7	1.0	<u>3.5</u>	0.21	0.024	0.065	
			226	433			(1.1, 0.97)	(3.7, 3.2)	(0.21, 0.20)	(0.024, 0.024)	(0.066, 0.064)	
			223	428								
Blackville, SC, USA Trial 02, 2008 (OK 69-72)	3	65 89 89	224	186	Stover [43]	7	1.2	<u>2.1</u>	1.1	0.31	0.011	
			224	181			(1.1, 1.3)	(1.9, 2.3)	(0.96, 1.2)	(0.31, 0.30)	(0.012, 0.01)	
			224	185								
Paris, ON, Canada Trial 03, 2008 (DeKalb 50-20)	3	R1 R5 R5-R6	215	200	Stover [48]	7	4.5	<u>8.6</u>	0.72	0.17	0.035	
			228	200			(4.6, 4.3)	(8.8, 8.3)	(0.73, 0.70)	(0.17, 0.16)	(0.033, 0.036)	
			217	200								
Branchton, ON, Canada Trial 04, 2008 (Pioneer 38A59)	3	R1 R5 R5-R6	213	200	Stover [62]	+0	6.4	17	0.19	0.035	0.035	
			213	200			(8.3, 4.4)	(22, 12)	(0.24, 0.14)	(0.044, 0.025)	(0.047, 0.022)	
			213	200								
							1	6.9	18	0.11	0.036	0.047
								(5.2, 8.5)	(14, 22)	(0.079, 0.15)	(0.026, 0.045)	(0.038, 0.055)
		3	1.7	4.4	0.079	0.013	0.016					
			(1.6, 1.7)	(4.2, 4.5)	(0.077, 0.081)	(0.014, 0.011)	(0.017, 0.014)					
		7	3.1	<u>8.2</u>	0.20	0.051	0.025					
			(3.1, 3.1)	(8.2, 8.2)	(0.21, 0.19)	(0.052, 0.05)	(0.024, 0.026)					
Richland, IA, USA Trial 05, 2008 (Middle Koop 5513)	3	R1 R6 R6	213	167	Stover [41]	6	1.9	<u>3.2</u>	0.24	0.057	0.023	
			224	162			(1.5, 2.3)	(2.5, 3.9)	(0.22, 0.26)	(0.046, 0.068)	(0.018, 0.027)	
			224	165			c0.00 4					
Wyoming, IL, USA Trial 06, 2008 (DKC60-18)	3	R1 R6 R6	224	193	Stover [29]	+0	8.5	11	0.13	0.060	0.032	
			224	188			(11, 6.0)	(14, 7.8)	(0.18, 0.073)	(0.076, 0.043)	(0.045, 0.019)	
			224	186			c0.00 5					
		1	10	13	0.15	0.098	0.035					

Location Trial no., Year (Variety)	Application				Sample [water %]	DAT ^b	Residues (mg/kg) ^c				
	No.	Growth stage	g ai/ha ^a	L/h a			Parent		IN- QDY62	IN- QDY63	IN- QDK50
							FW ^d	DW ^e			
							(9.6, 11)	(12, 14)	(0.16, 0.13)	(0.11, 0.085)	(0.03, 0.04)
						3	2.9 (2.9, 2.9)	3.8 (3.8, 3.8)	0.74 (0.71, 0.77)	0.090 (0.082, 0.098)	0.021 (0.023, 0.018)
						7	6.6 (6.1, 7.0)	<u>8.5</u> (7.9, 9.1)	2.0 (2.1, 1.8)	0.40 (0.43, 0.37)	0.032 (0.03, 0.033)
Paynesville, MN, USA Trial 08, 2009 (DKC35)	3	R1 R6 R6	215 217 215	143 142 143	Stover [47]	7	0.012 (0.00 9,0.0 15)	<u>0.023</u> (0.01 7, 0.028)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
Gardner, ND, USA Trial 09, 2008 (2K145)	3	R4 R5 R6	223 221 223	159 159 159	Stover [74]	7	0.57 (0.57, 0.57) c0.00 3	<u>2.2</u> (2.2, 2.2)	0.053 (0.055, 0.051) c0.003	0.012 (0.012, 0.012)	0.019 (0.016, 0.021)
Lenexa, KS, USA Trial 10, 2008 (08HYBBIO 8REM)	3	R1 87 87	220 221 220	134 135 137	Stover [62]	7	2.2 (2.0, 2.3)	<u>5.7</u> (5.3, 6.1)	0.46 (0.46, 0.46)	0.20 (0.19, 0.20)	0.028 (0.023, 0.033)
Delavan, WI, USA Trial 11, 2008 (DKC51-39)	3	R1 R5.5 R5.75	220 221 219	196 199 201	Stover [58]	7	2.5 (2.4, 2.6)	<u>6.0</u> (5.7, 6.2)	0.34 (0.34, 0.33)	0.17 (0.16, 0.18)	0.023 (0.022, 0.024)
Springfield, NE, USA Trial 12, 2008 (NK N38- 04)	3	R1 87 89	224 224 220	130 132 132	Stover [66]	7	1.3 (1.2, 1.3) c0.00 4	<u>3.8</u> (3.6, 3.9)	0.16 (0.16, 0.16)	0.039 (0.039, 0.038)	0.039 (0.037, 0.04)
Tipton, MO, USA Trial 13, 2008 (DeKalb DKC6423)	3	R1 R5 R5	224 224 224	262 256 259	Stover [70]	7	0.29 (0.31, 0.26)	<u>0.94</u> (1.0, 0.87)	0.038 (0.042, 0.034)	0.011 (0.011, < 0.01)	0.063 (0.072, 0.054)
Carlyle, IL, USA Trial 14, 2008 (Burrus 616)	3	R1 R6 R6	225 222 216	150 162 172	Stover [66]	7	0.32 (0.35, 0.29)	<u>1.0</u> (1.1, 0.88)	0.11 (0.094, 0.12)	0.027 (0.028, 0.025)	0.034 (0.041, 0.026)

Location Trial no., Year (Variety)	Application				Sample [water %]	DAT ^b	Residues (mg/kg) ^c				
	No.	Growth stage	g ai/ha ^a	L/h a			Parent		IN- QDY62	IN- QDY63	IN- QDK50
							FW ^d	DW ^e			
XLR)											
La Plata, MO, USA Trial 15, 2009 (LG 2540)	3	R1 R6 R6	221 221 223	159 195 191	Stover [56]	7	3.3 (3.5, 3.0)	<u>7.4</u> (8.0, 6.8)	1.6 (1.7, 1.5)	0.35 (0.36, 0.34)	0.029 (0.029, 0.029)
Hinton, OK, USA Trial 16, 2009 (DKC51-45)	3	75 87 89	222 224 219	178 189 190	Stover [65]	7	2.3 (2.6, 2.0)	<u>6.6</u> (7.4, 5.7)	0.083 (0.093, 0.072)	0.060 (0.069, 0.051)	0.044 (0.056, 0.032)

ND = not detected (< 0.003 mg/kg). ^a Individual application rates shown. ^b DAT = Days After Treatment. ^c Mean result shown, with individual results for analyses of duplicate samples from the same plot in brackets. ^d Fresh weight. ^e Dry weight. Residues detected in control samples are indicated with c preceding the reported residue value.

FATES OF RESIDUES IN PROCESSING

A high temperature processing study on the nature of residues of picoxystrobin in processes such as baking, boiling, brewing and pasteurisation was not provided to the 2012 JMPR.

Processing studies for picoxystrobin in barley, wheat, maize, soya bean and rape seed were provided.

Barley

A malting barley processing study based on a single field trial was conducted in Germany (Jones and Hill, 1998). Three treatment plots were established. Three applications were made at 750 g ai/ha at 9–18 day intervals. A large sample of grain was collected from each treated plot and from an untreated control area for processing, together, 31 days after the last application.

The barley grain was processed into beer using simulated commercial procedures. The grain was first cleaned by mechanical processes and screening. The cleaned grain was alternately wet- and dry-steeped (5 hours wet, 19 hours dry, 4 hours wet, 20 hours dry, 2 hours wet and 1 hour dry) for a total steeping time of 51 hours at a temperature of 12–15 °C. The steeped grain was germinated for 4 days at a mean temperature of 8.6–8.7 °C, with the germinating grain being turned once or twice daily. After germination, the grain was kiln dried over 30 hours using a stepped temperature program (50–85 °C) to obtain a moisture content < 10%. The malt was cleaned and germs were discarded. The cleaned malt was then brewed into a Pilsener style beer commencing with milling of the malt. The milled malt was mashed with soft water in a heated tun over 2 hours 20 minutes using a stepped temperature program from 35 to 77 °C. The resulting wort was separated from the spent grain, which was washed and the washings added to the wort. Hops were added to the wort, which was boiled for 90 minutes at atmospheric pressure. Trub (lees) was separated by centrifuging, the boiled wort was cooled and oxygen added until saturation was achieved. Yeast was added, and the wort was fermented over 7 days in a pilot scale (approx. 100L) fermentation vessel, and then transferred into casks for maturation at a temperature of 2.5 °C over 5 weeks. The spent yeast precipitated and was removed, with the rack beer being filtered to yield finished beer. Samples of grain before processing, malt, spent grain, trub (lees), young beer (after fermentation only), wort, spent yeast and finished beer were collected and frozen for analysis.

The maximum interval from barley grain harvest until extraction of the last samples was 9 months. Unprocessed grain for analysis shipped directly from the field to the laboratory was frozen for the entire period between sampling and analysis, while processing samples of grain were stored at ambient temperatures for about 2 months until processing was started. However, the picoxystrobin content of the grain sample collected at the processing facility is very similar to that for the grain shipped directly to the laboratory, indicating that ambient storage has not adversely affected the sample. All processed fraction samples were frozen after collection until analysis. Sample extracts were analysed within 8 days. Stability of picoxystrobin and metabolite residues in a variety of matrices including maize grain, dry peas, soya bean meal and potato has been verified over 24 months (Schierhoff, 2012). This covers the storage time for the study samples, and the samples are unlikely to have been adversely affected by storage.

Samples were analysed for parent compound only using GC/MS (method number RAM 288/01). This method was re-validated concurrently with the sample analysis giving acceptable recoveries (70–120%) and precision (RSD < 20%) in barley grain and processed fractions.

All untreated control samples contained picoxystrobin residues below the LOQ. Residues of picoxystrobin in barley grain and processed commodity samples are tabulated below.

Table 773 Residues of picoxystrobin and metabolites in German barley grain and processed fractions (Jones and Hill, 1998)

Location, Trial number, Year (Variety)	Application				Sample	PHI (days)	Picoxystrobin residues (mg/kg)	Processing factors
	No.	BBCH stage	g ai/ha	L/ha				
Nandlstadt- Wadensdorf, Bavaria, D-85405, Germany, RS-9708-G1, 1997, (Scarlett)	3	45	750	200	Grain	31	0.19	–
		55	750	200	Grain for processing	31	0.21	–
		77	750	200	Malt	–	0.10	0.48
					Spent grain	–	0.17	0.81
					Trub (lees)	–	0.01	0.05
					Young beer	–	< 0.01	< 0.05
					Spent yeast	–	0.03	0.14
					Beer	–	< 0.01	< 0.05
					Wort	–	< 0.01	< 0.05

Residues of picoxystrobin did not concentrate in any of the barley process fractions (malt, spent grain and yeast, lees, young beer, wort or finished beer). It is likely that significant portions of the residue would have been in the bran, germ or steeping water from the malting step, neither of which were sampled.

A further barley processing study involving two field trials was conducted in Scotland (Mason, 2000). Two plots were treated with three application of picoxystrobin at each site, at 11–24 day intervals. One plot received three applications at 250 g ai/ha, with the second receiving the first two at 250 g ai/ha, while the final application was at 500 g ai/ha. Barley grain was harvested at normal maturity 7–10 weeks after the last application.

Untreated control grain, both treated samples from the East Saulton site, and the higher rate treated grain from the Pathead site were processed into a lager style beer on a pilot scale (100 L) using simulated commercial processes. The barley grain was dried, cleaned, and malted by steeping (8 hours wet, 14 hours dry, 6 hours wet, 12 hours dry, 1 hour wet and 2 hours dry) at 15–17 °C, followed by germination over 4 days at a temperature rising from 15 to 18 °C. The malted barley was then kiln dried over approximately 24 hours at 40–85 °C or 50–95 °C, and then mashed at 64 °C for 60

minutes. Hops were added to the mash, which was boiled for 90 minutes, then fermented at a temperature of approximately 12 °C for 6 days. The young beer was matured (3 days at 13 °C, 1–2 days at 3 °C and a minimum of 7 days at 0 °C). Finally, the racked beer was filtered, bottled and pasteurised (15 minutes at 60 °C).

The barley was stored at ambient temperatures for 6–12 months between harvest and processing, while all process fractions and the unprocessed grain were extracted and analysed within 3 months of sampling/harvest. Process fractions (with the exception of beer which was chilled) were frozen between collection and analysis.

Samples were analysed using a GC/MS method (method number RAM 288/01).

Table 74 Residues of picoxystrobin and metabolites in Scottish barley grain and processed fractions (Mason, 2000)

Location, Trial number, Year (Variety)	Application				Sample	PHI (days)	Picoxystrobin residues (mg/kg)	Processing factors
	No.	BBCH stage	g ai/ha	L/ha				
East Saulton, East Lothian, Scotland, 392343/T1, 1998, (Prisma)	3	33	250	220	Grain	52	0.02	–
		59	250	220	Beer	–	< 0.01	< 0.5
		69	250	220				
	3	33	250	220	Grain	52	0.04	–
		59	250	220	Beer	–	< 0.01	< 0.25
		69	500	220				
Pathead, East Lothian, Scotland, 392343/T2, 1998, (Chariot)	3	32	250	220	Grain	70	0.02	–
		57	250	220	Malt	–	< 0.01	< 0.5
		65	500	220	Spent grain	–	0.01	0.5
					Spent yeast	–	< 0.01	< 0.5
					Beer	–	< 0.01	< 0.5

The barley processing study showed that residues of picoxystrobin did not concentrate in any of the barley process fractions tested (malt, spent grain and yeast, or beer). This is consistent with the earlier study conducted in Germany.

Wheat

A processing study in wheat was conducted in the USA and Canada (Rice, 2010). Field trials were established at two sites (one in Iowa and one in North Dakota). Plots were treated with three applications of picoxystrobin at a target individual application rate of 1120 g ai/ha, at a target interval of 14 days. Samples of treated and control grain were collected 45 days after the final application, frozen and shipped to a processing facility.

At the processing facility, the moisture content of the grain was tested and adjusted by drying if necessary at 54–71 °C to a moisture content of 11.0–13.5%, then cleaned by aspiration and screening. The cleaned wheat grain was tempered to a moisture content of 16% and milled in a disc mill and sifted (8-, 14- and 34-mesh sieves), and the material left on the 34-mesh sieve was aspirated to generate the germ fraction, which was reduction milled and sifted to separate germ and endosperm, with the germ being aspirated and further milled and sifted.

Further wheat tempered and milled (broken) and sifted to yield break flour (material passing through the 140 micron screen), middlings (material passing through the 800 micron screen) and coarse bran (material too large to pass through the 800 micron screen). A sample of middlings was collected, and the remainder reduction milled (once or twice as required) and sifted (160 micron) to

yield reduction flour (which was combined with the break flour to give the flour sample), and shorts. The bran from the breaking process was sifted (128 micron) and the material from the bran passing through the sieve added to the shorts sample, with the remainder retained on the sieve forming the bran fraction. Samples of processed fractions (germ, flour, bran, middlings and shorts) and unprocessed grain were frozen and shipped to the analytical laboratory. Untreated control samples were processed before the treated samples to minimise the risk of contamination.

The maximum interval for frozen storage from harvest until extraction for analysis (including processing time) was 12 months. Sample extracts were analysed within 4 weeks of extraction, although most were analysed within 4 days. Stability of picoxystrobin and metabolite residues in a variety of matrices including maize grain, dry peas, soya bean meal and potato has been verified over 24 months (Schierhoff, 2012). This covers the storage time for the study samples.

Analyses for parent compound and three metabolites (IN-QDK50, IN-QDY62 and IN-QDY63) were performed using an LC/MS/MS method (method number 24868). In concurrent method validations, most individual and all mean recoveries fell within the acceptable range (70–120%) and the relative standard deviations were < 20%.

Residues of picoxystrobin and metabolites were below the LOQ in all untreated control samples, and only residue was detected in one sample just above the LOD. Residues and processing factors for the treated samples are summarized below.

Table 75 Residues of picoxystrobin and metabolites in US wheat grain and processed fractions (Rice, 2010)

Location, Trial number, Year (Variety)	Application			Sample	PHI (days)	Residues (mg/kg)					PF (parent only)	
	No.	Growth stage (BBCH)	g ai/ha			Parent	IN- QDK5 0	IN- QDY6 2	IN- QDY6 3	Total		
Richland, IA, USA, Trial 01, 2008, (Wilcross 076V65- 733)	3	41–47	1131	Grain	45	0.014	ND	0.007	0.003		–	
			1086	Bran	45	0.030	0.007	0.016	0.005		2.1	
		69	1131	Flour	45	0.003	ND	ND	ND			0.21
				Middlings	45	0.013	ND	0.004	ND			0.93
				Shorts	45	0.021	0.004	0.007	ND			1.5
				Germ	45	0.053	0.007	0.012	0.004			3.8
Carrington, ND, USA, Trial 02, 2008, (Kelby)	3	31	1131	Grain	45	0.058	0.005	0.007	0.003		–	
			1120	Bran	45	0.11	0.016	0.016	0.010		1.9	
		73	1131	Flour	45	0.015	ND	ND	ND			0.26
				Middlings	45	0.025	ND	ND	ND			0.43
				Shorts	45	0.023	0.005	ND	ND			0.40
				Germ	45	0.15	0.023	0.013	0.006			2.6

Table 76 Processing factors for picoxystrobin in US wheat products (Rice, 2010)

Sample	Processing factors (picoxystrobin only)	Mean PF
Bran	1.9, 2.1	2.0
Flour	0.21, 0.26	0.24
Middlings	0.43, 0.93	0.68

Sample	Processing factors (picoxystrobin only)	Mean PF
Shorts	0.40, 1.5	0.95
Germ	2.6, 3.8	3.2

Residues of the metabolites were generally significantly lower than for the parent compound. Processing factors are therefore not reported here for the metabolites.

Residues of picoxystrobin concentrate in wheat bran and germ, but not in flour, middlings or shorts. The results indicate that the majority of the residue is found on the surface of the seed.

An earlier wheat processing study was conducted in Germany (Jones and Hill, 1999). Trials were conducted at two sites, with three treatment plots per site. Three applications were made at 750 g ai/ha, at 13–17 day intervals between BBCH stages 41 and 75. A large sample was collected from each treated plot and from an untreated control area at both sites for processing.

Grain was processed using simulated commercial procedures. The grain for processing was cleaned by aspiration and screening and samples of screenings and pre- and post-cleaning grain were collected. Cleaned grain was adjusted to a moisture content of 16% and tempered, then broken three times in a roller mill to reduce the bran percentage below 15%; wholemeal flour was sampled at this stage. A second portion of cleaned moisture-adjusted tempered grain was broken three times in a roller mill until the bran percentage was < 15% and screened through 710, 400 and 250 micron screens. The material passing through the 250 micron screen was flour type 550 (an all-purpose white flour) and was sampled. Further broken grain was sieved through 710, 400, 250 and 140 micron screens to yield bran (material on the 710 micron screen), middlings (material on the 400 and 250 micron screens), low grade flour (on top of the 140 micron screen) and patent flour (passing through the 140 micron screen). Once the bran was separated, the middlings were reduced in a mill, sieved and further reduced and screened to yield shorts (material on the 400 and 250 micron screens) and further patent flour. Bread was baked from wholemeal and type 550 flour.

Samples of grain for direct analysis were frozen shortly after collection, while samples for processing were transported at ambient temperatures to the processing facility, where they were frozen pending processing (within 5 days of harvest). Processed commodity samples were frozen shortly after collection for transport to the analytical laboratory. All sample extractions were completed within 12 months of harvest of the grain, and all samples were stored frozen from collection until extraction, with the exception of the processing itself and a period of around 3–5 days between harvest and arrival at the processing facility. Analyses were completed within 6 days of sample extraction. Stability of picoxystrobin residues in a range of samples including maize grain has been verified over 24 months, so it is unlikely that the samples will have been adversely affected by storage.

Samples were analysed for parent compound only using GC/MS (method number RAM 288/01). This method was re-validated concurrently with the sample analysis giving acceptable recoveries in wheat grain and processed fractions (70–120%) and precision (RSD < 20%).

With the exception of one of the samples of wholemeal bread, residues of picoxystrobin were < LOQ in all untreated control grain and processed fraction samples. Residues of picoxystrobin in grain and processed commodities are tabulated below.

Table 77 Residues of picoxystrobin and metabolites in German wheat grain and processed fractions (Jones and Hill, 1999)

Location, Trial number, Year (Variety)	Application				Sample	PHI (days)	Picoxystrobin residues (mg/kg)	Processing factors ^a
	No.	BBCH stage	g ai/ha	L/ha				
Haag an der Amper, Bavaria, D-85410, Germany, RS-9707-G1, 1997, (Astron)	3	43	750	200	Grain	37	0.11	–
		59–61	750	200	Grain pre clean	37	0.04	–
		71	750	200	Grain post clean	37	0.05	1.2
					Screenings	37	0.56	14
					Wholemeal flour	37	0.12	3.0
					Type 550 flour	37	0.12	3.0
					Bran	37	0.33	8.3
					Shorts	37	0.10	2.5
					Patent flour	37	0.12	3.0
					Wholemeal bread	37	0.05	1.2
					Type 550 bread	37	0.07	1.8
Axien, Sachsen- Anhalt, D-06922, Germany, RS-9707-K1, 1997, (Pegasso)	3	41	750	200	Grain	37	0.06	–
		61	750	200	Grain pre clean	37	0.03	–
		75	750	200	Grain post clean	37	0.03	1.0
					Screenings	37	0.10	3.3
					Wholemeal flour	37	0.08	2.7
					Type 550 flour	37	0.05	1.7
					Bran	37	0.23	7.7
					Shorts	37	0.05	1.7
					Patent flour	37	0.07	2.3
					Wholemeal bread	37	0.06	2.0
					Type 550 bread	37	0.04	1.3

^a Calculated based on the grain pre cleaning result.

Table 78 Processing factors for picoxystrobin in German wheat products (Jones and Hill, 1999)

Sample	Processing factors ^a	Mean PF	Processing factors ^b	Mean PF
Grain pre clean	–	–	–	–
Grain post clean	1.0, 1.2	1.1	–	–

Sample	Processing factors ^a	Mean PF	Processing factors ^b	Mean PF
Screenings	3.3, 14	8.7	1.7, 5.1	3.4
Wholemeal flour	2.7, 3.0	2.9	1.1, 1.3	1.2
Type 550 flour	1.7, 3.0	2.4	0.83, 1.1	0.97
Bran	7.7, 8.3	8.0	3.0, 3.8	3.4
Shorts	1.7, 2.5	2.1	0.83, 0.91	0.87
Patent flour	2.3, 3.0	2.7	1.1, 1.2	1.2
Wholemeal bread	1.2, 2.0	1.6	0.45, 1.0	0.73
Type 550 bread	1.3, 1.8	1.6	0.64, 0.67	0.66

^a Using the grain pre-cleaning result

^b Using the bulk grain result.

Soya bean

Soya bean processing studies were conducted in the USA (Shepard, 2009 and Rice, 2011). Plots were treated with three applications of picoxystrobin at a target individual application rate of 1120 g ai/ha. Seed samples (treated and untreated control) were collected at normal harvest, 14 days after the last application and shipped to a processing laboratory.

The seed was processed using simulated commercial procedures. Seed was first tested for moisture and dried at 54–71 °C to a moisture content of 10–13.5% if necessary, then cleaned by screening and aspiration. Cleaned seed was milled in a roller mill and aspirated to separate hull and kernel fractions. The moisture content of the kernel material was adjusted to 13.5% (with equilibration for 12 hours) if necessary. Kernel material was then heated to 71–79 °C, flaked and processed into collets by steam injection and compression. The collets reached a temperature of 93–121 °C, and were dried in an oven at 66–82 °C for 30–40 minutes. Crude oil was extracted from the collets by heating with hexane at 49–60 °C, with the crude oil and hexane separated by distillation (91–96 °C), and the crude oil being refined by heating (20–24 °C at high RPM for 90 minutes and 63–67 °C at low RPM for 20 minutes) with sodium hydroxide and separating the resulting refined oil and soapstock. The solvent was removed from the extracted collets by heating (99–104 °C) to yield meal. Samples of unprocessed seed, hulls, meal and refined oil were shipped to the laboratory for analysis. In the second study (Rice, 2011), in addition to the above processes, a batch of cleaned soya beans was processed mechanically by pressing to yield crude oil and mechanically pressed meal. The meal was sampled without further processing, while the crude oil was refined as for the solvent extracted oil.

Raw soya bean samples were stored frozen prior to processing or analysis, and processed samples were frozen after processing until analysis. The maximum interval from harvest to sample extraction (including processing time) was 7 months for the first study and 5 months for the second study, with all analyses being completed within 8 days of extraction. Stability of picoxystrobin and metabolite residues in soya bean seed, meal and oil matrices has been verified over 24 months for seed, meal and oil (with the exception of IN-QDY62 and IN-QDY63 in oil) (Schierhoff, 2012). This largely covers the storage time for the study samples.

Analyses for parent compound and three metabolites (IN-QDK50, IN-QDY62 and IN-QDY63) were performed using an LC/MS/MS method (method number 24868). In concurrent method validations, most individual and all mean recoveries fell within the acceptable range (70–120%) and the relative standard deviations were < 20%.

Residues and processing factors for the treated samples are summarized below.

Table 79 Residues of picoxystrobin and metabolites in soya bean seed and processed fractions

Location, Report no., Trial no., Year (Variety)	Application				Sample	PHI, days	Residues (mg/kg)					PF ^c	
	No.	BBCH stage	g ai/ha	L/ha			Parent	IN- QDY6 2	IN- QD Y63	IN- QD K50	Tot al		
Carlyle, IL, USA, 25488, Trial 01, 2008 (37N4)	3	61	1065	148	Seed	14	0.29	ND	ND	0.005		–	
		81	1100	126	Hulls	14	0.65	ND	ND	0.016		2.2	
		87–89	1082	163		14	0.008	ND	ND	0.005		0.03	
					Refined oil ^a	14	0.27	ND	ND	ND			0.93
Perley, MN, USA, 25488, Trial 02, 2008 (5B077RR)	3	61	1113	187	Seed	15	0.032	ND	ND	ND		–	
		85	1115	187	Hulls	15	0.18	ND	ND	0.009		5.6	
		87	1106	187		15	ND	ND	ND	ND		< 0.09	
					Refined oil ^a	15	0.050	ND	ND	ND			1.6
Tipton, MO, USA, 29661, Trial 01, 2010 (48-24 MorSoy)	3	61	1110	295	Seed	14	0.010	0.005	ND	ND		–	
		81	1121	297	Hulls	14	0.051	0.018	ND	ND		5.1	
		81	1166	304		14	0.011	0.006	ND	ND		1.1	
					Refined oil ^a	14	0.022	ND	ND	ND			2.2
					Refined oil ^b	14	0.034	ND	ND	ND			3.4
					Meal ^b	14	0.006	0.007	ND	ND			0.60
Springfield, NE, USA, 29661, Trial 02, 2010, Channel 3051 R	3	(R1)61	1113	164	Seed	13	0.050	0.009	ND	ND		–	
		(72, 6)	1132	179	Hulls	13	0.22	0.021	0.009	0.007		4.4	
		83	1001	176		13	0.003	0.012	ND	ND		0.06	
			<u>3246</u>		Refined oil ^a	13	0.050	ND	ND	ND			1.0
					Refined oil ^b	13	0.17	ND	ND	ND			3.4
					Meal ^b	13	0.018	0.011	0.004	ND			0.36

^a Solvent extracted^b Mechanically extracted^c Parent compound only ND = not detected (< 0.003 mg/kg)

In the soya bean supervised residue trial study (study number 24861: Shepard, 2010), aspirated grain fractions were generated from seed samples from two trial sites. Bulk samples of seed were collected at the same interval as the samples for analysis of the raw agricultural commodity and shipped to a processing facility. Seed samples were tested for moisture and dried if necessary at 43–57 °C to a moisture content of 10–13%. The sample was placed in a holding bin and moved

continuously through a screw conveyor and two bucket conveyors and back into the holding bin repeatedly for 120 minutes. Aspirated grain fractions were removed from the seed at several locations in the conveyors and the bin. The aspirated grain fractions were sorted by sieving and material passing through the 2360 micron sieve was collected and sampled for analysis. Seed was stored frozen prior to processing and aspirated grain fraction samples were stored frozen after generation. Samples were analysed using the same method as for the soya bean field trials. Residues are tabulated below.

Table 80 Residues of picoxystrobin and metabolites in soya bean aspirated grain fractions (study number 24861).

Location, Trial No., Year (Variety)	Application				Sample	DAT ^b	Residues (mg/kg) ^c			
	No.	Growth stage	g ai/ha ^a	L/ha			Parent	IN-QDY62	IN-QDY63	IN-QDK50
Carlyle, IL, USA Trial 14, 2008 (NK 37-N4)	3	(R1)61	213	148	Seed	17	0.012 (0.011, 0.013)	ND	ND	ND
		(R6-7)79-81	213	183						
		(R7)81	220	126						
					Process seed	17	<u>< 0.01</u>	ND	ND	ND
					AGF	17	<u>3.2</u> c0.005	0.015	0.098	0.024
Richland, IA, USA Trial 15, 2008 (Pioneer 93M11)	3	(R1)61	221	141	Seed	14	0.011 (0.012, 0.010)	ND	ND	ND
		(R7)81	224	163						
		(R7)81	224	165						
					Process seed	14	<u>0.010</u>	ND	ND	ND
					AGF	14	<u>1.9</u> c0.018	0.12	0.20	0.048

Table 81 Processing factors for picoxystrobin in soya bean products

Sample	Processing factors (picoxystrobin only)	Mean PF
Hulls	2.2, 4.4, 5.1, 5.6	4.3
Meal (solvent extracted)	0.03, 0.06, < 0.09, 1.1	0.32
Refined oil (solvent extracted)	0.93, 1.0, 1.6, 2.2	1.4
Refined oil (mechanically extracted)	3.4, 3.4	3.4
Meal (mechanically extracted)	0.36, 0.6	0.48
Aspirated grain fraction	1.9, 3.2	2.6

Residues of the metabolites were generally significantly lower than for the parent compound. Processing factors are therefore not reported here for the metabolites.

Residues of picoxystrobin concentrate in hulls and aspirated grain fractions, while those in meal (both solvent and mechanically extracted) were low. Residues of picoxystrobin in solvent extracted refined soya bean oil were slightly higher than those in the raw seed, while residues concentrated significantly in mechanically extracted refined oil. The results indicate that the majority of the residue is found on the surface of the seed and is removed with the hull.

Maize

A processing study for maize was conducted in the USA (Shepard, 2009). Plots were treated with three applications at a target application rate of 1120 g ai/ha, timed so that the first was applied around R1 (beginning of flowering/silking), with the second and third being made 7 days apart, close to harvest. Grain samples (treated and untreated control) were collected at normal harvest, and shipped to a processing laboratory.

The grain was processed using simulated commercial procedures by both wet and dry milling. The grain was first cleaned by aspiration and screening. Corn for dry milling was dried at 54–71 °C to a moisture content of 10–15% if necessary, then cleaned by aspiration and screening. Cleaned grain was moisture-conditioned to 21% moisture and tempered for approximately 2 hours. The tempered corn was fed into a disc mill to crack the kernel, and the resulting corn stock was dried in an oven at 54–71 °C for 30 minutes, before screening to separate bran/germ/large grits from grits/meal/flour. The bran/germ/large grits fraction was aspirated to remove the bran, screened to separate large grits + germ from germ + attached hull and endosperm. The latter fraction was disc milled, screened and the material on top of the screen aspirated to remove detached bran (material passing through the screen was large grits). These steps were repeated. The germ + large grits fraction was separated using a gravity separator. The germ fractions were combined and heated at 54–71 °C in an oven to achieve a moisture content of 8–12%.

The grits + meal + flour fraction was generated using a sifter equipped with 14 mesh and 62 mesh screens, with the material on top of the 14 mesh screen being grits, material on top of the 62 mesh screen being meal and the material passing through the 62 mesh screen being flour. The grit sample was a combination of the material from this step plus large grits from the gravity separator.

Germ was heated to 71–79 °C for 10 minutes, flaked and extracted in a batch process by heating with hexane at 49–60 °C for 30 minutes. The first batch of hexane/oil miscella was drained off and the extraction repeated twice more with 15 minute extraction times. The combined miscella was separated by distillation (91–96 °C), and the crude oil was refined by addition of sodium hydroxide, gentle heating (20–24 °C for 15 minutes at high RPM and 63–67 °C for 12 minutes at low RPM) and centrifuging to separate refined oil and soapstock. Refined oil samples were collected (soapstock was discarded).

Corn (dried and cleaned) for wet milling was steeped in hot (49–54 °C) water containing 0.1–0.2% sulphurous acid for 22–48 hours. After steeping, the corn was disc milled and the majority of the germ and hull were removed by water centrifuge, and then dried at 74–91 °C to 5–10% moisture. Germ and hull were separated by aspiration and screening. The remaining material (cornstock) was further disc milled and screened (50 micron screen). The material on top of the screen was discarded and the process water passing through centrifuged to separate into starch and gluten, with starch samples being collected, dried at 54–71 °C to a moisture content of \leq 15%.

The germ was conditioned to 12% moisture, heated (88–104 °C) and flaked, then pressed to expel some of the crude oil. Finally the press cake with residual oil was extracted with hexane using the same method as for the dry milling process. The combined miscella was separated by distillation, and the mechanically- and solvent extracted oil were combined for refining by the same method as for the dry milled crude oil. A sample of the wet milled oil was collected for analysis. Samples of unprocessed corn, meal, flour, starch, grits and refined oil (from both wet and dry milling) were shipped frozen to the laboratory for analysis.

Unprocessed grain samples and process fraction samples were frozen within 24 hours of collection and kept frozen until analysis, and grain awaiting processing was stored frozen between harvest and processing. The maximum interval from harvest until sample extraction (including processing and time awaiting processing) was 6 months. Sample extracts were analysed within 17 days of extraction. Stability of picoxystrobin and metabolite residues in maize grain and vegetable oil (soya bean) matrices has been verified over 24 and 6 months respectively (Schierhoff, 2012), which covers the storage time for the study samples.

Analyses for parent compound and three metabolites (IN-QDK50, IN-QDY62 and IN-QDY63) were performed using an LC/MS/MS method (method number 24868). In a concurrent validation, most individual recoveries and all mean values fell within the acceptable range (70–120%) and the relative standard deviations were < 20%.

Residues of picoxystrobin in the treated samples are summarized below.

Table 82 Residues of picoxystrobin and metabolites in maize grain and processed fractions

Location, Trial no., Year (Variety)	Application				Sample	PHI days	Residues (mg/kg)					PF ^a
	No.	BBCH stage	g ai/ha	L/ha			Parent	IN- QDY6 2	IN- QDY6 3	IN- QDK5 0	Total	
Carlyle, IL, USA Trial 01, 2008 (Burrus 616 XLR)	3	61 87-89 87-89	1137 1123 1106	150 163 159	Grain	7	0.12	ND	ND	ND		–
					Starch	7	0.003	ND	ND	ND		0.025
					Grits	7	0.061	ND	ND	ND		0.51
					Flour	7	0.12	ND	ND	ND		1.0
					Refined oil, wet milled	7	0.87	ND	ND	ND		7.3
					Meal	7	0.095	ND	ND	ND		0.79
					Refined oil, dry milled	7	0.65	ND	ND	ND		5.4
Richland, IA, USA Trial 02, 2008 (Middleko op 5513)	3	61 87-89 87-89	1098 1132 1098	167 167 167	Grain	7	0.044	ND	ND	ND		–
					Starch	7	ND	ND	ND	ND		< 0.07
					Grits	7	0.015	ND	ND	ND		0.034
					Flour	7	0.053	ND	ND	ND		1.2
					Refined oil, wet milled	7	0.28	ND	ND	ND		6.4
					Meal	7	0.034	ND	ND	ND		0.77
					Refined oil, dry milled	7	0.15	ND	ND	ND		3.4

^aParent compound only.

In the maize supervised residue trial study (study number 24864: Shepard, 2009), aspirated grain fractions were generated from grain samples from two trial sites. Bulk samples of grain were collected at the same interval as the samples for analysis of the raw agricultural commodity and shipped to a processing facility. Grain samples were tested for moisture and dried if necessary at 43–57 °C to a moisture content of 10–13%. The sample was placed in a holding bin and moved continuously through a screw conveyor and two bucket conveyors and back into the holding bin repeatedly for 120 minutes. Aspirated grain fractions were removed from the seed at several locations in the conveyors and the bin. The aspirated grain fractions were sorted by sieving and the material passing through the 2360 micron sieve was collected and sampled for analysis. Grain was stored frozen prior to processing and aspirated grain fraction samples were stored frozen after generation.

Samples were analysed using the same method as for the maize field trials. Residues are tabulated below.

Table 83 Residues of picoxystrobin and metabolites in maize aspirated grain fractions (study number 24864)

Location Trial no., Year (variety)	Application				Sample	DAT ^b	Residues (mg/kg) ^c			
	No.	BBCH stage	g ai/ha ^a	L/ha			Parent	IN-QDY62	IN-QDY63	IN-QDK50
Richland, IA, USA Trial 05, 2008 (Middle Koop 5513)	3	R1	213	167	Grain	6	ND	ND	ND	ND
		R6	224	162			(ND, ND)	(ND, ND)	(ND, ND)	(ND, ND)
		R6	224	165	Process grain	6	<u>0.012</u> (0.010, 0.014)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
Carlyle, IL, USA Trial 14, 2008 (Burrus 616 XLR)	3	R1	225	150	Grain	7	<u>< 0.01</u>	ND	ND	ND
		R6	222	162			(< 0.01, < 0.01)	(ND, ND)	(ND, ND)	(ND, ND)
		R6	216	172	Process grain	7	<u>< 0.01</u> (< 0.01, < 0.01)	ND (ND, ND)	ND (ND, ND)	ND (ND, ND)
					AGF	7	<u>0.17</u> (0.18, 0.13) c0.003	0.26 (0.27, 0.25)	< 0.01 (< 0.01, < 0.01)	ND (ND, ND)

Table 84 Processing factors for picoxystrobin in maize products

Sample	Processing factors (picoxystrobin only)	Mean PF
Starch	0.025, < 0.07	0.047
Grits	0.34, 0.51	0.43
Flour	1.0, 1.2	1.1
Wet milled refined oil	6.4, 7.3	6.9
Meal	0.77, 0.79	0.78
Dry milled refined oil	3.4, 5.4	4.4
Aspirated grain fractions	13, 17	15

No residues of IN-QDK50, IN-QDY62 or IN-QDY63 were detected in any of the unprocessed seed or process fraction samples. Processing factors are therefore not reported here for the metabolites.

Residues of picoxystrobin did not concentrate in maize starch, grits or meal, while residues in flour were virtually identical to those in grain. Picoxystrobin did concentrate in aspirated grain fractions, and in refined maize oil, both from the wet- and dry-milled maize, consistent with the organosoluble properties of the molecule.

Oilseed rape

A processing study for oilseed rape was conducted in North America (Thiel, 2010). Two field trials, in which picoxystrobin was applied twice at a target application rate of 1120 g ai/ha were conducted. Samples of treated and untreated control rape seed were collected at a pre-harvest interval of 21 days (although reported as 21 days, the dates given for the Saskatchewan site correspond to a 43 day PHI). The untreated and treated seed samples were shipped to a processing laboratory and processed using simulated commercial practices after retaining a sample of the raw seed.

After checking the moisture content and drying in an oven at 54–71 °C to 7–10% moisture if necessary, seed was aspirated and screened to remove impurities. Cleaned seed was flaked and heated (82–99 °C for 10–15 minutes) before pressing to remove a portion of the crude oil. The mechanically extracted presscake plus residual oil was then extracted with hexane at 49–60 °C for 30 minutes. The first batch of hexane/oil miscella was drained off and the extraction repeated twice more with 15–30 minute extraction times. The combined miscella was separated by distillation (91–96 °C).

The combined mechanically pressed and solvent extracted crude oil was placed in a water bath, pre-treated with phosphoric acid, and mixed for 30 minutes at 40–45 °C. The oil was refined by addition of sodium hydroxide, gentle heating (40–45 °C for 20 minutes and 49–70 °C for 10 minutes) and centrifuging to separate refined oil and soapstock. Refined oil samples were collected (soapstock was discarded). The refined oil was filtered, and bleached by heating at 40–50 °C and addition of diatomaceous earth. The mixture was placed under vacuum and the temperature increased to 85–100 °C for 12–15 minutes. The bleached oil was cooled and filtered then steam bathed for 45–60 minutes under vacuum with the temperature held between 234–250 °C. During cooling, citric acid was added as a deodoriser. The meal remaining after solvent extraction was mixed and heated to 99–104 °C to remove residual solvent.

Samples of press cake, crude solvent extracted and mechanically extracted oil, meal (solvent extracted) and refined oil were collected. Various other process fractions and by-products were collected, but results are not reported for these samples.

Analyses for parent compound and three metabolites (IN-QDK50, IN-QDY62 and IN-QDY63) were performed using an LC/MS/MS method (method number 24868). Unprocessed seed samples and process fraction samples were frozen after collection and kept frozen until analysis, with the maximum storage interval being 9 months for the seed samples and 3 months for the process fractions. Seed samples awaiting processing were stored frozen between harvest and processing. Stability of picoxystrobin and metabolite residues in oilseed and vegetable oil (soya bean) matrices has been verified over 24 and 6 months respectively (Schwartz, 2010), which covers the storage time for the study samples. Sample extracts were analysed within 43 days of extraction. Good method recoveries (70–120%) and precision (RSD < 20%) were achieved for the samples of rape seed and process fractions fortified concurrently with the sample analyses.

Table 85 Residues of picoxystrobin and metabolites in rape seed and processed fractions

Location, Trial no., Year, Variety	Application				Sample	PHI, days	Residues (mg/kg)					PF ^c
	No.	BBCH stage	g ai/ha	L/ha			Parent	IN- QDY6 2	IN- QDY6 3	IN- QD K50	Total	
Madras, OR, USA Trial 01, 2008 (Cracker Jack)	3	79	1107	188	Seed	7	0.13	ND	ND	ND		–
				190	Press cake	7	0.078	ND	ND	ND		0.60
		83	1119		Crude oil ^a	7	0.19	ND	ND	0.00 3		1.5
					Crude oil ^b	7	0.18	ND	ND	ND		1.4
					Refined	7	ND	ND	ND	ND		< 0.0

Location, Trial no., Year, Variety	Application				Sample	PHI, days	Residues (mg/kg)					PF ^c
	No.	BBCH stage	g ai/ha	L/ha			Parent	IN- QDY6 2	IN- QDY6 3	IN- QD K50	Total	
							oil					
				Meal ^b	7	0.069	ND	ND	ND		0.53	
Rosthern, SK, Canada Trial 02, 2008, (5020)	3	69–75	1093	198	Seed	7	0.42	ND	ND	0.00 4		–
		73–77	1065	188	Press cake	7	0.28	ND	ND	ND		0.67
					Crude oil ^a	7	1.3	ND	ND	ND		3.0
					Crude oil ^b	7	0.97	ND	ND	ND		2.3
					Refined oil	7	0.081	ND	ND	ND		0.20
					Meal ^b	7	0.085	ND	ND	0.00 4		0.20

^a Mechanically extracted

^b Solvent extracted

^c Parent compound only.

Table 86 Processing factors for picoxystrobin in oilseed rape products

Sample	Processing factors (picoxystrobin only)	Mean PF
Press cake (after mechanical extraction)	0.60, 0.67	0.63
Crude oil (mechanically extracted)	1.5, 3.0	2.2
Crude oil (solvent extracted)	1.4, 2.3	1.8
Refined oil	< 0.02, 0.20	0.11
Meal (after solvent extraction)	0.20, 0.53	0.37

No residues of IN-QDY62 or IN-QDY63 were detected in any of the unprocessed seed or process fraction samples, while residues of IN-QDK50 were mostly undetectable or very low (maximum of 0.004 mg/kg). Processing factors are therefore not reported here for the metabolites.

Residues of picoxystrobin did not concentrate in rape seed press cake or meal. Residues concentrated in crude oil (both mechanically and solvent extracted), consistent with the organosoluble properties of the molecule. Residues in refined oil were either very low or undetectable, indicating that the refining process destroyed the molecule.

Residues in animal commodities

Ruminants

A feeding study was conducted for picoxystrobin in lactating dairy cows (Wen, 2009). Fourteen Holstein/Friesian dairy cows aged 3 to 7 years and in mid to late lactation were selected for the study, based on health and behaviour. Body weights ranged from 470–629 kg on Day 1 and 496–657 kg on Day 28. Two animals formed the untreated control group (Group 1), three animals were included in

each of the low-, mid- and high-dose groups (Groups 2–4), and an additional three animals in Group 4 for the depuration phase. The doses were 40, 120 and 403 ppm dry weight in feed for Groups 2–4 respectively, or 1.35, 4.12, and 12.9 mg/kg bw/day. Picoxystrobin was administered to the treated cattle as a gelatine capsule given orally twice daily for 29 consecutive days, after an acclimatisation period of 14 days. The cattle were kept indoors and provided with fresh water *ad libitum*, together with a diet of grain, baled hay and corn silage. Food intake was monitored throughout the trial, and the previous week's feed intakes used to calculate the following week's doses. No adverse effects on cattle weight, feed consumption or milk yield were noted during the trial.

Milk was collected twice daily, with the sampled from the afternoon milking being refrigerated and proportionately combined with the next morning's sample to give a single daily sample from dosing day 1 until sacrifice. Additional milk samples from days 14 and 21 of dosing were separated into skim milk and cream. Within 23 hours of the final dose, one control animal, all animals from groups 2 and 3, and the three non-depuration animals from group 4 were sacrificed, and samples of liver, kidney, fat (perirenal, omental and subcutaneous), and muscle (a composite sample of equal amounts of round, flank and loin) were collected. The depuration cattle were sacrificed on days 32, 37 and 44 (after 3, 8 and 15 days of depuration respectively), and samples were collected as for the other animals. All samples were homogenised and frozen (–20 °C) for storage and transport to the laboratory.

Samples were analysed using an LC/MS/MS method (number 25997). The concurrent method recovery and precision was acceptable, with mean recoveries in the range 70–120% and relative standard deviations less than 20%.

Milk samples were stored for up to 34 days before analysis, while tissue samples were stored for up to 112 days. Fortified milk samples analysed after 36 days frozen storage had a recovery of 84%, while fortified tissue samples after 125 days storage showed recoveries of 81%, 82%, 92%, and 94% for liver, kidney, fat and muscle respectively. The samples are therefore unlikely to have been adversely affected by storage.

Table 87 Residues of picoxystrobin in milk (including depuration data)

Dose level (mg/kg in feed)	Sampling interval (days)	Picoxystrobin residues (mg/kg)
0	Residues of picoxystrobin were not detected in any milk samples from the control, low-, or mid-dose groups.	
40		
120		
400	1	ND, ND, ND, ND, ND, ND
	3	ND, ND, ND, ND, < 0.01 (0.004), < 0.01 (0.006)
	5	ND, < 0.01 (5); [0.004, 0.004, 0.004, 0.004, 0.006]
	7	ND, < 0.01 (5); [0.003, 0.004, 0.004, 0.004, 0.005]
	10	ND, < 0.01 (5); [0.003, 0.003, 0.004, 0.006, 0.006]
	14	< 0.01 (6); [0.004, 0.004, 0.004, 0.005, 0.006, 0.009]
	17	ND, ND, < 0.01 (4); [0.003, 0.004, 0.004, 0.007]
	21	ND, ND, < 0.01 (4); [0.004, 0.005, 0.005, 0.009]
	24	ND, ND, ND, < 0.01 (2); [0.005, 0.007], 0.014
	28	ND, ND, ND, ND, < 0.01 (0.005), 0.011
	30 (depuration day 1)	ND, ND, ND
	32 (depuration day 3)	ND, ND
	34 (depuration day 5)	ND, ND
36 (depuration day 7)	ND, ND	
39 (depuration day 10)	ND	

Picoxystrobin

Dose level (mg/kg in feed)	Sampling interval (days)	Picoxystrobin residues (mg/kg)
	43 (deuration day 14)	ND

ND = not detected. Limit of detection (LOD) = 0.003 mg/kg. Limit of quantification (LOQ) = 0.01 mg/kg.

Table 88 Residues of picoxystrobin in skim milk and cream

Matrix	Dose level (mg/kg in feed)	Sampling interval (days)	Picoxystrobin residues (mg/kg)
Skim milk	0	14	ND, ND
		21	ND, ND
	40	14	ND, ND, ND
		21	ND, ND, ND
	120	14	ND, ND, ND
		21	ND, ND, ND
	400	14	ND, ND, ND
		21	ND, ND, ND
Cream	0	14	ND, ND
		21	ND, ND
	40	14	ND, ND, ND
		21	ND, ND, ND
	120	14	< 0.01 (0.004), < 0.01 (0.004), < 0.01 (0.005)
		21	ND, < 0.01 (0.003), < 0.01 (0.004)
	400	14	0.016, 0.022, 0.033
		21	0.016, 0.019, 0.048

Table 89 Residues of picoxystrobin in cattle tissues (including deuration data)

Matrix	Dose level (mg/kg in feed)	Picoxystrobin residues (mg/kg)
Omental fat	0	ND
	40	< 0.01 (0.005), < 0.01 (0.006), < 0.01 (0.007)
	120	0.015, 0.021, 0.026
	400	0.041, 0.060, 0.077
	400 (deuration day 3)	0.049
	400 (deuration day 8)	ND
	400 (deuration day 15)	ND
Perirenal fat	0	ND
	40	< 0.01 (0.003), < 0.01 (0.003), < 0.01 (0.005)
	120	< 0.01 (0.009), 0.013, 0.017
	400	0.022, 0.056, 0.055
	400 (deuration day 3)	ND
	400 (deuration day 8)	ND
	400 (deuration day 15)	ND

Matrix	Dose level (mg/kg in feed)	Picoxystrobin residues (mg/kg)
Subcutaneous fat	0	ND
	40	ND, ND, ND
	120	< 0.01 (0.005), 0.016, 0.016
	400	< 0.01 (0.005), 0.029, 0.049
	400 (depuration day 3)	< 0.01 (0.008)
	400 (depuration day 8)	ND
	400 (depuration day 15)	< 0.01 (0.005)
Muscle	0	ND
	40	ND, ND, ND
	120	ND, ND, ND
	400	< 0.01 (0.003), < 0.01 (0.004), < 0.01 (0.008)
	400 (depuration day 3)	ND
	400 (depuration day 8)	ND
	400 (depuration day 15)	ND
Liver	0	ND
	40	ND, < 0.01 (0.005), < 0.01 (0.005)
	120	ND, 0.011, 0.017
	400	0.055, 0.087, 0.10
	400 (depuration day 3)	0.014
	400 (depuration day 8)	ND
	400 (depuration day 15)	ND
Kidney	0	ND
	40	ND, ND, ND
	120	ND, ND, ND
	400	< 0.01 (0.004), < 0.01 (0.005), 0.010
	400 (depuration day 3)	ND
	400 (depuration day 8)	ND
	400 (depuration day 15)	ND

Residues of picoxystrobin were not detected in whole milk samples from the control or low- and mid-dose groups. Low levels of picoxystrobin, mostly below the limit of quantification, were found in milk from the high-dose group. The mean residues in milk reached a plateau around day 14, while the maximum individual residue for the high dose group was 0.014 mg/kg on day 24.

No residues of picoxystrobin were detected in skim milk, while low levels were found in cream ND-< 0.01 mg/kg for the mid-dose group and 0.016–0.048 mg/kg for the high-dose group), indicating the fat solubility of the compound.

Only low levels of picoxystrobin were observed in muscle and kidney, with no detections for the control, low or mid-dose groups, and a maximum of < 0.01 mg/kg in muscle and 0.010 in kidney for the high dose group. Residues were detected in liver and omental and perirenal fat at all dose levels, while residues were detected in subcutaneous fat at the medium and high doses. A roughly linear relationship between dose and residue was observed for liver and fat. The maximum observed

residue for liver was 0.10 mg/kg at the high dose level and for fat the highest residue was 0.077 mg/kg in omental fat at the high dose level.

The depuration data indicated rapid clearance of picoxystrobin residues from milk and tissues. No residues were detected in milk, muscle, perirenal fat or kidney from the depuration animals. Liver and omental fat residues were undetectable by 8 days after the final dose, while residues of picoxystrobin below the limit of quantification were found in subcutaneous fat 3 and 15 days after the last dose.

Poultry

A feeding study was conducted for picoxystrobin in laying hens (Wen, 2010). Fifty-three white Leghorn hens (approximately 28 weeks old, and weighing 1.2–1.7 kg) were included in the study. Ten were designated as the untreated control group, and ten were assigned to each of the low- and mid-dose groups. In total, 23 birds were given the high dose including ten for the depuration phase, and three that were added at a later stage to generate additional samples due to one of the original sub-groups of three birds having abnormally high egg and tissue residues, thought to be the result of contamination. The birds in each dose group were further grouped in sub-groups of 3 or 4 individuals, with egg and tissue samples for the birds in each sub-group being pooled for analysis. Picoxystrobin was administered orally by capsule once daily for 36 days at doses of 15, 45 and 153 ppm dry weight in feed (or 0.97, 2.8 and 9.5 mg/kg bw/day), after an acclimatisation period of 14 days. For the additional sub-group of high dose hens, the dose was 160 ppm in feed (9.3 mg/kg bw/day). Untreated control birds received blank capsules. The hens were housed in pens kept indoors and provided with fresh water and layer poultry ration *ad libitum* (the daily feed consumption was monitored). Feeding of picoxystrobin did not appear to have any adverse effects on feed consumption, body weight or egg production.

The birds in each dose group were further grouped in sub-groups of 3 or 4, with egg and tissue samples for the birds in each sub-group being pooled for analysis. Eggs were collected twice daily and the eggs from the afternoon sampling were pooled with those from the next morning to provide a single daily egg sample. For the control and low-, mid- and high-dose groups (other than the depuration birds), eggs from three days prior to dosing, and from days 1, 3, 5, 7, 10, 14, 17, 21, 24 and 28 of dosing were analysed. Additionally, separate analyses of yolks and whites were performed for the day 14 and 21 samples. Eggs from the depuration birds from one day prior to cessation of dosing, on the day of the last dose, and 2, 4, 9, 11 and 14 days after the last dose were analysed.

Within 6 hours of the final dose, all birds from the untreated control group, the low- and mid-dose groups, and the non-depuration high-dose hens were sacrificed, and samples of liver, fat with skin, and muscle (equal amounts of leg and breast) were collected. Subgroups of 3 or 4 of the depuration hens were sacrificed 5, 10 and 15 days after the final dose, and samples were collected as described above. All samples were stored frozen until analysis.

Samples were analysed using an LC/MS/MS method (number 25997). The concurrent method recoveries and precision were acceptable, with mean recoveries in the range 70–120% and relative standard deviations less than 20%.

All egg samples were analysed within 43 days of collection, while all tissue samples (with the exception of some control samples, which were analysed 134 days after collection) were analysed within 37 days of collection. The stability of picoxystrobin residues in eggs stored frozen was verified over 54 days, with a recovery of 81% being achieved after storage. The stability of picoxystrobin residues in liver, fat and muscle was verified for samples stored frozen for 125 days as part of the cattle feeding study (see above). Residues of picoxystrobin the egg and tissue samples from the hen feeding study are therefore unlikely to have been adversely affected by storage.

Table 90 Residues of picoxystrobin in eggs (including depuration data)

Target dose level (mg/kg in feed)	Sampling interval (days)	Picoxystrobin residues (mg/kg)
0	Residues of picoxystrobin were not detected in any of the egg samples from the	

15	control, low- or mid-dose groups.	
45		
153	-3	ND, ND, ND
	1	ND, ND, ND
	3	ND, ND, ND
	5	ND, ND, ND
	7	ND, ND, ND
	10	ND, ND, < 0.01 (0.003)
	14	ND, ND, ND
	17	ND, ND, ND
	21	ND, ND, ND
	24	ND, ND, ND
	28	ND, ND, ND
	35 (deuration day -1)	ND, ND, ND
	36 (deuration day 0)	ND, 0.010, 0.014
	38 (deuration day 2)	ND, ND, ND
	40 (deuration day 4)	ND, ND, ND
	45 (deuration day 9)	ND, ND
	47 (deuration day 11)	ND
50 (deuration day 14)	ND	

ND = not detected. Limit of detection (LOD) = 0.003 mg/kg. Limit of quantification (LOQ) = 0.01 mg/kg.

Table 91 Residues of picoxystrobin in egg yolks and whites

Matrix	Dose level (mg/kg in feed)	Sampling interval (days)	Picoxystrobin residues (mg/kg)
Egg yolk	0	14	ND, ND, ND
		21	ND, ND, ND
	15	14	ND, ND, ND
		21	ND, ND, ND
	45	14	ND, ND, ND
		21	ND, ND, ND
	150	14	ND, < 0.01 (0.005), < 0.01 (0.005), < 0.01 (0.005)
		21	ND, ND, < 0.01 (0.003), < 0.01 (0.004)
Egg white	0	14	ND, ND, ND
		21	ND, ND, ND
	15	14	ND, ND, ND
		21	ND, ND, ND
	45	14	ND, ND, ND
		21	ND, ND, ND
	150	14	ND, ND, ND, ND
		21	ND, ND, ND, ND

Table 92 Residues of picoxystrobin in hen tissues (including depuration data)

Matrix	Dose level (mg/kg in feed)	Picoxystrobin residues (mg/kg)
Fat	0	ND, ND, ND
	15	ND, < 0.01 (0.004), < 0.01 (0.004)
	45	< 0.01 (0.004), < 0.01 (0.004), 0.010
	150	< 0.01 (0.007), 0.011, 0.016, 0.061 ^a
	150 (depuration day 5)	ND
	150 (depuration day 10)	ND
	150 (depuration day 15)	ND
Muscle	0	ND, ND, ND
	15	ND, ND, ND
	45	ND, ND, 0.024 ^a
	150	< 0.01 (0.003), < 0.01 (0.004), < 0.01 (0.006), 0.21 ^a
	150 (depuration day 5)	ND
	150 (depuration day 10)	ND
	150 (depuration day 15)	ND
Liver	0	ND, ND, ND
	15	ND, ND, 0.027 ^a
	45	ND, ND, ND
	150	ND, ND, < 0.01 (0.008), 0.41 ^a
	150 (depuration day 5)	ND
	150 (depuration day 10)	ND
	150 (depuration day 15)	ND

^a Indicates values to be discounted. The study author indicated that the results from subgroup 2 of the hens in the 150 ppm group showed anomalously high residues of picoxystrobin in tissues and that these results should be discounted. The 0.024 mg/kg muscle result at the 45 ppm feeding level has been discounted since it is anomalously high compared with the other 45 ppm results (both ND), the remaining 150 ppm results (0.003-0.006 mg/kg), and further, it is higher than any of the fat results at the 45 ppm feeding level despite picoxystrobin being fat soluble. The 0.027 mg/kg liver result for the 15 ppm feeding level has been discounted since both other results at 15 ppm and all results at the next highest feeding level (45 ppm) were ND.

No residues of picoxystrobin were detected in any of the untreated control egg samples, nor were any residues detected in the eggs from the low- and mid-dose group hens. Further, residues were undetectable in the separated white and yolk samples from these groups. In the non-depuration high dose group, no residues were detected other than one very low level detection (< 0.01 mg/kg) on day 10, while in the depuration group, two residues of 0.010 and 0.014 mg/kg were found on the last day of dosing. These residues cleared quickly, with no picoxystrobin being detected in eggs collected from depuration day 2 onwards. In egg yolk from the high dose group, residues of < 0.01 mg/kg picoxystrobin were detected on dosing days 14 and 21, while no detections were made in egg white. These results are consistent with the results for skim milk and cream for the lactating cattle study, where higher residues were found in cream.

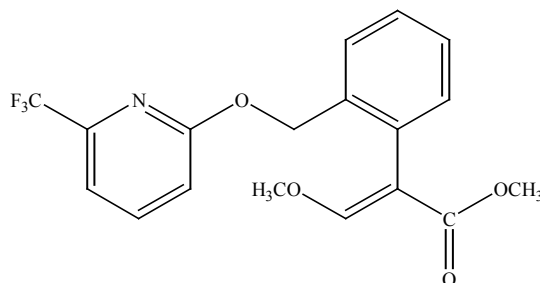
Tissue samples collected from the untreated control hens contained no residues of picoxystrobin. In fat, low levels of picoxystrobin were detected in the samples from the low dose group (< 0.01 mg/kg), while in the mid dose group, residues from < 0.01 to 0.010 mg/kg were found.

Fat residues for the high dose group ranged from < 0.01–0.016 mg/kg, with a residue of 0.061 mg/kg from sub-group 2 being discarded due to it being anomalously high. In muscle, no residues were detected for the low dose group. In the mid-dose group, two sub-groups did not have detectable residues in muscle, while the result of 0.024 mg/kg was discounted since it was higher than any of the values retained for the high dose group, and higher than the residues in fat for the mid-dose group despite picoxystrobin being fat soluble. In the high dose group, muscle residues of picoxystrobin were < 0.01 mg/kg in three of the sub-groups of hens, with the result of 0.21 mg/kg from sub-group 2 being discarded as an outlier due to probable sample contamination. In liver, residues of picoxystrobin were undetectable in two of the sub-groups of hens from the low dose group, and in all sub-groups from the mid dose hens. In the third sub-group of hens from the low dose group, picoxystrobin was quantified at 0.027 mg/kg. This result is regarded as anomalous, since no residues were detected in liver samples from the next highest dose group, and it was discarded. Finally for the high dose group, picoxystrobin was not detected in two of the sub-groups, while being found at < 0.01 mg/kg in the third sub-group, and at 0.41 mg/kg in the outlying sub-group (the latter was discarded due to probable contamination). Picoxystrobin cleared rapidly from hen tissues, with no residues being detected in any of the muscle, fat or liver samples in the depuration phase of the study.

APPRAISAL

Picoxystrobin (ISO common name) is a strobilurin type fungicide for use by foliar application in a range of broadacre crops including cereals, sweet corn, soya bean, rape and pulses. At the Forty-third Session of the CCPR, picoxystrobin was scheduled for evaluation as a new compound by the 2012 JMPR. Data was provided on the metabolism of picoxystrobin in food producing animals and plants, methods of analysis, stability of residues in stored analytical samples, GAP information, supervised residue trials, processing and animal feeding studies.

The IUPAC name for picoxystrobin is methyl (*E*)-3-methoxy-2-[2-(6-trifluoromethyl-2-pyridyloxymethyl)phenyl]acrylate



The 2012 JMPR established an ADI of 0–0.09 mg/kg bw for picoxystrobin and an ARfD of 0.09 mg/kg bw.

The following abbreviations are used for the metabolites discussed below:

Code	Chemical name	Structure
IN-QDK50	6-(Trifluoromethyl)-1 <i>H</i> -pyridin-2-one	
IN-QDY62	(<i>E</i>)-3-Methoxy-2-[2-(6-trifluoromethyl-2-pyridyloxymethyl)-phenyl]acrylic acid	

Code	Chemical name	Structure
IN-QDY63	2-[2-(6-Trifluoromethyl-2-pyridyloxymethyl)]benzoic acid	
IN-QCD12	Methyl (Z)-3-methoxy-2-[2-(6-trifluoromethyl-2-pyridyloxymethyl)-phenyl]acrylate	
IN-H8612	1,3-Dihydro-3-oxoisobenzofuran-1-carboxylic acid	
IN-QDY60	Methyl (E)-3-methoxy-2-(2-hydroxymethylphenyl)acrylate	
IN-QGS46	2-Hydroxy-2-[2-(6-trifluoromethyl-2-pyridyloxymethyl)phenyl]acetic acid	
IN-QGU72	2-Malonylglucosyl-6-trifluoromethylpyridine	
IN-K2122	Phthalic acid	
PAG3	2-(2-Hydroxymethylphenyl)-2-oxoacetic acid	
-	2-(2-Formylphenyl)-2-oxoacetic acid	
IN-QFA35	2-[2-(6-Trifluoromethyl-2-pyridyloxymethyl)phenyl]acetic acid	
IN-QGU73	Mixture of isomers, where n = 3, 4 or 6 2-{n-(3-Hydroxy-3-methylglutaryl)glucosyl}-6-trifluoromethylpyridine	

Code	Chemical name	Structure
R290447	Methyl (E)-3-methoxy-2-[n-hydroxy-2-(6-trifluoromethyl-2-pyridyloxymethyl)-phenyl]acrylate	
IN-QCD09	Methyl 2-[2-(6-trifluoromethyl-2-pyridyloxymethyl)-phenyl]acetate	
R290461	Methyl 2,3-dihydroxy-2-[2-(6-trifluoromethyl-2-pyridyloxymethyl)-phenyl]propionate	
PYST2	6-Trifluoromethyl-2-pyridylsulfuric acid	
R409665, metabolite 30	2-(6-Trifluoromethyl-2-pyridyloxy)acetic acid	

Metabolism in animals

The Meeting received information on the metabolism of radio-labelled picoxystrobin (separately ^{14}C -labelled at the pyridinyl and phenacrylate rings) in rats, lactating goats and laying hens.

The metabolism of picoxystrobin in rats was evaluated by the WHO panel of the JMPR at the present Meeting. It was concluded that picoxystrobin is extensively metabolised with over 30 identified metabolites. Significant biotransformation reactions include ester hydrolysis, oxidation, O-demethylation and glucuronide conjugation.

Picoxystrobin was administered to lactating goats by capsule twice daily immediately after milking for 7 days at 10 and 13.5 ppm in feed for the pyridinyl and phenacrylate labels respectively (0.244 and 0.296 mg/kg bw/day).

The majority of the dose was excreted in urine (46–49%), and faeces (27–36%).

Residues in milk reached a plateau by day 4 (maximum TRR of 0.010–0.012 mg parent equivalents/kg (mg eq/kg)). A total of 0.06–0.20 % of the administered dose was recovered in milk. Total residues in muscle were 0.006–0.010 mg eq/kg. In fat, total residues were 0.021–0.034 mg eq/kg. In liver, total residues were 0.12–0.34 mg eq/kg, and kidney residues were 0.057–0.15 mg eq/kg. A total of 0.11–0.20% of the administered dose was recovered in liver, with 0.01–0.02% recovered in kidney.

Residues in milk and muscle were not characterised due to the low total residues.

Parent was present in fat, liver and kidney. In fat, parent was the only significant residue, at 55–81% of TRR and 0.012–0.024 mg eq/kg. Other compounds (unidentified) ranged from 0.002–0.004 mg eq/kg (5.2–20% TRR) in fat.

In liver, parent was only present at 0.003 mg eq/kg (1.0–2.7% TRR). A number of components were found; only IN-QDY62 and IN-QFA35, at 0.017 and 0.013 mg eq/kg respectively, exceeded 0.01 mg eq/kg, and no component exceeded 10% TRR.

Parent was found in kidney at 0.002–0.004 mg eq/kg (2.5–3.8% TRR). The only significant component was IN-QFA35, at 14–15% TRR (0.008–0.020 mg eq/kg).

IN-QDY62, a rat metabolite, was found in the faeces, urine and bile. IN-QFA35, another rat metabolite, was found in bile.

Hens were dosed for 10 days, at a mean dose of 11.3 and 10.9 ppm in feed for the pyridinyl and phenacrylate labels respectively (0.947 and 0.883 mg/kg bw/day).

The majority (65–94%) of the administered dose was excreted.

Residues in egg yolks and whites reached a plateau at 8–10 days, at 0.19–0.21 mg eq/kg for yolks and 0.006–0.015 mg eq/kg for whites. Total residues in muscle, fat and liver were 0.019–0.023, 0.027–0.070 and 0.16–0.31 mg eq/kg respectively. In yolks, 0.08–0.10% of the administered dose was recovered, compared with 0.01–0.02% in white, 0.04–0.05% in muscle, 0.01–0.02% in fat, and 0.07–0.14% in liver.

Only day 10 yolks were extracted and characterised. Parent was found in yolk (0.003–0.005 mg eq/kg, or 1.3–2.2% of TRR), along with three metabolites IN-QDK50, IN-QFA35 and IN-QCD09, none of which exceeded 0.01 mg eq/kg or 10% of the TRR.

All three of these metabolites, IN-QDK50 (urine), IN-QFA35 (bile), and IN-QCD09 (bile), are metabolites found in rats.

The metabolism of picoxystrobin was similar in lactating goats and laying hens. Important metabolic pathways were:

- Oxidative cleavage of the molecule at the ether bridge to yield IN-QDK50 and IN-QDY60. Only IN-QDK50 was found in hens, while both metabolites were found in goats.
- Hydrolysis of the methyl ester to IN-QDY62.
- Loss of the methoxy methyl group, with subsequent hydroxylation of the carbon side chain, hydrolysis of the methyl ester, and further cleavage of the side chain yielding IN-QDY63 as a terminal metabolite.
- Cleavage of the acrylate side chain at the 2 position to yield phenyl acetate metabolites, with or without subsequent hydrolysis of the methyl ester, and/or hydroxylation at the 2 position, yielding IN-QGS46 and IN-QFA35.
- Hydroxylation of the phenyl ring (R290447).

Metabolism in plants

Metabolism of ¹⁴C-pyridinyl- and ¹⁴C-phenacrylate-picoxystrobin was investigated in wheat, rape seed and soya bean.

Wheat (field grown) was treated twice by foliar application at Zadok's stages 32 and 65–69 at 405–437 g ai/ha, giving a total seasonal rate of 842 and 817 g ai/ha for the pyridinyl and phenacrylate labels respectively. Forage was harvested 14 days after the second application, with straw and grain being collected at normal harvest.

Parent was identified in grain (3.5–7.6% of TRR, 0.006–0.011 mg eq/kg). The only other components identified in grain were phthalic acid, IN-H8612 and PAG3 at 7.4%, 15%, and 7.9% (0.023, 0.046, and 0.024 mg eq/kg) respectively. Parent was the largest residue in forage (50–56% of TRR, 2.0–3.3 mg eq/kg) and straw (20–21% and 2.0–2.4 mg eq/kg). No other residue components exceeded 10% TRR in forage or straw, although a number of metabolites exceeded 0.01 mg eq/kg.

Phthalic acid, IN-H8612, and PAG3 were not found in rats.

Rape (greenhouse grown) was treated with two late season foliar applications at BBCH growth stages 80 and 85 with either the pyridinyl or the phenacrylate label at individual rates of 403–483 g ai/ha. Forage was sampled 7 days after the first application and 14 days after the second application, with remaining plant material and seed collected at normal harvest 21 days after the second application.

In all cases, parent was the most significant residue, at 80–96% of the TRR (5.6–9.9 mg eq/kg) in forage, 70–72% of TRR (8.3–9.4 mg eq/kg) in foliage at harvest, and 89–94% of TRR (1.5–2.3 mg eq/kg) in seed. All metabolites were < 10% of the TRR. The only other component identified in seed was *Z*-isomer (IN-QC12), at 0.6% TRR (0.02 mg eq/kg). In forage and dry plant material at harvest, *Z*-isomer, IN-QDY62, IN-QDY63, IN-QDK50 and its glucose conjugate were identified (maximum 7.4% TRR or 0.96 mg eq/kg). The small extent of metabolism of picoxystrobin in rape compared with wheat and soya bean is likely the result of the late application and the fact that the experiment was conducted in a greenhouse rather than in the field.

IN-QDY62 (faeces, urine and bile), IN-QDY63 (bile) and IN-QDK50 (urine) are all rat metabolites.

Soya bean (field grown) was treated with ¹⁴C-pyridinyl or ¹⁴C-phenacrylate-labelled picoxystrobin. Two foliar applications were made at BBCH 69 and 73–75 to give target seasonal rates of 200 g ai/ha. Foliage (hay) samples were collected 14 days after the second application, with dry stalks and seed collected at normal harvest.

Parent was found in seed (1.5–5.9% TRR, or 0.002–0.004 mg eq/kg). In forage, parent was significant at 7.4–10% TRR (0.13–0.18 mg eq/kg). In seed, only phthalic acid (INK2122) and 2-(2-formylphenyl)-2-oxoacetic acid (R730529) were found at levels above 10% TRR and 0.01 mg eq/kg (21% TRR/0.030 mg eq/kg and 26% TRR/0.036 mg eq/kg respectively). Other significant residues in forage included the glucose conjugate of IN-QGS46 (8.4–14%, or 0.14–0.26 mg eq/kg, mixed glucose conjugates of R290461 (total 26–31%/0.44–0.55 mg eq/kg and malonyl glucose conjugate of R290461 (10%/0.18 mg eq/kg).

Phthalic acid and 2-(2-formylphenyl)-2-oxoacetic acid are not rat metabolites. IN-QGS46 (bile and urine) and R290461 (urine) are rat metabolites.

The major metabolic pathways for picoxystrobin in plants were:

- Oxidative cleavage of the molecule at the ether bridge to yield IN-QDK50 and IN-QDY60. IN-QDK50 was subsequently conjugated with glucose and malonic or glutaric acid, while the phenacrylate cleavage product was subject to further oxidation and cleavage giving phthalic acid or IN-H8612;
- Loss of the methoxy methyl group followed by reduction of the enol, further hydroxylation of the side chain, and conjugation of the hydroxyl groups with glucose and malonic acid (R290461 and conjugates); and
- Hydrolysis of the ester, followed by oxidation and cleavage of the acrylate moiety ultimately yielding the benzoic acid metabolite IN-QDY63 or a phenyl-acetic acid metabolite (IN-QFA35), with or without glucose conjugation of the hydroxyl or carboxylic acid functionalities.

Hydroxylation of the phenyl ring was also observed in wheat, while small amounts of the *Z*-isomer of picoxystrobin (IN-QCD12) were found in rape and wheat.

Environmental fate

The Meeting received information on the aerobic degradation of picoxystrobin in soil, photolysis on the soil surface, field dissipation in soil, hydrolysis, aqueous photolysis, and metabolism in rotational cropping (both field and confined).

Aerobic metabolism of picoxystrobin in the dark was studied in various soil types at 20 °C. The DT₅₀ values were 16–38 days, with DT₉₀ values of 76–337 days. The major degradation pathways

were ester hydrolysis, cleavage of the ether bridge to give IN-QDK50 (subsequently methylated), and mineralisation to carbon dioxide.

Picoxystrobin applied to thin layers of soil and irradiated for a period equivalent to 30 summer days at 50 ° latitude degraded rapidly with a DT_{50} of 7 days. The major degradation pathways were cleavage of the ether bridge and methyl acrylate moiety, yielding IN-QDK50 and phthalic acid, and finally mineralisation to carbon dioxide.

Microbial and photolytic degradation are both significant for picoxystrobin in/on soil.

Field dissipation studies for picoxystrobin were conducted in France, Germany, the UK, Canada and the USA. Degradation was relatively rapid (DT_{50} = 1.3–35 days, DT_{90} = 42–437 days). Metabolite levels were low, often below the limit of quantification, and less than parent. There was no evidence of accumulation of parent or metabolites.

Residues on succeeding crops

Rotational crop metabolism studies were conducted for ^{14}C -pyridinyl- and ^{14}C -phenacrylate-labelled picoxystrobin.

In one field rotation study, spring wheat, lettuce and carrot were sown 304–308 days after final application of radiolabelled compound at seasonal rates of 820–888 g ai/ha. The second field study involved winter wheat sown 107 days after the second of two foliar applications of labelled compound at a seasonal rate of 817–842 g ai/ha.

Picoxystrobin breaks down relatively rapidly in soil, and does not accumulate to a significant extent in following crops. Total residues did not exceed 0.01 mg eq/kg in wheat grain, lettuce and carrot roots from the field rotational studies. In wheat forage and straw and carrot leaves in the field studies, the most significant component was IN-QDK50 and conjugates, with a maximum total of 0.058 mg eq/kg (35–63% TRR), with free IN-QDK50 comprising only 0.002–0.006 mg eq/kg, or 2.0–6.9% TRR. No other components exceeded 0.01 mg eq/kg, or 10% TRR in any of the field rotational crop matrices. IN-QDK50 is a rat metabolite, found in urine.

Residues of picoxystrobin or its metabolites in following crops are therefore unlikely to be significant.

Methods of analysis

The Meeting received details of analytical methods for picoxystrobin residues in plant and animal matrices.

Analysis of picoxystrobin and the metabolites IN-QDK50, IN-QDY62 and IN-QDY63 in plant matrices involved extraction with acetonitrile/water, solid phase extraction clean-up, and GC/MS or LC/MS/MS analysis. LOQs are 0.01 mg/kg.

Methods were developed for analysis of parent in animal matrices. Samples were extracted with acetonitrile and in some cases cleaned up by solid phase extraction clean-up, with analysis by GC/MS or LC/MS/MS. LOQs are 0.01 mg/kg.

The suitability of the US FDA Pesticide Analytical Manual, Volume I (PAM I 3rd edition) protocols was assessed, with the GC method being found suitable for analysis of parent only in fatty and non-fatty plant matrices (apple and soya bean).

Suitable single residue analytical methods therefore exist for parent and the metabolites IN-QDK50, IN-QDY62 and IN-QDY63 in plant matrices, and for parent in animal matrices. A multi-residue method has been validated for the determination of parent only in plant matrices.

Stability of residues in stored analytical samples

Storage stability of picoxystrobin and the metabolites IN-QDK50, IN-QDY62 and IN-QDY63 in a range of plant commodities including high (apples, apple juice and lettuce), medium (wheat forage and apple pomace) and low (wheat straw and soya bean meal) water content, high acid (grapes), high

protein (dry pea), high starch (potato), and high oil (soya bean seed and refined oil) content was assessed for samples stored frozen for 24 months. With the exception of the metabolites IN-QDY62 and IN-QDY63 in soya bean oil, which were stable for 18 and 6 months respectively, all analyte/sample combinations were stable for 24 months frozen storage.

The stability of residues of picoxystrobin in animal commodity samples over the period of storage in the feeding studies was acceptable.

Definition of the residue

Total residues in milk and muscle were very low (≤ 0.012 mg eq/kg). In goat fat, parent was the only identified component, at 55–81% of the TRR and 0.012–0.024 mg/kg. In liver and kidney, parent was present at low levels (1.0–3.8% TRR; 0.002–0.004 mg eq/kg). The only components in liver > 0.01 mg eq/kg were IN-QDY62 and IN-QFA35 at 0.017 and 0.035 mg/kg respectively. No components exceeded 10% of the TRR in liver. In kidney, only IN-QFA35 (14–15% TRR, or 0.008–0.020 mg/kg) was significant. In egg yolks, no components were found at $> 10\%$ of TRR or 0.01 mg eq/kg. Both IN-QFA35 and IN-QDY62 are also metabolites found in rats.

As parent was the only identified residue in fat, and was found in all analysed animal tissues, it is a suitable marker compound for analysis. A residue definition of parent compound only is proposed for picoxystrobin in animal commodities for both compliance and risk assessment purposes.

The octanol-water partition coefficient ($\log_{10}K_{OW}$) for picoxystrobin is 3.7. In the cattle feeding study at the highest feeding level, mean residues of picoxystrobin were < 0.01 mg/kg in muscle, compared with 0.028 mg/kg in subcutaneous fat. Residues were undetectable in skim milk, with a mean level of 0.026 mg/kg in cream. The Meeting concluded that picoxystrobin residues are fat soluble.

In oilseed rape, the major component was parent at 89–94% of the TRR (1.5–2.3 mg eq/kg) in seed, and 70–96% of the TRR (5.6–9.9 mg eq/kg) in foliage. In wheat, parent was the only significant component in forage and straw (20–55% TRR, 2.0–3.3 mg eq/kg), and was found in grain (3.5–7.6% TRR, 0.006–0.011 mg eq/kg). In soya bean, parent was found at low levels in seed (1.5–5.9% TRR, 0.002–0.004 mg eq/kg). Parent was present at 0.13–0.18 mg eq/kg (7.4–10% TRR) in soya bean forage.

Other identified components in wheat grain were phthalic acid (IN-K2122) at 7.4% TRR, 0.023 mg eq/kg, PAG3 (7.9% of TRR, 0.024 mg eq/kg), and IN-H8612 (15% TRR, 0.046 mg eq/kg). In soya bean, only phthalic acid (21% TRR, 0.030 mg eq/kg) and 2-(2-formylphenyl)-2-oxoacetic acid (26% TRR, 0.036 mg eq/kg) were significant for seed. In soya bean forage, residue profiles were qualitatively similar to those for seed. IN-QGS46-glucoside was present at 0.26 mg eq/kg (14% TRR), with R290461-glucosides at 31% (0.55 mg eq/kg), and R290461 malonyl glucoside at 10% TRR (0.18 mg eq/kg).

Total residues did not exceed 0.01 mg eq/kg in wheat grain, lettuce and carrots from the field rotation studies. In wheat forage and straw and carrot leaves, the only significant (> 0.01 mg eq/kg, $> 10\%$ TRR) residue was IN-QDK50 and conjugates, which reached a total of 0.058 mg eq/kg (35–63% of TRR), with free IN-QDK50 comprising only 0.002–0.006 mg eq/kg, or 2.0–6.9% TRR. Picoxystrobin breaks down relatively rapidly in soil, and does not accumulate significantly in following crops. Residues of picoxystrobin or its metabolites in following crops are therefore unlikely to be significant, and inclusion of metabolites in the residue definition for rotational crops is not necessary, especially as IN-QDK50 is a metabolite found in rats.

The Meeting concluded that phthalic acid is not a toxicologically relevant metabolite, while PAG3 and IN-QDY63 were not of toxicological concern at the estimated dietary intake levels.

The International Estimate Daily Intake (IEDI) of IN-H8612 was above 0.15 $\mu\text{g}/\text{person}/\text{day}$, the Threshold of Toxicological Concern (TTC) for a compound with evidence of genotoxicity. The Meeting was unable to conclude on the toxicological relevance of the estimated intakes of IN-H8612.

2-(2-Formylphenyl)-2-oxoacetic acid is not supported by any toxicological studies but a structural alert for genotoxicity was identified. The IEDI was above 0.15 µg/person/day, the TTC for a compound with a structural alert for genotoxicity. The Meeting was unable to conclude on the toxicological relevance of the estimated intakes of 2-(2-formylphenyl)-2-oxoacetic acid.

Conjugated compounds (such as those of IN-QDK50, IN-QGS46 or R290461) are not suitable for inclusion in the residue definition, as their analysis requires specialised analytical methods incorporating enzymatic digestion or hydrolysis steps. IN-QDK50 is a metabolite in the rat.

Given that parent is the major component of the residue in many plant matrices (rape seed and forage, and wheat forage and straw), and was found in all other plant matrices tested, it is the most suitable marker compound for analysis of picoxystrobin residues. A residue definition of parent compound is proposed for plant matrices for the purposes of compliance.

Because the Meeting was unable to conclude on the toxicological relevance of the metabolites IN-H8612 and 2-(2-formylphenyl)-2-oxoacetic acid, the Meeting could not reach a conclusion on a residue definition for dietary risk assessment.

Residue definition for picoxystrobin in plant and animal commodities (for compliance with maximum residue levels): *picoxystrobin*.

Residue definition for picoxystrobin in plant and animal commodities (for dietary risk assessment): *a conclusion could not be reached*.

Picoxystrobin residue is fat-soluble.

Residues of supervised trials on crops

The Meeting received supervised trial data for application of picoxystrobin on sweet corn, peas (dry), beans (dry), soya bean (dry), wheat, barley and rape seed conducted in the USA and Canada. The OECD MRL calculator was used as a tool to assist in the estimation of maximum residue levels from the selected residue data set obtained from the supervised residue trials. As a first step, the Meeting reviewed all relevant factors related to each data set in arriving at a best estimate of the maximum residue level using expert judgement. Then the OECD MRL calculator was employed. If the statistical calculation spreadsheet suggested a different value from that estimated by the Meeting, a brief explanation of the deviation was supplied.

In all trials, duplicate field samples were collected at each sampling interval and separately analysed. The mean result of the duplicate analyses was taken as the best estimate of the residue.

Labels were available from Canada, describing the registered uses of picoxystrobin.

Sweet corn

Picoxystrobin is registered in Canada for use in sweet corn at a GAP of 4 × 0.22 kg ai/ha and a 7 day PHI. The Canadian use pattern constitutes the critical GAP for sweet corn.

Eleven trials were conducted in sweet corn at GAP in the USA and Canada. Residues in sweet corn cobs at the 7 day PHI were < 0.01 (11) mg/kg.

The meeting estimated a maximum residue level of 0.01* mg/kg for picoxystrobin in sweet corn (corn-on-the-cob), together with a median residue and a highest residue both at 0.01 mg/kg.

Pulses

Picoxystrobin is registered in pulses except soya bean (chickpea, lentil, guar bean, lablab bean, broad bean (dry), pigeon, pea, lupin, field bean, kidney bean, lima bean, navy bean, pinto bean, tepary bean, adzuki bean, black-eyed pea, catjang, cowpea, crowder pea, moth bean, mung bean, rice bean, southern pea, urd bean, and field pea) in Canada, at a maximum rate of 2 × 0.22 kg ai/ha with a 14 day PHI for harvest for human consumption.

Eleven trials were conducted in peas (dry) and eleven in beans (dry) in the USA and Canada and were evaluated against the Canadian GAP.

Residues in pea seed at the Canadian GAP were: < 0.01 (4), 0.010, 0.012, 0.013, 0.016 (2), 0.025 and 0.033 mg/kg. Residues in bean seed at the 14 day PHI were: < 0.01 (6), 0.011 (2), 0.016 and 0.038 (2) mg/kg.

Given the similarity of the data sets (confirmed by the Mann-Whitney U test), and the identical GAPs, the Meeting decided to combine the data sets for peas (dry) and beans (dry) for the purposes of determining a group maximum residue level. Residues were: < 0.01 (10), 0.010, 0.011 (2), 0.012, 0.013, 0.016 (3), 0.025, 0.033, and 0.038 (2) mg/kg.

The Meeting estimated a maximum residue level of 0.06 mg/kg for pulses (except soya bean), along with a median residue of 0.0105 mg/kg.

Picoxystrobin is registered in soya bean in Canada at a GAP of 3×0.22 kg ai/ha and a 14 day PHI. The Canadian use pattern represents the critical GAP for picoxystrobin in soya bean.

Twenty trials were conducted in soya bean in the USA and Canada and were assessed against the Canadian GAP. Residues in soya bean (dry) at the 14 day PHI were: < 0.01 (13), 0.010, 0.011, 0.012, 0.019, 0.031, 0.035, and 0.039 mg/kg.

The Meeting estimated a maximum residue level of 0.06 mg/kg for soya bean (dry), with a median residue of 0.01 mg/kg.

Cereal grains

In Canada, picoxystrobin is registered in cereal grains: wheat, barley, oats, rye, and triticale at a GAP of 3×0.22 kg ai/ha, with a PHI of 45 days.

Twenty-three trials were conducted in wheat in the USA and Canada and were assessed against the GAP of Canada. Residues in wheat grain from trials matching Canadian GAP were: < 0.01 (15), 0.010 (2), 0.013, 0.014, 0.019, 0.022, 0.025, and 0.028 mg/kg.

Seventeen trials were conducted in the USA and Canada in barley and were assessed against the Canadian GAP. Residues in barley grain from trials matching the Canadian GAP were: < 0.01 (4), 0.011, 0.014, 0.016 (2), 0.017, 0.022, 0.028 (2), 0.029, 0.047, 0.087, 0.12, and 0.22 mg/kg.

The Meeting decided that the residue data sets for wheat and barley were not sufficiently similar to combine for the purposes of establishing a group maximum residue level for cereal grains.

The Meeting estimated a maximum residue level of 0.04 mg/kg for picoxystrobin in wheat, with a median residue of 0.01 mg/kg.

Given the GAPs in Canada are the same for wheat, rye and triticale and the similarity of the crops, the Meeting decided to extrapolate from the wheat residue data to estimate maximum residue levels of 0.04 mg/kg for picoxystrobin in rye and triticale, with median residues of 0.01 mg/kg.

The Meeting estimated a maximum residue level of 0.3 mg/kg for picoxystrobin in barley, with a median residue of 0.017 mg/kg.

Given the GAPs are the same for barley and oats and the similarity of the crops, the Meeting decided to extrapolate from the barley residue data to estimate a maximum residue level of 0.3 mg/kg for picoxystrobin in oats, with a median residue of 0.017 mg/kg.

Picoxystrobin is registered in Canada for use in maize (field, seed and popcorn), with a GAP of 3×0.22 kg ai/ha, and a 7 day PHI.

Fifteen trials were conducted in maize at GAP in the USA and Canada. Residues in maize grain matching the Canadian GAP were: < 0.01 (13), 0.011, and 0.012 mg/kg.

The Meeting estimated a maximum residue level of 0.02 mg/kg for picoxystrobin in maize, together with a median residue of 0.01 mg/kg. The OECD MRL calculator yielded a value of

0.015 mg/kg. A higher limit than that generated by the calculator was chosen, noting the high level of censoring in the data set.

Rape seed

Seventeen trials were conducted in oilseed rape in the USA and Canada but were not according to a registered GAP. As a result the Meeting was unable to make a maximum residue level recommendation.

Animal feeds

Sweet corn forage

The GAP for sweet corn in Canada is 4×0.22 kg ai/ha, with a 0 day grazing interval. Residue data for sweet corn forage was collected for the USA and Canadian sweet corn trials. However, most samples were collected 7 days after treatment, which is not consistent with Canadian GAP.

Residues in sweet corn forage at 0 days after treatment (DAT) were 8.4 and 17 mg/kg.

The Meeting concluded that there were insufficient data points to estimate a highest residue and a median residue value for sweet corn forage.

Soya bean forage and hay

The Canadian GAP for soya bean (when forage is to be grazed or hay is to be harvested) is 1×0.22 kg ai/ha with a 14 day PHI.

Residue data for soya bean forage and hay were collected for the USA and Canadian soya bean residue trials.

At a 14 day PHI, residues of picoxystrobin in soya bean forage were: < 0.01, 0.25, 0.46, 0.57 (2), 0.80, 0.84, 0.88, 0.93, 1.4, 1.6 (3), 1.9, 2.0 (2), 2.1, 2.9, and 3.5 mg/kg (dry weight basis).

Residues of picoxystrobin in soya bean hay on a dry weight basis at the same interval were: < 0.01, 0.14, 0.39, 0.50, 0.51, 0.52, 0.59, 0.73, 0.81, 1.2, 1.6 (2), 1.7 (2), 1.8, 2.0, 2.1, 2.3 and 2.7 mg/kg.

The Meeting estimated a maximum residue level of 5 mg/kg for picoxystrobin in soya bean fodder, together with a median residue and a highest residue of 1.2 and 2.7 mg/kg respectively. The Meeting estimated a median residue and a highest residue of 1.4 and 3.5 mg/kg respectively for soya bean forage (dry weight).

Pea vines and hay

The GAP for picoxystrobin in pulses (except soya bean) in Canada is 2×0.22 kg ai/ha, with a 0 day PHI for vines (forage) and hay.

Data for pea vines and pea hay were collected for selected sites in the USA and Canadian pulse residue trials.

At a 0 day PHI, residues of picoxystrobin in pea vines were: 9.5, 14, 19, 22, 35 and 55 mg/kg (dry weight basis).

Residues of picoxystrobin in pea hay on a dry weight basis at the same interval were: 4.1, 7.1, 11, 14, 18, and 64 mg/kg.

The Meeting estimated a maximum residue level of 100 mg/kg for picoxystrobin in pea hay or pea fodder (dry), noting the value of 150 mg/kg estimated by the OECD MRL calculator. However, the Meeting agreed that 100 mg/kg represented a more realistic estimate of the maximum residue expected in pea fodder treated in accordance with GAP.

The highest residue and median residue values for pea hay are 64 and 12.5 mg/kg respectively (dry weight basis). The Meeting estimated a highest residue and a median residue value for pea vines of 55 and 20.5 mg/kg respectively (dry weight basis).

Wheat, barley, oat, rye and triticale forage, hay and straw

The Canadian GAP for wheat, barley, oat, rye and triticale forage is 1×0.22 kg ai/ha, with a 7 day grazing interval. The Canadian GAP for wheat, barley, oat, rye and triticale hay is 3×0.22 kg ai/ha, with a 14 day PHI. The Canadian GAP for wheat, barley, rye, oat and triticale straw is 3×0.22 kg ai/ha, with a 45 day PHI.

Residue data for wheat forage, hay and straw, and barley hay and straw were generated in the USA and Canada in accordance with the Canadian GAP.

Residues of picoxystrobin in wheat forage at a 7 day PHI were: 1.1, 1.3, 1.6, 1.7, 1.9, 2.2, 2.3, 3.6 (2), 3.7, 3.8, 3.9, 4.5, 4.6, 4.8, 6.3, 6.4, 7.0, 7.4, 8.9, 9.7, 11 (2), 12, and 31 mg/kg (dry weight basis).

Residues of picoxystrobin in wheat hay at a 14 day PHI were: 0.18, 0.19, 0.24, 0.41, 0.48, 0.51, 0.61, 0.68, 0.72, 0.78, 0.81, 0.90, 1.0, 1.1 (2), 1.4, 1.5, 1.7, 1.8, 2.4, 2.5, 2.8, 3.4, 3.6, and 4.0 mg/kg (dry weight basis).

Residues of picoxystrobin wheat straw at a 45 day PHI were: < 0.01, 0.016, 0.022 (2), 0.029, 0.033, 0.043, 0.079, 0.10 (2), 0.15, 0.28, 0.29, 0.32, 0.36, 0.49, 0.50, 0.52, 0.62, 0.86, 1.2 (2), and 1.7 mg/kg (dry weight basis).

Residues of picoxystrobin in barley hay at a 14 day PHI were: 0.20, 0.32, 0.34, 0.38, 0.39, 0.46, 0.55, 0.66, 0.77, 0.78, 0.86, 1.3, 1.4, 1.7 (2), 2.3, 2.4, 3.5, and 5.5 mg/kg (dry weight basis).

Residues of picoxystrobin in barley straw at a 45 day PHI were: 0.049, 0.050, 0.066, 0.069, 0.082, 0.087, 0.13, 0.22, 0.23, 0.24, 0.28, 0.35, 0.40, 0.41, 0.80, and 1.2 mg/kg (dry weight basis).

A median residue value and a highest residue value of 4.5, and 31 mg/kg respectively were estimated for wheat forage for use in livestock dietary burden calculations. The Meeting agreed that these values could be extrapolated to barley, oat, rye and triticale forage for the purposes of the livestock dietary burden calculations.

Hay and straw of different cereal grains are generally indistinguishable in trade.

The Meeting determined that the residue data sets for wheat and barley hay and for wheat and barley straw were similar (Mann-Whitney U-test).

The Meeting agreed to combine the data sets for wheat and barley hay for the purposes of estimating maximum residue levels for cereal fodders. The combined data set for wheat and barley hay were: 0.18, 0.19, 0.20, 0.24, 0.32, 0.34, 0.38, 0.39, 0.41, 0.46, 0.48, 0.51, 0.55, 0.61, 0.66, 0.68, 0.72, 0.77, 0.78 (2), 0.81, 0.86, 0.90, 1.0, 1.1 (2), 1.3, 1.4 (2), 1.5, 1.7 (3), 1.8, 2.3, 2.4 (2), 2.5, 2.8, 3.4, 3.5, 3.6, 4.0, and 5.5 mg/kg.

The Meeting agreed to combine the data sets for wheat and barley straw for the purposes of estimating median and highest residue values for cereal straws. The combined data set for wheat and barley straw were: < 0.01, 0.016, 0.022 (2), 0.029, 0.033, 0.043, 0.049, 0.050, 0.066, 0.069, 0.079, 0.082, 0.087, 0.10 (2), 0.11, 0.13, 0.15, 0.22, 0.23, 0.24, 0.28 (2), 0.29, 0.32, 0.35, 0.36, 0.40, 0.41, 0.49, 0.50, 0.52, 0.62, 0.80, 0.86, 1.2 (3), and 1.7 mg/kg.

Using the combined wheat and barley hay data set, the Meeting estimated maximum residue levels of 7 mg/kg for barley straw and fodder, dry and for wheat straw and fodder, dry, with median and highest residue values of 0.88 and 5.5 mg/kg (dry weight basis) respectively, for wheat and barley hay.

The Meeting agreed that the combined data set for barley and wheat hay could be extrapolated to the other cereal crops with the same GAP in Canada and estimated maximum residue

levels of 7 mg/kg for oat straw and fodder, dry, for rye straw and fodder, dry, and for triticale straw and fodder, dry.

The Meeting estimated median and highest residue values of 0.88 mg/kg and 5.5 mg/kg (dry weight basis) respectively for oat hay, rye hay and triticale hay, using the barley and wheat hay data set.

Using the combined wheat and barley straw data set, the Meeting estimated median and highest residue values of 0.225 and 1.7 mg/kg (dry weight basis) respectively, for wheat and barley straw.

The Meeting estimated median and highest residue values of 0.225 and 1.7 mg/kg (dry weight basis) for oat straw, rye straw and triticale straw, using the barley and wheat straw data set.

Maize forage and stover

The GAP for picoxystrobin in maize in Canada is 3×0.22 kg ai/ha, with a 0 day PHI for grazing of forage, and a 7 day PHI for grain and stover.

Residue data for maize forage and maize stover were collected for the USA and Canadian trials.

Residues in maize forage in accordance with the Canadian GAP were: 3.5, 4.6, 5.0, 5.7, 6.2, 6.3, 6.7, 7.1, 8.0, 8.5, 9.7, 11, 12, 13, and 14 mg/kg (dry weight basis).

Residues in maize stover in accordance with the Canadian GAP were: 0.023, 0.94, 1.0, 2.1, 2.2, 3.2, 3.5, 3.8, 5.7, 6.0, 6.6, 7.4, 8.2, 8.5 and 8.6 mg/kg (dry weight basis).

A median and a highest residue value of 7.1, and 14 mg/kg (dry weight) respectively were estimated for maize forage for use in livestock dietary burden calculations.

The Meeting determined a maximum residue level of 20 mg/kg for picoxystrobin in maize fodder, together with a median and a highest residue of 3.8 and 8.6 mg/kg (dry weight) respectively.

Processing studies

Processing studies were conducted in wheat, barley, soya bean, and maize. Processing factors are tabulated below.

Raw agricultural commodity (RAC)	Processed commodity	Processing factors	Best estimate processing factor	RAC median residue (mg/kg)	RAC MRL (mg/kg)	Processed commodity median residue (mg/kg)	PF × RAC MRL, where required
Barley	Beer	< 0.05, < 0.25 (2), < 0.5	0.26			< 0.01	–
	Spent grain	0.5, 0.81	0.66			0.011	–
Wheat	Bran	1.9, 2.1, 3.0, 3.8	2.7	0.01	0.04	0.027	0.108
	Germ	2.6, 3.8	3.2			0.032	0.128
	Wholemeal flour	1.1, 1.3	1.2			0.012	–
	Flour	0.21, 0.26	0.24			< 0.01	–
	Type 550 (white) flour	0.83, 1.1	0.97			< 0.01	–
	Patent flour	1.1, 1.2	1.2			0.012	–

Raw agricultural commodity (RAC)	Processed commodity	Processing factors	Best estimate processing factor	RAC median residue (mg/kg)	RAC MRL (mg/kg)	Processed commodity median residue (mg/kg)	PF × RAC MRL, where required
	Wholemeal bread	0.45, 1.0	0.73			< 0.01	–
	Type 550 (white) bread	0.64, 0.67	0.66			< 0.01	–
	Screenings	1.7, 5.1	3.4			0.034	–
Soya bean	Refined oil (solvent extracted)	0.93, 1.0, 1.6, 2.2	1.4	0.01	0.06	0.014	0.084
	Refined oil (mechanically extracted)	3.4, 3.4	3.4			0.034	0.204
	Meal (solvent extracted)	0.03, 0.06, < 0.09, 1.1	0.32			< 0.01	–
	Meal (mechanically extracted)	0.36, 0.60	0.48			< 0.01	–
	Aspirated grain fractions	190, 320	260			2.6	–
	Hulls	2.2, 4.4, 5.1, 5.6	4.3			0.043	–
Maize	Starch	0.025, < 0.068	0.047	0.01	0.02	< 0.01	–
	Grits	0.34, 0.51	0.43			< 0.01	–
	Flour	1.0, 1.2	1.1			0.011	–
	Refined oil (wet milled)	6.4, 7.3	6.9			0.069	0.138
	Refined oil (dry milled)	3.4, 5.4	4.4			0.044	0.088
	Meal	0.77, 0.79	0.78			< 0.01	–
	Aspirated grain fractions	13, 17	15			0.15	

Picoxystrobin concentrated significantly in wheat bran, wheat germ, soya bean refined oil, and maize refined oil.

The Meeting therefore estimated maximum residue levels of 0.15, 0.15, 0.2, and 0.15 mg/kg for wheat bran, processed, wheat germ, soya bean oil, refined, and maize oil, edible, respectively, based on the best estimate processing factors and the raw agricultural commodity maximum residue levels.

Residues in animal commodities

Farm animal dietary burden

The Meeting estimated the dietary burden of picoxystrobin in farm animals on the basis of the diets listed in Appendix IX of the FAO Manual 2009. Calculation from highest residue, median residue (some bulk commodities), and median processed commodity residue values provides levels in feed suitable for estimating maximum residue levels. The percentage dry matter is taken as 100% when the highest residue levels and median residue levels are already expressed on a dry weight basis.

	US/Canada, maximum	EU, maximum	Australia, maximum	Japan, maximum
Beef cattle	2.29	31.6	64 ^a	0.029
Dairy cattle	18.2	32.7	54.1 ^b	7.87
Poultry (broiler)	0.028	0.026	0.02	0.004
Poultry (layer)	0.028	9.52 ^{c,d}	0.02	0.02

^a Maximum calculated dietary burden for beef cattle, used for calculation of mammalian tissue maximum residue levels.

^b Maximum calculated dietary burden for dairy cattle, used for calculation of the milk maximum residue level.

^c Maximum calculated dietary burden for laying hens, used for calculation of egg maximum residue level.

^d Maximum calculated dietary burden for broiler hens, used for calculation of poultry tissue maximum residue levels.

The detailed dietary burden calculations are provided in Annex 6.

Animal feeding studies

Lactating cattle were dosed orally twice daily with picoxystrobin for 29 days at 39.7, 119.5, and 402.8 ppm in feed or 1.35, 4.12 and 12.9 mg/kg bw/day.

Picoxystrobin was not detected in whole milk from the low and mid-dose groups. Low levels (maximum 0.014 mg/kg), were found in some high-dose group samples. Milk residues reached a maximum around day 14. No residues were detected in skim milk, with levels in cream of 0.016–0.048 mg/kg for the high-dose group.

Picoxystrobin was not detected in muscle and kidney for the low or mid-dose groups, was found at < 0.01 mg/kg in muscle and 0.010 mg/kg in kidney for the high-dose group. Residues were detected in liver and fat at all doses. A roughly linear relationship between dose and residue was observed for liver and fat. The maximum residue at the high dose level was 0.10 mg/kg and 0.077 mg/kg for liver and fat (omental) respectively.

Depuration data indicated rapid clearance of residues from milk and tissues. No residues were detected in milk, muscle, perirenal fat or kidney from the depuration animals. Liver residues were undetectable by 8 days after the final dose, and were below the limit of quantification in fat (subcutaneous) by 3 and 15 days.

Laying hens were dosed orally daily with picoxystrobin for 36 days at 15.1, 45.4, 153 (main high-dose group) and 152 (depuration group) ppm in feed, or 0.97, 2.84, 9.49 and 9.53 mg/kg bw/day respectively. No residues were detected in eggs from the low and mid-dose group. In the high-dose group, residues in eggs reached a maximum of 0.014 mg/kg.

In fat, picoxystrobin was below the limit of quantification in the low dose group, while in the mid-dose group residues up to 0.010 mg/kg were found. Fat residues for the high-dose group reached a maximum of 0.016 mg/kg. In muscle, no residues were detected for the low or mid-dose groups, and were below the limit of quantification in the high-dose group. In liver, residues were undetectable in the low and mid-dose groups, and were below the limit of quantification in the high-dose group.

Picoxystrobin cleared rapidly from hen eggs and tissues, with no residues being detected in any samples after depuration day 2.

Animal commodity maximum residue levels

Mammals

The maximum dietary burdens for beef and dairy cattle are 64 and 54 ppm dry weight in feed respectively. Highest residue values calculated by interpolation or using transfer factors for picoxystrobin in mammalian animal matrices are tabulated below.

Feed level	Residues	Feed level	Residues (mg/kg)
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	(ppm) for milk residues	for (mg/kg) in milk	in (ppm) for residues	tissue	Muscle	Liver	Kidney	Fat
Highest residue determination (beef or dairy cattle)								
Feeding study	120	< 0.01	120		< 0.01	0.017	< 0.01	0.026
	40	< 0.01	40		< 0.01	< 0.01	< 0.01	< 0.01
Dietary burden and estimate of highest residue	54	0	64		0	0.012	0	0.015

Residues of picoxystrobin were not detected in milk from cattle at the two feeding levels bracketing the calculated maximum dietary burden for dairy animals. The Meeting therefore estimated a maximum residue level of 0.01* mg/kg for picoxystrobin in milk.

Residues of picoxystrobin were not detected in muscle or kidney from cattle at the two feeding levels bracketing the calculated maximum dietary burden for beef cattle. Residues were found at low levels above the LOQ in fat and liver of cattle at the next highest feeding level above the maximum dietary burden for beef cattle, and were below the LOQ for the next lowest feeding level.

The Meeting therefore estimated maximum residue levels of 0.02 mg/kg for edible offal (mammalian), meat (from mammals other than marine mammals) (fat), and mammalian fats (except milk fats).

Poultry

The maximum dietary burdens for broiler chickens and laying hens 9.5 ppm dry weight in feed. Highest residue values calculated by interpolation or using transfer factors for picoxystrobin in poultry animal matrices are tabulated below.

	Feed level	Residues	Feed level	Residues (mg/kg)			
	(ppm) for egg residues	(mg/kg) in egg	(ppm) for tissue residues	Muscle	Liver	Fat	
Highest residue determination (broiler or laying hens)							
Feeding study	15	< 0.01	15	< 0.01	< 0.01	< 0.01	
Dietary burden and estimate of highest residue	9.5	0	9.5	0	0	< 0.01	

Residues of picoxystrobin were not detected in the eggs, muscle or liver of hens fed at the next highest feeding level (15 ppm) above the maximum poultry dietary burden (9.5 ppm). Residues were detectable, but below the LOQ, in the fat of birds fed at 15 ppm.

The Meeting therefore estimated maximum residue levels of 0.01* mg/kg for picoxystrobin in eggs, poultry meat, and poultry, edible offal of. The Meeting estimated a maximum residue level of 0.01 mg/kg for picoxystrobin in poultry fats.

RECOMMENDATIONS

No maximum residue levels are recommended, nor are levels estimated for use for IEDI or IESTI assessment as the Meeting could not reach a conclusion on a residue definition for dietary risk assessment.

Definition of the residue for compliance with the MRL for animal and plant commodities: *picoxystrobin*.

The residue is fat soluble.

Residue definition for picoxystrobin in plant and animal commodities (for dietary risk assessment): *a conclusion could not be reached.*

DIETARY RISK ASSESSMENT

Because the Meeting was unable to conclude on the toxicological relevance of the metabolites IN-H8612 and 2-(2-formylphenyl)-2-oxoacetic acid, the Meeting could not reach a conclusion on a residue definition for dietary risk assessment.

As a result, long- and short-term dietary intake assessments could not be conducted.

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