CYPROCONAZOLE (239)

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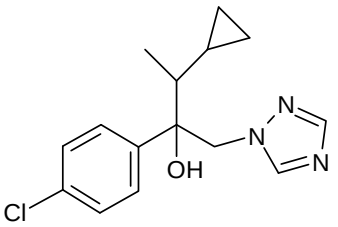
EXPLANATION

Cyproconazole is an azole fungicide used to control a wide range of fungi on cereal crops, coffee, sugar beet, fruit trees, grapes, including rust on cereal crops, powdery mildew on cereal crops, fruit tree and grapes, and scab on apple. It is both a prevention and treatment fungicide. At the 41st session of the CCPR (2009), it was scheduled for the evaluation as a new compound by the 2010 JMPR.

The residue studies were submitted by the manufacturer for support of the following commodities: almond, apple, barley, bean, maize, oat, pea, peanut, rice, sugar beet, soya bean, and wheat.

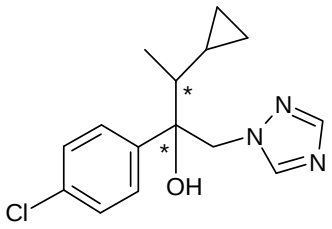
GAP information was also submitted by Japan and the Netherlands.

IDENTITY

ISO Common Name	Cyproconazole
Chemical name:	
IUPAC	(2RS,3RS;2SR,3SR)-2-(4-chlorophenyl)-3-cyclopropyl-1-(1H-1,2,4-triazol-1-yl)butan-2-ol
CA	alpha-(4-chlorophenyl)-alpha-(1-cyclopropyl-ethyl)-1H-1,2,4-triazole-1-ethanol
CAS No	94361-06-5
Manufacturers code No.	SAN619
Structural formula	
Molecular formula	C15H18ClN3O
Molecular mass	291.8

Stereochemistry:

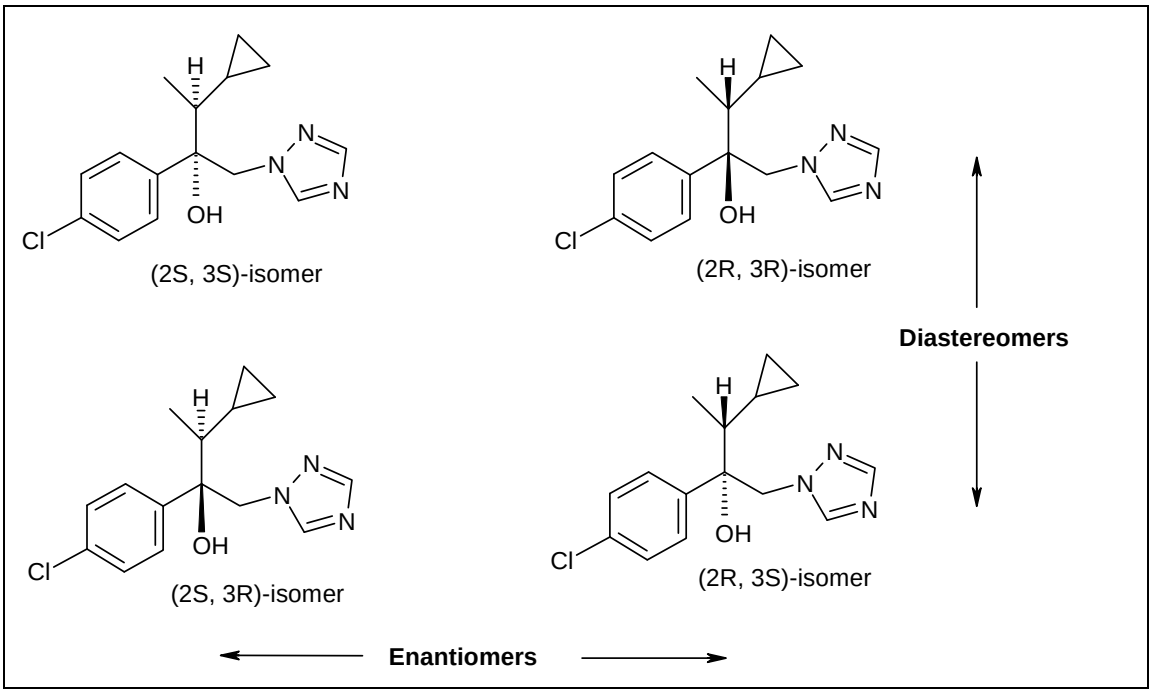
Cyproconazole possesses two chiral carbon atoms (C2 and C3 of the butane backbone) indicated in the structure with asterisks:



This structure exists in four stereoisomeric forms: two enantiomeric pairs of diastereoisomers. The following are defined:

"Diastereomer A": enantiomeric pair, where the 3-hydroxy group and 2-hydrogen are located on the same side.

"Diastereomer B": enantiomeric pair, where the 3-hydroxy group and 2-hydrogen are located on opposite sides.



Cyproconazole is an approximately 1:1 mixture of the two diastereomers, each of which is exactly a 1:1 mixture of the enantiomers. All four stereoisomers are present in similar amounts.

PHYSICAL AND CHEMICAL PROPERTIES

Property	Results	Method Test material	Reference
Melting point	106.2–106.9 °C	SAN619 pure ai (99.7%) OECD 102	Das, 1998 SAN619/0447
Boiling point	Thermal decomposition starts at about 300 °C before the boiling point is reached, and oxidative decomposition starts at about 115 °C	SAN619 pure ai (99.7%) OECD 103	Das, 2000 SAN619/6876

Property	Results	Method Test material	Reference
Temperature of decomposition or sublimation	Oxidative decomposition starts at about 115 °C, followed by thermal decomposition which starts at about 300 °C Cyproconazole shows neither without nor with air any peak between room temperature and the melting point (approx. 98 °C)	SAN619 pure ai (99.7%) OECD 103 SAN619 tech. ai (96.6%) OECD 113	Das, 2000 SAN619/6876 Angly, 2000 SAN619/6950
Relative density	1.25–103 kg / m^3 at 22 °C corresponding to a relative density of 1.25	SAN619 pure ai (99.7%) OECD 109	Füldner, 1998 SAN619/0503
Vapour pressure	Vapour pressure curve in the solid state: 10log P [Pa] = - 5868.9 · 1 / T [K] + 15.097 from fit of measurements between 45 and 75 °C vapour pressure at 25 °C : 2.6×10^{-5} Pa	SAN619 pure ai (99.7%) OECD 104	Widmer, 1998 SAN619/0532
Volatility	Henry's law constant at 25 °C : 5.0×10^{-5} Pa m^3/mol	calculation	Burkhard, 1999 SAN619/0099
Physical state and colour	Pure active substance: white fine powder Technical grade active substance: beige fine powder	SAN619 pure ai (99.7%) visual SAN619 tech. ai (96.6%) visual	Das, 1999a SAN619/6781 Das, 1999b SAN619/6780
Odour	Pure active substance: odourless Technical grade active substance: weak aromatic	SAN619 pure ai (99.7%) organoleptic SAN619 tech. ai (96.6%) organoleptic	Das, 1999a SAN619/6781 Das, 1999b SAN619/6780
Spectra active substance	UV Absorption Characteristics : The molar extinction were determined to be: solution / wavelength [nm] / molar extinction coefficient [mol^{-1} cm^{-1}] neutral 222 11515 266 233 acidic 222 12211 266 250 basic 222 10283 266 238 No absorption maximum between 350 nm and 750 nm was observed.	OECD 101 SAN619 pure ai (99.7%)	Oggenfuss, 2001 SAN619/7060
Spectra of impurities	None of the impurities present in the active substance as manufactured are of toxicological or environmental significance		
Solubility in water including effect of pH	The solubility of cyproconazole in buffer solutions was determined to be : 108 ± 8 mg/L at pH 4.1 and 22 °C 93 ± 18 mg/L at pH 7.1 and 22 °C 109 ± 4 mg/L at pH 10.0 and 22 °C	CIPAC MT 157.2 corresponding to OECD 105 SAN619 pure (98.9%)	Wisson, 1989 SAN619/6125
Solubility in organic solvents	The solubility in different organic solvents at 25 °C was determined to be : acetone 360 g/L dichloromethane 430 g/L ethyl acetate 240 g/L hexane 1.3 g/L methanol 410 g/L octanol 100 g/L toluene 100 g/L	CIPAC MT 157.3 SAN619 tech. (96.6%)	Stulz, 1998a SAN619/0522
Partition coefficient n-octanol / water	The octanol / water partition coefficient (Pow) and its logarithm to base 10 (log Pow) was determined to be: Pow = 1200 ± 61 log Pow = 3.1	OECD 107 SAN619 pure (99.7%)	Stulz, 1998b SAN619/0518

Property	Results	Method Test material	Reference
Hydrolysis rate	Cyproconazole was hydrolytically stable at pH 4, 5, 7 and 9 at 50 °C for 5 days. The ratios of the two isomers remained unchanged.	OECD 111 14C labelled SAN619, (98.9% radio-chemical purity)	Glänzel, 1999 SAN619/6849
Photochemical degradation	The molar extinction coefficients in aqueous buffer solutions at pH 5, 7 and 9 are below 10 l mol^{-1} cm^{-1} Therefore the investigation of photochemical degradation is not required	OECD 101 SAN619 pure (99.7%)	Oggenfuss, 2000 SAN619/7018
Dissociation constant	Cyproconazole does not have a dissociation constant within the range 3 to 10	OECD 112 SAN619 pure (98.9%)	Gampp, 1989 SAN619/6138
Stability in air, photo-chem. degradation, breakdown product(s)	Based on the calculation according to Atkinson the estimated half-life of cyproconazole in the atmosphere by hydroxyl radical oxidation is 0.661 days (1.5 · 106 OH-radicals / cm^3 and a 12 hour day)	calculation according to Atkinson	Glänzel, 1996 SAN619/5173
Flammability	Cyproconazole is not considered highly flammable	EEC A.10 SAN619 tech. (95.0%)	v. Helvoirt, 1994 SAN619/6239
Auto-flammability	Cyproconazole shows no self-ignition	EEC A.16 SAN619 tech. (95.0%)	v. Helvoirt, 1995 SAN619/6240
Flash point	Not required, cyproconazole, is a solid with a melting point > 40 °C		
Explosive properties	Cyproconazole is not considered an explosive in accordance with EEC Method A.14	EEC A.14 SAN619 tech. (95.7%)	Krips, 1996 SAN619/5160
Surface tension	Surface tension of aqueous suspensions at 20 °C by the Wilhelmy plate method was determined to be : σ = 65.2 mN / m (at 90% of the saturation concentration) Cyproconazole has to be regarded as a surface active substance because the surface tension is lower than 60 mN/m	OECD 115 SAN619 tech. (96.6%)	Martin, 1999 SAN619/6767
Oxidizing properties	Cyproconazole is not considered an oxidizing substance	EEC A.17 SAN619 tech. (95.0%)	Krips, 1995 SAN619/6238

FORMULATIONS

Formulation [a]	Active Ingredient Content
WG	300 g/kg
WG	100 g/kg
WG	160 g/kg
WG	400 g/kg
SL	100 g/L
SL	50 g/L
EC	240 g/L
SC	400 g/L
FU	1 g/part

[a] EC - emulsifiable concentrate, FU - smoke generator ; SC - suspension concentrate; SL - soluble concentrate; WG - wettable granule

Additionally there are numerous co-formulations with imazalil, difenoconazole, prochloraz, propiconazole, chlorothalonil, cyprodinil, fludioxonil, azoxystrobin, and copper.

Specification

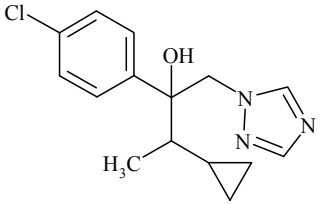
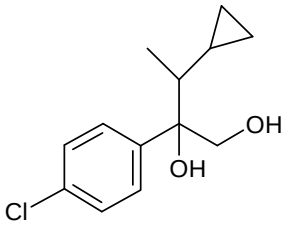
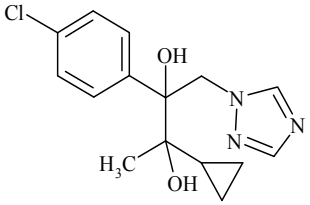
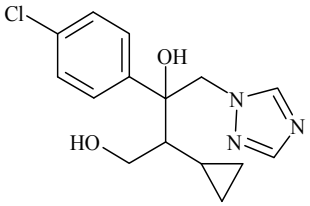
An FAO specification for cyproconazole has not been established by the JMPS under the new system.

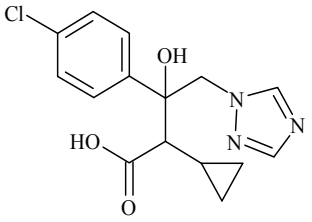
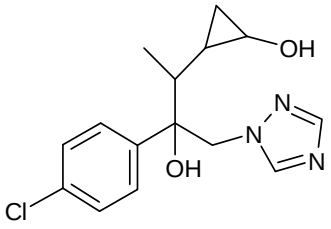
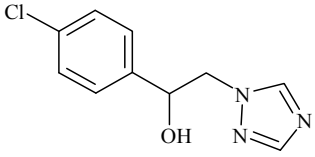
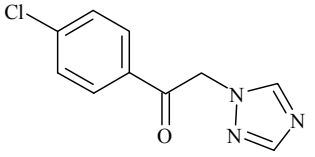
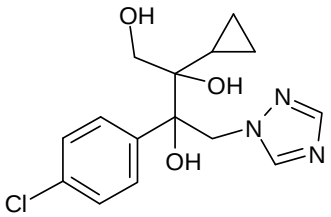
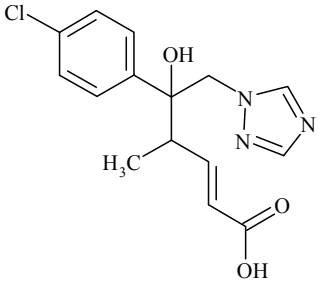
METABOLISM AND ENVIRONMENTAL FATE

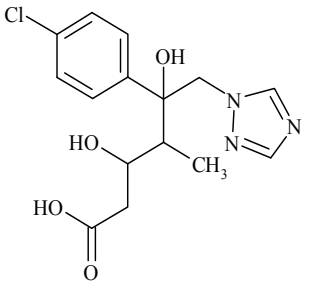
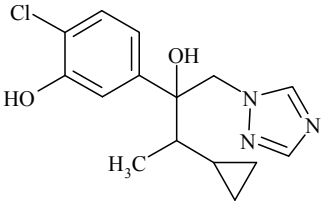
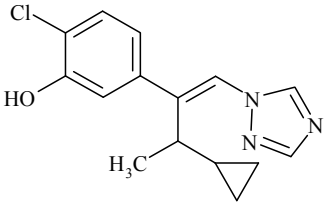
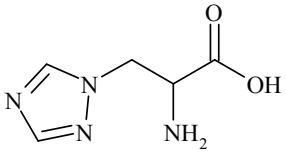
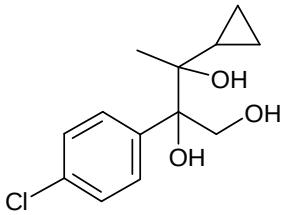
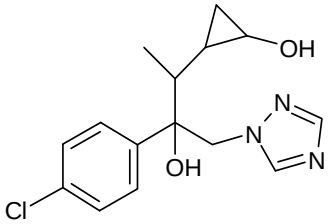
Metabolites are given various abbreviations and code numbers in the studies.

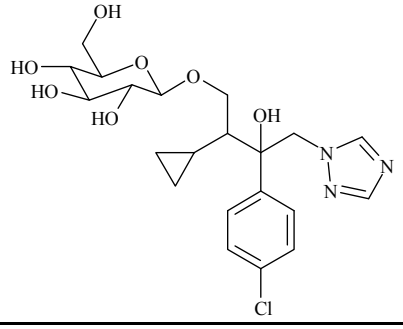
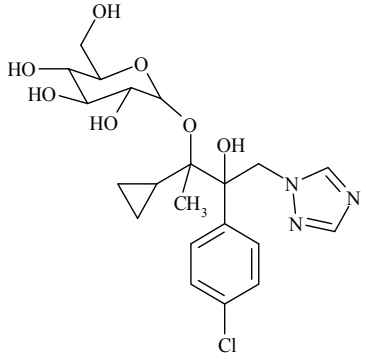
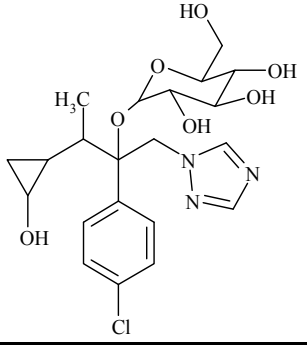
Abbreviations and codes, chemical names, and structures are shown below, along with information on the matrices in which the particular chemical was found.

Parent and metabolites identified in cyproconazole plant and livestock metabolism studies and environmental fate studies

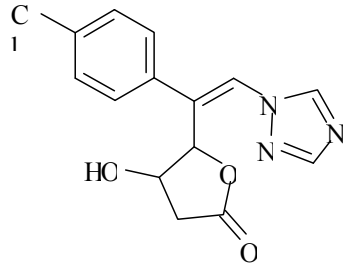
Common name/Code Metabolite#	Chemical name	Chemical structure	Matrices
Cyproconazole (CGA 221949) M1/M2	α-(4-chlorophenyl)-α-(1-cyclopropylethyl)-1H-1,2,4-triazole-1-ethanol		Apples, Grapes, and Wheat forage, grain and straw Poultry tissues, eggs and excreta Goat milk and tissues
M3/M4 NOA421152	2-(4-chlorophenyl)-3-cyclopropyl-1,2-butanediol		Rat (faeces, urine)
M9/M14 NOA 421153	2-(4-chlorophenyl)-3-cyclopropyl-1-[1,2,4]triazol-1-yl-butane-2,3-diol		Apples, Grapes, and Wheat forage, grain and straw Poultry tissues, eggs and excreta Goat kidney and liver
M11/M18 NOA 421154	3-(4-chlorophenyl)-2-cyclopropyl-4-[1,2,4]triazol-1-yl-butane-1,3-diol		Apples, Grapes, and Wheat forage, grain and straw Poultry tissues, eggs and excreta Goat liver

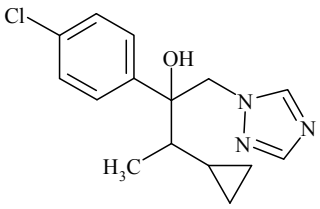
Common name/Code Metabolite#	Chemical name	Chemical structure	Matrices
M10/M10a NOA 452154	3-(4-chlorophenyl)-2-cyclopropyl-3-hydroxy-4-[1,2,4]triazol-1-yl-butyric acid		Poultry excreta, Grapes
M13 NOA451353	2-[2-(4-chloro-phenyl)-2-hydroxy-1-methyl-3-[1,2,4]triazol-1-yl-propyl]-cyclopropanol		Apples, grapes, wheat
M15 NOA 408616	1-(4-chlorophenyl)-2-[1,2,4]triazol-1-yl-ethanol		Apples, Grapes, and Wheat grain and straw Poultry tissues and eggs Soil photolysis
M16 CGA 123420	1-(4-chlorophenyl)-2-[1,2,4]triazol-1-yl-ethanone		Wheat forage and straw Poultry tissues, eggs and excreta Soil photolysis
M20 NOA452668	3-(4-chloro-phenyl)-2-cyclopropyl-4-[1,2,4]triazol-1-yl-butane-1,2,3-triol		Rat
M21/M21a NOA 405870	5-(4-chlorophenyl)-5-hydroxy-4-methyl-6-[1,2,4]triazol-1-yl-hex-2-enoic acid		Poultry tissues, eggs and excreta Goat milk

Common name/Code Metabolite#	Chemical name	Chemical structure	Matrices
M36 NOA 405872	δ-(4-chlorophenyl)-β,δ-dihydroxy-γ-methyl-1H-1,2,4-triazole-1-hexenoic acid		Goat milk and kidney
M31/M48 NOA 410714	2-chloro-5-(2-cyclopropyl-1-hydroxy-1-[1,2,4]triazol-1-ylmethyl-propyl)-phenol		Poultry excreta
M38 NOA 421155	1-[2-(4-chlorophenyl)-3-cyclopropyl-but-1-enyl]-1H-[1,2,4]triazole		Goat fat and liver
M39 CGA 131013	3-(1H-1,2,4-triazol-1-yl)-alanine		Wheat grain
M30/M33 NOA452669	2-(4-chloro-phenyl)-3-cyclopropyl-butane-1,2,3-triol		Rate urine/faeces
NOA451353	2-[2-(4-chloro-phenyl)-2-hydroxy-1-methyl-3-[1,2,4]triazol-1-yl-propyl]-cyclopropanol		Rat urine/faeces

Common name/Code Metabolite#	Chemical name	Chemical structure	Matrices
M41 (C3/C5)	glucoside of 3-(4-chlorophenyl)-2-cyclopropyl)-4-(1H-1,2,4-triazol-1-yl)-1,3-butanediol		Wheat forage and straw
M42	glucoside of 2-(4-chlorophenyl)-3-cyclopropyl-1-(1H-1,2,4-triazol-1-yl)-2,3-butanediol		Wheat forage and straw
M43	glucoside of α-(4-chlorophenyl)-α-[1-(2-hydroxycyclopropyl)ethyl]-1H-1,2,4-triazole-1-ethanol		Wheat forage and straw
M44/M45 (C4)	glucosides of α-(4-chloro3-hydroxyphenyl)-α-(1-cyclopropylethyl)-1H-1,2,4-triazole-1-ethanol		Wheat straw

Common name/Code Metabolite#	Chemical name	Chemical structure	Matrices
M46	malonic acid conjugate of M42		Wheat forage and straw
M47	malonic acid conjugate of M41		Wheat forage and straw
M50	Sulfuric acid mono-[1-(4-chlorophenyl)-2-[1,2,4]triazol-1-yl-ethyl] ester		Poultry liver and excreta
M51	Sulfuric acid mono-[3-(4-chlorophenyl)-2-cyclopropyl-2,3-dihydroxy-4-[1,2,4]triazol-1-yl-butyl] ester		Poultry excreta

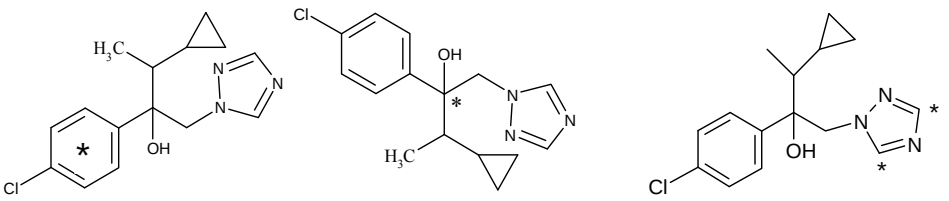
Common name/Code Metabolite#	Chemical name	Chemical structure	Matrices
M52 (M54) [a]	Sulfuric acid mono-[3-(4-chlorophenyl)-2-cyclopropyl-3-hydroxy-4-[1,2,4]triazol-1-yl-butyl] ester		Poultry liver and excreta
M53 (M54)[a]	Sulfuric acid mono-[3-(4-chlorophenyl)-2-cyclopropyl-3-hydroxy-4-[1,2,4]triazol-1-yl-butyl] ester		Poultry liver and excreta
M55 SYN 533911/SYN 533912	5-chloro-2-(1-hydroxy-2-4-[1,2,4]triazol-1-yl-ethyl) phenol or 2-chloro-5-(1-hydroxy-2-4-[1,2,4]triazol-1-yl-ethyl) phenol		Poultry excreta
M56 SYN 533921	5-[1-(4-chlorophenyl)-1-hydroxy-2-[1,2,4]triazol-1-yl-ethyl]-4-hydroxy-5-methyl-dihydro-furan-2-one		Poultry excreta

Common name/Code Metabolite#	Chemical name	Chemical structure	Matrices
M57 NOA 405870	(E)-5-(4-chlorophenyl)-4,5-dihydroxy-4-methyl-6-[1,2,4]triazol-1-yl-hex-2-enoic acid		Poultry excreta
M58 CGA 155705	4-chlorobenozic acid		Poultry excreta
M59	2-(4-chlorophenyl)-3-methyl-1-[1,2,4]triazol-1-yl-pentane-2,4-diol		Poultry liver
CGA71019 1,2,4-Triazole	1H-1,2,4-triazole		Soil Wheat grain Milk Cow liver Cow kidney
CGA142856 Triazole acetic acid	1,2,4-triazol-1-yl-acetic acid		Soil Cow liver Cow kidney Cow muscle

[a] M54 was identified as a stereoisomer of either M52 or M53.

Animal metabolism

The Meeting received studies on the metabolism of cyproconazole in lactating cows and laying Hens and rat. The metabolism of cyproconazole in plants and livestock was investigated using [triazole-^{14}C]-labelled, central α-labelled [^{14}C]-labelled, and/or [phenyl-U-^{14}C]-labelled cyproconazole.



[phenyl-U-^{14}C]-labelled [central α-^{14}C]-labelled [triazole-^{14}C]-labelled

Lactating goat. The Meeting received a study depicting the metabolism of [^{14}C]cyproconazole, labelled at the central alpha carbon, in lactating goats (1987; W.S. Skinner, *et al*.). A lactating goat was dosed orally for three consecutive days by gelatin capsule containing a mixture of [α-carbon ^{14}C]-cyproconazole (1.06 mg radiochemical purity 99.4%, specific activity 54.9 mCi/mmol) and technical grade cyproconazole (13.1 mg). Based on animal weight (14.2 kg), the dose level was 1 mg/kg body weight per day, or a dietary concentration of ~30 ppm. Urine and faeces were collected at 24-hour intervals and milk was collected twice daily. Blood was sampled at 2, 4, 8, 12, 24, 48, and 72 hours after the first dose. The animal was sacrificed approximately 24 hours after the last dose and samples of omental fat, renal fat, subcutaneous fat, kidney; liver, leg muscle, flank muscle, and shoulder muscle were taken. Other organs, bile and the gastrointestinal (GI) tract were also sampled.

Total radioactive residues (TRR) in samples were determined either directly by liquid-scintillation counting (LSC; milk and urine) or by combustion/LSC (tissues). Selected tissues were extracted with methanol (MeOH) and chloroform. Tissue extracts and milk were fractionated by column chromatography. Urine was methylated and fractionated by thin-layer chromatography (TLC). Extracts were analysed by TLC and reversed phase high-performance liquid chromatography (HPLC) and metabolites were identified by co-chromatography with authentic reference standards and mass spectrometry (MS). Conjugated radioactivity was investigated using base and enzyme (sulfatase and β-glucuronidase) hydrolyses.

The recoveries of applied radioactivity and the radioactive residues in excreta, milk, and tissues are shown in Table 1.

Table 1 Radioactivity and residues in tissues, milk, and excreta from a goat dosed with [α-carbon14C]-Cyproconazole for three consecutive Days at 30 ppm in the Diet.

Sample		Sampling period	% of applied dose	TRR (mg/kg)
Urine		Day 0-1	25.9	-
		Day 1-2	27.2	-
		Day 2-3	27.9[a]	-
		Total	81.0	-
Faeces		Day 0-1	3.5	-
		Day 1-2	3.6	-
		Day 2-3	3.2	-
		Total	10.3	-
Milk		Day 1 PM	0.05	0.590
		Day 2 AM	0.02	0.130
		Day 2 PM	0.04	0.660
		Day 3 AM	0.02	0.180
		Day 3 PM	0.05	0.830
		Day 4 AM	0.01	0.090
		Total	0.19	-
Fat	Omental	-	-	0.033
	Renal	-	-	0.035
	Subcutaneous	-	-	0.046
Kidney		-	0.02	0.092
Liver		-	0.95	1.170
Muscle	Flank	-	-	0.015
	Leg	-	-	0.007

Sample	Sampling period	% of applied dose	TRR (mg/kg)
Shoulder	-	-	0.009
Adrenal gland	-	< 0.001	0.072
Bladder	-	0.035	0.650
Brain	-	0.002	0.010
Gall Bladder	-	0.001	0.26
Heart	-	0.004	0.020
Hide	-	-	0.012
Lung	-	0.019	0.060
Ovaries	-	< 0.001	0.013
Pancreas	-	0.001	0.031
Spleen	-	< 0.001	0.018
Thyroid	-	< 0.001	0.018
Tongue	-	0.002	0.018
Udder	-	0.013	0.048
Uterus	-	0.001	0.024
Blood	-	-	0.014
GI tract contents	-	1.30	-
Total Recovery %	-	93.8	-

[a] Includes cage wash

Cyproconazole was present at levels of 8% and 21% TRR in urine and faeces, respectively and at 3.3% TRR (0.003 mg/kg) in milk. In tissues, cyproconazole was present in the liver, kidney, fat, and muscle at 21% (0.246 mg/kg), 5% (0.005 mg/kg), 27% (0.013 mg/kg), and 11% (0.002 mg/kg) of the TRR, respectively. Treatment of bound residues in liver released parent cyproconazole and M11/M18 (NOA421154) at levels up to 4% TRR (0.047 mg/kg). Characterisation and identification of residues are presented in Table 2.

Table 2 Characterisation and Identification of Residues in Tissues, Milk, and Excreta from a Goat Dosed with [α-carbon14C]-Cyproconazole for Three Consecutive Days at 30 mg/kg in the Diet.

Fraction/ component	Faeces	Urine	Milk [a]		Liver		Kidney		Fat		Muscle	
	% TRR	% TRR	% TRR	mg/kg[b]	% TRR	mg/kg[b]	% TRR	mg/kg[b]	% TRR	mg/kg[b]	% TRR	mg/kg[b]
Extractable	64	100	99.7	0.083	89	1.041	89	0.082	100	0.046	89	0.013
Cyproconazole	21	8	3.3	0.003	21	0.246	5	0.005	27	0.013	11	0.002
NOA421153; M9/M14	9	5	-	-	27	0.316	< 2	< 0.002	5	0.003	5	0.002
NOA421154; M11/M18	13	10	-	-	12	0.141	2	0.002	3	0.002	5	< 0.002
NOA452154; M10	< 1	3	8.3	0.007	-	-	-	-	-	-	-	-
NOA408616; M15	4	< 1	-	-	< 1	< 0.012	< 1	< 0.001	1	< 0.001	< 1	< 0.001
CGA123420; M16	5	< 1	-	-	3	0.035	< 1	< 0.001	< 2	< 0.001	4	0.001
NOA421155;M38	3	< 1	-	-	16	0.187	1	0.001	36	0.017	4	0.001
NOA405872; M36	< 1	2	68.5	0.057	-	-	-	-	-	-	-	-
Conjugates												
Cyproconazole	-	-	-	-	-	-	24	0.022	4	0.002	< 1	< 0.001
NOA421153; M9/M14	-	-	-	-	-	-	2	0.002	< 2	< 0.002	< 2	< 0.002
NOA421154; M11/M18	-	-	-	-	-	-	9	0.009	2	< 0.002	2	< 0.002
NOA408616; M15	-	-	-	-	-	-	3	0.003	< 1	< 0.001	< 1	< 0.001
Bound												
Non-extractable	36	-	0.3	< 0.001	11	0.129	11	0.010	-	-	11	0.002
Cyproconazole	-	-	-	-	1	0.012	< 2	< 0.002	-	-	-	-
NOA421154; M11/M18	-	-	-	-	4	0.047	< 4	< 0.004	-	-	-	-

[a] Composite sample from 0-3 day milk.

[b] Calculated from the highest quoted residue level where applicable.

A second study depicting the metabolism of [^{14}C]cyproconazole, labelled at the central alpha carbon, in lactating goats (1991; A. Guirguis, *et al*.) was available. Three goats were used for the study: Goat A was orally dosed with [14C]cyproconazole at 10 ppm in the diet for 4 consecutive days; Goat B was dosed with unlabeled cyproconazole at 1000 mg/kg in the diet for 2 days and then dosed with [^{14}C]cyproconazole at 1000 mg/kg for 2 days; and Goat C was not dosed with cyproconazole. Goat A was used to determine metabolite distribution and material balance, Goat B was used to generate metabolites for spectroscopic identification, and Goat C was used as a control. The goats were orally dosed with gelatin capsules once daily in the morning.

Milk was collected twice daily (in the a.m. and p.m.) and frozen immediately after collection. The petitioner provided sufficient information concerning daily feed intake, body weights, and milk production. Goats A and C were sacrificed 24 hours after the last dose and Goat B was sacrificed 7 hours after the last dose. The entire liver and kidneys and representative samples of muscle (longissimus dorsi and triceps) and fat (perirenal and omental) were collected and frozen immediately.

Radioactivity in tissue samples was determined by LSC following combustion and radioactivity in milk was determined directly by LSC. The TRR found in the milk and tissues of Goat A are presented in Table 3.

Table 3 TRR in milk and tissues from a lactating goat orally dosed with [α-carbon^{14}C]-Cyproconazole for 4 Days at 10 ppm (Goat A)

Matrix	TRR, expressed as mg/kg [^{14}C]cyproconazole
Milk: Day 1 - a.m.	0.208
Milk: Day 1 - p.m.	0.056
Milk: Day 2 - a.m.	0.140
Milk: Day 2 - p.m.	0.035
Milk: Day 3 - a.m.	0.161
Milk: Day 3 - p.m.	0.044
Milk: Day 4 - a.m.	0.173
Milk: Day 4 - p.m.	0.053
Fat	0.023
Kidney	0.066
Liver	0.212
Muscle	0.004

Samples of urine and faeces were also collected to estimate the extent of excretion of the test substance. About 51% and 28% of the applied dose was eliminated in the urine and faeces, respectively. M3 (NOA421152) was identified in the excreta (about 1% TRR), but not detected in milk or any tissue.

Milk and tissue samples from Goat A were subjected to extraction and hydrolysis procedures for residue characterisation and identification. During the fractionation procedures, aliquots of extracts, hydrolysates, and non-extractable residues were analysed for radioactivity by LSC or combustion/LSC. The general extraction procedures are summarised below. Because TRR in muscle were < 0.01 mg/kg, muscle tissue was not subjected to characterisation/identification procedures.

The various extracts of milk and tissues from Goat A were analysed by TLC on silica-gel plates with fluorescent indicator using four different solvent systems. Metabolites were identified by comparison of Rf values with those of nonlabelled standards of several potential cyproconazole metabolites. Selected extracts were also analysed by HPLC to confirm identification of metabolites.

Identification and/or confirmation of the identification of certain milk and liver metabolites was accomplished by MS. Milk extracts containing metabolites M21, M21a, and M36 were subjected

to spectroscopic identification by fast-atom bombardment (FAB) MS and nuclear magnetic resonance (NMR).

The identification of all metabolites by TLC was confirmed by HPLC, MS, and/or NMR with the exception of metabolites M38 (Z1) in fat (> 10% TRR), M14 in kidney (< 10% TRR), and M18 in liver (< 10% TRR). The identification of metabolite M38 (Z1) in fat was not confirmed. The confirmation of metabolite M36 in kidney was accomplished by comparison of the HPLC retention time with that of the same metabolite identified in urine by FAB-MS and NMR. A summary of the characterised/identified residues found in goat matrices is presented in Table 4.

Table 4 Summary of TRR Characterised/identified in milk and tissues from a Lactating Goat orally dosed with [α-carbon^{14}C]-Cyproconazole for 4 Days at 10 ppm (Goat A).

Fraction	Milk		Fat		Kidney		Liver	
	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
Identified								
Cyproconazole (M1)	5.50	0.006	24.92	0.006	20.90	0.014	12.24	0.026
Cyproconazole (M2)	3.61	0.004	22.33	0.005	10.83	0.007	7.13	0.015
NOA 2121153; M14	--	--	--	--	8.92	0.006	27.25	0.058
NOA421154; M18	--	--	--	--	--	--	8.53	0.018
NOA405870; M21	5.87	0.006	--	--	--	--	--	--
M21a	25.65	0.027	--	--	--	--	--	--
NOA405872; M36	46.95	0.050	--	--	12.43	0.008	--	--
NOA421155; M38 (Z1)	--	--	11.20	0.003	--	--	3.71	0.008
Total identified	87.58	0.093	58.45	0.013	53.08	0.035	58.86	0.125
Characterised								
Unknown (Rf=0.93)	0.36	< 0.001	--	--	--	--	--	--
Unknown (Rf=0.62)	--	--	--	--	0.08	< 0.001	--	--
Unknown (Rf=0.46)	--	--	--	--	--	--	7.10	0.015
Unknown (Rf=0.26)	--	--	3.44	0.001	--	--	--	--
Unknown (Rf=0.11-0.13)	--	--	3.18	0.001	8.95	0.006	--	--
Unknown (origin)	4.18	0.004	2.86	0.001	4.25	0.003	5.56	0.012
Hexane	1.13	0.001	17.04	0.004	--	--	--	--
Ethyl acetate	2.95	0.003	--	--	--	--	--	--
Acid-released	--	--	--	--	--	--	5.74	0.012
Aqueous	8.00	0.009	--	--	12.96	0.009	5.74	0.012
Precipitate	--	--	10.68	0.002	12.31	0.008	7.84	0.017
Total identified/characterised	104.20	0.110	95.65	0.022	91.63	0.060	90.84	0.193
Non-extractable	--	--	1.09	< 0.001	11.41	0.008	9.49	0.020

A third study depicting the metabolism of [^{14}C]cyproconazole, labelled at the central alpha carbon, in lactating goats (1991; Völlmin, S.) was provided to the Meeting.. Radioactive cyproconazole residues in milk were studied in two lactating goats dosed orally for 12 consecutive days by gelatin capsule containing [α-carbon 14C]-cyproconazole (radiochemical purity >99%, specific activity 54.9 mCi/mmol). The dose level was 0.048 mg/kg body weight per day, equivalent to a dietary concentration of 1 ppm. Samples of milk were taken twice daily and urine and faces were collected at 24-hour intervals. Blood was sampled at 2, 4, and 6 hours after the first dose and on Day 4, 8, and 12. The animal was sacrificed 2 hours after the last dose and samples of renal fat, kidney, liver, heart, and muscle were taken.

TRRs in samples were determined either directly by LSC or by LSC following solubilisation. Radioactivity in the milk was extracted with acetone (ACN) and purified by C18 solid-phase extraction (SPE) prior to analysis by TLC. Radioactive residues in milk are shown in Table 5.

Table 5 TRR in milk from a goat dosed with [α-carbon^{14}C]-Cyproconazole for 12 consecutive days at 1 ppm in the Diet.

Sampling period (days after 1st dose)	TRR (mg/kg) in milk		
	Goat 1	Goat 2	Mean
1	0.032	0.026	0.029
2	0.028	0.030	0.029
3	0.021	0.033	0.027
4	0.022	0.029	0.026
5	0.024	0.029	0.027
6	0.021	0.027	0.024
7	0.024	0.027	0.026
8	0.025	0.025	0.025
9	0.018	0.022	0.020
10	0.024	0.025	0.025
11	0.020	0.027	0.024
Mean	0.024	0.027	0.026

The recovery of applied radioactivity and radioactive residues in excreta and tissues are shown in Table 6.

Table 6 Recovery of Radioactivity and TRR in Tissues and Excreta from a Goat Dosed with [α-carbon^{14}C]-Cyproconazole for 12 Consecutive Days at 1 ppm in the Diet.

Sample	Goat 1		Goat 2		Average	
	% of dose	mg/kg	% of dose	mg/kg	% of dose	mg/kg
Urine [a] – total	62.8	-	67.0	-	64.9	-
Faeces [a] – total	22.7	-	22.1	-	22.4	-
Fat	-	0.025	-	0.019	-	0.022
Kidney	-	0.062	-	0.097	-	0.080
Liver	-	0.266	-	0.299	-	0.283
Muscle	-	0.006	-	0.005	-	0.005
Heart	-	0.008	-	0.011	-	0.010
Blood [b]	-	0.012	-	0.012	-	0.012

[a] Cumulative total.

[b] Measured 2 hours after the last dosing.

The majority of the radioactive dose was present in the excreta, with 64.9% and 22.4% of the applied radioactivity in the urine and faces, respectively.

Cyproconazole was not detected (< 0.001 mg/kg) in milk. Radioactivity was composed mainly of NOA405870 (17.2%) and NOA405872 (63.0%), with the remainder present mainly as polar compounds that included conjugates of the main metabolites. Radioactive residues in tissues and excreta were not analysed. Characterisation of the radioactive residue in milk is shown in Table 7.

Table 7 Characterisation and Identification of residues in Milk from a Goat Dosed with [α-carbon^{14}C]-Cyproconazole for 12 Consecutive Days at 1 ppm in the Diet.

Fraction and Identity	Percent of TRR [a]		
	Goat 1	Goat 2	Mean
NOA405870; M21	17.6	16.7	17.2
NOA405872; M36	61.2	64.7	63.0
Polar fraction [b]	11.0	12.0	11.5
Remainder	10.2	6.6	8.4

[a] Mean results from Day 1, 2, 3, 4, 7, 11, and 12 data.

[b] Includes conjugates of NOA405870 and NOA405872.

The proposed metabolism pathway of cyproconazole in goats is given in Figure 1 below.

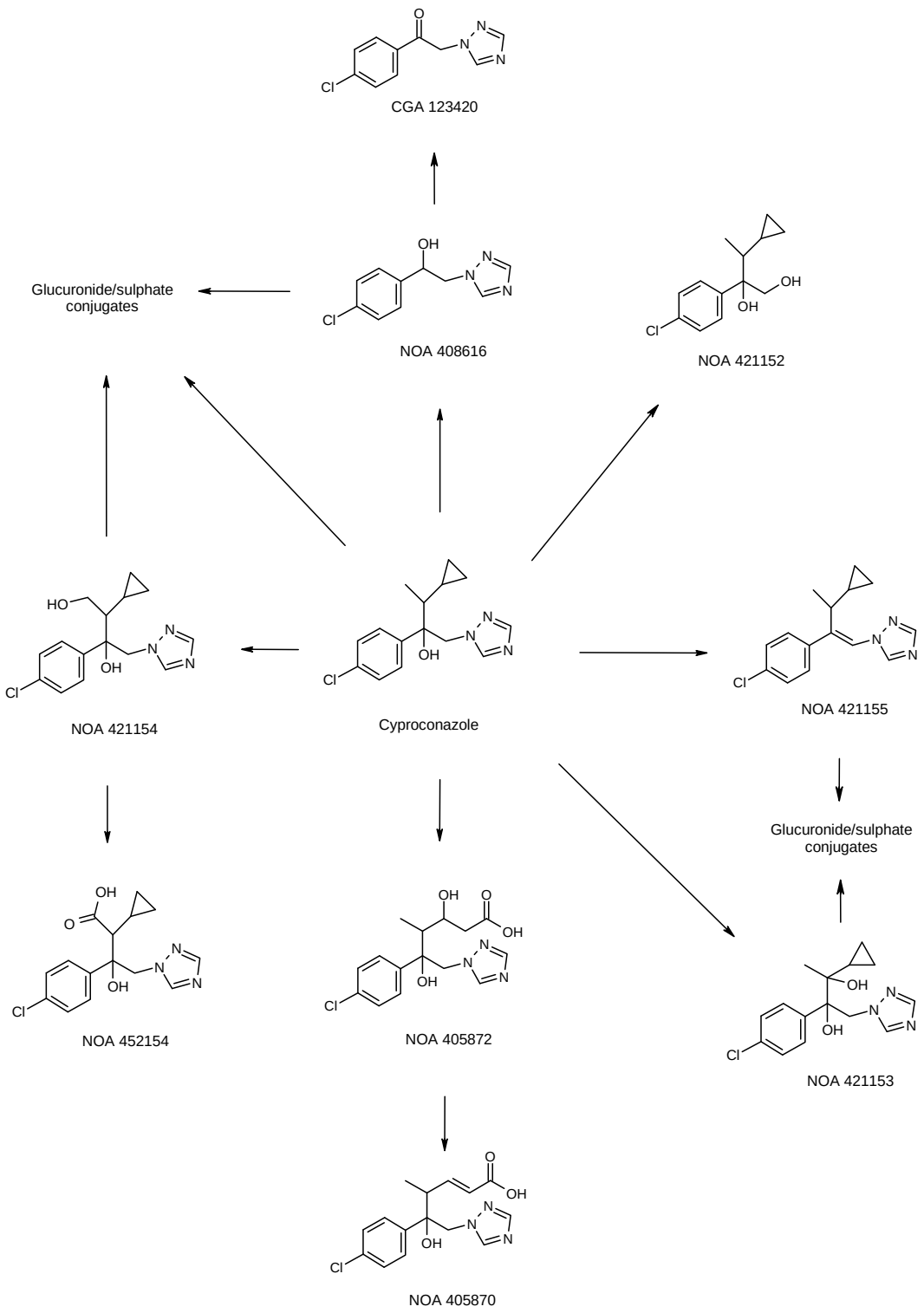


Figure 1 Pathway of cyproconazole metabolism in the goat

Laying hen

The Meeting received a study report depicting the metabolism of [^{14}C]cyproconazole, labelled at the central alpha carbon, in laying hens (1987; W.S. Skinner, *et al*.). The metabolic fate of cyproconazole was investigated in laying hens using [α-carbon^{14}C]-cyproconazole (radiochemical purity 99.4%, specific activity 54.9 mCi/mmol). Four laying hens were dosed orally via gelatin capsule for three consecutive days at a rate of 1 mg/kg of body weight per day, equivalent to a dietary concentration of approximately 30 ppm. A fifth hen received a single dose of 100 mg/kg of body weight, equivalent to a dietary concentration of 3000 ppm. Eggs and excreta were collected daily. Two of the low-dose hens and the high-dose hen were sacrificed 24 hours after the last dose. After sacrifice, muscle, fat, kidney and liver were taken. The two remaining low-dose hens were maintained for a total of 14 days to examine depletion of residues through daily egg production.

TRR in samples were determined either directly (egg white and yolk) or by combustion/LSC (tissues and excreta). Tissues were extracted with MeOH and chloroform and the extracts fractionated by column chromatography. Excreta were extracted with MeOH then methylated (diazomethane) and fractionated by TLC. Extracts were analysed by TLC and reversed phase HPLC and metabolites were identified by co-chromatography with authentic reference standards and MS. Conjugated radioactivity was investigated using potassium hydroxide (1M) and enzyme (sulfatase and β-glucuronidase) hydrolysis.

The recovery of applied radioactivity in the excreta, eggs and tissues is given in Table 8. Residue levels in tissues and eggs are shown in Table 9. Distribution of radioactivity and radioactive residues in eggs are shown in Table10.

Table 8 Recovery of Radioactivity from Hens Dosed with [α-carbon^{14}C]-Cyproconazole for One Day (100 ppm – high dose) or Three Consecutive Days (1 ppm – low dose)

Sample		Percent (%) of Applied Dose				
		Low dose				High dose
		Hen 1	Hen 2	Hen 3	Hen 4	Hen 5
Excreta						
0-1 day	Extract (MeOH)	26.8	24.6	-	-	-
	Non-extractable	3.1	3.6	-	-	-
1-2 day	Extract	28.7	28.5	-	-	-
	Non-extractable	3.1	3.5	-	-	-
2-3 day	Extract	29.8	30.5	-	-	-
	Non-extractable	3.0	2.8	-	-	-
3-7 day	Aqueous homogenate	2.3	2.0	-	-	-
0-3 day	Aqueous homogenate	-	-	94.7	94.0	-
0-1 day	Extract (MeOH)	-	-	-	-	11.3
	Non-extractable	-	-	-	-	0.8
Excreta total		96.8	95.5	94.7	94.0	12.1
Eggs						
0-14 day		1.1	1.5	-	-	-
0-3 day		-	-	0.9	0.4	-
Unlaid		-	-	-	-	1.4
Tissues						
Gizzard		-	-	-	-	16.5
Gastrointestinal Tract and contents		-	-	2.0	0.5	30.1
Selected tissues [a]		-	-	1.7	0.6	19.6
Carcass		1.4	0.5	1.4	0.5	16.1
Tissues total		1.4	0.5	5.1	1.6	82.3
Total recovery		99.3	97.5	101.0	96.0	95.8

[a] Animal sacrificed 24 hours after last dose.

Table 9 Distribution of radioactivity in tissues from hens dosed with [α-carbon^{14}C]-Cyproconazole for one day (100 mg/kg – high dose) or three consecutive days (1 ppm – low dose)

Sample	Low dose						High dose	
	Hen 3		Hen 4		Mean		Hen 5	
	mg/kg	% appl.	mg/kg	% appl.	mg/kg	% appl.	mg/kg	% appl.
Liver	0.305	0.3	0.128	0.1	0.217	0.2	91.6	2.1
Kidney	0.295	0.1	0.112	< 0.1	0.204	0.1	42.0	0.3
Lungs	0.158	< 0.1	0.063	< 0.1	0.111	< 0.1	19.9	0.1
Gizzard	0.107	< 0.1	0.052	< 0.1	0.080	< 0.1	740	16.5
Heart	0.154	< 0.1	0.064	< 0.1	0.109	< 0.1	36.4	0.1
Leg muscle	0.094	0.4	0.038	0.2	0.066	0.3	31.8	3.5
Breast muscle	0.063	0.2	0.020	0.1	0.042	0.1	15.6	1.7
Leg/breast fat	0.204	0.1	0.049	0.1	0.127	0.1	187	3.7
Abdominal fat	0.185	0.2	0.059	< 0.1	0.122	0.1	200	6.2
Skin	0.133	0.5	0.029	0.1	0.081	0.3	37.6	1.9
GI tract	0.188	2.1	0.086	0.5	0.137	1.1	369	30.1
Carcass	0.102	1.4	0.036	0.5	0.069	1.0	43.3	16.1

Table 10 Distribution of radioactivity in eggs from hens dosed with [α-carbon^{14}C]-Cyproconazole at 1 ppm (low dose) for Three Consecutive Days

Sampling interval (days)	Hen 1				Hen 2			
	Egg yolk		Egg white		Egg yolk		Egg white	
	mg/kg	% appl.	mg/kg	% appl.	mg/kg	% appl.	mg/kg	% appl.
0-1	0.091	< 0.1	0.176	-	n.a.	n.a.	n.a.	n.a.
1-2	n.a.	n.a.	n.a.	n.a.	0.172	0.1	0.212	0.2
2-3	0.407	0.1	0.198	0.1	0.414	0.2	0.223	0.2
3-4	0.569	0.2	0.057	0.1	0.591	0.2	0.095	0.1
4-5	0.562	0.2	0.020	< 0.1	0.643	0.2	0.023	< 0.1
5-6	0.484	0.1	0.007	< 0.1	n.a.	n.a.	n.a.	n.a.
6-7	0.343	0.1	0.003	< 0.1	0.538	0.2	0.006	< 0.1
7-8	0.235	0.1	0.001	< 0.1	0.315	0.1	0.006	< 0.1
8-9	n.a.	n.a.	n.a.	n.a.	0.134	0.1	< 0.001	< 0.1
9-10	0.028	< 0.1	< 0.001	< 0.1	n.a.	n.a.	n.a.	n.a.
10-11	0.008	< 0.1	< 0.001	< 0.1	n.a.	n.a.	n.a.	n.a.
11-12	0.002	< 0.1	< 0.001	< 0.1	0.004	< 0.1	< 0.001	< 0.1
12-13	0.002	< 0.1	< 0.001	< 0.1	0.002	< 0.1	< 0.001	< 0.1
13-14	0.002	< 0.1	< 0.001	< 0.1	< 0.001	< 0.1	< 0.001	< 0.1
n.a. = not applicable, no egg laid.								

Characterisation and identification of radioactive residues in excreta, tissues and eggs are shown in Tables 11, 12 and 13, respectively.

Table 11 Characterisation and identification of radioactivity in excreta from hens dosed with [α-carbon^{14}C]-Cyproconazole at 1 ppm (low dose) for three consecutive days

Fraction / Metabolite	% TRR
Extractable	90
Cyproconazole	2
NOA421153; M9/M14	6
NOA421154; M11/M18	4
NOA452154; M10	5
NOA408616; M15	7
Unknown (Z3)	1
Unknown	9
Non-extractable	10

Table 12 Characterisation and identification of radioactivity in tissues from hens dosed with [α-carbon^{14}C]-Cyproconazole at 1 ppm (low dose) for three consecutive days [a]

Fraction / Metabolite	Liver		Kidney		Muscle		Fat	
	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg
Extractable	70.5	0.153	83.5	0.170	92.5	0.061	98.0	0.124
Cyproconazole	4.0	0.009	2.5	0.005	3.5	0.002	41.0	0.052
NOA421153; M9/M14	20.0	0.044	12.5	0.025	20.5	0.014	37.0	0.047
NOA421154; M11/M18	4.5	0.009	< 2.0	< 0.004	-	-	-	-
NOA408616; M15	21.5	0.047	17.0	0.035	46.5	0.031	13.0	0.017
Conjugated NOA421154	< 4.0	< 0.009	< 3.0	< 0.006	-	-	-	-
Conjugated NOA408616	< 4.0	< 0.009	5.0	0.010	-	-	-	-
Conjugated OH-cyproconazole	< 4.0	< 0.009	6.5	0.013	7.0	0.005	< 1.0	< 0.001
Total Identified	< 62	-	< 48	-	< 78	-	< 92	-
Non-extractable	29.5	0.064	16.5	0.034	7.5	0.005	2.0	0.003
Released by KOH	10.5	0.023	15.0	0.031	-	-	-	-
Bound NOA421154	< 5.0	< 0.011	4.0	0.008	-	-	-	-
Bound NOA408616	< 2.0	< 0.004	4.0	0.008	-	-	-	-

[a] Hens were sacrificed 24 hours after last dose.

Results for the low-dose hen liver, kidney and muscle are mean data from two animals.

Liver TRR = 0.217 mg/kg, Kidney TRR = 0.204 mg/kg.

Muscle TRR = 0.066 mg/kg (origin of sample for analysis not stated in report, value for leg muscle used).

Fat TRR = 0.127 mg/kg (origin of sample for analysis not stated in report, value for leg/breast fat used).

Table 13 Characterisation and identification of radioactivity in eggs from hens dosed with [α-carbon^{14}C]-Cyproconazole at 1 ppm (low dose) for three consecutive days [a]

Fraction / Metabolite	3-4 day whole egg	2-3 day white		4-5 day yolk	
	%TRR	%TRR	mg/kg	%TRR	mg/kg
Extractable	98.0	97.5	0.206	97.5	0.496
Cyproconazole	16.5	10.0	0.021	21.5	0.109
NOA421153; M9/M14	7.0	35.0	0.074	14.5	0.074
NOA421154; M11/M18	2.5	< 1.0	< 0.002	< 1.0	< 0.005
NOA452154; M10	5.5	< 1.0	< 0.002	7.0	0.036
NOA408616; M15	2.5	36.0	0.076	3.5	0.018
Conjugated cyproconazole	1.0	-	-	2.5	0.013
Conjugated NOA421153	21.5	-	-	16.0	0.081
Conjugated NOA421154	4.0	-	-	< 2.0	< 0.010
Conjugated NOA408616	20.0	-	-	17.0	0.087
Non-extractable	2.0	2.5	0.005	2.5	0.013

[a] Hens were sacrificed 24 hours after last dose. 2-3 Day egg white TRR = 0.211 mg/kg (mean of two animals). 4-5 Day egg yolk TRR = 0.509 mg/kg (mean of two animals. Residue data (mg/kg) not given in the report for 3-4 day whole egg and high-dose unlaid eggs.

A second laying hen study depicting the metabolism of [^{14}C]cyproconazole, uniformly labelled in the phenyl ring (2001; Briswalter, C) was provided to the Meeting. Following a 7-day

acclimation period, five White Leghorn laying hens were dosed orally once a day in the morning for 4 consecutive days with [U-^{14}C-phenyl]-cyproconazole at a nominal dose of 12.5 mg ai/hen/day.

Excreta were collected daily from each animal, and cage wash samples (1:2 water:MeOH) were obtained prior to study initiation and at sacrifice. A composited cage debris sample from all hens was also collected at sacrifice. Eggs were collected twice a day from each hen and separated into whites and yolks. At sacrifice, whole eggs were also excised from 3 hens during necropsy. All samples were stored at -15 °C at the testing facility.

TRR in triplicate subsamples were determined directly by LSC for the cage wash samples, and by combustion with LSC for excreta, G.I. tract, gizzard, and blood. The remaining matrices were radioassayed by LSC following treatment with a tissue solubiliser. The limits of detection (LODs) for the radioassays ranged from 0.0002 mg/kg in egg whites to 0.0011 mg/kg in blood. The recovery of the administered dose was somewhat low, but was consistent between hens, ranging from 80.6–84.5% of the dose and averaging 82.4% of the dose (Table 14).

Table 14 Radioactive residues in eggs, tissue, and excreta

Matrix	Collection Timing (study hours)	Radioactive Residues [a]	
		% Administered dose	mg/kg
Excreta	0-24 h	18.7 ± 0.9	NA
	24-48 h	21.8 ± 1.6	NA
	48-72 h	22.6 ± 0.6	NA
	72-78 h	6.9 ± 0.7	NA
	Total (0-78 h)	70.0 ± 2.1	NA
Cage wash	78 h	0.05 ± 0.04	NA
Cage debris	78 h	< 0.01	NA
Whole Eggs	0-24 h	0.04 ± 0.02	0.421 ± 0.161
	24-48 h	0.23 ± 0.02	2.304 ± 0.353
	48-72 h	0.29 ± 0.03	2.817 ± 0.399
	72-78 h	0.41 ± 0.05	4.251 ± 0.933
	Total (0-78 h)	0.74 ± 0.21	2.209 ± 0.448
Egg whites	0-24 h	0.03 ± 0.02	0.446 ± 0.242
	24-48 h	0.15 ± 0.02	2.147 ± 0.361
	48-72 h	0.14 ± 0.03	1.985 ± 0.412
	72-78 h	0.14 ± 0.01	2.071 ± 0.407
	Total (0-78 h)	0.37 ± 0.09	1.606 ± 0.202
Egg yolks	0-24 h	0.01 ± 0.01	0.351 ± 0.155
	24-48 h	0.08 ± 0.01	2.648 ± 0.338
	48-72 h	0.15 ± 0.01	4.694 ± 0.504
	72-78 h	0.27 ± 0.04	9.011 ± 1.895
	Total (0-78 h)	0.37 ± 0.13	3.542 ± 1.024
Lean meat	78 h	1.96 ± 0.28	3.015 ± 0.279
Skin with attached fat	78 h	0.97 ± 0.17	8.660 ± 1.387
Peritoneal fat	78 h	1.31 ± 0.33	20.978 ± 2.274
Kidney	78 h	0.21 ± 0.02	10.609 ± 0.823
Liver	78 h	1.34 ± 0.07	15.249 ± 0.761
Blood	78 h	0.07	2.961 ± 0.554
Bile	78 h	0.06	147.723 ± 45.868
Gizzard	78 h	5.64	NA
% Recovered dose	NA	82.36 ± 1.57	

[a] Data are the average from radioassays of separate samples from 5 hens.

NA = not applicable.

Radioactive residues remaining in liver solids following solvent extraction (acetonitrile and acetonitrile/water) were subjected to microwave extraction in 2-propanol/water (80:20, v/v) at

temperatures ramping up to 180 ºC. The resulting solvent fraction was then analysed by TLC and HPLC. Liver residues released by the initial solvent extractions were also subjected to a microwave treatment. Solvent-extracted residues were concentrated, redissolved in 2-propanol/water (80:20, v/v) and microwaved at up to 180 ºC. The microwave treated fraction was then analysed by TLC and HPLC.

The aqueous ACN extracts from samples of liver and excreta were also utilized for isolation and detailed identification of individual metabolites. Solubilised ^{14}C-residues from liver and excreta were separated into 6-7 fractions with a C18 column eluted using a step gradient of 0.1% trifluoroacetic acid (TFA) to MeOH. Individual metabolites were then isolated from each fraction using preparative TLC and HPLC.

Extraction with ACN and aqueous ACN released 88.7–99.9% of the TRR from eggs and tissues, and PES accounted for only 0.03–4.6% of the TRR (Table 15). TLC and HPLC analyses of the solubilised ^{14}C-residues identified 79.9–94.8% of the TRR in poultry tissues and eggs (Table 16). Microwave treatment of the solubilized ^{14}C-residues from liver had only a slight quantitative effect on the residue profile.

Radioactive residues in solvent fractions were profiled and quantified by 2D-TLC and reverse-phase HPLC. The TLC system consisted of silica gel plates using one of three solvent systems. Reference standards were visualized by ultraviolet (UV) light (254 nm), and radioactive residues on plates were visualized and quantified using a Bio-Imaging Analyser. The reference standards were detected using a UV detector (220 nm) and 14C-residues were detected and quantified using an in-line radioactivity monitor.

The identity of metabolites was established by co-chromatography with reference standards and by LC-MS and LC-NMR analysis of individual fractions isolated from extracts of liver and excreta. Table 15 and Table 16 summarise the identifications.

Table 15 Summary of characterisation/identification of ^{14}C-residues in tissues and eggs from hens dosed for 4 days with [^{14}C-phenyl]Cyproconazole at levels equivalent to 114.2 ppm in the diet [a]

Compound [b] (fraction)	Muscle		Skin plus fat		Liver		Egg Whites		Egg Yolks	
	TRR = 3.020 mg/kg		TRR = 13.065 mg/kg		TRR = 15.232 mg/kg		TRR = 1.598 mg/kg		TRR = 3.542 mg/kg	
	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg
Cyproconazole (M1/M2)	44.4	1.341	67.1	8.767	27.1	4.128	29.6	0.473	50.1	1.775
NOA 421153 (M9/M14)	31.2	0.942	14.9	1.947	38.1	5.803	44.2	0.706	27.5	0.974
NOA 421154 (M18)	0.6	0.018	0.3	0.039	2.3	0.350	1.5	0.024	1.0	0.035
CGA 123420 (M16)	0.3	0.009	--	--	0.5	0.076	2.0	0.032	--	--
NOA 408616 (M15)	14.1	0.426	1.3	0.170	10.4	1.584	17.5	0.280	10.1	0.358
NOA 405870 (M21)	1.1	0.033	--	--	1.5	0.228	--	--	2.0	0.071
Total identified	91.7	2.769	83.6	10.922	79.9[c]	12.170	94.8	1.515	90.7	3.213
Minor unknowns [d]	1.9	0.057	0.8	0.105	10.5	1.599	3.6	0.058	1.3	0.046
Total characterised	93.6	2.827	84.4	11.027	90.4	13.770	98.4	1.572	92.0	3.259
Total extractable	93.5	2.824	88.7	11.589	90.4	13.770	98.3	1.571	99.9	3.538
Unextractable (PES)	1.8	0.054	1.2[e]	0.157	4.6	0.701	1.3	0.021	1.2	0.043
Accountability [f]	95.3		89.9		95.0		99.6		101.1	

[a] Values for each fraction/metabolite are not corrected for recoveries.

[b] M1 and M2 are diastereoisomers, as are M9 and M4.

[c] Other minor metabolites identified in liver included: M11 (1.4% TRR), M52 (0.7% TRR), M54 (0.8% TRR), and M59 (0.6% TRR).

[d] Unknowns each accounting for ≤ 7.3% of the TRR.

[e] Includes oil fraction (0.9% TRR) from hexane partitioning.

[f] Accountability = (Total extractable + Total unextractable)/(TRR from combustion analysis) × 100.

In excreta (24-78 hours), a total of 17 compounds were identified. The major ^{14}C-residues were comprised of sulfate conjugates (M50, M51, M52, and M53) of primary metabolites; these sulfate conjugates together accounted for 44.8% of the TRR in excreta. Parent accounted for only 2.6% of the TRR. Each of the remaining primary metabolites accounted for ≤ 4.8% of the TRR in excreta and included: M9, M14, M15, M16, M18, M10, M10a, M21, M57, M55, and M56.

Table 16 Summary of ^{14}C-residues in excreta from hens dosed with [^{14}C-phenyl]-Cyproconazole

Fraction/Metabolite	**% TRR** [a]
M50	10.5
M55	1.4
M53	5.5
M15 (NOA 408616)	2.7
M51	9.6
M16 + M10 + M56	3.0
M52 + (M57)	19.2
M21 (NOA 405870)	4.8
M10a	2.2
M18 (NOA 421154)	0.8
M9/14 (NOA 421153)	1.5
M1/M2 (cyproconazole)	2.6
Total Identified	63.9[b]
Minor Unknown Fractions, each ≤ 5.0% TRR	31.8
Total Characterised	94.7
Total extractable	95.8
Residual Solids (PES)	4.2
Accountability	100

[a] Values are corrected to 100%; the actual recovery from extraction of excreta was 106.2%.

[b] Other minor metabolites identified in excreta included: M31 or M48 (0.01% TRR), M38 (0.05% TRR), and M58 (0.25% TRR).

The proposed pathway of metabolism of cyproconazole in laying hens is shown in Figure 2.

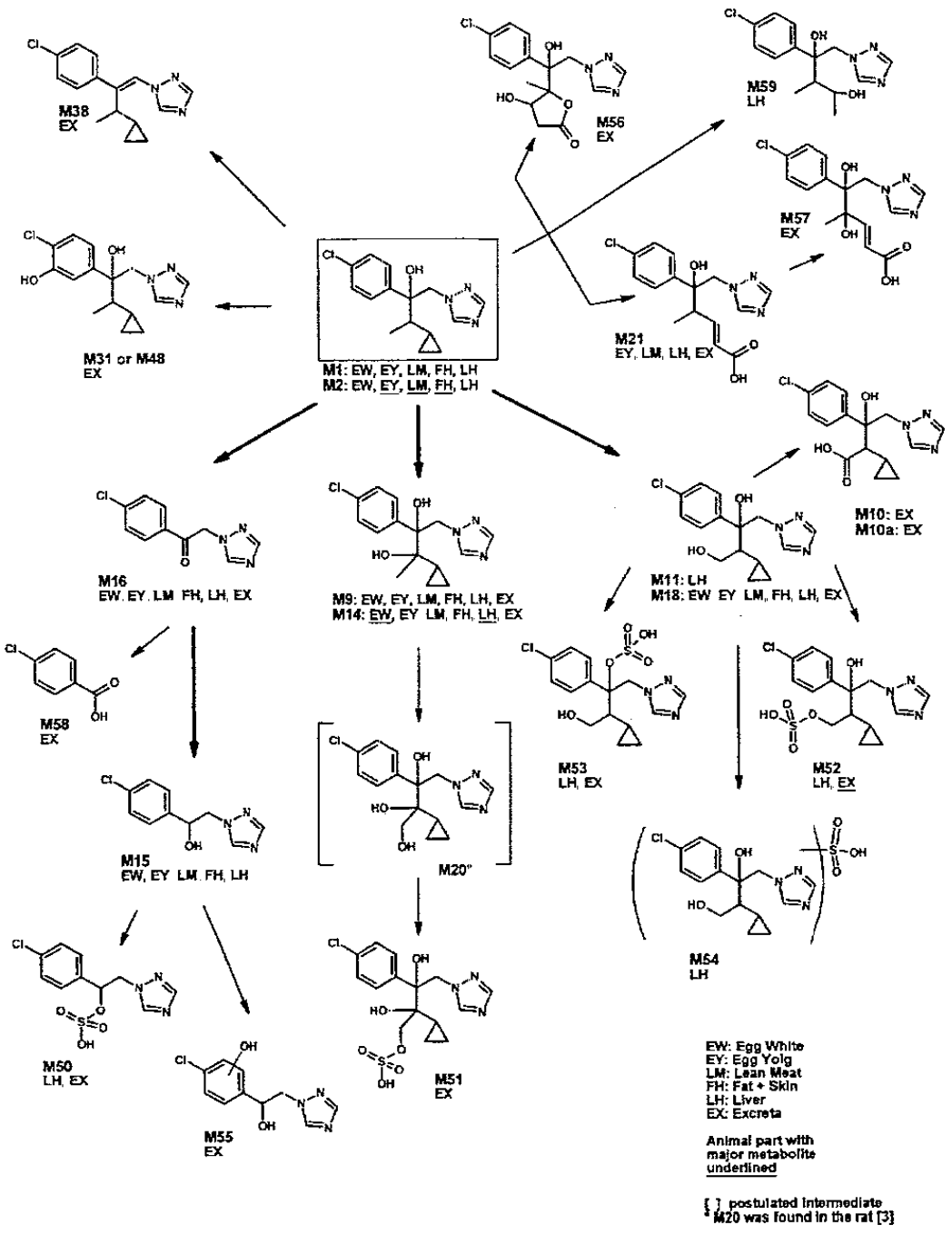


Figure 2 Metabolic profile of cyproconazole in laying hens

Metabolism in rats

Several rat metabolism studies for cyproconazole were supplied to the Meeting (Schweitzer, 1987, Report No. CBK 11738/87, Syngenta File No. SAN619/6086;Karapally, Völlmin, Spielman, 1987, Report No. 31302, CBK 11816/87, Syngenta File No. SAN619/6085). Cyproconazole was found to be extensively metabolised in the male and female rats under all dose regimens. In addition to unchanged parent compound, 35 metabolites were detected in the extracts, among which the 13 more significant ones were isolated. All the other metabolites were only found in very low quantity (≤ 1%). Among the more prominent fractions in urine were NOA421152 (M3 & M4), NOA408616, NOA421154 (M18) and NOA452669 (M30/33). In faeces, NOA421152 (M3 & M4) and NOA421153 (M14) were the major metabolites beside parent. Further metabolites at significant amounts were NOA421152 (M4), NOA421153 (M9), NOA452154 (M10/M10a), NOA451353 (M13), NOA421154 (M18) and NOA452668 (M20).

Plant metabolism

Metabolism studies in which [α-^{14}C]cyproconazole was applied to apples, grapes and grapevine seedlings, peanuts, and wheat; in which [triazole-^{14}C]cyproconazole was applied to wheat; and in which [phenyl-^{14}C]cyproconazole was applied to wheat were made available to the Meeting.

Peanuts (Skinner, et a, 1987, SAN619/608.)

Five peanut plants were grown under greenhouse conditions, and were treated by various schedules. Two plants were treated with an EC formulation once at 100 g ai/ha; one ("time 0") was harvested immediately, and the other ("A") two weeks after the dose, 6 weeks before maturity. Two plants ("B" and "C") were treated 4 times each at 2-week intervals, at a rate of 100 g/hectare, and were harvested at maturity, 2 weeks after the last dose. The fifth plant ("D") was treated once at a rate of 1 kg/hectare three weeks before maturity, and was harvested at maturity. The test substance was applied to the entire aerial portion of the plants with a sable hair brush.

The foliage, seeds, and shells were analysed separately. Extractions were performed sequentially with MeOH, $CHCl_3$, and MeOH. Extracted residues from Plant A were analysed by TLC and LC of certain TLC zones. Slow-eluting fractions were subjected to enzymatic hydrolysis to further release residues. For Plants B, C, and D, residues were analysed by LC. Unextractable residual solids from these plants were refluxed in base, acidified, and partitioned between water and ethyl acetate. The ethyl acetate phase was analysed by LC. Residues which remained unextractable in all plants were analysed by combustion to ^{14}CO followed by LSC. Seeds and shells were weighed, and the ^{14}C residues quantified by LSC of combusted aliquots. Results of the analysis are presented in Tables 17 and 18.

Table 17 Distribution of metabolites in foliage of peanut plants Tteated with ^{14}C-Cyproconazole

% of Total ^{14}C	Time 0	Plant A	Plant B	Plant C	Plant D
Foliage					
(M1,M2) cyproconazole	(98)	(36,43)	(32,35)	(28,32)	(36,32)
M9	0	< 1	2	< 1	2
M14	"	< 1	1	1	< 1
M18	"	< 1	1	1	< 1
Conj. 1	"	3	< 1	< 1	< 2
Conj. 2	"	2	< 2	< 1	< 2
Unextractable		8	14	21	15
Bound (M1/M2)	"	-	8	10	9
Bound M18	"	-	< 1	1	1
Dead Leaves					
extractable	-	-	88	88	99
M1 + M2 cyproconazole	-	-	71	77	91

% of Total ^{14}C	Time 0	Plant A	Plant B	Plant C	Plant D
unextractable	-	-	12	12	1

Table 18 Distribution of radioactivity into peanut foliage, shells, and seeds. after application of ^{14}C-Cyproconazole

Matrix	Plant A		Plant B		Plant C		Plant D	
	mg/kg equiv.	% appl. dose	mg/kg equiv.	% appl. dose	mg/kg equiv.	% appl. dose	mg/kg equiv.	% appl. dose
Foliage	2.72	80.0	4.34	25.4	3.0	15.4	7.17	17.0
Dead leaves	-	-	-	39.1	-	46.8	-	33.4
Seeds	0.004	0.002	< 0.01	< 0.007	< 0.01	< 0.007	< 0.03	< 0.009
Shells	0.001	0.004	0.007	0.004	0.023	0.014	0.11	0.03
Total Recovery		80		65		62		50

The proposed metabolism pathway of cyproconazole in peanut, based on the limited results of the peanut study, is given in Figure 3.

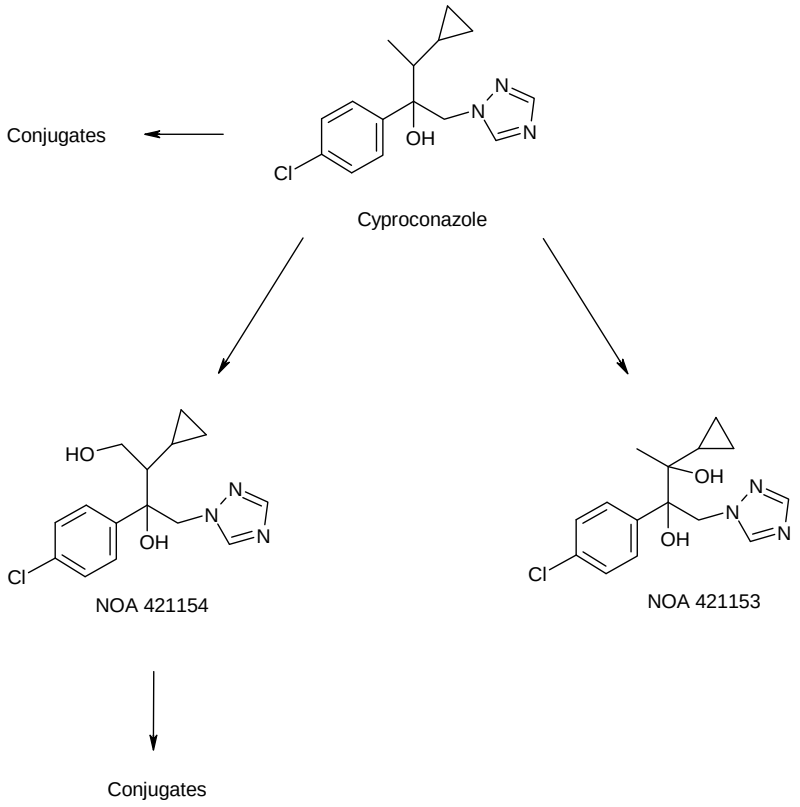


Figure 3 Metabolic Pathways of Cyproconazole in Peanuts

Grapevine Seedlings (Schächtele and Karapall, 1987, SAN619/6078)

Twenty-one 3-week-old grape seedlings grown in a growth chamber were treated one time with a 0.01% solution of ^{14}C-cyproconazole applied to the lower surface of the leaves. Each plant received approximately 25 µg of radioactive chemical, corresponding to 10.8 × 106 disintegrations per minute (dpm) for three plants, and 11.4 × 106 dpm for the other plants. Samples were taken at pre-harvest

intervals (PHIs) of 0, 1, 3, 7, 14, 28, and 49 days, and consisted of treated leaves, untreated leaves and new growth, and stems and roots.

In addition, several glass plates were "treated" with ^{14}C-cyproconazole, each plate receiving 2.5 µg of chemical. The plates were kept under the same conditions as the treated seedlings and were analysed for cyproconazole residues at the same times. Any loss of the chemical from these plates would be due to evaporation. The results were used to estimate the evaporative loss of cyproconazole from the treated leaves.

The three sampling groups were analysed for surface and internal residues. Surface residues from each group were collected by rinsing the plant part with water. The rinse liquid was partitioned with methylene chloride; conjugated residues were subsequently removed from the aqueous partition by enzyme hydrolysis. Remaining residues were collected by homogenizing the sample with MeOH, filtering, and extracting with methylene chloride. Remaining polar and conjugated components in the aqueous layer were released by enzyme hydrolysis. All solutions were analysed for their ^{14}C contents. The amount of radioactivity (as percentage of applied dose) was measured as a function of time on leaves, stems, and roots. Results are summarised in Table 19.

Table 19 Time Dependence of recovered activity after treatment of grapevine seedlings with ^{14}C-Cyproconazole (in % of applied dose)

Days After Application		0	1	3	7	14	28	49
Treated Leaves	Rinse	95.4%	87.0%	78.7%	44.5%	15.3%	7.0%	2.5%
	Extract-able	0.2	3.9	4.3	14.8	18.8	23.4	18.6
	Non-extractable	< 0.1	0.1	0.1	0.5	1.4	2.3	3.5
	Total Residues	95.6	91.0	83.2	59.9	35.5	32.7	24.6
Stems and Roots	Rinse	-	-	-	-	-	-	-
	Extract-able	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	0.2	0.1
	Non-extractable	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	0.1	0.1
	Total Residues	< 0.1	< 0.1	< 0.1	< 0.1	0.1	0.3	0.2
Un-treated Leaves & New Growth	Rinse	< 0.1	< 0.1	< 0.1	0.1	0.1	0.1	0.1
	Extract-able	< 0.1	< 0.1	< 0.1	0.3	0.5	0.6	0.5
	Non-extractable	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	0.1	0.1
	Total Residues	< 0.1	< 0.1	< 0.1	0.4	0.6	0.8	0.7

There was little translation of the chemical to the untreated leaves or the stems and roots. The predominant fate of the chemical was loss through evaporation; only 25% of the total applied radioactivity remained after 49 days. The predominance of this mode for the loss of the chemical was corroborated by the observations from the studies involving the treated glass plates, in which only 10.7% of the applied dose remained after 49 days.

The composition of the surface and internal residues found in the treated leaves is summarised in Tables 20 and 21.

Table 20 Composition of internal residues from grapevine seedling leaves treated with ^{14}C-cyproconazole (in % of applied dose)

DAA [a]	0		1		3		7		14		28		49	
Code	free	conj.	free	conj.	free	conj.	free	conj.	free	conj.	free	conj.	free	conj.
M1	nd	nd	1.5	nd	1.6	nd	3.9	nd	4.1	nd	2.5	nd	0.9	nd
M2	"	"	1.8	"	2.1	"	6.0	0.2	7.2	"	6.7	0.2	3.4	0.2
M9	"	"	nd	"	nd	"	0.2	nd	0.6	"	1.3	0.2	0.9	0.2

DAA [a]	0		1		3		7		14		28		49	
Code	free	conj.	free	conj.	free	conj.	free	conj.	free	conj.	free	conj.	free	conj.
M10	"	"	"	"	"	"	nd	"	nd	"	nd	0.1	nd	0.1
M11	"	"	"	"	"	"	"	0.1	"	0.1	"	0.3	"	0.4
M13[b]	"	"	"	"	"	"	"	0.5	"	0.5	"	1.2	"	1.2
M14	"	"	"	"	"	"	0.4	0.3	1.0	0.5	1.4	0.6	0.7	0.5
M15	"	"	"	"	"	"	0.1	0.6	nd	0.5	nd	1.0	nd	0.7
M18	"	"	"	"	"	"	nd	1.1	"	1.5	"	2.5	"	1.9
M31[b]	"	"	"	"	"	"	nd	nd	"	0.1	"	nd	"	0.1
Polar CH2Cl2 Sol.	"	"	"	"	"	"	0.4	0.2	1.1	0.4	2.5	0.8	3.7	0.9
Plate	"	"	"	"	"	"	nd	nd	nd	nd	nd	nd	0.2	nd
Polar H2O Sol.	"		"		"		0.2		0.4		1.2		1.8	
Tot. Res.	nd	nd	3.3	nd	3.9	nd	11.0	3.0	14.0	3.6	14.5	6.9	9.8	6.2

[a] Days After Application.

[b] Structure not elucidated.

Table 21 Composition of free surface residues from grapevine seedling leaves treated with ^{14}C-cyproconazole[a] (in % of applied dose)

Code	0	1	3	7	14	28	49
M1	42.3	39.4	36.4	20.8	7.4	2.7	0.6
M2	51.7	45.6	41.1	22.1	6.3	2.5	0.6
Polar CH2Cl2 Solubles	< 0.1	0.2	0.2	0.1	< 0.1	0.3	< 0.1
Unk. 1	0.2	0.3	0.2	0.3	0.2	0.2	"
Unk. 2	< 0.1	0.4	< 0.1	0.2	0.1	< 0.1	"
"Plate" Residues	0.5	0.4	0.3	0.1	0.1	"	"
Polar Water Solubles	0.1	0.2	0.1	0.3	0.4	0.5	0.5
Total Residues	94.8	86.5	78.3	44.0	14.5	6.2	1.7

[a] No conjugated metabolites were found. Numbers represent average of three plants per testing.

Grapes (Schächtele and Karapall, 1988, SAN619/6079)

The metabolism of [α-carbon^{14}C]-cyproconazole (radiochemical purity 98%, specific activity 188.3 µCi/mg) was studied in three year old grape vines (variety: Riesling × Silvaner) maintained outdoors in a protective enclosure. The vines were treated four times at 2-week intervals. All vines were sprayed with [α-carbon^{14}C]-cyproconazole formulated as an SL (soluble concentrate) formulation at a rate of 20 g ai/ha. Approximately 20% of the treatment solution was applied directly to the soil surrounding the vines. Twenty-nine days after the final application, separate samples of leaves and fruit were harvested from the vines.

Fruits were rinsed with a surfactant solution and water to remove surface residues and then extracted with MeOH. Non-extracted residues were determined by combustion/LSC. MeOH extracts were partitioned with dichloromethane (DCM) after removal of the solvent under vacuum. The remaining aqueous phase was treated with cellulase enzyme before re-partitioning with DCM. Extracted radioactivity was characterised by two-dimensional TLC and metabolites identified by co-chromatography with reference standards. Residue levels in grapes are presented in Table 22.

Table 22 Radioactive residues in grapes (fruit) following foliar application of [α- carbon^{14}C] Cyproconazole

Grape fraction	TRR		Organic soluble		Aqueous soluble		Conjugated	
	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR
Surface rinse	0.12	27.9	0.11	25.6	0.01	2.3	-	-
Extractable	0.24	55.8	0.18	41.9	0.03	7.0	0.03	7.0
Unextracted	0.08	18.6	-	-	-	-	-	-
Total	0.43	-	0.29	67.5	0.04	9.3	0.03	7.0

Treatment of aqueous soluble radioactivity with cellulase enzyme released small amounts (up to 2.0% TRR) of sugar conjugated cyproconazole and metabolites NOA421152, NOA451353, NOA421153, NOA421154, NOA408616, and M5 in addition to polar organic and water soluble material (up to 4.1% TRR). Characterisation of the radioactive residues in grape fruits is summarised in Table 23.

Table 23 Characterisation of radioactive residues in grapes (fruit) following foliar application of [α-carbon^{14}C]-Cyproconazole

Metabolite	Grape Fruit (Percent of TRR)								
	Surface			Sub-surface			Whole Fruit		
	Free	Conj.	Total	Free	Conj.	Total	Free	Conj.	Total
Cyproconazole	23.5	n.a.	23.5	39.5	0.2	39.7	63.0	0.2	63.2
NOA421152/M5[a]	< 0.1	n.a.	< 0.1	< 0.1	0.3	0.3	< 0.1	0.3	0.3
NOA421153/ NOA451353[a]; M9/M14, M13	0.3	n.a.	0.3	1.3	0.2	1.4	1.5	0.2	1.6
NOA408616; M15	< 0.1	n.a.	< 0.1	0.4	1.8	2.2	0.4	1.8	2.2
NOA421154; M11/M18	0.3	n.a.	0.3	0.5	2.0	2.4	0.6	2.0	2.6
Polar/CH2Cl2 soluble	0.2	n.a.	0.2	2.0	1.9	3.9	2.2	1.9	4.1
Unknown 1	0.2	n.a.	0.2	< 0.1	< 0.1	< 0.1	0.2	< 0.1	0.2
Unknown 2	< 0.1	n.a.	< 0.1	0.1	< 0.1	0.1	0.1	< 0.1	0.1
TLC1	< 0.1	n.a.	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
TLC2	0.5	n.a.	0.5	1.0	0.1	1.1	1.5	0.1	1.6
TLC3	0.3	n.a.	0.3	0.5	0.2	0.7	0.8	0.2	1.0
TLC4	0.3	n.a.	0.3	0.4	< 0.1	0.4	0.7	< 0.1	0.7
Polar/H2O soluble	-	1.2	1.2	-	2.3	2.3	-	3.4	3.4
Non-extractable	-	-	-	-	-	-	-	-	18.7
Total	25.4	1.2	26.6	45.6	8.9	54.5	71.0	10.1	99.8

n.a. = not analysed.

[a] Metabolite structures not identified in this study.

The proposed metabolism pathway of cyproconazole in grapes is given in Figure 4.

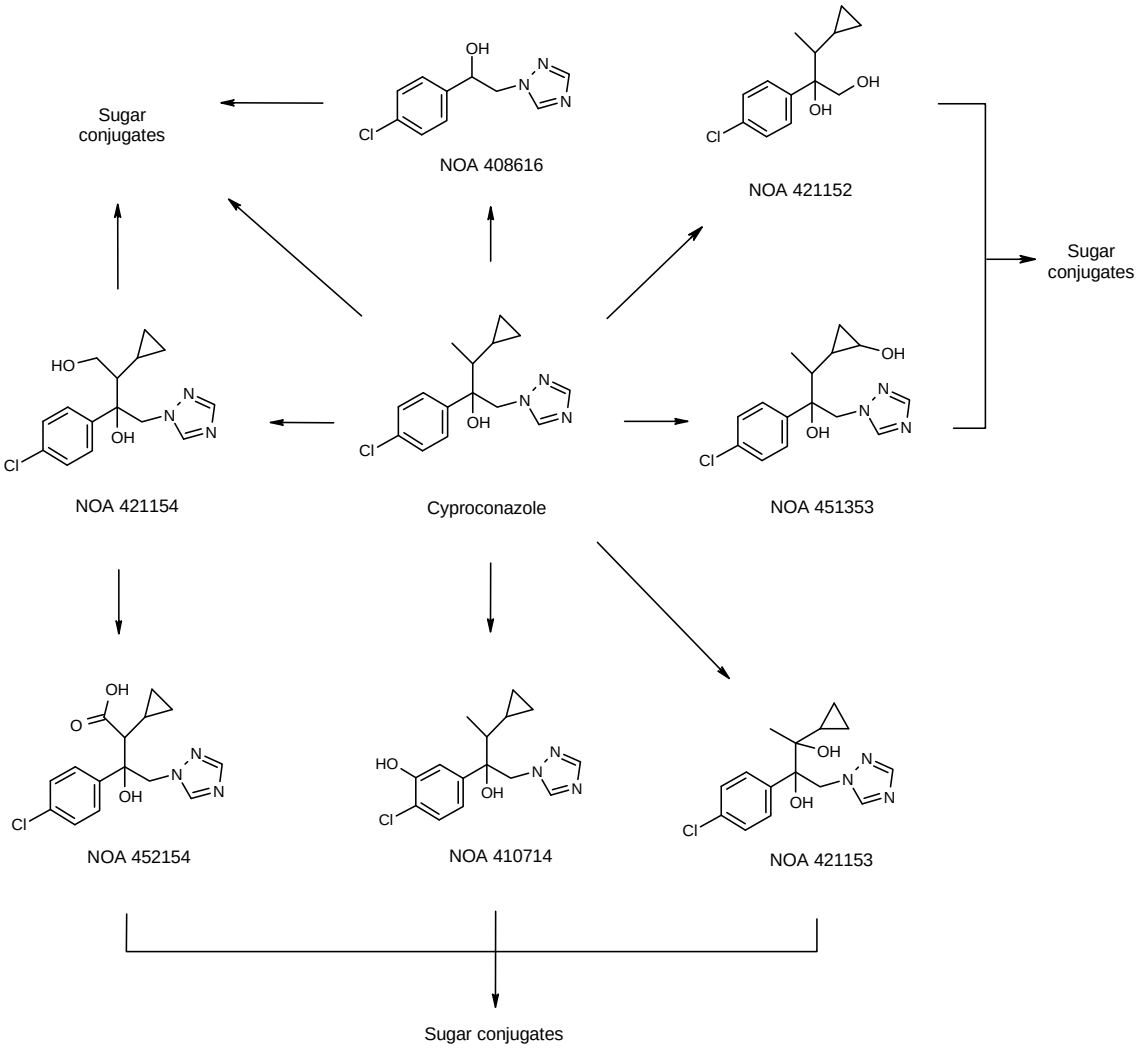


Figure 4 Metabolic Pathways of Cyproconazole in Grapes

Apples (Schächtele and Karapally, 1988, SAN 619/6081))

The metabolism of [α-carbon14C]-cyproconazole (radiochemical purity 98%, specific activity 188.3 µCi/mg) was studied in three year old apple trees (variety: Glockenapfel) grown outdoors in a protective enclosure. The trees were treated four times at 2-week intervals. All trees were sprayed with [α-carbon^{14}C]-cyproconazole as an SL formulation at a rate of 40 g ai/ha. Approximately 20% of the treatment solution was applied directly to the soil surrounding the trees. Twenty-eight days after the final application, leaves and apples were harvested and analysed using methods similar to those previously described for grapes.

Analysis of apples showed that surface residues contained 17% of the TRR, with extracted and unextracted residues containing 69.6 and 11.9% of the TRR, respectively. Surface residues were composed primarily of cyproconazole and minor amounts of unidentified metabolites and polar water soluble radioactivity. Parent cyproconazole was the major residue in apple extracts, accounting for a total of 76.4% of the TRR. Treatment of aqueous soluble radioactivity with cellulase enzyme released a small amount (0.3% TRR) of sugar conjugated NOA421154, in addition to polar organic and water

soluble material totalling 1.8% of the TRR. Characterisation of radioactive residues in apple fruits is summarised in Table 24.

Table 24 Characterisation/identification of radioactivity in apples (fruits) following foliar application of [α-carbon^{14}C]-Cyproconazole

Metabolite	Apple Fruit (Percent of TRR [a])								
	Surface			Sub-surface			Whole Fruit		
	Free	Conj.	Total	Free	Conj.	Total	Free	Conj.	Total
Cyproconazole	16.7	n.a.	16.7	59.7	< 0.2	59.7	76.4	< 0.2	76.4
NOA421152/M5[b]; M13	< 0.1	n.a.	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
NOA421153/NOA451353[b]; M9/M14, M13	< 0.2	n.a.	< 0.2	2.8	< 0.2	2.8	2.8	< 0.2	2.8
NOA421154; M11/M18	< 0.2	n.a.	< 0.2	0.9	0.3	1.1	0.9	0.3	1.1
NOA408616; M15	< 0.1	n.a.	< 0.1	0.5	< 0.1	0.5	0.5	< 0.1	0.5
Polar/CH2Cl2 soluble	< 0.1	n.a.	< 0.1	2.3	0.5	2.8	2.3	0.5	2.8
Unknown 1	0.3	n.a.	0.3	0.8	< 0.1	0.8	1.1	< 0.1	1.1
TLC1	< 0.1	n.a.	< 0.1	0.1	< 0.1	0.1	0.1	< 0.1	0.1
TLC2	0.1	n.a.	0.1	0.4	< 0.1	0.4	0.5	< 0.1	0.5
TLC3	0.2	n.a.	0.2	0.3	< 0.1	0.3	0.5	< 0.1	0.5
TLC4	0.1	n.a.	0.1	0.4	< 0.1	0.4	0.5	< 0.1	0.5
Polar/H2O soluble	-	0.6	0.6	-	0.7	0.7	-	1.3	1.3
Non-extractable	-	-	-	-	-	-	-	-	11.9
Total	17.4	0.6	18.0	68.2	1.4	69.6	85.6	2.0	99.5

n.a. = not analysed.

[a] Mean of fruit from 2 trees.

[b] Metabolite structures not identified in this study

The proposed metabolism pathway of cyproconazole in apples is given in Figure 5.

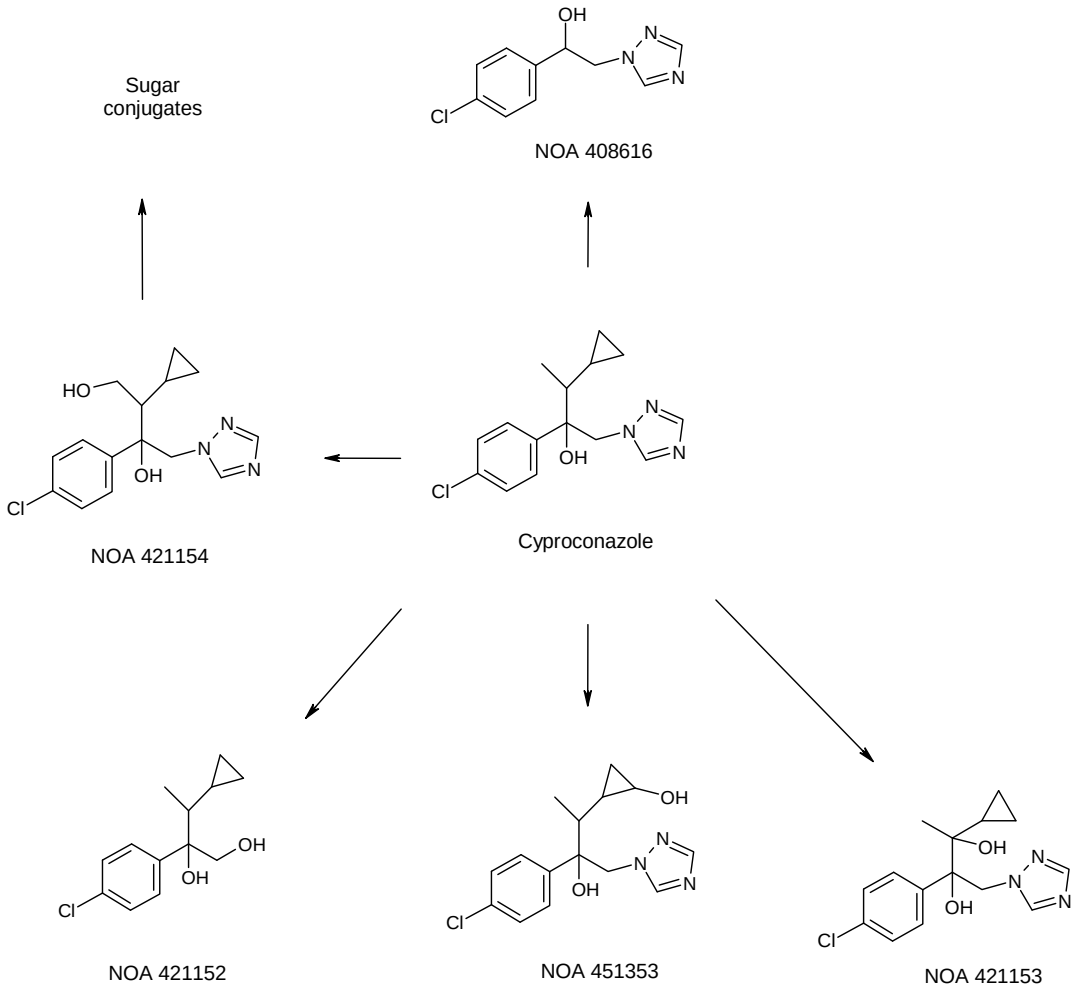


Figure 5 Metabolic Pathways of Cyproconazole in Apples

Wheat: central α-labelled [^{14}C]-labelled (Schächtele and Karapally, 1987, SAN619/5523)

Two plots of 14-week-old wheat plants were transferred to a container in an "outdoor shed." There, they were treated twice at an interval of 4 weeks with a soluble concentrate formulation of ^{14}C-cyproconazole at a rate of 80 g ai/hectare (32.4 g ai/A). The plants were harvested 34 days after the final application and separated into their various components (leaves, stems, ears, and grain). Only the results for the grain were reported. Extraction and analysis procedures for the surface and internal residues of the grain were identical to those for apples and grapes (see above). Combined analysis results for the two plots are presented in Table 25.

Table 25 Composition of recovered radioactivity in wheat grain (expressed as % of total recovered activity)

Code [a]	Surface (%)			Internal (%)			Total (%)		
	free	conj.	total	free	conj.	total	free	conj.	total
M1	18.0	n.a.	18.0	6.8	< 0.1	6.8	24.8	< 0.1	24.8
M2	14.1	"	14.1	6.6	0.1	6.7	20.7	0.1	20.8
M3/4/5	< 0.1	"	< 0.1	0.4	0.4	0.8	0.4	0.4	0.8
M9	"	"	"	2.1	< 0.1	2.1	2.1	< 0.1	2.1
M11	"	"	"	0.5	0.4	0.9	0.5	0.4	0.9

Code [a]	Surface (%)			Internal (%)			Total (%)		
	free	conj.	total	free	conj.	total	free	conj.	total
M13[b]/14	"	"	"	2.1	1.8	3.9	2.1	1.8	3.9
M15	"	"	"	0.3	0.3	0.6	0.3	0.3	0.6
M18	"	"	"	0.5	1.4	1.9	0.5	1.4	1.9
Polar CH2Cl2 Solubles	0.4	"	0.4	1.8	1.4	3.2	2.2	1.4	3.6
Polar Water Solubles	-	2.4	2.4	-	3.9	3.9	-	6.5	6.5
Bound Residues	-	-	-	-	-	-	-	-	22.7
"Plate" Activity	4.0	"	4.0	4.5	2.8	7.3	8.5	2.8	11.3
Total Residues									99.9

[a]M1 and M2 are diastereomers of parent compound.

[b] Structure not elucidated.

A significant portion of the "recovered" residues (44.1%) could not be identified (i.e., bound, polar, or "plate" activity).

Wheat: [U^{14}C]phenyl-labelled (1994; Smith and Wisson)

Winter wheat plants were grown in the greenhouse. [Phenyl(U)-^{14}C]-cyproconazole (1760 mCi/mg) was applied as a 100 g/L SL formulation to winter wheat plants grown in a greenhouse at a rate of 200 g ai/ha. Two applications were performed. Forage samples were taken 11 days after the first application and grain and straw samples were harvested 41 days after the second application.

Tissue samples were ground to a powder and the TRR was determined by combustion (Table 26).

Tissues were washed with water, ground in MeOH and the debris extracted with hot water. The debris was hydrolysed in boiling 1N HCl and 10N NaOH. The crude aqueous extracts were partitioned with DCM or pentane. The TRR was thus separated into three fractions: aqueous-soluble, organic-soluble and bound (Table 26).

Table 26 Distribution of TRR in wheat RACs Treated with phenyl-labelled cyproconazole

RAC	TRR (mg/kg)	Aqueous-Soluble		Organic-Soluble		Bound	
		mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR
Forage	1.76	0.476	27.	1.19	68	0.022	1.2
Straw	5.51	1.87	34.	2.89	52.	0.088	1.6
Grain	0.094	0.074	79.	0.009	9.6	0.001	1.1

The organic and aqueous extracts were analysed by 2-D and 1-D TLC, GC/MS and/or HPLC and the elution profiles compared with that of reference standards. Identifications are summarised in Table 27. The bound residues were characterised as associated with cellulose and lignin

Table 27 Identification of residues in wheat RACs treated with phenyl-labelled cyproconazole

Metabolite	Forage		Straw		Grain	
	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR
Cyproconazole	1.064	60.3	2.436	44.2	0.014	14.9
M9/M14	0.059	3.3	0.184	3.3	0.013	13.8
M15	< 0.001	< 0.1	0.017	0.3	< 0.001	< 0.1
M16	< 0.001	< 0.1	0.010	0.2	< 0.001	< 0.1
C3/C5	0.094	5.3	0.383	7.0	0.014[a]	14.9
C6	0.043	2.4	0.192	3.5	-	-
Very Polar	0.082	4.6	0.182	3.3	0.007	7.4
Others	0.234	13.3	1.248	22.7	0.025	26.6
Total Identified	1.260	71.4	3.212	58.7	0.041	43.6

[a] Total Conjugates.

Wheat: [U^{14}C]triazole-labelled (1994; S. Völlmin)

[^{14}C]Cyproconazole uniformly labelled in the triazole ring (~1:1 mixture of diastereomers, radiochemical purity of > 98%, specific activity of 53.0 mCi/mmol) was diluted with MeOH and mixed with unlabeled cyproconazole and blank formulation (100 SL). Wheat plants which had been sown in an open field and then transferred to six containers (~300 plants per container) which were placed in a greenhouse were treated with [^{14}C]cyproconazole at rates equivalent to 100 g ai/ha or 0.089 lb ai/A (two containers) and 160 g ai/ha or 0.14 lb ai/A (three containers). The sixth container was not treated. Application was repeated 18 days later.

Samples of forage were collected 11 days following the first treatment. Samples of grain and straw (entire plant remaining after collection of the grain) were collected 42 days following the second treatment. Samples were stored frozen (-20 ºC) prior to analysis.

Samples of forage, grain, and straw were ground into small pieces and then analysed for total radioactive residues by combustion/LSC; forage and straw were lyophilized prior to grinding. The TRR in samples of wheat commodities are presented in Table 28.

Table 28 TRR in/on Wheat Forage, Grain, and Straw Following One (forage) or Two (grain and straw) Treatments with [U^{14}C]triazole-Cyproconazole

Wheat matrix	TRR, mg/kg [^{14}C]cyproconazole equivalents [a]	
	0.10 kg ai/ha	0.16 kg ai/ha
Forage	0.971, 1.169 (1.070)	2.328, 2.341, 2.343 (2.337)
Grain	0.201, 0.217 (0.209)	0.257, 0.314, 0.356 (0.309)
Straw	4.039, 4.699 (4.369)	4.905, 5.576, 7.322 (5.934)

[a] Each value represents one sample; average residues in parentheses.

Samples of wheat commodities were subjected to extraction and hydrolysis procedures for residue characterisation and identification. During the fractionation procedures, aliquots of extracts, hydrolysates, and non-extractable residues were analysed for radioactivity by LSC or combustion/LSC.

DCM extracts were analysed by two-dimensional TLC on pre-coated silica-gel plates with fluorescent indicator. Metabolites were identified by co-chromatography with non-labelled reference standards of cyproconazole, M9, M11, M14, M15, M16, M18, M39 (triazole alanine), and M40 synthesized by Sandoz. Radioactivity was visualized using a radioactivity imager and quantified by integration using imager software.

Extracts were analysed by reversed-phase HPLC for the determination of polar metabolites. The HPLC was equipped with an RP 18 or RP 100-8 column, a UV detector, and a radioactivity monitor. Some extracts were cleaned up on Sephadex or BondElut columns prior to HPLC analysis.

Several polar and unknown metabolites were observed in forage and straw. To identify these metabolites, they were isolated from samples of forage and straw from the high-treatment rate samples. The samples were extracted three times with MeOH and the MeOH was removed by evaporation. The aqueous residue was partitioned with DCM under neutral and acidic conditions, and then several times with EtOAc under neutral, acidic, and alkaline conditions. All EtOAc extracts were combined, evaporated to dryness, and redissolved in water. Based on TLC and LSS analyses, it was determined that the EtOAc extracts obtained under neutral and acidic conditions contained the major amounts of polar metabolites; these extracts were subjected to preparative TLC and HPLC separation procedures for isolation. The isolated metabolites were then identified by spectroscopic procedures including 1H NMR and LC/MS.

The proposed structures of conjugated metabolites were confirmed by hydrolysis of the isolated metabolites with enzymes and hydrolysis of samples with HCl. Enzyme hydrolysis was conducted by incubating the metabolite with β-glucosidase in pH 5.5 phosphate buffer for 15 hours at

37 ºC. Chemical hydrolysis was conducted by incubating the samples in 0.5 N HCl for up to 96 hours at 37 ºC. The hydrolysates were partitioned with DCM and EtOAc and the hydrolysis products were identified by co-chromatography with reference standards using two-dimensional TLC.

A summary of the characterised/identified residues found in wheat commodities is presented in Table 29.

Table 29 Summary of Radioactive Residues Characterised/Identified in Wheat Commodities Treated with [U^{14}C]triazole-Cyproconazole

Fraction	Forage – 0.10 kg ai/ha		Grain – 0.10 kg ai/ha		Grain – 0.16 kg ai/ha		Straw – 0.16 kg ai/ha	
	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
Identified								
Cyproconazole (M1)	32.0	0.344	1.3	0.003	1.0	0.003	21.5	0.943
Cyproconazole (M2)	40.9	0.440	2.2	0.005	1.7	0.004	30.3	1.327
M9	2.5	0.027	1.9	0.004	1.9	0.006	2.5	0.110
M11/M14	1.3	0.014	1.8	0.004	1.8	0.005	1.7	0.074
M15/M18	--	--	< 0.5	< 0.001	< 0.5	< 0.001	1.3	0.055
M16	< 0.5	< 0.01	--	--	--	--	< 0.5	0.015
M39	--	--	61.5	0.128	63.0	0.194	--	--
M41	3.3	0.037	--	--	--	--	9.5	0.419
M42	2.9	0.029	--	--	--	--	4.2	0.249
M43	< 0.5	< 0.01	--	--	--	--	2.4	0.099
M44	--	--	--	--	--	--	0.8	0.037
M45	--	--	--	--	--	--	1.0	0.043
M46	4.2	0.044	--	--	--	--	1.8	0.078
M47	4.8	0.052	--	--	--	--	3.4	0.153
Total identified	91.9	0.983	68.7	0.144	69.4	0.214	80.4	3.513
Characterised								
Unknowns	9.2[a]	0.098	< 0.5	< 0.001	< 0.5	< 0.001	13.6[b]	0.594
TLC origin	2.5	0.027	8.3	0.018	7.5	0.022	1.4	0.060
Aqueous	1.0	0.011					2.3	0.102
DCM-soluble at pH 2	--	--	2.3	0.005	1.5	0.005	--	--
Total identified/characterised	104.6	1.119	79.3	0.166	78.4	0.242	97.7	4.269
Non-extractable	1.6	0.018	9.0	0.015	8.9	0.021	0.7	0.029

[a] Each < 1.7% TRR (< 0.02 mg/kg).

[b] Each < 1.6% TRR (< 0.07 mg/kg).

[c] Sum of radioactivity in aqueous fractions following extraction with EtOAc or DCM.

A metabolic pathway for cyproconazole in wheat is given in Figure 6. Cyproconazole may be metabolized by: (i) oxidative elimination of the triazole ring and conversion to triazole alanine; (ii) hydroxylation of the methyl- and cyclopropyl-substituted carbon to form M9/M14; (iii) oxidation of the methyl group to form M11/M18; (iv) elimination of the cyclopropyl-substituted carbon to form the benzylic alcohol (M15) and further oxidation to the ketone (M16); (v) hydroxylation of the cyclopropyl ring and the phenyl ring followed by conjugation to form M43 and M44/M45; and (vi) conjugation of metabolites to form glucosides.

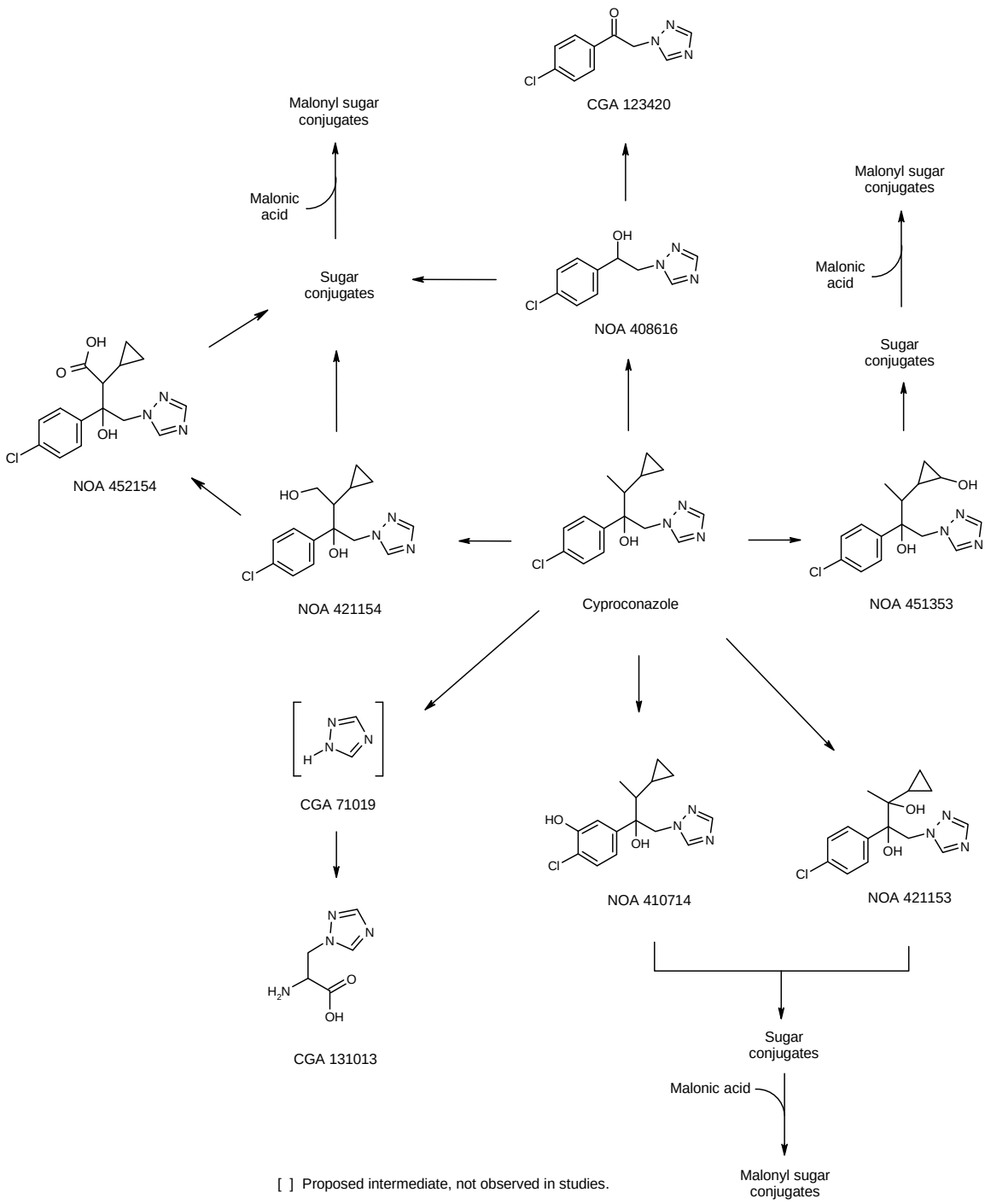


Figure 6 Metabolic Pathways for Cyproconazole in Wheat

Sugar beet: [U^{14}C]triazole-labelled (Völlmin, 1997, SAN619/0396)

The metabolism of [U-triazole^{14}C]-cyproconazole (radiochemical purity 98%, specific activity 90.1 µCi/mg following isotopic dilution) was studied in sugar beet plants (variety: Gala) grown outdoors in protected containers. Initial application was made at growth stage BBCH 41 with a second application made 21 days later at BBCH 48. One set of plants was sprayed at 80 g ai/ha per application, with a separate set of plants sprayed at 120 g ai/ha per application. In each case, [U-triazole^{14}C]-cyproconazole was formulated as Alto 100 SL. Treated plants were harvested 28 days after the last treatment and separated into roots and leaves with tops.

TRR in roots and leaves with tops were determined by combustion/LSC. Samples were extracted with MeOH/water (8:2 v/v) and partitioned with DCM after removal of the solvent under vacuum. Residues in the leaves with tops samples were further extracted with hot water and hydrolysed with 0.1M HCl (80°C, 24 hours). Extracts were analysed by 2D-TLC and HPLC and metabolites were identified by co-chromatography with authentic reference standards. Residue levels and the distribution of radioactivity in sugar beets are shown in Table 30.

Table 30 TRR in Sugar Beets Following Foliar Treatment with [U-triazole^{14}C]-Cyproconazole

Fraction	TRR (mg/kg)	
	2 x 80 g ai/ha	2 × 120 g ai/ha
Roots	0.022	0.107
Leaves with Tops	2.953	4.223

Characterisation of radioactive residues in sugar beet plants is shown in Table 31.

Table 31 Characterisation and Identification of Radioactivity in Sugar Beets Following Foliar Application of [U-triazole^{14}C]-Cyproconazole

Fraction / Metabolite	Roots				Leaves with Tops			
	2 × 80 g ai/ha		2 × 120 g ai/ha		2 × 80 g ai/ha		2 × 120 g ai/ha	
	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
Organic soluble:	79.9	0.018	90.8	0.097	81.9	2.419	83.1	3.509
Cyproconazole	78.6	0.017	80.8	0.086	76.2	2.249	76.8	3.240
NOA421153; M9/M14	0.8	< 0.001	4.0	0.005	1.5	0.045	2.5	0.104
NOA421154; M11/M18	-	-	1.9	0.002	-	-	-	-
NOA408616; M15	-	-	0.9	0.001	0.9	0.027	0.9	0.036
CGA123420; M16	-	-	n.d.	n.d.	0.5	0.014	0.4	0.018
Unknown 1	-	-	1.1	0.001	0.1	0.004	0.4	0.016
Unknown 2	-	-	0.5	< 0.001	0.2	0.005	-	-
Unknown 3	-	-	0.9	0.001	2.6	0.077	2.2	0.095
Unknown 4	0.5	< 0.001	0.7	< 0.001	-	-	-	-
Water-soluble:	23.7	0.005	17.2	0.018	8.4	0.248	13.1	0.551
PRP-1 column: fraction 1	19.5[a]	0.004	-	-	0.9	0.027	-	-
PRP-1 column: fraction 2	0.1	< 0.001	-	-	0.2	0.005	-	-
PRP-1 column: fraction 3	4.1	< 0.001	-	-	7.3[b]	0.216	-	-
Hot water extract	-	-	-	-	0.8	0.023	-	-
0.1M HCl hydrolysis	-	-	-	-	0.3[c]	0.008	-	-

Fraction / Metabolite	Roots				Leaves with Tops			
	2 × 80 g ai/ha		2 × 120 g ai/ha		2 × 80 g ai/ha		2 × 120 g ai/ha	
	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
Non-extractable	3.2	< 0.001	4.6	0.005	1.3	0.038	-	-
Total	106.7	-	112.7	-	92.6	-	98.9	-

[a] Represents 10 polar compounds, each containing < 10% TRR (< 0.002 mg/kg)

[b] Represents cyproconazole, NOA421153, NOA421154 and at least 7 polar components, each < 1% TRR (< 0.03 mg/kg).

[c] Represents mainly cyproconazole.

The proposed metabolism pathway of cyproconazole in sugar beets is given in Figure 7.

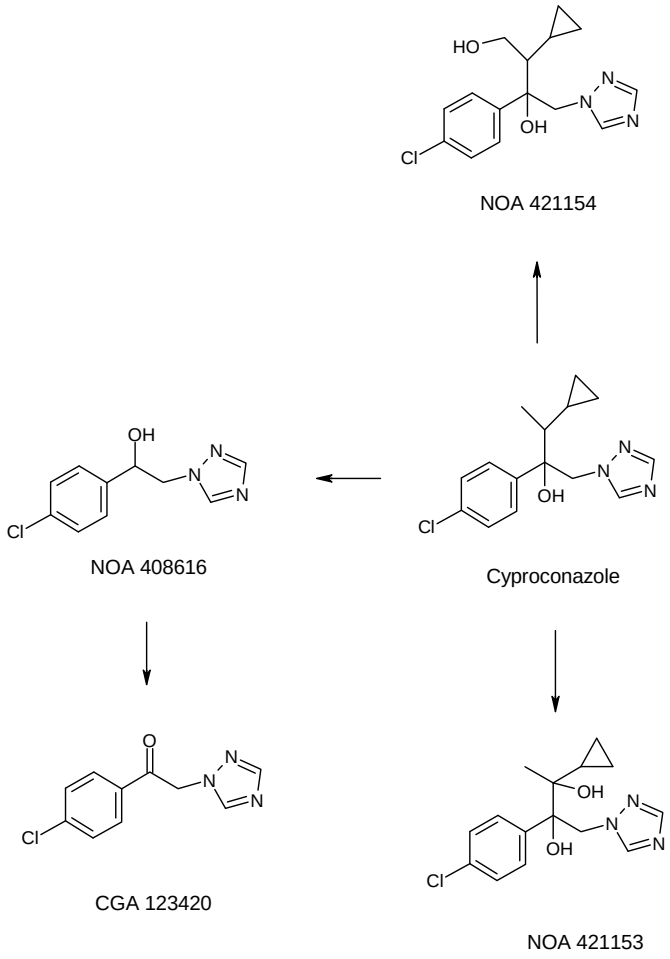


Figure 7 Pathways of Cyproconazole Metabolism in Sugar Beets

Plant Metabolism following Seed Treatment

Wheat: [U^{14}C]triazole-labelled (Caley and Kingsley, 1997, SAN619/0357)

The metabolism of [U-triazole^{14}C]-cyproconazole (radiochemical purity > 97%, specific activity 181.5 µCi/mg was studied in spring wheat (variety: Baldus). Wheat seeds were treated with [U-triazole^{14}C]-cyproconazole formulated as a seed treatment (0.5% w/v) at a rate of 1 g ai/100 kg seed

applied by rotating the seeds in a flask containing the formulation. Sub-samples of treated seed were analysed to confirm the application rate and homogeneity. Seed was sown outdoors in a field plot (5 m×5 m) at a rate of 15 g seed/m2.

Plants were harvested at the beginning of heading (BBCH growth stage 51) and at maturity. Mature plants were separated into straw and grain and the total residues in each sample were determined by combustion/LSC.

Wheat forage, grain, and straw each contained total radioactive residues less than 0.001 mg/kg, indicating transport of residues from the treated seed to the plant was negligible. No attempt was made to characterise residues due to the low levels of radioactivity in the harvested samples.

Summary of plant metabolism studies

Cyproconazole was identified as the major residue in apples, grapes, wheat forage and straw, accounting for 44–76% of the TRR, and accounting for 5–45% of the TRR in wheat grain. The major identified residues in wheat grain were TA (62% TRR) in the ^{14}C-triazole-labeled study, and M9/M14 (14% TRR) and glycoside conjugates of M11/M18 (15% TRR) in the ^{14}C-phenyl-labeled study. In addition, a number of minor metabolites have been identified in plants, including M9/M14, M11/M18, M15, M16 and various glycoside conjugates of parent and these primary metabolites.

The data from these studies indicate that cyproconazole is metabolized in plants by: (i) hydroxylation of the methyl- and cyclopropyl-substituted carbon to form M9/M14; (ii) oxidation of the methyl group to form M11/M18; (iii) elimination of the cyclopropyl-substituted carbon to form the benzylic alcohol (M15) and further oxidation to the ketone (M16); (iv) hydroxylation of the cyclopropyl ring and the phenyl ring; (v) conjugation of parent and hydroxylated metabolites to form various glycosides; and (vi) oxidative elimination of the triazole ring and its subsequent conversion to triazole alanine. See Figure 8.

Summary overall animal and plant metabolism

The situation is summarised in Figure 8.

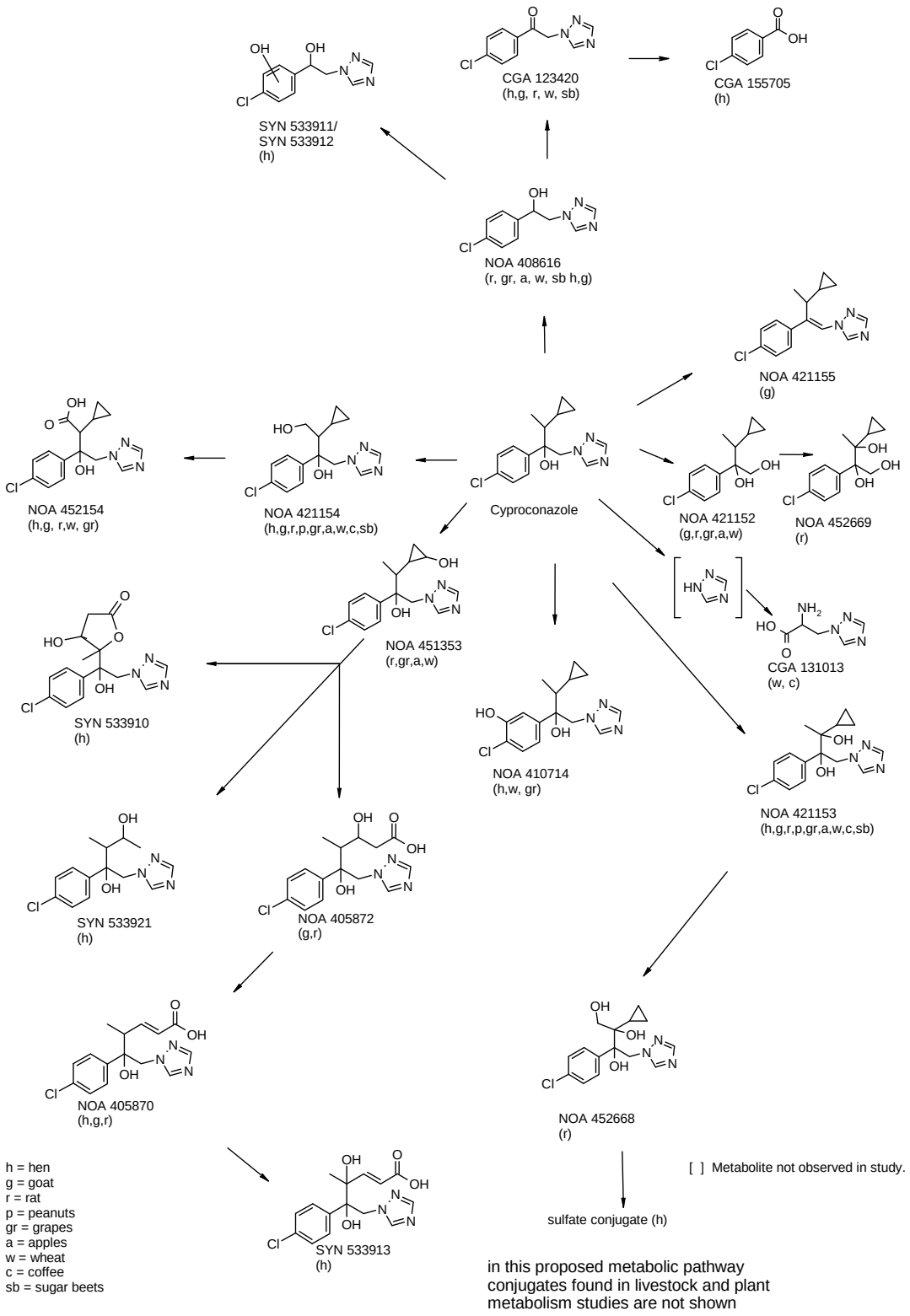


Figure 8 Metabolic Pathways of Cyproconazole in Animals and Plants

Environmental Fate in Soil

The Meeting received information on confined (radiolabelled) rotational crops, field crop rotation, aerobic degradation in soil, anaerobic degradation in soil, soil photolysis, rate of degradation in soil, adsorption and desorption in soil, mobility in soil, hydrolysis, aqueous photolysis, biological degradation, and bioaccumulation in fish. Only those data relevant to the current evaluation are reported below (FAO Manual 2009).

Confined Rotational Crop Studies

A confined rotational crop study (Skinner, Collier, Quisted, 1987, PA-B86-06, SAN 619/6030) was conducted in the USA. . Clay pots (8" dia.) containing a sandy loam soil were treated with [^{14}C]cyproconazole and aged in the greenhouse. No primary crop was grown in the treated soil, but the pots were watered routinely. After 30 and 90 days of aging, lettuce, wheat and sugar beets were planted as representative rotational crops and grown to maturity. Greenhouse temperatures ranged from 18-27 ºC and artificial light was used to provide a 12-hour light/12-hour dark cycle.

Prior to application the [α-^{14}C]cyproconazole was isotopically diluted with non-radiolabelled cyproconazole formulated as a soluble concentrate. The final specific activity of the test material was 126,064 dpm/µg. The ^{14}C-test material was diluted with ACN:water (1:1) for application.

Samples of lettuce (roots and leaves) and wheat (grain and straw plus chaff) were harvested at normal maturity, and sugar beets were harvested when the roots were ~10 cm in length. For both the 30- and 90-day plantback intervals (PBIs), the planting to harvest intervals (DAP) were 42–48 days for lettuce, 70–76 days for wheat, and 89–91 days for sugar beets. Soil samples were also collected immediately after treatment [0 days after treatment (DAT)], at each planting (30 and 90 DAT), and at each harvest (78–181 DAT). The report stated that samples were stored in a walk-in freezer.

Levels of total radioactivity in minced plant samples and homogenized soil samples were determined by combustion with LSC. The LOD for the radioassays was not reported. TRR in soil were 0.046 mg/kg immediately after treatment and remained relatively constant from 30 to 160 DAT at 0.034–0.046 mg/kg (Table 32). A slight decline in soil residues was noted in the final soil sample at 181 DAT (0.026 mg/kg). In the rotational crops, TRR were higher at the 30-day PBI than at the 90-day PBI for all matrices with the exception of lettuce roots.

Table 32 TRR in Soil and Lettuce, Wheat, and Sugar Beet Commodities following a Single Soil Application of [^{14}C]Cyproconazole at 0.10 kg ai/ha

Crop Matrix	Plant-back interval (days)	Harvest interval [a]		TRR (mg/kg)[b]
		DAT	DAP	
Lettuce Foliage	30	78	48	0.040
	90	132	42	0.032, 0.035, 0.027 [0.031]
Lettuce Root	30	78	48	0.032
	90	132	42	0.048, 0.057, 0.075 [0.060]
Wheat Straw	30	106	76	0.52, 0.46, 0.35 [0.44]
	90	160	70	0.17, 0.16, 0.21 [0.18]
Wheat Grain	30	106	76	0.010, 0.07, 0.011 [0.009]
	90	160	70	0.003, 0.004, 0.004 [0.004]
Sugar Beet Foliage	30	119	89	0.091, 0.110, 0.078 [0.092]
	90	181	91	0.035, 0.018, 0.032 [0.028]
Sugar Beet Root	30	119	89	0.010, 0.008, 0.008 [0.009]
	90	181	91	0.009, 0.006, 0.009 [0.008]
Soil - 0 days post-treatment	NA	0	--	0.046
30 days post-treatment	NA	30	--	0.042
Lettuce harvest (30-day PBI)	30	78	--	0.034
90 days post-treatment	NA	90	--	0.039
Wheat harvest (30-day PBI)	30	106	--	0.042
Sugar beet harvest (30-day PBI)	30	119	--	0.042
Lettuce harvest (90-day PBI)	90	132	--	0.046
Wheat harvest (90-day PBI)	90	160	--	0.037
Sugar beet harvest (90-day PBI)	90	181	--	0.026

[a] DAT = days after soil treatment; DAP = days after planting.

[b] Selected samples were analysed in triplicate; average TRR values are reported in brackets.

With the exceptions of lettuce root and wheat grain samples from the 90-day PBI, which were only radioassayed, plant samples were sequentially extracted with MeOH, chloroform, and MeOH. ^{14}C-Residues in sample extracts from lettuce samples were analysed directly by HPLC, and extracts from wheat grain (30-day PBI) were analysed directly by 1D-TLC. Extracted ^{14}C-residues from these commodities were further fractionated using a silica-gel column eluted sequentially with EtOAc and MeOH. The ^{14}C-residues in the EtOAc fractions were analysed by HPLC. The MeOH fraction was concentrated and treated with sulfatase and β-glucuronidase (unspecified conditions). The resulting hydrolysate was acidified and partitioned with EtOAc, and ^{14}C-residues in the EtOAc phase were analysed by HPLC. The post-extraction solids (PES) from wheat straw and sugar beet tops and roots were further extracted by treatment with 1M KOH at 80 ºC for 1 hour. The resulting hydrolysate was acidified and partitioned with EtOAc, and ^{14}C-residues in the EtOAc fraction were analysed by HPLC.

In addition to the plant samples, soil samples from 0–181 DAT were extracted sequentially with MeOH, ACN and MeOH, and the resulting extracts were pooled and analysed by HPLC.

Radioactive residues in plant and soil extracts were analysed by 1D-TLC or HPLC. The TLC analysis was conducted using silica-gel plates with ethyl acetate as the developing solvent. The HPLC analyses consisted of a reverse-phase column (C8) using mobile phase gradients of ACN to MeOH, each containing 0.1% TFA. HPLC retention times for standards were determined using a UV detector (228 nm), and radioactivity was determined by LSC of collected fractions.

Results are summarised in Tables 33–35.

Table 33 Summary of Characterisation and Identification of ^{14}C-Residues in Rotational Crops from a 30-day PBI Following a Soil Application of [α-^{14}C]Cyproconazole at 010 kg ai/ha

Fraction/Metabolite [a]	Lettuce foliage (0.040 mg/kg)		Lettuce root (0.032 mg/kg)		Wheat Straw (0.440 mg/kg)		Wheat Grain (0.009 mg/kg)		Sugar Beet Tops (0.092 mg/kg)		Sugar Beet Root (0.009 mg/kg)	
	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg
Cyproconazole (M1/M2)	72.5	0.029	50.0	0.016	15.9	0.07	33.3	0.003	39.1	0.036	44.4	0.004
M9/M14	--	--	--	--	9.1	0.04	--	--	8.7	0.008	< 11.1	< 0.001
M18	12.5	0.005	--	--	2.3	0.01	--	--	--	--	< 11.1	< 0.001
Conjugated M11/182	--	--	--	--	13.6	0.06	--	--	8.7	0.008	--	--
Conjugated Cyproconazole [b]	--	--	--	--	6.8	0.03	--	--	3.3	0.003	22.2	0.002
Total identified	85.0	0.034	50.0	0.016	47.7	0.21	33.3	0.003	59.8	0.055	66.7	0.006
Total characterised	85.0	0.034	50.0	0.016	47.7	0.21	33.3	0.003	59.8	0.055	66.7	0.006
Total extractable	95.0	0.038	50.0	0.016	86.4	0.38	66.7	0.006	91.3	0.084	88.9	0.008
Unextractable (PES)[c]	7.5	0.003	50.0	0.016	13.6	0.06	33.3	0.003	9.7	0.009	11.1	0.001
Accountability [d]	103		100		100		100		100		100	

[a] Diastereomers of each compound are reported together; e.g., M1 + M2 = Cyproconazole.

[b] Conjugated residues were released by either enzymatic treatment of solubilized ^{14}C-residues or base hydrolysis of the PES fraction.

[c] Residues remaining after exhaustive extractions, including base hydrolysis.

[d] Accountability = Total extracted + Total unextractable.

Table 34 Summary of Characterisation and Identification of ^{14}C-Residues in Rotational Crops from a 90-Day PBI Following a Soil Application of [α-^{14}C]Cyproconazole at 0.10 kg ai/ha

Fraction/Metabolite [a]	Lettuce foliage (0.031 mg/kg)		Wheat Straw (0.180 mg/kg)		Sugar Beet Tops (0.028 mg/kg)		Sugar Beet Root (0.008 mg/kg)	
	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg
Cyproconazole	77.4	0.024	27.8	0.05	57.1	0.016	50.0	0.004
M9/M14	--	--	5.6	0.01	3.6	0.001	--	--
M18	3.2	0.001	--	--	--	--	--	--
Conjugated M9[b]	--	--	5.6	0.01	--	--	--	--
Conjugated M11/M18[b]	--	--	11.1	0.02	3.6	0.001	--	--
Conjugated Cyproconazole2	--	--	16.7	0.03	--	--	12.5	0.001
Total identified	80.6	0.025	66.8	0.12	64.3	0.018	62.5	0.005
Total characterised	80.6	0.025	66.8	0.10	64.3	0.018	62.5	0.005
Total extractable	93.5	0.029	88.9	0.16	89.3	0.025	87.5	0.007
Unextractable (PES)[c]	6.5	0.002	11.1	0.02	10.7	0.003	12.5	0.001
Accountability [d]	100		100		100		100	

[a] Diastereomers of each compound are reported together; e.g., M1 + M2 = Cyproconazole.

[b] Conjugated residues were released by either enzymatic treatment of solubilized 14C-residues or base hydrolysis of the PES fraction.

[c] Residues remaining after exhaustive extractions, including base hydrolysis.

[d] Accountability = Total extracted + Total unextractable.

Table 35 Summary of ^{14}C-Residues in Soil Samples Following an Application of [α-^{14}C]Cyproconazole at 0.10 kg ai/ha

Fraction	0 DAT (0.046 mg/kg)		30 DAT (0.042 mg/kg)		90 DAT (0.039 mg/kg)		119 DAT (0.037 mg/kg)		181 DAT (0.026 mg/kg)	
	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
Solvent extracted	93.5	0.043	69.0	0.029	59.0	0.023	51.4	0.019	80.8	0.021
Cyproconazole	91.3	0.042	66.7	0.028	59.0	0.023	48.6	0.018	69.2	0.018
Unextracted	4.3	0.002	31.0	0.013	41.0	0.016	48.6	0.018	23.1	0.006

Field crop rotational studies

Two field rotational crop tests were conducted between 1990 and 1992 in GA and NC, USA, depicting the potential for residue accumulation of cyproconazole in/on the commodities of several rotational crops (Ali, 1994, DP-301530; Ali, 1994, DP-301581). The 100 g/L SL formulation (ALTO® 100 SL) was applied seven times, at 13- to 17-day retreatment intervals, to a primary crop of peanuts at 0.099 kg ai /ha/application in 140–150 L/ha of finished spray using ground equipment. Peanuts were harvested 14 and 15 days after the final treatment. Each plot at the NC site was planted with mustard greens and/or spinach or collard greens, radishes and/or carrots, and wheat 30, 182, 360, and 536 days after the last treatment. Each plot at the GA site was planted with collard greens, carrots, radishes and wheat or sorghum 38, 160, 425, and 588 days after the last treatment. The petitioner reported that spinach and carrots from the 30-day PBI at the NC site and carrots from the 425-day PBI at the GA site did not grow, and did not report any harvest of spinach samples from the 182-day PBI or collard greens and carrots from the 360-day PBI at the NC site, or of radish samples from the 160-day and 360-day PBIs at the GA site. In addition, no grain or straw samples were collected from wheat plants from the 180-day PBI at either site because plants failed to produce seed heads.

Wheat and sorghum forage samples were collected at the 20-30 cm growth stage (40–152 and 59 days after planting, respectively) and at the milk dough growth stage (112–249 and 102 days after planting, respectively). The remaining rotational crops were harvested at full maturity: 40–59 days after planting for mustard greens and radishes and 60–172 days after planting for collard greens, 81–

206 days after planting for carrots, 124–288 days after planting for wheat grain and hay, and 128 days after planting for sorghum grain and hay.

Samples of rotational crop commodities were analysed for residues of cyproconazole using method AM-0842-0790-0 (above). The results of the field rotational crop trials are presented in Table 36. Residue data were not corrected for concurrent method recoveries or apparent residues in/on untreated samples.

Table 36 Residues of Cyproconazole in/on the Commodities of Various Rotational Crops Following Seven Applications at 0.099 kg ai/ha/application

PBI [a] (days)	Commodity	Test States	Residue Value (mg/kg)[b]
Leafy vegetable			
30	Mustard greens	NC	0.13
38	Collard greens	GA	0.034
160	Collard greens	GA	0.021
182	Mustard greens	NC	0.046, 0.049[c], 0.050[c]
360	Mustard greens	NC	< 0.01
425	Collard greens	GA	< 0.01
536	Mustard greens	NC	0.011
588	Collard greens	GA	< 0.01
Root crop			
30	Radish, tops	NC	0.062
	Radish, roots	NC	0.021
38	Carrot, tops	GA	< 0.01
	Carrot, roots	GA	0.021
160	Carrot, tops	GA	0.019
	Carrot, roots	GA	0.014
182	Carrot, tops	NC	0.042
	Carrot, roots	NC	0.045, 0.054 3, 0.059
360	Radish, tops	NC	< 0.01
	Radish, roots	NC	< 0.01
536	Radish, tops	NC	< 0.01
	Radish, roots	NC	< 0.01
588	Carrot, tops	GA	< 0.01
	Carrot, roots	GA	< 0.01
Grain crop			
30	Wheat, forage (20-30 cm)	NC	0.033
	Wheat, forage (milk dough)	NC	0.016
	Wheat, grain	NC	< 0.01
	Wheat, straw	NC	0.081
38	Wheat, forage (20-30 cm)	GA	0.022
	Wheat, forage (milk dough)	GA	< 0.01
	Wheat, grain	GA	< 0.01
	Wheat, straw	GA	0.017
160	Wheat, forage (20-30 cm)	GA	< 0.01
	Wheat, forage (milk dough)	GA	< 0.01
182	Wheat, forage (20-30 cm)	NC	0.031
360	Wheat, forage (20-30 cm)	NC	< 0.01
	Wheat, forage (milk dough)	NC	< 0.01
	Wheat, grain	NC	< 0.01
	Wheat, straw	NC	< 0.01
425	Wheat, forage (20-30 cm)	GA	< 0.01
	Wheat, forage (milk dough)	GA	< 0.01
	Wheat, grain	GA	< 0.01
	Wheat, straw	GA	0.011
536	Wheat, forage (20-30 cm)	NC	0.011
	Wheat, forage (milk dough)	NC	< 0.01
	Wheat, grain	NC	< 0.01
	Wheat, straw	NC	< 0.01
588	Sorghum, forage (20-30 cm)	GA	< 0.01

PBI [a] (days)	Commodity	Test States	Residue Value (mg/kg)[b]
	Sorghum, forage (milk dough)	GA	< 0.01
	Sorghum, grain	GA	< 0.01
	Sorghum, straw	GA	< 0.01

[a] PBI = plantback interval; the interval between final application of cyproconazole to the primary crop and planting the rotated crop.

[b] Residue values were not corrected for concurrent method recovery or residues in untreated samples.

[c] This sample reflects reanalysis after 20 months of frozen storage for storage stability determinations.

The Meeting received interim results from an additional two limited field rotational crop trials conducted in Zones 1 and 5 of the USA during 2004/2005 (Oakes, 2006, T003259/03). At each site, a 100 g/L SC/L formulation of cyproconazole was applied to a primary crop of soya beans as two broadcast foliar applications during vegetative development (BBCH 12-36) at rates of 0.04 kg ai/ha and at retreatment intervals (RTI) of 14–15 days, for a total of 0.082 kg ai/ha/season. Applications were made using ground equipment at volumes of 122–196 L/ha, and did not include the use of any spray adjuvants. The primary soya bean crop was grown to normal maturity and removed, and representative rotational crops of spinach, radish, and winter wheat were planted at each site at 60 or 61 days after the last application (60-day PBI). No unusual weather conditions were noted for either trial site during the study period. Supplemental irrigation was provided as needed.

Single control and duplicate treated samples of each rotational crop commodity were harvested at the typical stage of maturity for the given commodity. Spinach samples were harvested at 50–64 DAP, and radish roots and tops were harvested at 44–64 DAP. Samples of fall forage were collected from wheat at 60–61 DAP. The following spring additional forage samples were collected at jointing (225–246 DAP) and hay samples were collected at the boot or soft dough stages (258–275 DAP). Samples of grain and straw were collected at maturity (289–308 DAP). Samples were not cleaned or washed after collection, but were stored frozen.

Residues of cyproconazole were determined in rotational crop commodities using a gas chromatograph/mass-selective detection (GC/MSD) method (Method #AM-0842-0790-0). The method was validated in conjunction with the analysis of field trial samples, using control samples fortified with cyproconazole at 0.01–0.5 mg/kg. The validated LOQ is 0.01 mg/kg for each matrix, and the LOD was not reported. Average recoveries of cyproconazole were 78–103% from rotational crop commodities, with standard deviation of 0–14% (Table 37). Apparent residues were < LOQ in/on all control samples.

Table 37 Cyproconazole Residues in Rotational Crops from Application of Cyproconazole to the Primary Crop at a Total Rate of 0.082 kg ai/ha (1× USA Seasonal Rate for Soya bean)

Trial ID (City, State; Year)	Zones	Crop; Variety	Commodity	Total Rate (kg ai/ha)	Harvest (DAP)[a]	PBI [b] (days)	Residues [c, e] (mg/kg)
Hudson, NY 2004, 2005 5680	1	Spinach; Champion	Leaves	0.082	50	61	< 0.01, < 0.01
		Radish; Geneva	Tops		44		< 0.01, < 0.01
			Roots		44		< 0.01, < 0.01
		Winter Wheat; Tyee	Forage, fall		61		< 0.01, < 0.01
			Forage, spring		246		< 0.01, < 0.01
			Hay		275		< 0.01, < 0.01
			Straw		308		< 0.01, < 0.01
			Grain		308		< 0.01, < 0.01
Champaign, IL 2004, 2005 5684	5	Spinach; Bloomsdale	Leaves	0.082	64	60	< 0.01, < 0.01
		Radish; Cherry Belle	Tops		64		0.010[d], 0.012[d]
			Roots		64		< 0.01, < 0.01
		Winter Wheat; Kaskaskia	Forage, fall		60		< 0.01, < 0.01
			Forage, spring		225		< 0.01, < 0.01
			Hay		258		< 0.01, < 0.01
			Straw		289		< 0.01, < 0.01
			Grain		289		< 0.01, < 0.01

[a] DAP = days after planting.

[b] PBI = plant-back interval.

[c] The LOQ is 0.01 mg/kg for cyproconazole in each matrix, and the LOD was not reported.

[d] Values are the average of triplicate analyses of each sample.

[e] The LOQ is 0.01 mg/kg for cyproconazole in each matrix. For calculating the median, mean and standard deviation, the LOQ was used for residue values of <LOQ.

Aerobic Soil Degradation

The aerobic soil metabolism of ^{14}C-triazole labelled cyproconazole was investigated in Flaach sandy loam soil (Glänzel, 1994, Report No. 41323, Syngenta File No. SAN619/5321). The application rate of cyproconazole was 0.303 mg per kg soil (dry weight), equivalent to a field rate of 227 g ai/ha assuming homogeneous distribution in the upper 5 cm soil layer and a soil bulk density of 1.5 kg/L. The treated soils, in flasks, were incubated in the dark, over a period of 140 days at about 20 °C, with soil moisture of 40 % of the maximum water holding capacity (MWHC). Solid adsorbents were attached to each flask to trap volatile organics and carbon dioxide. Samples were taken for analysis at intervals up to 140 days after application.

Each sample was extracted with methanol followed by methanol/water (8:2, twice) and two or three extractions with water. Radioactivity extracted from the soil was quantified by liquid scintillation counting (LSC).

The methanol extracts were combined and the methanol evaporated. The concentrated extract was passed through a silica clean-up column, the eluate (fraction R2) was retained and the column was washed with methanol/water (fraction R3) and the bulk of the radioactivity eluted with methanol/water (8:2, extract B). A final wash was made with methanol (fraction R4). Extract B and fraction R2 were finally concentrated and analysed by thin layer chromatography (TLC) and high performance liquid chromatography (HPLC).

Water extracts were combined and concentrated prior to analysis by TLC and HPLC.

Selected samples were further extracted twice with acetonitrile/water/acetic acid (140:60:10) and once with acetonitrile/water/25% ammonia (140:60:2.5) to release quantities of the bound residue.

Samples of soil residues after the primary extractions were dried and the radiocarbon remaining determined by oxidation.

Distribution of the radioactivity in extracts as a function of incubation time are summarised in Table 38. Identification and quantitation of degradates as a function of incubation time are given in Table 39. Several additional degradation products exhibiting polar properties were observed amounting to ≤ 1% of AR in the methanol/water extraction and to ≤ 2% AR in the water extraction. The extraction of the soil bound radioactivity reduced the unextracted fraction to ≤ 8% of AR. Small amounts of active ingredient (≤ 3% of AR) were found in these harsh extracts. The remainder of the released radiocarbon exhibited very polar properties. Triazole and triazolyl acetic acid were not detected in these extracts.

Cyproconazole was the major component in the extracts of soil at all times. The calculated half-life (DT_{50}, first order kinetics) of cyproconazole was 148 days.

Table 38 Recovery and distribution of radioactivity in soil treated with ^{14}C-triazolyl cyproconazole (values as % Applied Radioactivity)

Incubation time (days)	Methanol /water extract	Extract B	Fraction R2	Fraction R3	Fraction R4	Water extract	$^{14}CO_2$	Unextracted	Total recovery [a]
0	95.71	91.84	1.47	0.82	1.53	-	-	0.76	96.47
14	91.45	79.18	2.37	0.83	1.07	1.37	0.05	4.64	97.51
28	85.96	77.78	4.85	1.15	0.86	3.42	0.03	7.63	97.05
56	86.60	75.96	6.47	0.97	0.53	4.25	0.16	8.38	99.39
84	82.92	72.84	6.58	1.07	0.66	5.82	0.15	10.95	99.85

Incubation time (days)	Methanol /water extract	Extract B	Fraction R2	Fraction R3	Fraction R4	Water extract	$^{14}CO_2$	Unextracted	Total recovery [a]
112	73.41	57.84	11.72	0.80	0.64	12.26	0.10	13.10	98.86
140	62.80	46.42	14.72	0.93	0.44	17.27	0.33	16.19	96.59

[a] Sum of methanol/water extract, water extract, 14CO2 and unextracted radioactivity

Table 39 Identification of degradates (values as % AR) [a]

Incubation time (days)	Cyproconazole	1H-1,2,4-Triazole	1,2,4-Triazol-1-yl-acetic acid
0	89.05	0.0	0.0
14	80.05	0.35	0.09
28	72.31	0.81	0.56
56	71.84	6.36	1.35
84	67.37	6.22	1.78
112	50.55	13.15	2.56
140	39.97	17.36	6.70

[a] Sum of compounds in methanol/water (extract B and fraction R2) and water extracts

In a second aerobic soil metabolism study, the degradation of ^{14}C-benzyl cyproconazole was investigated in three different soil types (Flaach, Louisiana and North Carolina) in the laboratory (Wisson, 1992, Report No. 41321, Syngenta File No. SAN619/5362). The soils were maintained at a moisture content of 75% at 0.33 bar. Cyproconazole was added to each soil at a concentration of 0.25 mg/kg (0.25mg ai/kg dry soil) and incubated at 21.6 ± 3.2 °C in the dark for either 112 days (Flaach and Louisiana) or 210 days (North Carolina). Traps were fitted to collect any evolved carbon dioxide.

Samples were taken after 0, 14, 28, 56, 84 and 112 days (all soils) and after 140 and 210 days (North Carolina only) for analysis. Soils were extracted twice with methanol/water (8:2), radioactivity in the extracts was quantified by LSC. After removal of the methanol, extracts were partitioned with dichloromethane and the organic phase analysed for cyproconazole and degradates by TLC. Unextracted radioactivity remaining in the soil was determined by oxidation.

The total radioactive recoveries were between 85 and 105% of (AR), with the majority greater than 90%. Only a small amount of the test substance was mineralised to $^{14}CO_2$ (2% of AR) from the North Carolina soil but 33 and 27% AR was evolved from the Flaach and Louisiana soils respectively in the same time. The calculated half-life (DT_{50}, first order kinetics) of cyproconazole was 104 days, 124 days and > 1 year in the Flaach, Louisiana and North Carolina soils respectively. No degradates were identified. Results are summarised in Table 40.

Table 40 Distribution of radioactivity in soil treated with ^{14}C-benzyl cyproconazole (values as % AR)

Incubation time (days)	Cypro-conazole	Non-polar	Polar	Very polar	Aqueous phase	$^{14}CO_2$	Unextracted	Total recovery
Flaach Soil (Sandy clay loam)								
0	83.9	0.1	2.4	0.5	0.1	0.0	0.7	100.7
14	54.2	0.1	0.7	4.6	0.1	7.3	15.1	86.2
28	64.4	0.2	1.2	1.0	0.2	13.5	17.1	92.1
56	52.2	0.3	2.4	0.5	0.4	26.7	16.3	95.2
84	46.3	0.3	2.4	0.8	0.9	32.4	17.1	101.6
112	42.3	0.6	2.8	1.6	1.0	32.9	23.9	102.7
Louisiana Soil (Silt loam)								
0	84.3	0.3	0.8	0.6	0.1	0.0	1.0	99.6
14	54.0	0.2	2.2	2.7	0.0	10.9	15.5	93.1
28	55.9	0.7	1.3	0.5	0.2	25.2	12.2	93.0
56	38.3	1.2	2.0	0.5	0.9	44.2	12.8	98.0
84	56.6	0.6	0.9	1.2	1.4	26.1	12.0	101.2

Incubation time (days)	Cypro-conazole	Non-polar	Polar	Very polar	Aqueous phase	$^{14}CO_2$	Unextracted	Total recovery
112	45.9	0.3	1.6	1.1	1.5	26.8	13.0	91.7
North Carolina Soil(Loamy sand)								
0	91.9	0.0	0.6	1.5	0.1	0.0	0.3	99.7
14	66.7	0.4	0.8	3.1	0.0	0.5	9.2	85.0
28	77.3	0.1	2.8	0.6	0.2	1.2	12.0	89.6
56	83.1	0.5	0.6	0.9	0.8	1.2	4.2	90.4
84	82.5	0.3	0.7	0.7	1.2	0.6	4.1	100.5
112	82.0	0.0	0.9	1.3	0.3	2.9	14.0	100.5
140	82.2	0.1	1.4	1.7	0.9	2.3	7.8	104.1
210	83.1	0.0	0.9	1.4	0.5	2.2	20.4	104.6

Non-polar – sum of radioactive components eluting significantly up the TLC plate

Polar – sum of radioactive components eluting marginally up the TLC plate

Very polar – sum of radioactive components not eluting off the origin of the TLC plate

Aqueous phase – radioactivity remaining in the aqueous phase after partitioning the methanol/water extracts with dichloromethane

A third aerobic soil metabolism study was reported with [U-^{14}C-Phenyl]-Cyproconazole (Glanzeil and Wisson, 1994, Report No. 41322, Syngenta File No. SAN619/5288). Cyproconazole was applied at 0.252 mg/kg soil (dry weight) to sandy loam soil, equivalent to a field rate of 189 g ai/ha. Samples were incubated at 40% MWHC in the dark for up to 140 days at 21 ± 4 °C either in Erlenmeyer flasks closed with cotton wool ('open' test system, experiment A) or with adsorption tower to trap carbon dioxide and volatile compounds ('closed' test system, experiment B).

Treated soils were extracted at intervals successively with methanol followed by methanol/water; extracted radioactivity was quantified by LSC and the distribution of cyproconazole and metabolites determined by TLC after a C18 column clean up of the extracts.

Residual radioactivity in the soil after extraction was determined by oxidation. Selected extracted soil samples were treated successively with methanol/water, water, acetonitrile/water/acetic acid and acetonitrile/water/ammonia mixtures in an attempt to remove additional residues. Samples of this extracted soil were mixed with either fresh or sterilised Flaach soil and incubated in a closed test system for up to 60 days. Samples of this re-incubated soil were analysed as above after 30 and 60 days incubation.

A summary of the study data is given in Tables 41 and 42. The total radiocarbon recovery for the open system declined from 98% to 52% of AR after 140 days, for the closed system the recovery averaged 96%. The rate of degradation of cyproconazole was fastest in the open test system with a half-life (DT_{50}, first order kinetics) of 97 days. No degradates were identified.

Table 41 Distribution of radioactivity in soil treated with ^{14}C-phenyl cyproconazole in an 'open' system (values as % AR)

Incuba-tion time (days)	Cypro-conazole	Non-polar meta-bolites	Polar meta-bolites	Very polar meta-bolites	Rinses R1-R5	Unextrac-ted radio-activity	Extract A	Extract B	Total recovery
0	93.7	0.5	0.3	0.1	0.9	3.4	94.3	94.6	97.7
14	83.6	0.4	< 0.1	0.1	1.6	10.6	85.0	84.0	95.6
28	77.5	0.1	0.5	< 0.1	2.0	8.5	77.6	76.0	86.1
56	61.2	0.1	0.9	< 0.1	1.8	13.3	64.4	62.1	77.7
84	53.7	0.2	1.1	0.2	2.2	13.6	57.0	55.2	70.6
112	30.4	0.4	1.1	0.2	1.8	19.4	34.6	32.1	54.2
140	27.3	0.1	1.1	0.2	1.5	21.5	30.1	28.7	51.6

Non-polar – sum of radioactive components eluting significantly up the TLC plate

Polar – sum of radioactive components eluting marginally up the TLC plate

Very polar – sum of radioactive components not eluting off the origin of the TLC plate

Rinses R1-R5 – radioactivity recovered from the clean-up column after elution of radioactivity

Extract A – methanol/water extract of soil prior to clean-up

Extract B – extract of soil after column clean-up (used to determine amounts of cyproconazole and metabolites)

Table 42 Distribution of radioactivity in soil treated with ^{14}C-phenyl cyproconazole in a 'closed' system (values as % AR)

Incubation time (days)	Cyproconazole	Total metabolites	Rinses R1-R5	Volatiles	CO2	Unextracted radioactivity	Extract A	Extract B	Total recovery
0	89.7	1.6	1.4	NA	NA	3.5	99.3	91.3	102.7
14	83.6	0.6	2.3	< 0.1	0.5	9.9	88.8	84.2	99.2
28	82.5	1.5	1.8	< 0.1	2.8	8.6	87.7	84.0	99.2
56	68.7	1.2	1.7	< 0.1	6.3	17.2	75.7	69.9	99.2
84	63.0	1.1	1.7	< 0.1	2.1	21.5	63.4	64.1	87.0
112	57.5	0.7	2.4	< 0.1	11.2	21.5	64.6	58.1	97.2
140	58.0	0.7	1.2	< 0.1	1.9	20.8	64.0	58.7	86.8

Rinses R1-R5 – radioactivity recovered from the clean-up column after elution of radioactivity

Extract A – methanol/water extract of soil prior to clean-up

Extract B – extract of soil after column clean-up (used to determine amounts of cyproconazole and metabolites)

Soil Photolysis

The Meeting received a report on the photolysis of 14C-phenyl labelled cyproconazole on soil (Adam, 2000, Report No. 99DA05, Syngenta File No. SAN619/6887). The^{14}C-phenyl cyproconazole was applied at a rate of 90 g ai/ha on to the surface of moist (75% field capacity) Gartenacker loam/silt loam soil and irradiated with a xenon arc light source filtered to remove UV-light below 290 nm in a 12 hour dark light cycle. Further treated samples were maintained wholly in the dark, as controls. The temperature of the soil layers was kept at 19.5 ± 0.1 °C. The duration of the experimental light exposure was adjusted to the corresponding light exposure at latitude 30 °N to 50 °N. During the incubation air was drawn over the samples and trapped in NaOH to retain any carbon dioxide produced (proved by reaction of exposed NaOH with barium carbonate).

Soil samples were removed at intervals and extracted with cold acetonitrile/water (8:2), followed by soxhlet extraction with acetonitrile alone. Radioactivity in the extracts was quantified by LSC. The combined extracts were concentrated and analysed for cyproconazole and its degradation products, by HPLC and TLC.

The overall recovery of radioactivity was 101% (98.1 to 103%) for all samples. Under irradiation (12 hours light/dark cycle) the extractable radioactivity decreased from 100% to 96% after 30 days (equivalent to 20 days summer sunlight at 30°N to 50°N). Correspondingly, non-extractable radioactivity increased to 3% (0.8% in dark controls) by the end of the study. Volatile radioactivity in form of $^{14}CO_2$ amounted to 1% (0.1% in dark controls) at the end of the study.

In the dark control samples no degradation of the compound was observed after 30 days. In the irradiated samples only, about 5% of cyproconazole was degraded during the experiment resulting in a half-life of approximately 296 days. Two minor photoproducts formed in quantities below or equal to 0.9% AR were identified as NOA408616, (1-(4-chloro-phenyl)-2-[1,2,4]triazole-1-yl-ethanol) and CGA123420, (1-(4-chloro-phenyl)-2-[1,2,4]triazol-1-yl-ethanone).

Hydrolysis

The water hydrolysis of ^{14}C-triazole labelled cyproconazole was reported to the Meeting (Glänzel, 1999, Report No. 99AG04. Syngenta File No. SAN619/6849). The study was performed under sterile conditions in a sterile hood. ^{14}C-cyproconazole was added to buffers at pH 4, 5, 7 and 9 at approximately 5 mg/kg and incubated for up to 5 days at 50 °C. Samples of the buffers were taken

daily and analysed for total radioactive content by LSC and for cyproconazole and its degradates by HPLC and/or 2-dimensional (2D) TLC.

The test samples remained sterile during the test. The total recovery of radioactivity was between 92 and 103% AR for all samples. ^{14}C-cyproconazole accounted for 100% of the radioactivity in all buffer solutions at all sampling times

METHODS OF RESIDUE ANALYSIS

Analytical methods have been developed and validated for the determination of cyproconazole in plant and animal commodities. Table 43 summarises the analytical methods which have been developed to support the submitted studies.

Table 43 Methods for the Determination of Residues of Cyproconazole in Commodities of Plant and Livestock Origin

Substrate	Matrix	Method	LOQ	Author (Year)
Crops/ Soil/ Livestock	Various	GC-NPD / CG-ECD CBK 11032/86011	0.01 mg/kg	Ko (1986)
Crops	Various	GC-NPD / GC-MSD AM-0842-0790-0 ILV to method	0.01 mg/kg	Ali (1990) Oakes (2006a) Oakes (2006b) Wassell, Gilles (1991)
Crops	Coffee bean, soya bean seed	GC-NPD AM-0822-0994-3	0.01 mg/kg (coffee bean) 0.02 mg/kg (soya bean seed)	Ali (1994) Lin (2004)
Crops/ Livestock	Various	GC-NPD / GC-MSD BS 2598/BS 8058 ILV to method	0.01 mg/kg, except: 0.002-0.005 mg/kg (milk) 0.05 mg/kg bovine liver, kidney, muscle	Bourry, Gasser (1991) Bourry, et al. (1996) Joubert (1994) Hertl (1995) Gasser, Hertl (1994) Maxwell, et al. (1994) Krennhuber, Pfarl (1996)
Crops	Barley grain, straw, whole plant	GC-NPD REM SDZ/CYPROCONAZOLE-CEREALS/JM/92/1	0.01 mg/kg	McKenzie (1993)
Crops	Sugar beet leaf, root	GC-NPD AGRI 064 rev. 2	0.01 mg/kg (root) 0.02 mg/kg (leaf)	Baravelli (2004)
Crops	Wheat grain, straw	GC-NPD REM 130.02	0.02 mg/kg (grain) 0.04 mg/kg (straw)	Ryan, Clark (2006a) Stack (1997)
Crops	Cereal grain, straw	LC-MS/MS SOP RAM 397/01	0.01 mg/kg	Crook (2002)
Crops	Pea seed, OSR seed, lettuce, apple	LC-MS/MS SOP RAM 297/02	0.01 mg/kg	Crook (2004) Elliott (2004)
Crops	Wheat grain, OSR seed, melon, apple	LC-MS/MS DFG S19 (extended revision) ILV to method	0.01 mg/kg	Schulz (2004) Lakaschus (2004)
Livestock	Bovine liver, kidney, fat, muscle, milk	HPLC/UV (M36, M21a) GC/NPD (cyproconazole) HPLC/MS (M14) (data collection in feeding study)	M14, 0.01 mg/kg M21a, 0.005 M36, 0.05 Cyproconazole: 0.005 milk 0.01 fat 0.05 kidney, liver	Oakes (1994)
Livestock	Bovine liver, kidney, fat, milk; Hen egg	LC-MS/MS SOP RAM 499/01 ILV to method	0.01 mg/kg	Crook (2006a) Ryan, Clark (2006b) Bour (2006)

Substrate	Matrix	Method	LOQ	Author (Year)
Livestock	Bovine liver, fat, milk; Hen egg	LC-MS/MS SOP RAM 499/02	0.01 mg/kg	Crook (2006b)
Livestock	Bovine liver, kidney, fat, muscle, milk; Hen egg	LC-MS/MS DFG S19 (extended revision) ILV to method	0.01 mg/kg	Klimmek (2004) Lakaschus (2005)

Analytical methods for plant matrices

Residue analytical method CBK 11032/86011 (Ko, 1986, SAN619/5034.)

The method is applicable for the determination of cyproconazole residues in apples, grapes, peanuts and soil matrices. Samples are Soxhlet extracted with acetonitrile (ACN) overnight. Following rotary evaporation to dryness, the aqueous residue is passed through Extrelut column, washed with methylene chloride and evaporated to dryness. For gel-permeation chromatography (GPC) cleanup, the sample is redissolved in cyclohexane: methylene chloride (85:15) and passed through GPC columns packed with Bio-Beads S-X3. For C-18 (Bond Elute) cleanup, the sample is redissolved in methylene chloride (100%) and passed through C-18 column. Following appropriate cleanup, the sample is evaporated to dryness and redissolved in toluene. Cyproconazole is then quantitated by gas-liquid chromatography using either a capillary (preferred) or packed column with either a nitrogen-phosphorous detection (NPD, preferred) or electron-capture detection (ECD).

Recovery data of cyproconazole are summarised in Table 44. The mean recovery of cyproconazole from fortified samples of all matrices (n = 15) at 0.025–0.5 mg/kg was 95% (RSD 14.3%).

Table 44 Recovery Values for Analytical Method CBK 11032/86011 for the Determination of Cyproconazole in Crops Using Capillary Column with NPD.

Matrix	Fortification level (mg/kg)	Cleanup Method	Recovery		n
			mg/kg	%	
Grape	0.025	GPC	0.021	84	1
Grape	0.050	GPC	0.056	112	1
Grape	0.050	GPC	0.048	96	1
Grape (wet pomace)	0.050	GPC	0.045	90	1
Grape	0.100	GPC	0.112	112	1
Apple	0.100	GPC	0.084	84	1
Apple	0.100	GPC	0.095	95	1
Peanut (nutmeat)	0.100	GPC	0.086	86	1
Peanut (vine)	0.100	C-18	0.089	89	1
Grape (dry pomace)	0.200	C-18	0.148	74	1
Grape	0.250	C-18	0.185	74	1
Apple	0.250	GPC	0.277	111	1
Peanut (hull)	0.250	GPC	0.260	104	1
Soil	0.250	GPC	0.247	99	1
Soil	0.500	GPC	0.575	115	1
Mean				95	15
RSD				14.3	

Using a capillary column the linearity range was studied on both NP and EC detectors with seven cyproconazole fortification levels ranging from 0.25–10.0 mg/kg. The regression coefficient (r^2) for NP and EC detectors was 0.99985 and 0.99928, respectively. An additional study on NP detectors only with cyproconazole fortification levels ranging from 0.05–2.0 mg/kg showed a regression coefficient (r^2) of 0.99986.

The stated limit of detection of the method is 0.01 mg/kg for all matrices investigated, using both NPD and ECD.

Residue analytical method AM-0842-0790-0 (Ali, 1990, SAN619/5141)

A GC/MSD method (Method #AM-0842-0790-0) for gathering data on cyproconazole (free and conjugated) residues in plant commodities was provided (1990; Ali, S.). This method is a more recent version of the above method, but includes use of MSD for detection and quantitation of cyproconazole, instead of NPD.

To release free and conjugated residues, plant samples are mixed with 1N HCl and hydrolysed for 1 hour at 95 °C. For oil samples, the sample is first dissolved in hexane and residues are extracted into 1N HCl prior to hydrolysis. The resulting hydrolysates are cooled to room temperature, homogenized with ethanol, mixed with Celite and filtered. Residues in the resulting filtrate are concentrated to an aqueous remainder, and cleaned up by SPE using C18 and NH2 cartridges. Residues are loaded onto the C18 and washed with water, MeOH water (1:9, v:v), and hexane. Residues are then eluted with 5% IPA in toluene and sodium sulfate is added to remove any water. Residue are then loaded onto the NH2 cartridge and eluted with 5% IPA in toluene. Residues are determined by GC/MSD using an external standard and the m/z 220 ion for quantitation. The ratio of the m/z 83, 139 and 222 ions can be used for confirmation. The reported LOQ is 0.01 mg/kg for all substrates.

Method AM-0842-0790-0 was validated using samples of apples, grapes, raisins and peaches fortified at 0.01–0.5 mg/kg and samples of nectarines, wheat commodities and peanut commodities fortified at 0.10 mg/kg. Recoveries are summarised in Table 45.

Table 45 Recovery Results from Method Validation on Apple, Grape, Raisin, Peach, Nectarine, and Peanut and Wheat Commodities using GC/MSD Method AM-0842-0790-0

Crop/matrix	Spiking Level (mg/kg)	Recoveries (%)	Mean Recovery (±SD)	CV (%)
Apple	0.01	120, 120, 90, 60	98 ± 29	29
	0.05	82, 88, 98	89 ± 8	9
	0.10	110, 114, 78, 99	100 ± 16	16
	0.20	115, 112	114 ± 2	2
	0.50	92, 106, 90	96 ± 9	9
	Total	60-120	98 ± 17	17
Grape	0.01	160, 130, 100, 60	113 ± 43	38
	0.05	82, 74	78 ± 6	7
	0.10	100, 89	95 ± 8	8
	0.20	98, 115	107 ± 12	11
	0.50	92, 89	91 ± 2	2
	Total	60-160	99 ± 26	27
Raisin	0.01	90, 80, 70, 60	75 ± 13	17
	0.05	78, 76	77 ± 1	2
	0.10	86, 87	87 ± 1	1
	0.20	84	NA	0
	0.50	84, 107, 90	94 ± 12	13
	Total	60-107	83 ± 12	14
Peach	0.01	120, 90	105 ± 21	20
	0.05	110, 84, 94, 84	93 ± 12	13
	0.10	76, 119, 105	98 ± 30	31
	0.20	80, 75	78 ± 4	5
	0.50	83, 106, 124, 98, 115	105 ± 16	15
	Total	75-124	98 ± 17	18
Nectarine	0.10	79, 79, 87, 82, 85	82 ± 4	5
Peanut Forage	0.10	102, 109, 90, 101, 81	97 ± 11	11
Peanut Hay	0.10	100, 99, 95, 102, 95	98 ± 3	3
Peanut meat	0.10	90, 79, 85, 86, 83	85 ± 4	5
Peanut Hulls	0.10	73, 79, 51, 75, 71	70 ± 11	16
Wheat Forage	0.10	84, 99, 95, 96, 99	95 ± 6	7

Crop/matrix	Spiking Level (mg/kg)	Recoveries (%)	Mean Recovery (±SD)	CV (%)
Wheat Hay	0.10	80, 83, 62, 85, 83	79 ± 9	12
Wheat Grain	0.10	108, 97, 106, 63, 79	91 ± 19	21

Independent laboratory validation (ILV) of residue analytical method AM-0842-0790-0

The residue analytical method for the analysis of cyproconazole by GC-NPD, method AM-0842-0790-0, was successfully validated by an independent laboratory (Wassell and Gilles, 1991, Report No. B9007-CN1. Syngenta File No. SAN619/6406). The method was validated for peanut hay and peanut meat. Results are summarised in Table 46.

Table 46 ILV Data for Analytical Method AM-0842-0790-0 for the Determination of Cyproconazole in Crops

Matrix	Fortification level (mg/kg)	Recovery (%)		RSD (%)	n
		Mean	Range		
Peanut Hay	0.01	114	104–128	11	3
	0.05	94	80–105	14	3
	Overall	104	80–128	15	6
Peanut Nutmeat	0.01	70	63–75	9	3
	0.05	71	65–76	8	3
	Overall	70	63–76	8	6

Residue analytical method 180E.00 (Adams, 2003, Syngenta File No. SAN619_10077)

Cereal foliage, grain and straw specimens were extracted by maceration with aqueous MeOH. An aliquot of the extract was evaporated, dissolved in aqueous ACN, and loaded on to a C18 SPE cartridge. After washing with aqueous ACN, the compound was eluted with 80% ACN/water. The eluate was diluted with water and loaded on to an Envicarb SPE cartridge. After washing with aqueous ACN both compounds were eluted from Envicarb SPE cartridge with ACN. The evaporated fraction was dissolved in mobile phase, divided into two HPLC vials and injected onto two separate column-switching HPLC systems with UV detection.

Recovery data of cyproconazole are summarised in Table 47.

Table 47 Recovery Values for Analytical Method 180E.00 for the Determination of Cyproconazole in Crops.

Matrix	Fortification level (mg/kg)	Recovery (%)		RSD (%)	n
		Mean	Range		
Wheat Foliage/Straw	0.01	105	91-122	11	9
	0.02	99	–	–	1
	0.05	71	88-102	8.0	14
	0.10	102	100-104	2.0	3
	0.20	97	83-119	17	7
	0.50	77	–	–	1
	1.0	97	93-102	4.7	3
	2.0	91	78-99	13	3
	5.0	91	85-88	1.8	3
	Overall	97	78-122	12.1	44
Wheat Grain	0.02	97	91-108	11	14
	0.05	97	–	–	1
	0.1	94	79-105	8.7	10
	Overall	96	79-108	9.9	25

The LOQ of the method is 0.02 mg/kg for cereal grain and 0.01 mg/kg for cereal straw/foliage.

Method AM-0822-0994-3 (Ali, 1994, Syngenta File No. SAN619/6409; Lin, 2004, Report No. 001901-03)

Method AM-0822-0994-3 is applicable for the determination of cyproconazole residues in dry coffee beans and soya bean seed and is largely identical to method AM-0842-0790-0 described above. Samples are hydrolysed in HCL for 1 hour at 95 °C. The hydrolysates are cooled to room temperature and ethanol is added. The samples are blended for 3 minutes and then suction filtered. After removal of the ethanol using rotary evaporation, aliquots of the extracts are cleaned-up using reversed-phase (C-18) and normal-phase aminopropyl SPE columns. Cyproconazole is then quantitated by GC-NPD using a 30 m × 0.53 mm (I.D.) 5% methyl silicone fused silica column.

Recovery data of cyproconazole are summarised in Table 48.

Table 48 Recovery Values for Analytical Method AM-0822-0994-3

Matrix	Fortification level (mg/kg)	Recovery (%)		RSD (%)	n
		Mean	Range		
Coffee Dry Green Beans	0.01	105	100-110	-	2
	0.05	82	75-94	10	4
	0.10	71	63-91	18	4
	0.20	81	75-86	-	2
	0.50	78	78	-	2
	Overall	83	63-230[a]	15	14
Coffee Dry Roasted Beans	0.10	75	67-83	-	2
	0.20	70	70	-	2
	Overall	73	67-83	10	4
Soya bean Dry Seed	0.02	82	72-88	9.1	4
	1.0	96	81-105	12	4
	Overall	90	72-105	14	8

[a] Mean does not include recovery of 230%.

Residue analytical method BS 2598/BS 8058 (Bourry and. Gasse, 1991, SAN619/5407)

Method BS 2598 was developed for the analysis of cyproconazole in plant and soil matrices. Method BS 2598 was later updated to include the analysis of cyproconazole in livestock tissues and re-named BS 5058. Earlier versions of method BS2598/BS 8058 include CBK 11837/87, CBK 12014/88, CBK 12751/89, CBK 11600/86, and AGR/MOA/CYPROC-1. All these methods are essentially the same.

In method BS 2598/BS 8058 (routine method 1), homogenized plant samples are extracted by blending or shaking with aqueous ACN or ACN. The extract is filtered and the organic phase is removed by evaporation prior to clean-up. Purification is achieved by a modular system of column clean-up using a selection of Extrelut, silica and/or C-18 columns. The eluate is concentrated and dissolved in toluene. Determination of cyproconazole is by packed, mega-bore or capillary-column GC-NPD or by GC-MSD using m/z 222 for quantification and m/z 139 for qualification.

Recovery data of cyproconazole from crops matrices that were obtained during method validation of BS 2598/BS 8058 are summarised in Table 49. Recovery data generated during crop field trial studies are summarised in Tables 50–51.

Recovery data of cyproconazole from livestock matrices obtained during method validation of BS 2598/8058 are summarised in Table 52.

Table 49 Recovery Data for Analytical Method BS 2598/BS 8058 for the Determination of Cyproconazole in Crops

Matrix	Fortification level (mg/kg)	Recovery (%) Mean	RSD (%)	n
Method BS 2598				
Cereal Grain	0.01-4.0	93	4.5	20
Cereal Plant	0.01-4.0	94	1.7	5
Cereal Straw	0.01-4.0	94	5.6	16
Grapes (Berries)	0.01-4.0	90	7.8	17
Sugar Beet Leaves	0.01-4.0	96	9.4	16
Sugar Beet Roots	0.01-4.0	87	10.3	16
Peaches	0.01-4.0	91	19	5
Coffee, Green, Dried	0.01-4.0	78	10.3	5
Coffee, Roasted	0.01-4.0	75	-	2
Oil Seed Rape Plants	0.01-4.0	97	15	4
Oil Seed Rape Pods	0.01-4.0	91	9.6	4
Oil Seed Rape Seeds	0.01-4.0	90	7.1	4
Peanut Shells	0.01-4.0	90	15	4
Peanuts Seeds	0.01-4.0	90	17	5
Tea Leaves	0.01-4.0	81	4.0	4
Apples	0.01-4.0	88	15	10
Overall	0.01-4.0	91	9.1	142

Table 50 Cyproconazole Recovery Data for Analytical Method BS 2598/BS 8058 Used in Leek, Sugar Beet and Rye Residue Field Trials

Matrix	Fortification level (mg/kg)	Recovery (%) Mean	Range	RSD (%)	n
Report 92013/ 333022					
Leek	0.01	105	98-110	6.0	3
	0.1	92	90-95	2.4	4
	0.3	91	91	-	1
	1.0	108	108	-	1
	Overall	98	90-110	8.2	9
Report TDS BS-5177/IF-93/21887-01					
Sugar Beet Leaves	0.01	105	79-127	14	9
	0.11	81	78-82	2.9	3
	0.22	94	84-103	-	2
	0.55	88	65-92	4.1	4
	0.88	82	80-84	-	2
	1.1	104	100-108	-	2
	Overall	94	65-127	17	22
Sugar Beet Root	0.01	79	53-107	20	13
	0.22	64	62-66	-	2
	1.1	84	63-107	18	7
	Overall	79	53-107	19	22
Report TDS BS-4112/SAN-9213 Az. 94160/92					
Rye	0.01	98	98	-	1

Matrix	Fortification level (mg/kg)	Recovery (%)		RSD (%)	n
		Mean	Range		
Ears	0.05	101	101	-	1
	0.10	100	96-103	-	2
	Overall	100	96-103	3.1	4
Rye Plant w/o Ears	0.01	74	74	-	1
	0.03	107	107	-	1
	0.10	89	86-92	-	2
	Overall	90	74-107	15	4
Rye Grain	0.01	98	92-104	-	2
	0.10	68	68	-	1
	Overall	88	68-104	21	3
Rye Straw	0.01	80	80	-	1
	0.1	89	79-95	9.6	3
	1.0	93	83-103	-	2
	Overall	89	79-103	11	5

Table 51 Cyproconazole Recovery Data for Analytical Method BS 2598/BS 8058 Used in Wheat Residue Trials

Matrix	Fortification level (mg/kg)	Recovery (%)			RSD (%)	n
		Individual Values	Mean	Range		
Wheat Whole Plant	0.01	117	-	-	-	1
	0.05	103, 87, 114, 66, 131, 89, 77, 109	97	66–131	22	8
	0.10	100, 81, 98	93	81-100	11	3
	1.0	109, 86, 98, 68, 101, 97, 98, 82, 112, 64, 99, 85, 91	92	64-112	16	13
	1.2	72	72	72	-	1
	2.0	115	115	115	-	1
	Overall	-	94	64-131	18	27
Wheat Ears	0.01	101, 105, 101, 116, 126	110	101-126	10	5
	0.05	109, 88, 91, 91, 100, 84, 95, 87, 114, 85, 107	96	84-114	11	11
	0.10	88, 101, 96, 101, 98, 107	99	88-107	6	6
	0.20	91, 87	89	87-91	-	2
	1.0	105, 103, 99, 88	99	88-105	8	4
	10	89	89	89	-	1
	Overall	-	98	84-126	10	29
Wheat Straw	0.01	138, 47, 108, 101	99	47-138	38	4
	0.02	95	95	95	-	1
	0.04	105, 85, 81, 85, 126, 84, 90, 94, 100, 82, 85, 87	92	81-126	14	12
	0.05	107, 81, 71, 84, 92, 95, 104	91	71-107	13	7
	0.10	82, 109, 97, 79, 116, 115	100	79-116	16	6
	0.40	112	112	112	-	1
	0.50	99, 92, 95, 101, 108,	101	92-115	8	9

Matrix	Fortification level (mg/kg)	Recovery (%)			RSD (%)	n
		Individual Values	Mean	Range		
		115, 98, 106, 93				
	0.667	73, 88	81	73-88	-	2
	0.80	96, 91, 94, 73, 95, 93, 85, 97, 89, 99, 104, 83, 103, 95	93	73-104	9	14
	1.0	98, 92	95	92-98	-	2
	1.5	98	98	98	-	1
	2.0	105	105	105	-	1
	Overall	-	95	47-138	15	60
Wheat Grain	0.01	94, 91, 121, 74, 89, 116, 95, 121, 102, 125, 102, 93, 136, 101, 118, 98, 99, 96, 91, 99, 97, 111, 78, 106, 107	102	74-136	14	25
	0.02	107, 73, 75, 95, 89, 103, 105, 74, 70	88	70-107	17	9
	0.04	91, 97, 73, 82, 107, 105, 91, 75	90	73-107	14	8
	0.05	106, 123, 104, 105, 99, 97, 105, 115, 101, 68	102	68-123	14	10
	0.10	101, 98, 96, 106, 109	102	96-109	5	5
	0.20	109, 101, 82, 65, 92, 85, 96, 80	89	65-109	15	8
	0.40	71	71	71	-	1
	0.50	96	96	96	-	1
	1.0	90, 94	92	90-94	-	2
	Overall	-	97	65-136	15	69

Table 52 Recovery Data for Analytical Method BS 2598/BS 8058 for the Determination of Cyproconazole in Livestock Matrices

Matrix	Fortification level (mg/kg)	Recovery (%)	RSD (%)	n
		Mean		
Method BS 2598				
Bovine Liver	0.05-0.50	84	5.2	11
Bovine Kidney	0.05	74	-	2
Bovine Muscle	0.05	78	-	2
Bovine Fat	0.01-0.10	75	24	11
Bovine Milk	0.002-0.033	88	1.4	10
Hens Eggs	0.01-0.10	92	16	16
Hens Muscle	0.01-0.10	105	17	16
Hens Liver	0.01-0.10	94	16	13

ILV of residue analytical method BS 2598/BS 8058 (Krennhuber and Pfarl, 1996, SAN619/0063)

Method BS 8058 was independently validated for plant material (apples, beans, cereal grains, rape seed), food of livestock origin (eggs, meat, milk), and soil. The validation was done by fortifying untreated samples with cyproconazole at levels equivalent to the limit of determination and ten times that level. The validation was performed by two different laboratory operators for plant and soil matrices. During validation, the working procedure described in residue analytical method BS 8058

was followed with minor modifications. Cyproconazole residues in sample extracts were quantified using GC-MSD monitoring ions m/z 222 (quantification) and m/z 139 (confirmation).

Recovery data of cyproconazole from crop matrices are summarised in Table 53.

Table 53 Recovery Data for ILV of Residue Analytical Method BS 2598/BS 8058 for the Determination of Cyproconazole in Crop and Livestock Matrices.

Matrix	Fortification level (mg/kg)	Recovery (%)		RSD (%)	n
		Mean	Range		
Apples	0.01	100	86-107	7	10
	0.10	94	80-110	12	10
	Overall	97	80-110	10	20
Beans	0.01	90	85-97	5	10
	0.10	71	59-77	8	10
	Overall	81	59-97	11	20
Cereal Grains	0.01	94	78-117	15	10
	0.10	78	63-94	12	10
	Overall	86	63-117	14	20
Oil Seed Rape Seed	0.01	99	86-113	11	10
	0.10	88	52[a]-103	22	10
	Overall	94	52-113	17	20
All crops	Overall	89	52-117	16	80
Eggs	0.01	92	89-99	4	5
	0.10	88	81-90	5	5
	Overall	90	81-99	5	10
Meat	0.01	94	91-97	3	5
	0.10	97	93-99	2	5
	Overall	95	91-99	3	10
Milk	0.003	95	88-100	6	5
	0.03	91	86-94	3	5
	Overall	93	86-100	5	10
All livestock matrices	Overall	93	81-100	5	30

[a] Two samples yielded low recoveries (52% and 53%, respectively) due to insufficient phase separation at the liquid/liquid extraction of the defatting step.

Residue analytical method REM SDZ/CYPROCONAZOLE-CEREALS/JM/92/1 (McKenzie, 1993. SAN619/5227)

This method was used for the analysis of cyproconazole in cereal grain, straw, and whole plant. After extraction by maceration with organic solvent followed by purification using liquid-liquid partitions, the extracts were purified further by elution through silica Bond Elute cartridges (all matrices) and/or C18 Bond Elute cartridges (straw). Quantitation of cyproconazole was via GC-NPD.

Recovery data of cyproconazole from crop matrices are summarised in Table 54.

Table 54 Cyproconazole Recoveries on Samples of Barley Determined by Residue Analytical Method SDZ/CYPROCONAZOLE-CEREALS/JM/92/1

Matrix	Fortification level (mg/kg)	Recovery (%)		RSD (%)	n
		Mean	Range		
Barley Whole Plant	0.01	77	70-80	7.5	3
	0.25	87	73-94	12	5
	5.0	78	76-77	-	2

Matrix	Fortification level (mg/kg)	Recovery (%)		RSD (%)	n
		Mean	Range		
	Overall	82	70-94	12	10
Barley Grain	0.01	100	90-100	-	2
	0.10	86	85-86	-	2
	Overall	93	85-110	13	4
Barley Straw	0.01	86	70-100	17	3
	0.25	84	75-100	14	4
	5.0	70	70	-	1
	Overall	83	70-100	15	8
All matrices	Overall	84	70-110	13	22

Residue analytical method AGRI 064 Rev. 2 (Baravelli, 2004, ICI5504/2624)

A gas chromatographic method for the determination of cyproconazole residues was validated for sugar beet leaves and roots. Homogenized sugar beet samples are extracted through the blending of grinded frozen sample with diatomaceous earth (Extrelut column) followed by elution with DCM. Quantification of residues of cyproconazole in sugar beet samples is achieved by capillary-column GC-NPD using an external standardization technique.

Recovery data of cyproconazole from sugar beet root and leaves are summarised in Table 55.

Table 55 Cyproconazole Recoveries on Root and Leave Samples of Sugar Beets Determined by Residue Analytical Method AGRI 064 Rev. 2.

Matrix	Fortification level (mg/kg)	Recovery (%)		RSD (%)	n
		Mean	Range		
Sugar Beet Root	0.01	79	89-93	0.84	1 (5[a])
	0.02	99	-	-	1
	0.05	100	-	-	1
	0.10	83	-	-	1
	0.20	93	-	-	1
	Overall	91	79-100	10	9
Sugar Beet Leaves	0.02	96	89-93	1.0	1 (5[a])
	0.04	99			1
	0.08	101			1
	0.10	94			1
	0.20	89			1
	Overall	96	89-101	4.9	9

[a] Mean value of 5 different injections (from one sample).

Residue analytical method REM 130.02 (Ryan and Clark, 2006,CGA64250/5088)

Method REM 130.02 (modified) was originally developed for the analysis of propiconazole. The method was used for the analysis of residues of cyproconazole in winter wheat in SAN619/0199. Cyproconazole is extracted from crop samples by shaking with MeOH:water (80:20, v/v). After filtering, an aliquot of the extract is evaporated to low volume, diluted with water, and partitioned into hexane: t-butyl-methyl-ether (50:50, v/v). After evaporation to dryness, the residue is re-dissolved in hexane. Final clean-up is by SPE using an aluminium oxide packing. Final determination is by GC-NPD. Small modifications in comparison to the original method were applied: cyproconazole was eluted from the aluminium column with additional TBME and a different column was used for the separation of cyproconazole from propiconazole. Furthermore, an additional GC-run was performed and final volume for injection increased from 2 to 5 µL.

Recovery data of cyproconazole from crop matrices are summarised in Table 56.

Table 56 Cyproconazole Recoveries on Root and Leave Samples of Sugar Beets Determined by Residue Analytical Method REM 130.02

Matrix	Fortification level (mg/kg)	Recovery (%)		RSD (%)	n
		Mean	Range		
Wheat Grain	0.02	119	-	-	1
	0.2	104	-	-	1
	Overall	112	104-119	-	2
Wheat Straw	0.04	85	-	-	1
	0.4	89	-	-	1
	Overall	87	85-89	-	2
All matrices	Overall	99	85-119	16	4

Residue analytical method SOP RAM 397/01 (Crook, 2002, SAN619/7281)

In method SOP RAM 397/01, homogenized plant samples are extracted by shaking with aqueous ACN. After centrifugation and dilution with water, samples are filtered to remove particulate. Determination of cyproconazole is by high-performance liquid chromatography with triple quadrupole mass spectrometric detection (LC-MS/MS) monitoring m/z 292 (Q1) as the ion source and m/z 70 as daughter ion (Q3).

Recovery data of cyproconazole from crop matrices are summarised in Table 57.

Table 57 Recovery Values for Residue Analytical Method SOP RAM 397/01 for the Determination of Cyproconazole in Crop Matrices

Matrix	Fortification level (mg/kg)	Recovery (%)		RSD (%)	n
		Mean	Range		
Cereal Grain	0.01	110	107-111	1	5
	0.1	108	107-111	2	5
	Overall	109	107-111	2	10
Cereal Straw	0.01	105	103-109	3	5
	0.1	109	108-112	1	5
	Overall	107	103-112	3	10

Residue analytical method SOP RAM 397/02 (Crook, 2004, SAN619/7521)

The analytical procedure described is closely based upon method RAM 397/01. It is suitable for the determination of residues of cyproconazole in crop samples using an external standardization procedure. Samples are extracted by homogenization with ACN:ultra-pure water, aliquots are diluted with ultra-pure water :ACN with thorough ultrasonication. Samples are filtered through a filter disc (25 mm GHP GF 0.45 µm) prior to final determination LC-MS/MS, monitoring m/z 292 (Q1) as the ion source and m/z 70 as daughter ion (Q3) used for quantification.

Recovery data of cyproconazole from crop matrices are summarised in Table 58.

Table 58 Recovery Data for Analytical Method SOP RAM 397/02 for the Determination of Cyproconazole in Crops.

Matrix	Fortification level (mg/kg)	Recovery (%)		RSD (%)	n
		Mean	Range		
Dried Peas Dried Seed	0.01	106	101-109	3	6
	0.10	108	106-111	2	6
	Overall	107	101-111	2	12
Oilseed Rape Seed	0.01	101	94-106	5	6
	0.10	100	93-104	4	5
	Overall	100	93-106	4	11
Lettuce	0.01	103	99-109	4	5

Matrix	Fortification level (mg/kg)	Recovery (%)		RSD (%)	n
		Mean	Range		
Heads	0.10	108	104-110	3	6
	Overall	106	99-110	4	11
Apples Fruit	0.01	104	102-107	2	6
	0.10	105	104-106	1	6
	Overall	105	102-107	1	12

Residue analytical enforcement method DFG S19 (extended revision) for plant matrices (Schulz, 2004, SAN619/7489)

Method DFG S19 (extended revision)[13] was validated for the analysis of cyproconazole in the representative crop matrices wheat grain (dry crop), oil seed rape seed (high-fat crop), whole melons (high-water crop), and apples (acidic crop). For extraction, module E1 is used for melons (whole fruit) and apples, and modules E2 and E7 for wheat grain and oilseed rape (seed), respectively. Clean-up is carried out according to module GPC. Cyproconazole is quantified by LC-MS/MS. Ion transition m/z 291.97 to m/z 69.88 was monitored for quantification.

Recovery data of cyproconazole from crop matrices are summarised in Table 59.

Table 59 Recovery Data from Representative Crop Matrices for Enforcement Method DFG S19 (extended revision) for the Determination of Cyproconazole in Crops

Matrix	Fortification level (mg/kg)	Recovery (%)		RSD (%)	n
		Mean	Range		
Wheat Grain	0.01	94	91-96	2.8	5
	0.1	97	93-101	3.7	5
	Overall	95	91-101	3.5	10
Oilseed rape Seed	0.01	71	62-79	11	5
	0.1	102	93-108	5.8	5
	Overall	89	62-108	20	10
Melon Whole fruit	0.01	89	83-98	6.9	5
	0.1	83	79-89	4.9	5
	Overall	86	79-98	7.1	10
Apple Fruit	0.01	93	89-96	2.9	5
	0.1	97	92-101	3.6	5
	Overall	95	89-101	3.7	10

ILV of residue analytical enforcement method DFG S19 (extended revision) for plant matrices (Lakaschus, 2004, SAN619/7559)

The purpose of this study was to perform an ILV of the DFG Method S 19 (extended revision), for the determination of residues of cyproconazole in wheat grain and melon. Cyproconazole is quantified using LC-MS/MS. Ion transitions m/z 291.97 to m/z 69.88 (primary transition) and m/z 291.97 to m/z 124.89 (secondary transition) were both and was monitored for qualification (first transition). This slightly deviates from the original method validation described above. However, since both transitions yield equivalent results, this has no impact on the validity of the study.

Recovery data of cyproconazole from wheat grain and melon are summarised in Table 60.

[13] DFG S 19. Modular Multiple Analytical Method for the Determination of Pesticide Residues in Foodstuffs, L 00.00-34 of the Collection of Official Test Methods according to § 35 LMBG (German Federal Food Act), extended and revised version of DFG Method S19, November 1999.

Table 60 Recovery Values for the ILV of Enforcement Method DFG S19 (extended revision) for the Determination of Cyproconazole in Crops

Matrix	Fortification level (mg/kg)	Recovery (%)		RSD (%)	n
		Mean	Range		
Secondary transition m/z 292 → m/z 125					
Wheat Grain	0.01	94	88-97	3.8	5
	0.1	95	93-98	2.3	5
	Overall	95	88-98	3.1	10
Melons	0.01	96	93-100	2.7	5
	0.1	95	92-102	4.6	5
	Overall	95	92-102	3.7	10

Animal commodities (see also method BS2598/BS8058 and Table 52 above)

Data collection methods for cyproconazole and some metabolites are included with the ruminant feeding studies (Blanz, 1995, TDS BS5217; Ali, 1995, TDS DP-301816; Oakes, 1994, T021566-04). Samples of milk and cattle tissue from the cattle feeding study were analysed for residues of cyproconazole using a GC/NPD method. Briefly, tissue and milk samples were extracted (twice for tissue samples) with acetone and centrifuged. The acetone was removed from the extract by rotary evaporation, water was added, and the extract was cleaned up on an Extrelut solid-phase extraction (SPE) column and a silica column (in series). Residues were eluted from the SPE column using toluene and were eluted from the silica column using ethyl acetate. For tissue samples, the eluate was evaporated to dryness and the residue redissolved in toluene for injection on a GC equipped with Supelco SPB5 capillary column and a nitrogen-phosphorus detector. For milk samples, the eluate was evaporated to dryness, redissolved in methanol and water, and cleaned up on a C18 SPE column. Residues were eluted from the column using methanol:water (8:2, v:v) and the eluate was evaporated to dryness and redissolved in toluene for GC analysis as described above. The stated limit of detection was 0.003 ppm.

Samples of milk, cream, and skimmed milk from the cattle feeding study were analysed for residues of metabolites M21a and M36 using an HPLC/UV method. Samples were extracted with acetone and centrifuged. The acetone was removed from the extract by rotary evaporation and the aqueous phase was acidified with 0.1 M HCl and partitioned three times with ethyl acetate. The combined ethyl acetate phases were evaporated to dryness and the residue was redissolved in ethanol and water for injection onto an HPLC equipped with two C18 columns and a UV detector (220 nm). An isocratic mobile phase of acetonitrile:0.02 M NaH2PO4 (25:75 on first column, 20:80 on second column; v:v) was used. The stated limit of detection was 0.003 ppm for each compound.

Samples of kidney and liver from the cattle feeding study were analysed for residues of metabolite M14 using an HPLC/MS method. Briefly, samples were homogenized, 0.1% trifluoroacetic acid was added, and the mixture was extracted three times with acetonitrile. Following isolation of the extract by centrifugation, the extracts were neutralized with phosphate buffer (pH 7). Acetonitrile was then removed from the extracts under vacuum and the aqueous phase was cleaned up on a C18 SPE column; residues were eluted from the column using methanol:water (95:5, v:v). After overnight storage in the refrigerator, the eluate was centrifuged and then diluted with water:methanol (9:1, v:v) for analysis. Samples were injected onto an HPLC equipped with a Zorbax Rx-C18 column and an MS detector with an atmospheric pressure chemical ionization interface. The reported limit of detection was 0.0003 ppm and the demonstrated limit of quantitation was 0.01 ppm.

The animal commodity methods were validated by fortifying untreated samples of milk and tissues with cyproconazole and metabolites M21a, M36, and M14. The results of the validation studies are presented in Table 61.

Table 61 Method recoveries of cyproconazole and metabolites M14, M21a, and M36 from fortified samples of untreated milk and tissues

Commodity	Fortification level (mg/kg)	Percent recovery [a]
Cyproconazole		
Milk, whole	0.005	70.7-117 (6)
	0.05	49.1-63.5 (8); 71.0-118 (9)
Milk, cream	0.05	74.7
	0.2	105
Milk, skimmed	0.05	108
	0.2	82.4
Fat	0.01	63.1; 72.6
	0.05	47.9, 62.1; 90.0
	0.1	89.9, 95.8
Kidney	0.05	66.0; 81.0
Liver	0.05	75.3, 80.5
	0.1	84.3-88.3 (3)
	0.5	78.8-89.9 (6)
Muscle	0.05	71.3, 85.4
M14		
Kidney	0.01	97-112 (5); 140
	0.05	93-110 (6)
Liver	0.01	111-123 (5)
	0.05	101-105 (3)
	1	86-93 (5)
M21a		
Milk	0.005	72.2
	0.05	50.7-62.1 (8); 70.7-93.1 (22)
	0.2	48.7-69.9 (9)
Milk, cream	0.05	50.6
	0.2	57.0
Milk, skimmed	0.05	76.1-116 (3)
	0.2	48.5, 61.8
M36		
Milk	0.005	48.5
	0.05	48.6-69.7 (15); 71.1-106 (15)
	0.2	57.2-68.0 (5); 72.0-79.8 (4)

[a] Each recovery value represents one sample unless otherwise indicated in parentheses; recoveries outside the 70-120% range are listed separately.

Residue analytical method SOP RAM 499/01 (Crook, 2006, SAN619/8212)

The Meeting received study on an analytical method description and validation data for a LC/MS/MS method (Syngenta Method RAM 499/01) for determining residues of free and conjugated cyproconazole in livestock commodities. This method is also being proposed for enforcing tolerances of cyproconazole in livestock commodities. For this method, free and conjugated cyproconazole residues are extracted with ACN:water (80:20 v/v) and centrifuged. Residues in the resulting extracts are then hydrolysed using either concentrated ammonia (eggs and tissues) or 2M HCl (milk) under gentle reflux for 2 hours. After cooling, subsamples are diluted sequentially with ACN and water, and the resulting fractions were analysed by LC/MS/MS. The HPLC system consisted for a C18 column run at 40 °C with a mobile phase gradient of 0.2% acetic acid in water to ACN. The HPLC was coupled to a triple-quadrupole mass spectrometer run in the multiple-reaction-monitoring (MRM) mode. The retention time for cyproconazole is ~1.6 minutes. The primary transition used for quantitation is m/z 292→70, and the secondary transition used for confirmation is m/z 292→125. Calibration is with external standards. The validated LOQ is 0.01 mg/kg for cyproconazole in each livestock commodity, and the estimated LOD is 0.002 mg/kg.

Recovery results for various fortified livestock commodities are given in Table 62. Apparent residues of cyproconazole were < 0.01 mg/kg in all control samples. The LC/MS/MS method was also adequately validated in conjunction with the analysis of samples from the cattle and poultry feeding studies (Table 63).

Table 62 Recovery Results from Method Validation of Livestock Matrices using LC/MS/MS Method RAM 499/01[a]

Matrix	Cyproconazole Spiking Level (mg/kg)	Recoveries Obtained (%)	Mean Recovery ± SD [CV] (%)
Bovine Liver	0.01	106, 102, 99, 95, 104	101 ± 4 [4]
	0.10	107, 110, 103, 96, 98	103 ± 6 [6]
	Overall	95-110	102 ± 5 [5]
Bovine Kidney	0.01	92, 102, 94, 117, 104	102 ± 10 [10]
	0.10	100, 110, 92, 87, 109	100 ± 10 [10]
	Overall	92-110	101 ± 10 [9]
Bovine Muscle	0.01	101, 102, 108, 99, 98	102 ± 4 [4]
	0.10	107, 105, 104, 93, 101	102 ± 5 [5]
	Overall	93-108	102 ± 4 [4]
Bovine Fat	0.01	92, 99, 92, 90, 90	93 ± 4 [4]
	0.10	96, 92, 96, 89, 96	94 ± 3 [3]
	Overall	89-99	93 ± 3 [4]
Bovine Milk	0.01	118, 104, 104, 111, 105	108 ± 6 [6]
	0.10	113, 117, 110, 105, 100	109 ± 7 [6]
	Overall	100-118	109 ± 6 [6]
Eggs	0.01	82, 106, 110, 102, 113	103 ± 12 [12]
	0.10	106, 109, 102, 93, 102	102 ± 6 [6]
	Overall	82-110	102 ± 9 [9]

[a] Standards for fortification were prepared in ACN

Table 63 Summary of Concurrent Recoveries of Cyproconazole from Livestock Feeding Studies [a]

Matrix	Spike level (mg/kg)	Sample size (n)	Recoveries (%)	Mean ± SD [CV] (%)
Milk	0.01-0.20	18	99-116	107 ± 6 [6]
Cattle liver	0.01-0.50	6	90-112	101 ± 10 [10]
Cattle kidney	0.01, 0.05	6	99-108	103 ± 4 [4]
Cattle fat	0.01-0.10	16	82-103	95 ± 5 [5]
Cattle muscle	0.01-0.05	6	81-115	103 ± 14 [14]
Eggs	0.01-0.10	12	83-106	95 ± 6 [6]
Poultry Skin/fat, Fat	0.01, 0.05	4	74-102	91 ± 12 [13]
Poultry Liver	0.01, 0.05	2	108, 112	110
Poultry Muscle	0.01, 0.05	2	99, 104	102

[a] Data were obtained from the cattle and poultry feeding studies.

Independent Laboratory Validation of Method RAM 499/01 (Bour, 2006, SNY/CYP/06001, SAN619/8053)

An ILV trial of Method RAM 499/01 was conducted by ADME Bioanalyses of Vergèze, France. Commercially obtained samples of eggs, milk, bovine liver, and bovine fat were fortified with cyproconazole at the reported LOQ (0.01 mg/kg) and at 10x the LOQ (0.10 mg/kg). For each commodity, two control samples and five samples at each fortification level were analysed. The method was conducted as written without modification, and recoveries were determined using both the primary (m/z 292→70) and secondary (m/z 292→125) transitions. No communication with the developing laboratory was reported.

The method was successfully validated in the first trial. See Table 64.

Table 64 Recovery Results Obtained by an Independent Laboratory Validation using LC/MS/MS RAM 499/01

Matrix	Monitored Transition	Spiking Level (mg/kg)	Recoveries (%)	Mean Recovery ± SD [CV] (%)
Bovine Liver	292.1→70.0	0.01	88, 88, 77, 91, 95	88 ± 7 [8]
		0.10	87, 84, 85, 86, 79	84 ±3 [4]
		Overall	77-91	86 ±5 [6]
	292.1→125.0	0.01	91, 86, 78, 96, 91	88 ±7 [8]
		0.10	87, 82, 88, 87, 79	85 ±4 [5]
		Overall	78-96	87 ±6 [6]
Bovine Fat	292.1→70.0	0.01	83, 88, 84, 79, 83	83 ± 3 [4]
		0.10	82, 84, 82, 84, 80	82 ±2 [2]
		Overall	79-88	83 ± 2 [3]
	292.1→125.0	0.01	80, 79, 77, 88, 81	81 ± 4 [5]
		0.10	86, 84, 82, 83, 80	83 ± 2 [3]
		Overall	77-88	82 ± 3 [4]
Bovine Milk	292.1→70.0	0.01	87, 88, 93, 90, 82	86 ± 5 [6]
		0.10	90, 93, 90, 90, 87	90 ±2 [2]
		Overall	82-93	88 ± 4 [5]
	292.1→125.0	0.01	83, 71, 93, 77, 66	78 ± 11 [14]
		0.10	91, 95, 90, 91, 91	92 ± 2 [2]
		Overall	66-95	85 ± 10 [12]
Hen Eggs	292.1→70.0	0.01	100, 87, 87, 85, 84	89 ± 7 [7]
		0.10	85, 82, 96, 76, 85	85 ± 7 [9]
		Overall	76-100	87 ± 7 [8]
	292.1→125.0	0.01	90, 96, 91, 83, 86	89 ± 5 [6]
		0.10	86, 81, 98, 77, 90	86 ± 8 [9]
		Overall	77-96	88 ± 7 [7]

Radiovalidation of Method RAM 499/01 (2006; Chen and Wilson, 2006, T027741/04)

A radiovalidation study using ^{14}C-labeled milk and tissue samples from a goat dosed with [triazole-U-^{14}C]cyproconazole (98.6% radiochemical purity) at a dose of 2.56 mg/kg body weight/day (72 ppm) to determine the extractability of free and conjugated residues of cyproconazole from livestock matrices was provided.

For analysis, samples of milk and kidney were extracted once with ACN (100%) and then repeatedly with ACN:water (80:20). Samples of liver, muscle and fat were extracted repeatedly with ACN:water (80:20). All extracts were combined by matrix. Radioactivity in the combined extracts and the residual solids were determined by LSC and combustion/LSC. The ACN:water extractions released 92.2–99.3% of the TRR from milk and tissues, and 0.7–7.9% of the TRR remained in the residual solids.

To assess the suitability of different hydrolytic procedures for recovering conjugated and free cyproconazole residues, subsamples of milk and tissue extracts were subjected to enzyme, acid and base hydrolyses. For the enzyme hydrolysis, concentrated extracts were incubated overnight at 37ºC with β-glucuronidase in a 0.2M acetate buffer (pH 5). For the acid hydrolysis, concentrated extracts were hydrolysed in 1M HCl at 95 ºC for 1 hour. For the base hydrolysis, concentrated extracts were hydrolysed by refluxing in concentrated NH4OH (6M) for 2 hours. After cooling the resulting hydrolysates were partitioned with EtOAc. Cyproconazole residues in the resulting organic fractions were then quantified using 2D-TLC or 1D-TLC (acid hydrolysed residues).

The TLC analyses used silica gel plates with two solvent systems: SS1-diethyl ether:ethanol:formic acid (95:5:05); and SS2 - DCM:MeOH (95:5). Radioactive residues on TLC plates were detected and quantified using a Fuji FLA 5000 Bio-imaging analyser system, and reference standards of parent (M1 and M2) were visualized with UV light. The identity of cyproconazole was confirmed by co-chromatography with standards using reverse-phase HPLC.

The extraction procedures (ACN:water) used in the proposed enforcement method (RAM Method 499/01) will adequately extract residues from livestock commodities. Although enzymatic hydrolysis would clearly be preferable for liver samples, the specified acid or base hydrolyses will adequately release conjugated residues of cyproconazole. Results are summarised in Tables 65 and 66.

Table 65 Distribution of ^{14}C-Residues in Milk and Tissues from Goats Dosed with [^{14}C-triazole]-Cyproconazole

Fraction	Kidney		Liver		Leg Muscle		Omental Fat		Milk (Day 4)	
	TRR = 3.928 mg/kg		TRR = 9.559 mg/kg		TRR = 0.430 mg/kg		TRR = 0.544 mg/kg		TRR = 2.059 mg/kg	
	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg
ACN/H20 extracts	96.5	3.787	92.2	8.802	96.8	0.416	99.3	0.540	99.2	2.043
1M HCl hydrolysis	92.0	3.613	90.9	8.672	87.0	0.374	90.2	0.490	98.8	2.035
EtOAc	50.6	1.989	58.6	5.589	42.5	0.183	73.2	0.398	81.8	1.686
cyproconazole (TLC)	17.0	0.668	26.3	2.504	19.5	0.084	50.1	0.273	9.9	0.204
6M NH4OH hydrolysis	97.0	3.809	92.4	8.816	94.7	0.407	100.6	0.547	98.9	2.037
EtOAc	55.6	2.185	51.8	4.941	54.1	0.233	84.9	0.462	77.9	1.605
cyproconazole (TLC)	30.2	1.189	31.2	2.970	25.3	0.109	59.2	0.323	5.3	0.111
β-glucuronidase treatment	88.8	3.487	91.9	8.770	94.1	0.405	96.0	0.522	101.3	2.085
EtOAc	68.5	2.689	81.4	7.768	49.6	0.214	90.2	0.490	80.6	1.659
cyproconazole (TLC)	37.9	1.487	50.7	4.839	25.7	0.111	57.0	0.310	9.6	0.199
Residual Solids (PES)	3.6	0.141	7.9	0.757	3.2	0.014	0.7	0.004	0.8	0.016

Table 66 Recovery of Free and Conjugated Cyproconazole from Milk and Goat Tissues Using Enzymatic, Basic and Acidic Hydrolyses

Matrix	Cyproconazole Residues					
	Enzyme hydrolysis (β-glucuronidase)		Base hydrolysis (6.0M NH4OH)		Acid hydrolysis (1.0M HCl)	
	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
Kidney	37.9	1.487	30.2	1.189 (80%)[a]	17.0	0.668 (45%)
Milk	9.6	0.199	5.3	0.111 (56%)	9.9	0.204 (103%)
Liver	50.7	4.839	31.2	2.970 (61%)	26.3	2.504 (52%)
Fat	57.0	0.310	59.2	0.323 (104%)	50.1	0.273 (88%)
Muscle	25.7	0.111	25.3	0.109 (98%)	19.5	0.084 (76%)

[a] Values in parentheses (%) represent the amount of cyproconazole recovered by base or acid hydrolyses compared to the enzyme hydrolysis.

Residue analytical method SOP RAM 499/02 (Crook, 2006, SAAN69/8212)

Method SOP RAM 499/02 is an update to method SOP RAM 499/01 using modified extraction conditions. Samples are extracted by homogenization with ACN:water (80:20 v/v). Extracts are centrifuged and aliquots (5.0 mL = 0.5 g) are hydrolysed with concentrated ammonia solution or 1 M hydrochloric acid solution (milk only). Aliquots of the hydrolysate are diluted and final determination

is by LC-MS/MS as described in method SOP RAM 499/01. For validation of the method see data obtained for method SOP RAM 499/01.

Residue analytical enforcement method DFG S19 (extended revision) for livestock matrices (Klimmek, 2004, SAN619/7504)

The residue analytical method for cyproconazole in livestock matrices is based on the multi residue method DFG-method S19 (extended revision: DFG S 19. Modular Multiple Analytical Method for the Determination of Pesticide Residues in Foodstuffs, L 00.00-34 of the Collection of Official Test Methods according to § 35 LMBG (German Federal Food Act), extended and revised version of DFG Method S19, November 1999). For the extraction, module E6 was used for fat containing matrices and module E8 for milk and meat. The clean-up procedure is carried out according to module GPC. Cyproconazole is quantified by LC-MS/MS. Primary transition m/z 291.97→m/z 69.88 was used for quantification. Minor modifications were made in order to adapt the method to the laboratory situation.

Recovery data of cyproconazole are summarised in Table 67.

Table 67 Recovery Data for Method DFG S19 (extended revision) for the Determination of Cyproconazole in Livestock Matrices

Matrix	Fortification level (mg/kg)	Recovery (%)		RSD (%)	n
		Mean	Range		
Primary transition m/z 292→70					
Bovine Fat	0.01	91	88–92	1.8	5
	0.05	91	89–91	0.5	5
	Overall	91	88–92	1.3	10
Bovine Muscle	0.01	94	90–98	3.4	5
	0.1	96	92–99	2.7	5
	Overall	95	90–99	3.0	10
Bovine Kidney	0.01	97	95–100	2.2	5
	0.1	103	102–105	0.9	5
	Overall	100	95–105	3.8	10
Bovine Liver	0.01	90	85–94	4.5	5
	0.1	89	87–95	4.1	4[a]
	Overall	90	85–95	4.1	9
Milk	0.01	89	86–92	3.4	5
	0.1	95	91–100	4.2	5
	Overall	92	86–100	4.7	10
Eggs	0.01	85	81–88	4.0	5
	0.1	88	81–91	4.5	5
	Overall	86	81–91	4.4	10

[a] Due to an outlier (identified by Grubbs test), only 4 specimens were used for the statistical calculations.

Independent laboratory validation of residue analytical enforcement method DFG S19 (extended revision) for livestock matrices (Lakaschus, 2005, SAN619/7870.)

The purpose of this study was to perform an independent laboratory validation of DFG Method S 19 (extended revision) for the determination of residues of cyproconazole in livestock matrices (milk, meat, fat). Cyproconazole is quantified by LC-MS/MS. Ion transitions m/z 291.97 to m/z 69.88 (primary transition) and m/z 291.97 to m/z 124.89 (secondary transition) were both monitored.

Recovery data of cyproconazole from milk, meat and fat are summarised in Table 68.

Table 68 Recovery Data for the ILV of Enforcement Method DFG S19 (extended revision) for the determination of cyproconazole in livestock matrices

Matrix	Fortification level (mg/kg)	Recovery (%)		RSD (%)	n
		Mean	Range		
Primary transition m/z 292→70					
Milk	0.01	102	98–107	3.3	5
	0.1	103	97-105	4.0	5
	Overall	102	97-107	3.5	10
Meat	0.01	95	93–95	1.6	5
	0.1	101	98–103	1.9	5
	Overall	98	93–103	3.5	10
Fat	0.01	97	94–99	4.3	5
	0.1	97	96–99	1.4	5
	Overall	97	94–99	3.0	10
Secondary transition m/z 292→125					
Milk	0.01	102	96–109	5.0	5
	0.1	104	99-107	4.1	5
	Overall	103	96–107	4.5	10
Meat	0.01	95	92–97	2.2	5
	0.1	101	97–104	2.9	5
	Overall	98	92-104	3.9	10
Fat	0.01	98	92–106	5.6	5
	0.1	99	97 – 103	2.5	5
	Overall	99	92-103	4.1	10

Stability of Residues in Stored Analytical Samples

The registrant submitted data depicting the stability of residues of cyproconazole in/on various raw agricultural commodities and processed commodities during frozen storage (Ali, 1994, SAN619/0289; Ali, 1994, SAN619/0035; Cameron, 1994, SAN619/5273). Samples of untreated grapes, raisins, nectarines, peaches, peanut commodities (forage, hay, hulls, nutmeat, refined oil, soapstock, and expelled presscake), and wheat commodities (forage, grain, and hay) were placed in glass bottles and fortified with cyproconazole at 0.5 mg/kg (peanut refined oil and soapstock) or 0.1 mg/kg (all other commodities). The samples of peanut forage and hay and wheat forage and hay were coarsely chopped then mixed in a food chopper, samples of peanut hulls, peanut nutmeat, and wheat grain were ground to a fine powder in a blender, and samples of grapes, nectarines, and peaches were chopped with a scalpel prior to fortification. The samples were then stored frozen at ≤-12 ºC and removed at various intervals for analysis. Samples were analysed using the GC/NPD method AM-0842-0790-0. The results of the storage stability studies are presented in Table 69.

Concurrent storage stability studies were conducted with the cattle feeding study to demonstrate the stability of residues of cyproconazole in milk and tissues, of residues of metabolites M21a and M36 in milk, and of residues of metabolite M14 in kidney and liver during frozen storage. Samples of control tissues and milk were fortified with cyproconazole (milk, fat, kidney, and liver), M21a and M36 (milk), or M14 (kidney and liver) and stored under the same storage conditions as the samples from the feeding study. For cyproconazole, samples were removed for analysis after approximately 1, 3, 9, and 12 months (milk only), for metabolites M21a and M36, samples were removed for analysis after 12 months, and for metabolite M14, samples were removed for analysis after 20 months of storage. Samples were analysed using the livestock-commodity methods discussed in the "Residue Analytical Methods" section. The results of the storage stability study are presented in Table 70.

Table 69 Storage Stability and Fresh Fortification Recoveries of Residues of Cyproconazole from Samples of Various Plant Commodities Fortified with Cyproconazole and Stored Frozen at ≤-12 °C

Commodity [a]	Storage interval (months)	Fresh fortification recovery (%)[b]	Storage stability remaining (%)	Corrected storage stability remaining (%)[c]
Grapes (0.1 mg/kg)	0	75-104 (3); 126, 130	--	--
	9	93, 116, 119	86, 95, 102, 110	79, 87, 93, 101
	42	87, 96, 99	86, 93, 93, 95	91, 99, 99, 101
Raisins (0.1 mg/kg)	0	74-100 (5)	--	--
	11	76, 77, 78	69, 70, 82, 82	90, 91, 106, 106
	43	87, 95, 109	84, 101, 106, 112	87, 104, 109, 115
Nectarines (0.1 mg/kg)	0	79-87 (5)	--	--
	11	90, 91	76, 77, 86, 115	84, 85, 95, 127
	42	79, 90, 91	76, 77, 78, 82	88, 89, 90, 95
Peaches (0.1 mg/kg)	0	72-89 (5)	--	--
	9	84, 99, 107	76, 78, 93, 116	79, 81, 96, 120
	42	81, 82, 83	81, 82, 86, 91	99, 100, 105, 111
Peanut, forage (0.1 mg/kg)	0	81-109 (5)	--	--
	9	93, 99, 117	78, 97, 109, 112	76, 94, 106, 109
	38	80, 86, 105	81, 98, 102, 111	90, 108, 113, 123
Peanut, hay (0.1 mg/kg)	0	97-104 (5)	--	--
	10	89, 100, 102	100, 102, 107, 114	103, 105, 110, 118
	41	87, 97, 104	92, 103, 107, 109	96, 107, 111, 114
Peanut, hulls (0.1 mg/kg)	0	51; 71-79 (4)	--	--
	9	70, 82, 91	70, 80, 81, 87	86, 99, 100, 107
	39	69, 75, 86	68, 70, 71, 85	89, 91, 93, 111
Peanut, nutmeat (0.1 mg/kg)	0	79-90 (5)	--	--
	10	84, 91, 97	79, 79, 94, 96	87, 87, 104, 106
	40	87, 92, 95	92, 98, 105, 105	101, 107, 115, 115
Peanut, expelled presscake (0.1 mg/kg)	0	75-105 (9)	--	--
	28	75, 86, 92	75, 78, 79, 82	89, 92, 94, 97
Peanut, refined oil (0.5 mg/kg)	0	81-105 (8)	--	--
	27	73, 78, 83	65, 84, 85, 85	83, 108, 109, 109
Peanut, soapstock (0.5 mg/kg)	0	81-99 (5)	--	--
	28	85, 85, 86	81, 88, 89, 92	99, 103, 105, 107
Wheat, forage (0.1 mg/kg)	0	84-99 (5)	--	--
	10	82, 88, 89	72, 96, 97, 99	83, 111, 112, 115
	39	100, 113, 115	102, 106, 112, 128	93, 97, 102, 117
Wheat, grain (0.1 mg/kg)	0	63; 79-108 (4)	--	--
	9	82, 83, 99	84, 84, 88, 91	95, 95, 100, 103
	39	71, 83, 92	79, 87, 101, 102	96, 106, 123, 124
Wheat, hay (0.1 mg/kg)	0	62; 80-85 (4)	--	--
	10	86, 86, 94	71, 76, 90	80, 86, 102
	41	97, 106, 108	106, 113, 116, 119	102, 109, 112, 115

[a] Fortification level in parentheses.

[b] Number of samples in parentheses; recoveries outside the 70-120% range are listed separately.

[c] Calculated by dividing the storage stability remaining by the average fresh fortification recovery.

Table 70 Storage Stability and Fresh Fortification Recoveries of Residues of Cyproconazole and Metabolites M14, M21a, and M36 from Samples of Livestock Commodities Fortified with Cyproconazole at 0.01–10 mg/kg and Stored Frozen at -20 °C

Commodity [a]	Storage interval (months)	Fresh fortification recovery (%)[b]	Storage stability recovery (%)	Corrected storage stability recovery (%)[c]
Cyproconazole				
Milk (0.01 mg/kg)	12	67.4, 73.7	78.2, 98.2	111, 139
Fat (0.05 mg/kg)	1	48.2	61.1, 92.6	127, 192
	3	74.3	50.8, 68.4	68.4, 92.1

Commodity [a]	Storage interval (months)	Fresh fortification recovery (%)[b]	Storage stability recovery (%)	Corrected storage stability recovery (%)[c]
	9	135	51.5, 78.2	38.1, 57.9
Fat (1 mg/kg)	1	75.4	64.2, 79.5	85.1, 105
	3	79.2	46.3, 70.8	58.5, 89.4
	9	76.7	62.9, 67.3	82.0, 87.7
Kidney (0.1 mg/kg)	1	88.3	99.4, 103	113, 117
	3	111	75.2, 97.1	67.7, 87.5
	9	68.1	84.6, 104	124, 153
Kidney (1 mg/kg)	1	82.1	76.5, 89.0	93.2, 108
	3	99.5	86.7, 89.8	87.1, 90.3
	9	76.1	62.9, 67.3	82.7, 88.4
Liver (0.1 mg/kg)	1	85.4, 92.0	78.9, 82.8, 89.6	89.0, 93.3, 101
	9	79.5	79.6, 100	100, 126
Liver (1 mg/kg)	1	92.0	83.4, 85.4	90.7, 92.8
	3	73.7	74.1, 79.9	101, 108
	9	60.9	69.1, 71.4	113, 117
Liver (10 mg/kg)	1	74.7	82.1, 83.3	110, 112
	3	89.4	64.3, 70.7	71.9, 79.1
	9	67.6	66.7, 66.8	98.7, 98.8
M14				
Kidney (0.05 mg/kg)	20	92.86, 101.45 (0.01)	77.7, 102.2, 147.1	80.0, 105, 151
Liver (0.1 mg/kg)	20	115.56 (0.01)	60.9, 97.3	52.7, 84.2
Liver (1.0 mg/kg)	20	100.73, 101.09 (0.05)	73.7, 87.3	73.0, 86.5
M21a				
Milk (0.01 mg/kg)	12	77.0, 84.6	68.4, 71.6	84.7, 88.6
Milk (0.2 mg/kg)	12	86.4, 89.2	74.7, 77.3	85.1, 88.0
M36				
Milk (0.01 mg/kg)	12	65.4, 81.3	44.7, 45.1	60.9, 61.5
Milk (0.2 mg/kg)	12	81.9, 92.7	51.0, 55.5	58.4, 63.6

[a] Fortification level in parentheses.

[b] Fresh fortification level and the fortification level of the stored sample are the same unless otherwise indicated in parentheses.

[c] Calculated by dividing the storage stability remaining by the average fresh fortification recovery.

USE PATTERN

Cyproconazole-based products are recommended for foliar application to control airborne diseases, including powdery mildew and rusts. Cyproconazole may also be used as a seed treatment fungicide. The GAPs are summarised by Codex crop group and country in Table 71 and have been assembled from labels or translations of labels provided by the manufacturer. Additional use information was provided by Japan and by the Netherlands. The GAPs are generally for supervised field trials considered by the Meeting and do not represent all available GAPs worldwide. Additionally considerable use pattern information was presented without labels and was therefore not summarised.

Table 71 Summary of National GAPs (Labels) for Use of Cyproconazole on Food and Feed Crops, Arranged by Crop Group

Crop	Country	Formulation	Application					PHI Days/ Comment
			Method	kg as/ha	kg as/hL	Retreatment interval (days)	No. or max (kg ai/ha/ season)	
Pome Fruits (FP002)								
Apple	Italy	WG, 10%	foliar spray	-	0.02	10-14	4	7
Legume vegetables (VP014)								
Pea	France	SC, 375 g/L chlorothalonil and 40 g/L cyproconazole)	foliar spray	0.07	-	15	2	21 Food pea 40 Feed pea
Pulses (VP015)								
Bean	Great Britain (UK)	SC, 375 g/L chlorothalonil and 40 g/L cyproconazole)	foliar spray	0.08	0.04	14-21	2	42
Pea	Great Britain (UK)	SC, 375 g/L chlorothalonil and 40 g/L cyproconazole	foliar spray	0.08	0.02-0.04	14-21	2	42
Root and Tuber Vegetable (VR016)								
Sugar beet	Italy	SC (200 g/L azoxystrobin and 80 g/L cyproconazole)	foliar spray	0.08	0.016-0.04	18-21	2	21
Sugar beet	The Netherlands	SC (160 g/L in combination with azoxystrobin)	foliar spray	0.06	0.015 – 0.03	21	2	45
Cereal Grains (GC020)								
Wheat	Germany	EC (240 g/L)	foliar spray	0.096	0.024-0.048	-	2	35 Spray until BBCH 61 (early flowering)
Wheat	Japan	SC(20% azoxystrobin/80% cyproconazole)	foliar spray	-	0.0037	-	2	30
Wheat	The Netherlands	SC (80 g/L and 160 g/L, in combination with azoxystrobin)	foliar spray	0.08	0.02 – 0.04	-	2	42 - 45
Rye	Germany	EC (240 g/L)	foliar spray	0.096	0.024-0.048		2	35 Spray until BBCH 61 (early flowering)
Rye	The Netherlands	SC (80 g/L in combination with azoxystrobin)	foliar spray	0.08	0.02 – 0.04	-	1	42
Triticale	France	SL (100 g/L)	foliar spray	0.1	0.1- 0.017	na	1	-
Triticale	The Netherlands	SC (80 g/L and 160 g/L in combination with azoxystrobin)	foliar spray	0.08	0.02 – 0.04	-	1	42 - 45
Barley	Germany	EC (240 g/L)	foliar spray	0.096	0.024-0.048		2	35 Spray until BBCH 61 (early flowering)

Crop	Country	Formulation	Application					PHI Days/ Comment
			Method	kg as/ha	kg as/hL	Retreatment interval (days)	No. or max (kg ai/ha/ season)	
Oats	Great Britain (UK)	SC (200 g/L azoxystrobin and 80 g/L cyproconazole)	foliar spray	0.08	0.04	unclear	2	GS59 (emergence of ear)
Maize	United States	SL (100 g/L or 0.83 lbs ai/gal)	foliar spray, incl sprinkler irrigation	0.04	0.04	7-14	2 and 0.04	30 21 silage
Oilseed (SO023)								
Rape	Great Britain (UK)	SC (20 g/L azoxystrobin and 80 g/L cyproconazole)	foliar spray	0.08	0.04	-	2	30 Or BBBCH 79, whichever occurs first
Soya bean	United States	SL (100 g/L or 0.83 lbs ai/gal)	foliar spray, incl sprinkler irrigation	0.04	0.042 ground; 0.21 aerial	14-28	2	30 14 forage
Peanut	Australia	SL (100 g/L)	foliar spray	0.06	0.015-0.06 ground; 0.3 aerial	< 14	3 consecutive (5 per season)	14

RESIDUES RESULTING FROM SUPERVISED TRIALS

Supervised field trials for cyproconazole were received by the Meeting for the crops summarised below:

Crop	Table No
Apple	73
Pea (succulent)	73
Pea and Bean (dry)	74
Sugar beet (roots)	75
Wheat (grain)	76
Rye (grain)	77
Barley (grain)	78
Maize (field corn) (grain)	79
Rape seed (canola)	80
Soya bean (dry)	81
Peanut	82
Pea vines (green)	83
Pea and bean fodder	84
Sugar beet tops	85
Wheat straw	86

Crop	Table No
Rye straw	87
Barley straw	88
Maize fodder/stover	89
Maize forage	90
Rape seed forage	91
Soya bean forage	92
Soya bean hay	93
Peanut hulls	94
Peanut forage	95
Peanut fodder	96

Results for trials conducted in accordance with the maximum GAP as reflected in a relevant approved national label are underlined. Where a residue value is greater at a PHI interval longer than the GAP PHI value, the longer PHI value is selected (underlined). Where replicate samples are collected and analysed, the highest value is selected. Where replicate analyses are conducted on a sample, the average result is selected.

Pome Fruits

Apple

A total of six supervised residue trials were conducted on apples between 1993 and 1994, three in Southern Europe and three in South America.

Table 72 Apple Field Trials

Location/ Year/ Variety	Application					PHI (days)	Cypro-conazole (mg/kg)	Study/ Report
	Formulation	Method	Rate (kg ai/ha)	Rate (kg ai/hL)	Growth Stage (BBCH)			
Lerida, Spain 1994 Golden Delicious	10% WG	Foliar	0.027 0.029 0.028 0.029 0.033	1500 L/ha	65 – 67 77 - 79	0 3 8 10 14	0.06 0.06 0.03 0.03 0.03	R10301
Lerida, Spain 1994 Golden Delicious	10% WG	Foliar	0.030 0.028 0.028 0.027 0.030	1500 L/ha	65 -67 77 - 79	0 3 8 10 14	0.08 0.07 0.05 0.05 0.05	R10301
Lerida, Spain 1994 Belleza de Roma	10% WG	Foliar	0.028 0.029 0.030 0.028 0.028	1500 L/ha	65 77 - 79	0 3 8 10 14	0.05 0.03 0.03 0.03 0.03	R10301
Guaravera. Brazil 1993 Ana	100 g/L SL	Foliar		0.002 0.002 0.002 0.002	50% mature Maturation	14	< 0.01	12/93-R305 SAN948
				0.004 0.004 0.004 0.004	50% mature Maturation	14	0.03	

Location/ Year/ Variety	Application					PHI (days)	Cypro-conazole (mg/kg)	Study/ Report
	Formulation	Method	Rate (kg ai/ha)	Rate (kg ai/hL)	Growth Stage (BBCH)			
Sitio Sao Roque-Valinhos- SP, Brazil 1993 Ohio Beat	100 g/L SL	Foliar		0.002 0.002 0.002 0.002	Diameter>60 mm Mature fruit	21	0.01	12/93-R305
				0.004 0.004 0.004 0.004	Diameter>60 mm Mature fruit	21	0.04	
				0.002 0.002 0.002 0.002	Diameter>60 mm Mature fruit	0 7 14 21 28	0.03 0.02 0.01 0.01 0.01	R137B/ SAN945

Legume vegetables

A total of eight supervised residue trials were conducted on fresh peas between1994 and 2004. The trials were conducted on outdoor crops, five in Northern Europe and three in Southern Europe. In six of the trials, samples were harvested both at the succulent pea stage (about BBCH 75–79) and at the dry pea (pulse) stage (>BBCH 80).

Table 73 Fresh Pea (Legume) Trials

Location/ Year/ Variety	Application					PHI (days)	Cypro-conazole (mg/kg)	Study/ Report
	Formulation	Method	Rate (kg ai/ha)	Rate (kg ai/hL)	Growth Stage (BBCH)			
St. Aubin, France (North) 1994 Nina	40 g/L SL	Foliar	0.080 0.082	398 L/ha 408 L/ha	61 65 - 67	14 pod 21 pod 14 pea 21 pea	0.02 < 0.01 < 0.01 < 0.01	R10295/ BS9427
Bonnencontre, France (north) 1994 Valette	40 g/L SL	Foliar	0.076 0.080	382 L/ha 400 L/ha	61 65 - 67	14 pod 20 pod 14 pea 20 pea	0.02 0.02 < 0.01 < 0.01	R10295 BS9427
Birkin, UK (North) 2004 Solara	80 g/L SC	Foliar	0.082 0.082	306 L/ha 306 L/ha	61 – 63 69 - 73	7 pea (BBCH 75 14 pea 21 pea (BBCH 79) 30 pea 43 pea	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	04-0422
Hummanby, Yorkshire, UK (North) 2004 Samson	80 g/L SC	Foliar	0.078 0.085	291 L/ha 318 L/ha	61 67 -71	14 pea 21 pea (BBCH 79) 30 pea 43 pea	< 0.01 < 0.01 < 0.01 < 0.01	04-0422
Montbellet, France (North) 2004 Atos	80 g/L SC	Foliar	0.080 0.080	301 L/ha 299 L/ha	63 75	0 pea 7 pea (BBCH 75)	0.01 < 0.01	04-0422

Location/ Year/ Variety	Application					PHI (days)	Cypro-conazole (mg/kg)	Study/ Report
	Formulation	Method	Rate (kg ai/ha)	Rate (kg ai/hL)	Growth Stage (BBCH)			
						14 pea 21 pea 30 pea	< 0.01 < 0.01 < 0.01	
Epennes, France (South) 2004 NC	80 g/L SC	Foliar	0.080 0.081	300 L/ha 304 L/ha	61 72	0 pea 7 pea (BBCH 77) 14 pea 21 pea 29 pea	0.01 0.01 0.01 0.01 0.01	04-0421
Finhan, France (South) 2004 Austin	80 g/L	Foliar	0.080 0.080	300 L/ha 301 L/ha	61 69 – 71	7 pea (BBCH 76) 14 pea 21 pea 30 pea	< 0.01 < 0.01 < 0.01 < 0.01	04-0421
Meauzac, France (South) 2004 Bacara	80 g/L SC	Foliar	0.080 0.080	300 L/hg 299 L/ha	61 67 – 71	7 pea (BBCH 75) 14 pea 21 pea 30 pea	< 0.01 < 0.01 < 0.01 < 0.01	04-0421

A total of 29 supervised residue trials were conducted on dry peas and dry beans between 1993 and 2006. The trials were conducted on outdoor crops, twenty-one in Northern Europe and eight in Southern Europe.

Table 74 Pulses (Dry Peas and Beans) Trials

Location/ Year/ Variety	Application					PHI (days)	Cypro-conazole (mg/kg)	Study/ Report
	Formulation	Method	Rate (kg ai/ha)	Rate (kg ai/hL) or Volume (L/ha)	Growth Stage (BBCH)			
Pea								
Mesnil la Comtesse, France (North) 1993 Solara (field pea)	240 g/L SL	Foliar	0.076 0.080	385 406	61 69	29 37	< 0.01 < 0.01	R93003F/ BS 9888
Viapre le Petit, France (North) 1993 Therise (field pea)	240 g/L SL	Foliar	0.079 0.077	400 390	61 69	28 37	< 0.01 < 0.01	R93003F BS 9888
Chalons, France (North) 1993 Alex (field pea)	240 g/L SL	Foliar	0.078 0.075	396 380	61 69	29	< 0.01	R93003F/ BS 9888
Cuperly, France (North) 1993 Messire (field pea)	140 g/L SL	Foliar	0.080 0.075	404 380	61 69	29 38	< 0.01 < 0.01	R93003F/ BS 9888

Location/ Year/ Variety	Application					PHI (days)	Cypro-conazole (mg/kg)	Study/ Report
	Formulation	Method	Rate (kg ai/ha)	Rate (kg ai/hL) or Volume (L/ha)	Growth Stage (BBCH)			
St. Aubin, France (North) 1994 Nina (green pea)	40 g/L SC	Foliar	0.080 0.082	398 408	61 65 – 67	14 pod 21 pod 14 pea 21 pea	0.02 < 0.01 < 0.01 < 0.01	R10295/ BS 9427
Bonnencontre, France (North) 1994 Valette (green pea)	40 g/L SC	Foliar	0.076 0.080	382 400	61 65 – 67	14 pod 20 pod 14 pea 21 pea	0.02 0.02 < 0.01 < 0.01	R10295/ BS 9427
Penhoet Kevignac, France (North) 1994 Cador (green pea)	40 g/L SC	Foliar	0.081 0.076	405 382	60 – 61 66	21 pod 28 pod 21 pea 28 pea	< 0.01 < 0.01 < 0.01 < 0.01	R10295/ BS 9427
Penhoet Kevignac, France (North) 1994 Cador (green pea)	40 g/L SC	Foliar	0.084 0.084	420 420	61 67	21 pod 28 pod 21 pea 28 pea	< 0.01 < 0.01 < 0.01 < 0.01	R10295/ BS 9427
Rouvres St Jean, France (North) 2004 Canyon (dry pea)	80 g/L SC	Foliar	0.081 0.080	303 301	71 75	Pea: 0 7 14 21 30	 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	04-0422
Montbellet, France (North) 2004 Atos (dry pea)	80 g/L SC	Foliar	0.080 0.080	301 299	63 75	Pea: 0 7 14 21 30	 0.01 < 0.01 < 0.01 < 0.01 < 0.01	04-0422
Epennes, France (South) 2004 NC (farm seed)	80 g/L SC	Foliar	0.080 0.081	300 304	61 72	Pea: 0 7 14 21 29	 0.01 0.01 0.01 0.01 0.01	04-0421
Biziat, France (South) 2004 Atos (dry pea)	80 g/L SC	Foliar	0.081 0.079	304 297	67 77	Pea: 0 7 14 21 30	 0.01 0.01 0.03 0.01 0.01	04-9421
Finhan, France (South) 2004 Austin (dry pea)	80 g/L SC	Foliar	0.080 0.080	300 301	61 69 -71	Pea: 7 14 21 30	 < 0.01 < 0.01 < 0.01 < 0.01	04-0421
Meauzac, France (South) 2004 Bacara (dry pea)	80 g/L SC	Foliar	0.080 0.080	300 299	61 67 – 71	Pea: 7 14 21 30	 < 0.01 < 0.01 < 0.01 < 0.01	04-0421

Location/ Year/ Variety	Application					PHI (days)	Cypro-conazole (mg/kg)	Study/ Report
	Formulation	Method	Rate (kg ai/ha)	Rate (kg ai/hL) or Volume (L/ha)	Growth Stage (BBCH)			
Whatton, Nottinghamshire, UK (North) 1994 Baccara (combining pea)	40 g/L SC	Foliar	0.080 0.080	200 200	Knott 203 Knott 207	27 pea	< 0.02	BS 5558
Woodhall Spa, Lincolnshire, UK (North) 1994	40 g/L SC	Foliar	0.080 0.080	200 200	Knott 203 Knott 205/6	26 pea	< 0.02	BS 5558
Goole, N Humberside, UK (North) 1994 Solara (combining pea)	40 g/L SC	Foliar	0.080 0.080	200 200	Knott 203 Knott 206	30 pea	< 0.02	BS 6668
Chatteris, Cambridgeshire, UK (North) 1994 Bacchus (combining pea)	40 g/L SC	Foliar	0.080 0.080	200 200	Knott 203 Knott 205/6	26 pea	< 0.02	BS 5558
Hinton, Worcester, UK (North) 1994 Baroness (combining pea)	40 g/L SC	Foliar	0.080 0.080	200 200	Knott 203/4 Knott 206	17 pea	< 0.02	BS 5558
Birkin, UK (North) 2004 Solara (dry pea)	80 g/L SC	Foliar	0.082 0.082	306 308	61 – 63 69 – 73	0 plant Pea: 7 14 21 30 43	1.40 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	04-0422
Hummanby, Yorkshire, UK (North) 2004 Samson (dry pea)	80 g/L SC	Foliar	0.078 0.085	291 318	61 67 – 71	Pea: 14 21 30 43	 < 0.01 < 0.01 < 0.01 < 0.01	04-0422
Bean								
Boce, France (North) 2006 Castel (dry bean)	80 g/L SC	Foliar	0.053 0.080	197 299	62 87	26 bean	< 0.01	05-0415
Monteux, France (South) 2005 Big Borloto (dry bean)	80 g/L SC	Foliar	0.083 0.081	208 203	69 – 71 75 – 77	30 bean	0.01	05-0607
Grisolles, France (South) 2005 Linex (dry bean)	80 g/L SC	Foliar	0.079 0.079	200 200	62 76	31 bean	0.02	05-0607
Estillac, France (South)	80 g/L SC	Foliar	0.087 0.083	200 200	64 73 – 75	29 bean	0.05	05-0607

Location/ Year/ Variety	Application					PHI (days)	Cypro-conazole (mg/kg)	Study/ Report
	Formulation	Method	Rate (kg ai/ha)	Rate (kg ai/hL) or Volume (L/ha)	Growth Stage (BBCH)			
2005 Linex (dry bean)								
Marsillargues, France (South) 2005 Linex (dry bean)	80 g/L SC	Foliar	0.080 0.080	300 300	61 67	28 bean	0.01	05-0607
Horston, UK (North) 2005 Clipper (dry bean)	80 g/L SC	Foliar	0.081 0.081	303 302	61 70	30 bean	< 0.01	05-0415
Draycott, UK (North) 2005 Quattro (dry bean)	80 g/L SC	Foliar	0.082 0.083	308 310	61 79	Bean: 30 36	 < 0.01 < 0.01	05-0415
Woodhouse Eves, UK (North) 2006 Castel (dry bean)	80 g/L SC	Foliar	0.080 0.081	300 303	59 – 62 78 – 82	30 bean	< 0.01	T014143-05/ T014143-05-REG

Root and tuber vegetables

Sugar beet

A total of thirty-two supervised residue trials were conducted on sugar beet between 1986 and 2002. The trials were conducted on outdoor crops, sixteen in Northern Europe and sixteen in Southern Europe.

Table 75 Sugar Beet (Roots) Field Trials

Location/ Year/ Variety	Application					PHI (days)	Cypro-conazole (mg/kg)	Study/ Report
	Formulation	Method	Rate (kg ai/ha)	Rate (kg ai/hL) or Volume (L/ha)	Growth Stage (BBCH)			
Wigoltingen, Switzerland (North) 1986 Kadutschka	? WG	Foliar	0.080 0.080	500 500	-	0 15 28 36	0.02 0.02 0.02 0.01	R8836
Wigoltingen, Switzerland (North) 1986	40 g/L SC	Foliar	0.060 0.060	500 500	-	0 15 28 36	0.03 0.02 0.01 0.02	R8834
Portes Conches en Ouche, France (North) 1987 Allyx	32 g/kg WG	Foliar	0.064 0.064	400 400	-	0 17 31 37	0.01 0.01 0.01 < 0.01	R9141
Chepoix, France (North) 1988 Montpeso	40 g/L SC	Foliar	0.060 0.060	400 400	-	0 14 30	0.06 0.02 < 0.01	811072/ R 9335
Chepoix, France (North)	40 g/L SC	Foliar	0.060 0.060	400 400	-	29	< 0.01	811072/ R 9336

Location/ Year/ Variety	Application					PHI (days)	Cypro-conazole (mg/kg)	Study/ Report
	Formulation	Method	Rate (kg ai/ha)	Rate (kg ai/hL) or Volume (L/ha)	Growth Stage (BBCH)			
1988 Montpeso								
Livery-Louvercy, France (North) 1987 Allyx	100 g/L SL	Foliar	0.060 0.060	-	-	0 14 26 48	< 0.01 < 0.01 < 0.01 < 0.01	R9030
Bucy le Long, France (North) 1987 Gala	? WG	Foliar	0.060 0.060	400 400	-	0 13 28 42	< 0.01 0.01 0.01 < 0.01	R9033
Guerbigny, France (North) 1988 Allyx	40 g/L SC	Foliar	0.060 0.060	400 400	-	0 16 29 49	0.02 0.02 < 0.01 < 0.01	R9334/ 811071
Marsillargues, France (South) 1999 Nevada	80 g/L EC	Foliar	0.056 0.056 0.056	400 400 400	39 39 30	0 22 30 43	< 0.02 < 0.02 < 0.02 < 0.02	9911001
Escatalens, France (South) 1999 Nevada	80 g/L EC	Foliar	0.056 0.056 0.056	300 300 300	19 31 39	0 21 30 45	0.02 0.02 0.02 0.02	9911002
Roquecourbe, France (South) 2000 Agora	80 g/L EC	Foliar	0.055 0.055 0.054	975 983 967	31 38 – 39 39 – 49	0 20	0.03 < 0.02	NOV/RES/00061
Mauguio, France (South) 2000 Agora	80 g/L EC	Foliar	0.057 0.059 0.054	408 423 388	39 39 39	0 20	0.03 < 0.02	NOV/RES/00062
Magdeburg, Sachsen-Anhalt, Germany (North) 1993	100g /L SL	Foliar	0.081 0.076	202 189	- 48	0 14 21 28 35 65	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	BS-5177
Burgwedel-Thonse, Niedersachsen, Germany (North) 1993	100 g/L SL	Foliar	0.080 0.081	200 202	- 47	0 14 21 28 35	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	
Hilgermissen, Niedersachsen, Germany (North) 1993	100 g/L SL	Foliar	0.082 0.082	204 204	- 47	0 14 21 28 35 52	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	BS-5177
Pforzheim, Baden-Wurttenberg, Germany (North) 1993 Hilma	100 g/L SL	Foliar	0.083 0.082	206 204	- 41 – 42	0 14 21 28 35 69	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	BS-5177
Coldham, Cambridgeshire, UK (North) 1995	100 g/L SL	Foliar	0.060 0.060	250 250	26 >30	0 3 7 10	< 0.02 < 0.02 < 0.02 < 0.02	R95-033/ BS 7525

Location/ Year/ Variety	Application					PHI (days)	Cypro-conazole (mg/kg)	Study/ Report
	Formulation	Method	Rate (kg ai/ha)	Rate (kg ai/hL) or Volume (L/ha)	Growth Stage (BBCH)			
Jackpot						14	< 0.02	
Bury, St. Edmonds, UK (North) 1995 Aztec	100 g/L SL	Foliar	0.060 0.060	250 250	-	0 3 7 10 14	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	R95-033/ BS 7525
Bramston, Liemeorm, UK (North) 1995 Druid	100 g/L SL	Foliar	0.060 0.060	250 250	26 29-35	0 14	< 0.02 < 0.02	R95-033/ BS 7525
Bury, St. Edmunds, UK (North) Zulu	100 g/L SL	Foliar	0.060 0.060	250 250	15 15-40	0 14	< 0.02 < 0.02	R95-033/ BS 7525
Marmorta, Italy (South) 1987 Monohill	100 g/L SL	Foliar	0.080 0.080 0.080	800 800 800	-	0 14 28	0.04 0.01 < 0.01	R9052
Molinella, Italy (South) 1987 Rizor	100 g/L SL	Foliar	0.080 0.080 0.080	800 800 800	-	0 14 28	0.02 0.02 < 0.01	R9053
Portonovo, Italy (South) 1987 Monofort	100 g/L SL	Foliar	0.080 0.080 0.080	800 800 800	-	27	0.02	R9054
Filo, Italy (South) 1987 Monofort	100 g/L SL	Foliar	0.080 0.080 0.080	800 800 800	-	27	0.02	R9055
Bagnarola di Budrio, Italy (South) 2002 Extra Brio Saros	80 g/L SC	Foliar	0.076 0.074	475 463	44 45	21 28	0.04 0.01	0224R/25R
Pesaro, Italy (South) 2002 Fiamma	80 g/L SC	Foliar	0.082 0.081	515 607	43 45	21 28	0.03 0.01	0224R/25R
Marano di Castenaso, Italy (South) 2002 Sucrosaros	80 g/L SC	Foliar	0.085 0.082	529 511	39 45	21 28	< 0.01 < 0.01	AGRI 004/03
S. Petro in Casale, Bologna, Italy (South) 2002 Gea	80 g/L SC	Foliar	0.087 0.081	543 509	39 46	21 28	0.02 < 0.01	AGRI 004/03
Unita Periferica di Ronchi Di Villafrnaca-Pd, Italy (South) 1999 Azzurro	80 g/L EC	Foliar	0.056 0.056 0.056	600 600 600	45 47 48-49	0 7 14 21 28	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	2074/99
Unita Periferica di Serravalle a Po-Mantova, Italy (South)	80 g/L EC	Foliar	0.056 0.056 0.056	600 600 600	44-45 45-46 46-48	21	< 0.02	2075/99

Location/ Year/ Variety	Application					PHI (days)	Cypro-conazole (mg/kg)	Study/ Report
	Formulation	Method	Rate (kg ai/ha)	Rate (kg ai/hL) or Volume (L/ha)	Growth Stage (BBCH)			
1999 Puma								
Badajoz, Spain (South) 1999 Autopoly (Mezano)	80 g/L EC	Foliar	0.055 0.055 0.057	980 987 1016	42-43 44-46 49	21	< 0.02	2060/99
Valladolid, Spain (South) 1999 Cima	890 g/L EC	Foliar	0.057 0.055 0.056	1010 958 1008	33-39 39-43 43-47	0 7 14 21	< 0.02 < 0.02 < 0.02 < 0.02	2061/99

Cereal grains

A total of forty-seven supervised residue trials were conducted on wheat and rye between 1986 and 2001. The trials were conducted on outdoor crops, thirty-nine in Northern Europe and eight in Southern Europe. A total of forty-two supervised residue trials were conducted on barley between 1986 and 2001. The trials were conducted on outdoor crops, twenty-eight in Northern Europe and fourteen in Southern Europe. A total of twenty-two supervised residue trials on maize (corn) were conducted in fifteen states in the United States.

Table 76 Wheat Grain Field Trials

Location/ Year/ Variety	Application					PHI (days)	Cypro-conazole (mg/kg)	Study/ Report
	Formulation	Method	Rate (kg ai/ha)	Rate (kg ai/hL) or Volume (L/ha)	Growth Stage (BBCH)			
Cote-d Or, France (North) 1991 Recital	240 g/L SL	Foliar	0.10 0.082	405 412	61 – 69	55	< 0.01	91034M/5_7D/ BS 9358
Izy, France (North) 1997 Sideral	53 g/kg WG	Foliar	0.080 0.080	400 400	31-32 71-73	44	< 0.01	2210/97
Izy, France (North) 1997 Sideral	80 g/L EC	Foliar	0.080 0.080	400 400	32-32 71-73	44	< 0.01	2238/97
Izy, France (North) 1997 Sideral	160 g/L EC	Foliar	0.080 0.080	400 400	32-32 71-73	46	< 0.02	2201/97
Cadenac, France (South) 1986	100 g/L SL	Foliar	0.10 0.080	500 500	59	48	0.01	
Saintes, France (South) 1986 Talent	100 g/L SL	Foliar	0.10 0.080	400 400	59	48	< 0.01	CBK11719/86/ 8714
Saintes, France (South) 1987	100 g/L SL	Foliar	0.080 0.10	400 400	59	30 58	< 0.01 < 0.01	CBK II-1202/88/ 9004

Location/ Year/ Variety	Application					PHI (days)	Cypro-conazole (mg/kg)	Study/ Report
	Formulation	Method	Rate (kg ai/ha)	Rate (kg ai/hL) or Volume (L/ha)	Growth Stage (BBCH)			
Talent								
Mauguio, France (South) 1998 Arstar	45 g/kg WG	Foliar	0.082 0.083	411 418	51 65	47	< 0.02	9812901
Lansargues, France (South) 1998 Primadur	45 g/kg WG	Foliar	0.080 0.081	402 407	51 65	45	< 0.02	9812902
Mauguio, France (South) 1998	80 g/L EC	Foliar	0.073 0.084	367 418	32 65	37 47	< 0.02 < 0.02	9813103
Lansargues, France (South) 1998 Primadur	80 g/L EC	Foliar	0.082 0.084	409 422	32 65	36 45	< 0.02 < 0.02	9813104
Marsanne, France (South) 2001	80 g/L SC	Foliar	0.080 0.080	300 300	33 65	42	< 0.02	0112902
Rodinghausen, Germany (North) 1996 Pegasos	100 g/L SL	Foliar	0.10 0.10	400 400	49 61	35 54	0.05 < 0.01	R96-007
Rodinghausen, Germany (North) 1996 Pegasos	240 g/L EC	Foliar	0.096 0.096	400 400	49 61	35 54	0.04 < 0.01	R96-007
Rodinghausen, Germany (North) 1996 Pegasos	160 k/kg WG	Foliar	0.096 0.096	400 400	49 61	35 54	0.04 < 0.01	R96-007
Sandelsbronn, Germany (North) 1996 Contra	100 g/L SL	Foliar	0.10 0.10	300 300	49 61	35 65	0.01 < 0.01	R96-007
Sandelsbronn, Germany (North) 1996 Contra	240 g/L EC	Foliar	0.096 0.096	300 300	49 61	35 65	0.01 < 0.01	R96-007
Sandelsbronn, Germany (North) 1996 Contra	160 g/kg WG	Foliar	0.10 0.10	300 300	49 61	35 65	< 0.01 < 0.01	R96-007
Axien, Germany (North) 2001 Aristos	80 g/L SC	Foliar	0.080 0.080	300 300	32-33 69	35 50	< 0.01 < 0.01	gwh 32401
Gross-Niendorf, Germany (North) 2001	80 g/L SC	Foliar	0.080 0.080	300 300	31-32 69-73	42	< 0.01	gwh92401
Mehlbek, Germany (North) 1991 Kraka	100 g/L SL	Foliar	0.082 0.11 0.10	307 106 103	31 49 61	37	0.02	R10176/ BS 2781
Hilgermissen, Germany (North) 1991 Orestis	100 g/L SL	Foliar	0.078 0.093 0.10	300 280 320	31-32 52 61	35 43	0.05 0.04	R10177/ BS 2158

Location/ Year/ Variety	Application					PHI (days)	Cypro-conazole (mg/kg)	Study/ Report
	Formulation	Method	Rate (kg ai/ha)	Rate (kg ai/hL) or Volume (L/ha)	Growth Stage (BBCH)			
Upstedt, Germany (North) 1991 Ibis	100 g/L SL	Foliar	0.089 0.088 0.091	330 270 290	31 49 61	35 38	0.02 0.05	R10178/ BS 2767
Aarbergen-Panrod, Germany (North) 1991 Orestis	100 g/L SL	Foliar	0.079 0.10 0.10	400 100 100	31 49 61	35 42 50	< 0.01 0.02 < 0.01	R10178/ BS 2767
Riedstadt-Crumstadt, Germany (North) 1991 Bert	100 g/L SL	Foliar	0.082 0.10 0.11	410 410 430	31 49 61	53	< 0.01	R10180/ BS 2787
Niddatal-Ilbenstadt, Germany (North) 1991 Kanzler	100 g/L SL	Foliar	0.075 0.10 0.10	380 410 400	31 49 61	42 48	< 0.01 0.02	R10181/ BS 2772
Hilgermissen, Germany (North) 1992 Kontrast	100 g/L SL	Foliar	0.088 0.11 0.11	440 440 440	37 52 69	35 42	0.01 0.02	R10249/ BS 3641
Hilgermissen, Germany (North) 1992 Kontrast	40 g/L SC	Foliar	0.081 0.087 0.080	410 430 400	37 52 69	35 42	< 0.01 0.01	R10249/ BS 3641
Jemstorf-Lute, Germany (North) 1992 Orestis	100 g/L SL	Foliar	0.088 0.11 0.097	440 430 390	37 55 61	35 42	< 0.01 < 0.01	R10249/ BS 3641
Jemstorf-Lute, Germany (North) 1992 Orestis	40 g/L SC	Foliar	0.081 0.080 0.080	400 400 400	37 55 61	35 42	< 0.01 < 0.01	R10249/ BS 3641
Bockenem 13, Germany (North) 1992 Astron	100 g/L SL	Foliar	0.084 0.093 0.088	420 370 350	36-37 51 61	36 42	0.02 < 0.01	R10249/ BS 3641
Bockenem 13, Germany (North) 1992 Astron	40 g/L SC	Foliar	0.084 0.078 0.076	420 390 380	36-37 51 61	36 42	< 0.01 < 0.01	R10249/ BS 3641
Goch-Nierswalde, Germany (North) 1992 Orestis	100 g/L SL	Foliar	0.078 0.097 0.10	390 390 410	37 49-51 61	35 42	< 0.01 0.01	R10249/ BS 3641
Nierswalde, Germany (North) 1992 Orestis	40 g/L SC	Foliar	0.074 0.081 0.083	370 400 410	37 49-51 61	35 42	< 0.01 < 0.01	R10249/ BS 3641
Dettelbach-Schernau, Germany (North) 1992 Agronom	100 g/L SL	Foliar	0.080 0.10 0.10	300 300 300	38 49 62	35 42	0.02 < 0.01	R10249/ BS 3641
Dettelbach-Schernau,	40 g/L SC	Foliar	0.080 0.080	300 300	38 49	35 42	< 0.01 < 0.01	R10249/ BS 3641

Location/ Year/ Variety	Application					PHI (days)	Cypro-conazole (mg/kg)	Study/ Report
	Formulation	Method	Rate (kg ai/ha)	Rate (kg ai/hL) or Volume (L/ha)	Growth Stage (BBCH)			
Germany (North) 1992 Agronom			0.080	300	62			
Hirblingen, Germany (North) 1992 Ares	100 g/L SL	Foliar	0.080 0.10 0.10	300 300 300	39 53 61	34 41	0.05 0.02	R10249/ BS 3641
Hirblingen, Germany (North) 1992 Ares	40 g/L SC	Foliar	0.080 0.080 0.080	300 300 300	39 53 61	34 41	0.02 < 0.01	R10249/ BS 3641
Bex/VD, Switzerland (North) 2001 Albis	80 g/L SC	Foliar	0.080 0.080	400 400	37 69	35 47	< 0.02 < 0.02	2075/01
	80 g/L SC	Foliar	0.080 0.080	400 400	37 69	35 47	< 0.02 < 0.02	2075/01
Vouvry/VS, Switzerland (North) 2001 Galaxy	80 g/L SC	Foliar	0.080 0.080	400 400	33 69	35 47	< 0.02 < 0.02	2076/01
	80 g/L SC	Foliar	0.080 0.080	400 400	33 69	35 47	< 0.02 < 0.02	2076/01

Table 77 Rye Field Trials

Location/ Year/ Variety	Application					PHI (days)	Cypro-conazole (mg/kg)	Study/ Report
	Formulation	Method	Rate (kg ai/ha)	Rate (kg ai/hL) or Volume (L/ha)	Growth Stage (BBCH)			
Hilgermissen, Germany (North) 1992 Rapid	100 g/L SL	Foliar	1.1 1.1	460 420	54 64	35 41	0.20[a] 0.01	R10250/ BS 4112
Braunschweig-Hondelage, Germany (North) 1992 Ammando Hybrid	100 g/L SL	Foliar	0.10 0.089	400 360	54 61	35 42	0.01 0.01	R10250/ BS 4112
Goch-Kessel, Germany (North) 1992 Halo	100 g/L SL	Foliar	0.11 0.98	440 390	37-51 61	40	0.03	R10250/ BS 4112
Ivenrode, Germany (North) 1998 Hacada	23 g/kg WG	Foliar	0.080 0.080	400 400	32 69-71	49 57	< 0.02 < 0.02	gr 39298
Wallersdorf, Germany (North) 1998 Borellus	53 g/kg WG	Foliar	0.080 0.080	400 400	31-32 71	49 56	< 0.02 < 0.02	gr 40498

[a] The 35 day PHI was at BBCH 83, which is early dough and not a mature harvest stage. PHI 41 is acceptable because the last application was made around BBCH 61 per the GAP.

Table 78 Barley Field Trials

Location/ Year/ Variety	Application					PHI (days)	Cypro-conazole (mg/kg)	Study/ Report
	Formulation	Method	Rate (kg ai/ha)	Rate (kg ai/hL) or Volume (L/ha)	Growth Stage (BBCH)			
Pinson, France (North) 1986 Illia	100 g/L SL	Foliar	0.10 0.10	100 100	-	44	0.02	R8730
	100 g/L SL	Foliar	0.10 0.10	100 100	-	44	0.03	R8730
Montalzat, France (South) 1997 Sonja	45.3 g/kg WG	Foliar	0.080 0.080	400 400	32 71	43	< 0.01	2186/97
Realville, France (South) 1997 Baraka	45.3 g/kg WG	Foliar	0.080 0.080	400 400	31-32 71	45	0.03	2187/97
	45.3 g/kg WG	Foliar	0.080 0.080	400 400	32 69	45	< 0.01	2187/97
Lansargues, France (South) 1997 Alpha	45.3 g/kg WG	Foliar	0.080 0.080	400 400	37 69	42	0.05	2189/97
Montalzat, France (South) 1997 Sonja	80 g/L EC	Foliar	0.080 0.080	400 400	32 71	43	< 0.01	2228/97
Realville, France (South) 1997 Baraka	80 g/L EC	Foliar	0.080 0.080	400 400	31-32 71	45	0.03	2228/97
Divajeu, France (South) 1997 Orelie	80 g/L EC	Foliar	0.080 0.080	400 400	43 71	45	0.01	2229/97
Lasargues, France (South) 1997 Alpha	80 g/L EC	Foliar	0.080 0.080	400 400	37 69	45	0.04	2231/97
Dange, France (South) 2001 Ludine	160 g/L EC	Foliar	0.079 0.078	390 390	31 69	41	0.04	0110901
Ingrandes, France (South) 2001	160 g/L EC	Foliar	0.073 0.083	370 410	32 69	41	0.04	0110902
Cayrac, France (South) 2001 Sonja	80 g/L SC	Foliar	0.080 0.080	400 400	33 69-71	42	< 0.02	0112701
	80 g/L SC	Foliar	0.080 0.080	400 400	33 69-71	42	< 0.02	0113201
Marsillargues, France (South) 2001 Baraka	80 g/L SC	Foliar	0.080 0.080	400 400	34 71-73	42	0.05	0112702

Location/ Year/ Variety	Application					PHI (days)	Cypro-conazole (mg/kg)	Study/ Report
	Formulation	Method	Rate (kg ai/ha)	Rate (kg ai/hL) or Volume (L/ha)	Growth Stage (BBCH)			
	80 g/L SC	Foliar	0.080 0.080	400 400	32-33 69-71	42	0.03	0113202
Denges/VS, Switzerland (North) 1986	100 g/l SL	Foliar	0.10 0.10	1000 1000	61	42	0.04	R8811
Hugelshofen/TG, Switzerland (North) 1986 Gerbel	100 g/L SL	Foliar	0.10 0.10	500 500	59	42	0.03	R8812
Hugelshofen/TG, Switzerland (North) 1986 Gerbel	400 g/L SL	Foliar	0.10 0.10	500 500	59	42	0.03	R8814
Bottens/VS, Switzerland (North) 1987 Gerbel	80 g/L EC	Foliar	0.060 0.080 0.080	500 500 500	61	35 42	0.02 0.02	R9136
Ottoberg/TG, Switzerland (North) 1987 Mammut	80 g/L EC	Foliar	0.060 0.060 0.060	500 500 500	-	34 41	0.04 0.03	R9137
Burgwedel-Thonse, Germany (North) 1992 Catinka	100 g/L SL	Foliar	0.11 0.10	460 410	58-59 63	35 42	0.07 0.05	R10250/ TDS BS4112
Anhausen (Augsburg), Germany (North) 1992 Loreley	100 g/L SL	Foliar	0.10 0.10	300 300	55 62	35 42	0.02 0.01	R10250/ TDS BS4112
Ostkilver, Germany (North) 1996 Loreley	100 g/L SL	Foliar	0.095 0.095	400 400	59 61-63	35 48	0.04 0.02	R96-007
Ostkilver, Germany (North) 1996 Loreley	240 g/L EC	Foliar	0.097 0.097	400 400	59 61-63	35 48	0.04 0.03	R96-007
Ostkilver, Germany (North) 1996 Loreley	160 g/kg WG	Foliar	0.097 0.097	400 400	59 61-63	35 48	0.03 0.02	R96-007
Sandelsbronn, Germany (North) 1996 Astrid	100 g/L SL	Foliar	0.10 0.096	400 400	55-59 61	35 54	0.02 < 0.01	R96-007
Sandelsbronn, Germany (North) 1996 Astrid	240 g/L EC	Foliar	0.094 0.091	400 400	55-59 61	35 54	0.02 < 0.01	R96-007
Sandelsbronn, Germany (North) 1996	160 g/kg WG	Foliar	0.090 0.090	400 400	55-59 61	35 54	0.01 < 0.01	R96-007

Location/ Year/ Variety	Application					PHI (days)	Cypro-conazole (mg/kg)	Study/ Report
	Formulation	Method	Rate (kg ai/ha)	Rate (kg ai/hL) or Volume (L/ha)	Growth Stage (BBCH)			
Astrid								
Ostkilver, Germany (North) 1996 Loreley	2 × 0.080 g/L SC 1 × 100 g/L SL	Foliar	0.10 0.097 0.077	410 390 390	32 49 61	35 49	0.02 0.01	R96-022
Leiston, UK (North) 1987 Pipkin	100 g/L SL	Foliar	0.080 0.080 0.080	200 200 200	 60	69	0.02	R9237
Sibton, UK (North) 1987 Igri	100 g/L SL	Foliar	0.080 0.080 0.080	200 200 200	 61	70	0.04	R9240
Duns Tew, Oxon., UK (North) 1991 Igri	100 g/L SL	Foliar	0.080 0.080 0.080	200 200 200	 31 61	?	< 0.01	BS 5553
Bucknell, Oxon. , UK (North) 1991 Plaisant	100 g/L SL	Foliar	0.080 0.080 0.080	200 200 200	13-14 32 61-65	51	0.01	SDZ 0292/2/ BS 5553
Dalderby, Hornsastle, Lincs., UK (North) 1991 Puffin	100 g/L SL	Foliar	0.080 0.080 0.080	200 200 200	12-13 31 61	59	0.01	SDA 0292/2/ BS 5553
Chudleigh, Devon., UK (north) 1992 Frolic	100 g/L SL	Foliar	0.080 0.080 0.080	200 200 200	Z12 Z31 Z59	53	0.01	SDZ 0692/ BS 5573
Aynho, Oxon., UK (North) 1992 Gypsy	100 g/L SL	Foliar	0.080 0.080 0.080	200 200 200	Z23 Z31 Z55-59	54	< 0.01	SDZ 0692/ BS 5573
Newton St. Cyres, Devon., UK (North) 1992 Fighter	100 g/L SL	Foliar	0.080 0.080 0.080	200 200 200	Z13 Z31 Z59	52	< 0.01	SDZ 0692/ BS 5573
Haltham, Lincs., UK (North) 1992 Pipkin	100 g/L SL	Foliar	0.080 0.080 0.080	200 200 200	Z13-21 Z31 Z59-60	42	0.02	SDZ 0692/ BS 5573
St. Madoes, Scotland, UK (North) 1992 Plaisant	100 g/L SL	Foliar	0.080 0.080 0.080	200 200 200	Z24 Z31 Z59-65	52	0.01	SDZ 0692/ BS 5573

Maize (Field corn)

A total of twenty-two supervised residue trials were conducted in fifteen states in the United States

Table 79 Maize Field Trials

Location/ Year/ Variety	Application					PHI (days)	Cypro-conazole (mg/kg)	Study/ Report
	Formulation	Method	Rate (kg ai/ha)	Rate (kg ai/hL) or Volume (L/ha)	Growth Stage (BBCH)			
Hudson, NY, USA (Region 1) 2004 DKC53-54RR	100 g/L SC	foliar	0.040 0.041	20 20	75 988-93	30	< 0.01	T002814-03
Rose Hill, NC, USA (Region 2) 2004 Pioneer 34B97	100 g/l SC	Foliar	0.040 0.040	230 150	R2 R5-R6	30	< 0.01	T002814-03
Richlans, IA, USA (Region 5) 2004 Golden Harvest H-9247 BT	100 g/L SC	Foliar	0.040 0.041	180 160	R3-R4 R5	7 14 21 30 37	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	T002814-03
	100 g/L SC	Foliar	0.040 0.040	160 160	R3 R5	30	< 0.01	T002814-03
Chatsworth, IA, USA (Region 5) 2004 Midwest 7X088	100 g/L SC	Foliar	0.040 0.040	140 140	R3 R5	30	< 0.01	T002814-03
Searsville, IA, USA (Region 5) 2004 DKC 5145	100 g/L SC	Foliar	0.040 0.041	150 150	R4 R5	30	< 0.01	T002814-03
Champaign, Il, USA (Region 5) 2004 Pioneer 34H31	100 g/L SC	Foliar	0.041 0.043	100 140	- 91	7 14 21 30 37	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	T002814-03
Carlyle, Il, USA (Region 5) 2004	100 g/L SC	Foliar	0.041 0.040	170 91	75 79	30	< 0.01	T002814-03
Chemung Township/ Harvard, WI, USA (region 5) 2004 Hughes 5172RR	100 g/L SC	Foliar	0.041 0.039	170 160	75 79	30	< 0.01	T002814-03
York, NE, USA (Region 5) 2004 DKC60-19	100 g/L, SC	Foliar	0.040 0.040	190 150	71 86	30	< 0.01	T002814-03
Osceola, NE, USA (Region 5) 2004 DKC60-19	100 g/L SC	Foliar	0.040 0.040	190 190	69 88	30	< 0.01	T002814-03
Geneva, MN, USA (Region 5) 2004 Pioneer 36N18	100 g/L SC	Foliar	0.040 0.040	150 150	R4 R5	30	< 0.01	T002814-03
Paynesville, MN, USA (Region 5) 2004 Pioneer 36B08	100 g/L SC	Foliar	0.041 0.040	190 190	85 85	30	< 0.01	T002814-03
Noblesville, IN, USA (Region 5)	100 g/L SC	Foliar	0.041 0.041	130 130	R3 R5-R6	30	< 0.01	T002814-03

Location/ Year/ Variety	Application					PHI (days)	Cypro-conazole (mg/kg)	Study/ Report
	Formulation	Method	Rate (kg ai/ha)	Rate (kg ai/hL) or Volume (L/ha)	Growth Stage (BBCH)			
2004 5322								
New Holland, OH, USA (Region 5) 2004 8627R	100 g/L SC	Foliar	0.041 0.042	200 150	Early dough R6	30	< 0.01	T002814-03
Centerville, SD, USA (Region 5) 2004 Dairyland Stealth 1608	100 g/L SC	Foliar	0.041 0.040	130 140	75 99	30	< 0.01	T002814-03
Fitchburg, WI, USA (Region 5) 2004 Pioneer 38H67	100 g/L SC	Foliar	0.041 0.040	280 280	86 86	30	< 0.01	T002814-03
LaPlata, MO, USA (Region 5) 2004 LG 2540	100 g/L SC	Foliar	0.041 0.040	160 160	77 R4-R5	30	< 0.01	T002814-03
Conklin, MI USA (Region 5) 2004 36B92	100 g/L SC	Foliar	0.040 0.040	200 190	R2 R5	7 14 21 30 37	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	T002814-03
Highland, KS, USA (Region 5) 2004 Pioneer 32P75	100 g/L SC	Foliar	0.040 0.040	130 94	R2 R5	30	< 0.01	T002814-03
Clay, TX, USA (Region 6) 2004 DKC-66-80	100 g/L EC	Foliar	0.040 0.040	19 19	- 85	30	< 0.01	T002814-03
Madera, CA, USA (Region 10) 2004	100 g/L SC	Foliar	0.04 0.041	280 280	Formed ears Ears with silk	30	< 0.01	T002814-03

Oilseeds

Rape seed (Canola)

A total of thirteen supervised residue trials were conducted on oilseed rape during 2004 and 2005. The trials were conducted on outdoor crops, nine in Northern Europe and four in Southern Europe.

Table 80 Rape Seed Field Trials

Location/ Year/ Variety	Application					PHI (days)	Cypro-conazole (mg/kg)	Study/ Report
	Formulation	Method	Rate (kg ai/ha)	Rate (kg ai/hL) or Volume (L/ha)	Growth Stage (BBCH)			
Vouvry/VS, Switzerland (North) 2004 Panther electra	80 g/L SC	Foliar	0.082 0.080	610 600	69 79-80	29	0.05	04-0312
La Moriniere, Nouzilly, France (North) 2004 Saturnin	80 g/L SC	Foliar	0.080 0.082	310 320	77 79-80	32	0.23	04-0414
Prunay, France (North) 2005 Aviso	80 g/L SC	Foliar	0.077 0.081	300 300	67 80	30	0.04	05-0409
La Chapelle de Guinchay, France (North) 2005 Hearty	80 g/L SC	Foliar	0.074 0.081	280 310	61-63 78-80	33	0.03	05-0410
La Chapelle de, France (North) 2005 Standing	80 g/L SC	Foliar	0.081 0.083	310 310	65 79-80	34	0.03	05-0410
Beine Nauroy, France (North) 2005 Aviso	80 g/L SC	Foliar	0.083 0.083	300 300	69 80	30	0.03	05-0409
La Sence Septoutre, France (North) 2005 Savana	30 g/L SC	Foliar	0.082 0.080	300 300	69 78	30	0.08	05-0409
Platia, Monferran-Saves, France (South) 2004	80 g/L SC	Foliar	0.080 0.082	310 320	79 83	28	0.09	04-0413
Taize, France (South) 2004 Saturnin	80 g/L SC	Foliar	0.083 0.084	320 320	77 79-83	31	0.21	04-0413
Monferran-Saves, France (South) 2005 Standing	80 g/L SC	Foliar	0.080 0.085	300 320	67 80	30	0.10	05-0310
Monferran-Saves, France (South) 2005 Oliris	80 g/L SC	Foliar	0.081 0.074	310 280	67 80	30	0.08	05-0310
Niederstriegis-Littdorf, Germany (North) 2004 Aviso	80 g/L SC	Foliar	0.080 0.080	300 300	67 80	37	0.01	gra230004

Location/ Year/ Variety	Application					PHI (days)	Cypro-conazole (mg/kg)	Study/ Report
	Formulation	Method	Rate (kg ai/ha)	Rate (kg ai/hL) or Volume (L/ha)	Growth Stage (BBCH)			
Wessin Germany (North) 2004 Smart	80 g/L SC	Foliar	0.080 0.080	300 300	67 78-80	49	< 0.01	gra230004

Soya bean

Twenty trials were conducted in the United States for outdoor use on soya beans. Seeds (mature dry), hay, and forage samples were analysed.

Table 81 Field Trials for Soya bean Seed in the USA

Location/ Year/ Variety	Application					PHI (days)	Cypro-conazole (mg/kg)	Study/ Report
	Formulation	Method	Rate (kg ai/ha)	Rate (kg ai/hL) or Volume (L/ha)	Growth Stage (BBCH)			
Champaign, IL, USA (Region 5) 2003 Golden Harvest	100 g/l SC	Foliar	0.040 0.043	160 160	79 89	28	< 0.02	T001901-03
Attica, IN, USA (region 5) 2003 Becks's 306 100 g/L SC	100 g/L SC	Foliar	0.041 0.039	160 150	79 89	27	< 0.02	T0001901-03
Leland, MS, USA (Region 4) 2003 NKX248R	100 g/L SC	Foliar	0.042 0.040	190 170	77 85	31	0.05	T0001901-03
Kingston, NC, USA (Region 2) 2004 S57-A4	100 g/L SC	Foliar	0.042 0.040	150 140	Pod fill Pod fill	16 23 30 37	0.01 0.01 0.01 0.01	T002037-03
Elko, SC, USA (Region 2) 2004 S73-Z5	100 g/L SC	Foliar	0.040 0.040	26 26	77 79	30	< 0.01	T002037-03
Proctor, AR, USA (Region 4) 2004 Pioneer 94B73RR	100 g/L SC	Foliar	0.040 0.040	140 140	69 R6	30	0.04	T002037-03
Richland, IA, USA (Region 5) 2004 Pioneer 93M80	100 g/L SC	Foliar	0.040 0.040	130 160	R5 R5-R6	9 16 23 30 37	0.01 0.02 0.02 0.02 0.02	T002037-03
Richland, IA, USA (Region 5) 2004 Pioneer 93M87	100 g/L SC	Foliar	0.039 0.040	130 130	R4 R5	30	0.03	T002037-03
Carlyle, IL, USA (Region 5)	100 /L SC	Foliar	0.042 0.042	130 99	69 R6	30	0.03	T002037-03

Location/ Year/ Variety	Application					PHI (days)	Cypro-conazole (mg/kg)	Study/ Report
	Formulation	Method	Rate (kg ai/ha)	Rate (kg ai/hL) or Volume (L/ha)	Growth Stage (BBCH)			
2004 B-T 383CR								
Geneva, MN, USA (region 5) 2004 Pioneer 91M50	100 g/L SC	Foliar	0.040 0.040	150 160	R5 R6	30	0.03	T002037-03
Noblesville, IN, USA (Region 5) 2004 387NRR	100 g/L SC	Foliar	0.041 0.041	130 130	R5-R6 R6-R7	30	< 0.01	T002037-03
Kirksville, MO, USA (Region 5) 2004 Asgrow 3302	100 g/L SC	Foliar	0.040 0.040	180 160	71 R5	30	0.02	T002037-03
York, NE, USA (Region 5) 2004 NC+2A44RR	100 g/L SC	Foliar	0.040 0.040	190 190	77 81	9 16 21 30 37	0.02 0.02 0.02 0.02 0.02	T002037-03
New Holland, OH, USA (Region 5) 2004 SC 9373RR	100 g/L SC	Foliar	0.041 0.041	150 150	71 75	30	0.02	T002037-03
Lesterville, SD, USA (Region 5) 2004 Mustang M-222RR	100 g/L SC	Foliar	0.040 0.040	140 130	71 75	30	0.01	T002037-03
Highland, KS, USA (Region 5) 2004 Pioneeer 93M80	100 g/L SC	Foliar	0.042 0.041	120 120	R5 R6	30	0.03	T002037-03
Conklin, MI, USA (Region 5) 2004 Pioneeer 92B38	100 g/L SC	Foliar	0.040 0.040	19 19	R5 R6	30	< 0.01	T002037-03
Gardner, ND, USA (Region 5) 2004 0332132	100 g/L SC	Foliar	0.042 0.041	160 170	75 77	30	< 0.01	T002037-03
Fitchburg, WI, USA (Region 5) 91M50	100 g/L SC	Foliar	0.041 0.040	220 210	65 71	30	0.01	T002037-03

Peanut

A total of eight supervised trials were reported from Australia (three trials), Brazil (one trial), and the United States (four trials).

Table 82 Peanut Field Trials (Nut Meat)

Location/ Year/ Variety	Application					PHI (days)	Cypro-conazole (mg/kg)	Study/ Report
	Formulation	Method	Rate (kg ai/ha)	Rate (kg ai/hL) or Volume (L/ha)	Growth Stage (BBCH)			
Queensland, Australia 1990 Menzies	80 g/L SC	Foliar	0.060 0.060 0.060 0.060	250 250 250 250	10 day interval	2 6 13	< 0.02 < 0.02 (ND) < 0.02 (ND)	AUS 03/08
	80 g/L SC	Foliar	0.12 0.12 0.12 0.12	250 250 250 250	10 day interval	2	< 0.02	AUS 03/08
	80 g/L SC + BS1000 (0.15%)	Foliar	0.060 0.060 0.060 0.060	250 250 250 250	10 day interval	6	< 0.02 (ND)	AUS 03/08
Queensland, Site 2, Australia 1990 SO95	80 g/L SC	Foliar	0.060 0.060 0.060 0.060	250 250 250 250	8 – 13 day interval	3 7 14	< 0.02 < 0.02 < 0.02	AUS 03/08
	80 g/L SC	Foliar	0.12 0.12 0.12 0.12	250 250 250 250	8 – 13 day interval	3 7 14	< 0.02 < 0.02 (ND) 0.05	AUS 03/08
	80 g/L SC + BS1000 (0.15%)	Foliar	0.060 0.060 0.060 0.060	250 250 250 250	7 – 8 day interval	7	< 0.02 (ND)	AUS 03/08
Queensland, Australia 1987 Virginia Bunch	100 g/L SL	Foliar	0.060 0.060 0.060 0.060 0.060 0.060	370 370 370 370 370 370	21 day interval	18	< 0.02	CBK 13130/90
	100 g/L SL	Foliar	0.080 0.080 0.080 0.080 0.080 0.080	370 370 370 370 370 370	21 day interval	18	< 0.02	CBK 13130/90
	100 g/L SL	Foliar	0.20 0.20 0.20 0.20 0.20 0.20	370 370 370 370 370 370	21 day interval	18	< 0.02	
Eakley, OK, USA 1987 Spanco	100 g/L SL	Foliar	0.062 0.062 0.062 0.062 0.062 0.062 0.062 0.062	140 140 140 140 140 140 140 140	13 day interval Begin maturity	21	< 0.01	433018-9
Hartford, Geneva County, AL, USA 1988	100 g/L SL	Foliar	0.086 0.086 0.086 0.086	140 130 140 140	13 day interval	19	< 0.01	433018-9

Location/ Year/ Variety	Application					PHI (days)	Cypro-conazole (mg/kg)	Study/ Report
	Formulation	Method	Rate (kg ai/ha)	Rate (kg ai/hL) or Volume (L/ha)	Growth Stage (BBCH)			
Florunner			0.086 0.086 0.086 0.086	140 140 140 140	 Begin maturity			
Suffolk, Suffolk County, VA, USA 1988 NC-7	100 g/L SL	Foliar	0.086 0.086 0.086 0.086 0.086 0.086 0.086 0.086	86 86 86 86 86 86 86 86	-	16	0.016	433018-9
Enfield, Halifax County, NC, USA 1988	100 g/L SL	Foliar	0.086 0.086 0.086 0.086 0.086 0.086 0.086 0.086	200 200 200 200 200 200 200 200	12-17 interval Mature	0	< 0.01	433018-9

Livetock feed commodities

Pea vines (green)

Table 83 Field trials for pea vines (green)

Location/ Year/ Variety	Application					PHI (days)	Cypro-conazole (mg/kg)	Study/ Report
	Formulation	Method	Rate (kg ai/ha)	Rate (kg ai/hL)	Growth Stage (BBCH)			
St. Aubin, France (North) 1994 Nina	40 g/L SL	Foliar	0.080 0.082	398 L/ha 408 L/ha	61 65 - 67	0 plant	1.5	R10295/ BS9427
Bonnencontre, France (north) 1994 Valette	40 g/L SL	Foliar	0.076 0.080	382 L/ha 400 L/ha	61 65 - 67	0 plant	0.78	R10295 BS9427
Birkin, UK (North) 2004 Solara	80 g/L SC	Foliar	0.082 0.082	306 L/ha 306 L/ha	61 – 63 69 - 73	0 plant 7 plant w/o pea 14 plant w/o pea 21 plant w/opea 30 plant w/o pea 43 plant w/o pea	1.4 0.26 0.28 0.07 0.21 0.24	04-0422
Hummanby, Yorkshire, UK (North) 2004	80 g/L SC	Foliar	0.078 0.085	291 L/ha 318 L/ha	61 67 -71	0 plant 7 plant 14 plant w/o pea	2.10 0.21 0.07	04-0422

Location/ Year/ Variety	Application					PHI (days)	Cypro-conazole (mg/kg)	Study/ Report
	Formulation	Method	Rate (kg ai/ha)	Rate (kg ai/hL)	Growth Stage (BBCH)			
Samson						21 plant w/o pea 30 plant w/o pea 43 plant w/o pea	0.02 0.04 0.12	
Montbellet, France (North) 2004 Atos	80 g/L SC	Foliar	0.080 0.080	301 L/ha 299 L/ha	63 75	0 plant w/o pea 7 plant w/o pea 14 plant w/o pea 21 plant w/o pea 30 plant w/o pea	1.60 0.30 0.28 0.43 0.68	04-0422
Epennes, France (South) 2004 NC	80 g/L SC	Foliar	0.080 0.081	300 L/ha 304 L/ha	61 72	0 plant w/o pea 7 plant w/o pea 14 plant w/o pea 21 plant w/o pea 29 plant w/o pea	1.10 0.47 0.31 0.83 0.50	04-0421
Finhan, France (South) 2004 Austin	80 g/L	Foliar	0.080 0.080	300 L/ha 301 L/ha	61 69 – 71	0 plant 7 plant w/o pea 14 plant w/o pea 21 plant w/opea 30 plant w/o pea	0.71 0.21 0.22 0.35 0.20	04-0421
Meauzac, France (South) 2004 Bacara	80 g/L SC	Foliar	0.080 0.080	300 L/hg 299 L/ha	61 67 – 71	0 plant 7 plant w/o pea 14 plant w/o pea 21 plant w/o pea 30 plant w/o pea	1.20 0.02 0.62 0.34 0.84	04-0421

Pea hay or fodder (dry) and Bean fodder

Table 84 Field Trials for Pea and Bean Fodder

Location/ Year/ Variety	Application					PHI (days)	Cypro-conazole (mg/kg)	Study/ Report
	Formulation	Method	Rate (kg ai/ha)	Rate (kg ai/hL) or Volume (L/ha)	Growth Stage (BBCH)			
Pea								
St. Aubin, France (North) 1994 Nina (green pea)	40 g/L SC	Foliar	0.080 0.082	398 408	61 65 – 67	0 plant	1.50	R10295/ BS 9427
Bonnencontre, France (North) 1994 Valette (green pea)	40 g/L SC	Foliar	0.076 0.080	382 400	61 65 – 67	0 plant	0.78	R10295/ BS 9427
Penhoet Kevignac, France (North) 1994 Cador (green pea)	40 g/L SC	Foliar	0.081 0.076	405 382	60 – 61 66	0 plant 14 plant	1.70 0.05	R10295/ BS 9427
Penhoet Kevignac, France (North) 1994 Cador (green pea)	40 g/L SC	Foliar	0.084 0.084	420 420	61 67	0 plant 14 plant	2.00 0.02	R10295/ BS 9427
Rouvres St Jean, France (North) 2004 Canyon (dry pea)	80 g/L SC	Foliar	0.081 0.080	303 301	71 75	Plant w/o pea: 0 7 14 21 30	 0.92 0.44 0.66 1.1 1.0	04-0422
Montbellet, France (North) 2004 Atos (dry pea)	80 g/L SC	Foliar	0.080 0.080	301 299	63 75	Plant w/o pea: 0 7 14 21 30	 1.6 0.30 0.28 0.43 0.68	04-0422
Epennes, France (South) 2004 NC (farm seed)	80 g/L SC	Foliar	0.080 0.081	300 304	61 72	Plant w/o pea: 0 7 14 21 29	 1.1 0.47 0.31 0.83 0.50	04-0421
Biziat, France (South) 2004 Atos (dry pea)	80 g/L SC	Foliar	0.081 0.079	304 297	67 77	Plant w/o pea: 0 7 14 21 30	 2.2 1.1 1.1 1.2 2.1	04-9421
Finhan, France (South) 2004 Austin (dry pea)	80 g/L SC	Foliar	0.080 0.080	300 301	61 69 -71	Plant w/o pea: 0	 0.71	04-0421

Location/ Year/ Variety	Application					PHI (days)	Cypro-conazole (mg/kg)	Study/ Report
	Formulation	Method	Rate (kg ai/ha)	Rate (kg ai/hL) or Volume (L/ha)	Growth Stage (BBCH)			
						7 14 21 30	0.21 0.22 0.35 0.20	
Meauzac, France (South) 2004 Bacara (dry pea)	80 g/L SC	Foliar	0.080 0.080	300 299	61 67 – 71	Plant w/o pea: 0 7 14 21 30	 1.2 0.20 0.62 0.34 0.84	04-0421
Whatton, Nottinghamshire, UK (North) 1994 Baccara (combining pea)	40 g/L SC	Foliar	0.080 0.080	200 200	Knott 203 Knott 207	27 haulm	0.36	BS 5558
Woodhall Spa, Lincolnshire, UK (North) 1994	40 g/L SC	Foliar	0.080 0.080	200 200	Knott 203 Knott 205/6	27 haulm	0.17	BS 5558
Goole, N Humberside, UK (North) 1994 Solara (combining pea)	40 g/L SC	Foliar	0.080 0.080	200 200	Knott 203 Knott 206	30 haulm	< 0.02	BS 6668
Chatteris, Cambridgeshire, UK (North) 1994 Bacchus (combining pea)	40 g/L SC	Foliar	0.080 0.080	200 200	Knott 203 Knott 205/6	26 haulm	0.51	BS 5558
Hinton, Worcester, UK (North) 1994 Baroness (combining pea)	40 g/L SC	Foliar	0.080 0.080	200 200	Knott 203/4 Knott 206	17 haulm	1.20	BS 5558
Birkin, UK (North) 2004 Solara (dry pea)	80 g/L SC	Foliar	0.082 0.082	306 308	61 – 63 69 – 73	Plant w/o pea: 0 7 14 21 30 43	 1.4 0.26 0.28 0.07 0.21 0.24	04-0422
Hummanby, Yorkshire, UK (North) 2004 Samson (dry pea)	80 g/L SC	Foliar	0.078 0.085	291 318	61 67 – 71	Plant w/o pea: 0 7 14 21 30	 2.10 0.21 0.07 0.02 0.04	04-0422

Location/ Year/ Variety	Application					PHI (days)	Cypro-conazole (mg/kg)	Study/ Report
	Formulation	Method	Rate (kg ai/ha)	Rate (kg ai/hL) or Volume (L/ha)	Growth Stage (BBCH)			
						43	0.12	
Bean								
Boce, France (North) 2006 Castel (dry bean)	80 g/L SC	Foliar	0.053 0.080	197 299	62 87	plant w/o bean	0.52	05-0415
Monteux, France (South) 2005 Big Borloto (dry bean)	80 g/L SC	Foliar	0.083 0.081	208 203	69 – 71 75 – 77	30 plant w/o bean	0.05	05-0607
Grisolles, France (South) 2005 Linex (dry bean)	80 g/L SC	Foliar	0.079 0.079	200 200	62 76	31 plant w/o bean	0.05	05-0607
Estillac, France (South) 2005 Linex (dry bean)	80 g/L SC	Foliar	0.087 0.083	200 200	64 73 – 75	29 plant w/o bean	0.10	05-0607
Marsillargues, France (South) 2005 Linex (dry bean)	80 g/L SC	Foliar	0.080 0.080	300 300	61 67	28 plant w/o bean	0.04	05-0607
Horston, UK (North) 2005 Clipper (dry bean)	80 g/L SC	Foliar	0.081 0.081	303 302	61 70	30 plant w/o bean	< 0.01	05-0415
Draycott, UK (North) 2005 Quattro (dry bean)	80 g/L SC	Foliar	0.082 0.083	308 310	61 79	Plant w/o bean: 30 36	 0.06 0.09	05-0415
Woodhouse Eves, UK (North) 2006 Castel (dry bean)	80 g/L SC	Foliar	0.080 0.081	300 303	59 – 62 78 – 82	30 Plant w/o bean	0.05	T014143-05/ T014143-05-REG

Sugar beet leaves or tops

A total of thirty-two supervised residue trials were conducted on sugar beet between 1986 and 2002. The trials were conducted on outdoor crops, sixteen in Northern Europe and sixteen in Southern Europe.

Table 85 Sugar Beet leaves or tops Field Trials

Location/ Year/ Variety	Application					PHI (days)	Cypro-conazole (mg/kg)	Study/ Report
	Formulation	Method	Rate (kg ai/ha)	Rate (kg ai/hL) or Volume (L/ha)	Growth Stage (BBCH)			
Wigoltingen, Switzerland (North)	? WG	Foliar	0.080 0.080	500 500		0 15 28	0.75 0.14 0.12	R8836

Location/ Year/ Variety	Application					PHI (days)	Cypro-conazole (mg/kg)	Study/ Report
	Formulation	Method	Rate (kg ai/ha)	Rate (kg ai/hL) or Volume (L/ha)	Growth Stage (BBCH)			
1986 Kadutschka						36	0.14	
Wigoltingen, Switzerland (North) 1986	40 g/L SC	Foliar	0.060 0.060	500 500	-	0 15 28 36	1.5 0.44 0.25 0.17	R8834
Portes Conches en Ouche, France (North) 1987 Allyx	32 g/kg WG	Foliar	0.064 0.064	400 400	-	0 17 31 37	2.2 0.64 0.29 0.23	R9141
Chepoix, France (North) 1988 Montpeso	40 g/L SC	Foliar	0.060 0.060	400 400	-	0 14 30	1.5 0.36 0.26	811072/ R 9335
Chepoix, France (North) 1988 Montpeso	40 g/L SC	Foliar	0.060 0.060	400 400	-	29	0.37	811072/ R 9336
Livery-Louvercy, France (North) 1987 Allyx	100 g/L SL	Foliar	0.060 0.060	-	-	0 14 26 48	2.3 0.35 0.20 0.35	R9030
Bucy le Long, France (North) 1987 Gala	WG	Foliar	0.060 0.060	400 400	-	0 13 28 42	2.4 0.19 0.29 0.07	R9033
Guerbigny, France (North) 1988 Allyx	40 g/L SC	Foliar	0.060 0.060	400 400	-	0 16 29 49	1.9 0.12 0.10 0.15	R9334/ 811071
Marsillargues, France (South) 1999 Nevada	80 g/L EC	Foliar	0.056 0.056 0.056	400 400 400	39 39 30	0 22 30 43	0.70 0.08 0.12 0.05	9911001
Escatalens, France (South) 1999 Nevada	80 g/L EC	Foliar	0.056 0.056 0.056	300 300 300	19 31 39	0 21 30 45	1.0 0.13 0.10 < 0.05	9911002
Roquecourbe, France (South) 2000 Agora	80 g/L EC	Foliar	0.055 0.055 0.054	975 983 967	31 38 – 39 39 – 49	0 20	1.2 0.27	NOV/RES/00061
Mauguio, France (South) 2000 Agora	80 g/L EC	Foliar	0.057 0.059 0.054	408 423 388	39 39 39	0 20	0.38 0.07	NOV/RES/00062
Magdeburg, Sachsen-Anhalt, Germany (North) 1993	100g /L SL	Foliar	0.081 0.076	202 189	- 48	0 14 21 28 35 65	1.4 0.57 0.44 0.29 0.36 0.09	BS-5177
Burgwedel-Thonse, Niedersachsen, Germany	100 g/L SL	Foliar	0.080 0.081	200 202	- 47	0 14 21 28	< 0.01 1.4 0.84 0.51	

Location/ Year/ Variety	Application					PHI (days)	Cypro-conazole (mg/kg)	Study/ Report
	Formulation	Method	Rate (kg ai/ha)	Rate (kg ai/hL) or Volume (L/ha)	Growth Stage (BBCH)			
(North) 1993						35	0.39 0.20	
Hilgermissen, Niedersachsen, Germany (North) 1993	100 g/L SL	Foliar	0.082 0.082	204 204	- 47	0 14 21 28 35 52	1.8 0.45 0.71 0.27 0.31 0.24	BS-5177
Pforzheim, Baden-Wurttenberg, Germany (North) 1993 Hilma	100 g/L SL	Foliar	0.083 0.082	206 204	- 41 – 42	0 14 21 28 35 69	1.6 0.44 0.24 0.20 0.14 0.09	BS-5177
Coldham, Cambridgeshire, UK (North) 1995 Jackpot	100 g/L SL	Foliar	0.060 0.060	250 250	26 >30	0 3 7 10 14	1.7 0.71 0.48 0.45 0.42	R95-033/ BS 7525
Bury, St. Edmonds, UK (North) 1995 Aztec	100 g/L SL	Foliar	0.060 0.060	250 250	-	0 3 7 10 14	1.4 0.45 0.32 0.23 0.16	R95-033/ BS 7525
Bramston, Liemeorm, UK (North) 1995 Druid	100 g/L SL	Foliar	0.060 0.060	250 250	26 29-35	0 14	0.81 0.20	R95-033/ BS 7525
Bury, St. Edmunds, UK (North) Zulu	100 g/L SL	Foliar	0.060 0.060	250 250	15 15-40	0 14	1.1 0.34	R95-033/ BS 7525
Marmorta, Italy (South) 1987 Monohill	100 g/L SL	Foliar	0.080 0.080 0.080	800 800 800	-	0 14 28	1.4 0.47 0.34	R9052
Molinella, Italy (South) 1987 Rizor	100 g/L SL	Foliar	0.080 0.080 0.080	800 800 800	-	0 14 28	2.7 0.61 0.36	R9053
Portonovo, Italy (South) 1987]Monofort	100 g/L SL	Foliar	0.080 0.080 0.080	800 800 800	-	27	0.59	R9054
Filo, Italy (South) 1987 Monofort	100 g/L SL	Foliar	0.080 0.080 0.080	800 800 800	-	27	0.82	R9055
Bagnarola di Budrio, Italy (South) 2002 Extra Brio Saros	80 g/L SC	Foliar	0.076 0.074	475 463	44 45	21 28	0.54 0.82	0224R/25R
Pesaro, Italy (South) 2002 Fiamma	80 g/L SC	Foliar	0.082 0.081	515 607	43 45	0 14 21 28	1.1 0.05 0.06 < 0.02	0224R/25R

Location/ Year/ Variety	Application					PHI (days)	Cypro-conazole (mg/kg)	Study/ Report
	Formulation	Method	Rate (kg ai/ha)	Rate (kg ai/hL) or Volume (L/ha)	Growth Stage (BBCH)			
Marano di Castenaso, Italy (South) 2002 Sucrosaros	80 g/L SC	Foliar	0.085 0.082	529 511	39 45	0 14 21 28	1.3 0.54 0.34 0.21	AGRI 004/03
S. Petro in Casale, Bologna, Italy (South) 2002 Gea	80 g/L SC	Foliar	0.087 0.081	543 509	39 46	0 14 21 28	1.6 0.64 0.29 0.12	AGRI 004/03
Unita Periferica di Ronchi Di Villafrnaca-Pd, Italy (South) 1999 Azzurro	80 g/L EC	Foliar	0.056 0.056 0.056	600 600 600	45 47 48-49	0 7 14 21 28	0.36 0.38 0.12 0.07 0.07	2074/99
Unita Periferica di Serravalle a Po-Mantova, Italy (South) 1999 Puma	80 g/L EC	Foliar	0.056 0.056 0.056	600 600 600	44-45 45-46 46-48	21	0.05	2075/99
Badajoz, Spain (South) 1999 Autopoly (Mezano)	80 g/L EC	Foliar	0.055 0.055 0.057	980 987 1016	42-43 44-46 49	21	0.21	2060/99
Valladolid, Spain (South) 1999 Cima	890 g/L EC	Foliar	0.057 0.055 0.056	1010 958 1008	33-39 39-43 43-47	0 7 14 21	1.4 0.63 0.53 0.43	2061/99

Straw and fodder of cereal grains, and Maize forage

A total of forty-seven supervised residue trials were conducted on wheat and rye between 1986 and 2001. The trials were conducted on outdoor crops, thirty-nine in Northern Europe and eight in Southern Europe. A total of forty-two supervised residue trials were conducted on barley between 1986 and 2001. The trials were conducted on outdoor crops, twenty-eight in Northern Europe and fourteen in Southern Europe. A total of twenty-two supervised residue trials on maize (field corn) were conducted in fifteen states in the United States.

Table 86 Wheat straw field trials

Location/ Year/ Variety	Application					PHI (days)	Cypro-conazole (mg/kg)	Study/ Report
	Formulation	Method	Rate (kg ai/ha)	Rate (kg ai/hL) or Volume (L/ha)	Growth Stage (BBCH)			
Cote-d Or, France (North) 1991 Recital	240 g/L SL	Foliar	0.10 0.082	405 412	61 – 69	55	1.7	91034M/5_7D/ BS 9358
Izy, France	53 g/kg WG	Foliar	0.080	400	31-32	44	0.17	2210/97

Location/ Year/ Variety	Application					PHI (days)	Cypro-conazole (mg/kg)	Study/ Report
	Formulation	Method	Rate (kg ai/ha)	Rate (kg ai/hL) or Volume (L/ha)	Growth Stage (BBCH)			
(North) 1997 Sideral			0.080	400	71-73			
Izy, France (North) 1997 Sideral	80 g/L EC	Foliar	0.080 0.080	400 400	32-32 71-73	44	0.16	2238/97
Izy, France (North) 1997 Sideral	160 g/L EC	Foliar	0.080 0.080	400 400	32-32 71-73	46	0.23	2201/97
Cadenac, France (South) 1986	100 g/L SL	Foliar	0.10 0.080	500 500	 59	48	0.37	
Saintes, France (South) 1986 Talent	100 g/L SL	Foliar	0.10 0.080	400 400	 59	48	0.23	CBK11719/86/ 8714
Saintes, France (South) 1987 Talent	100 g/L SL	Foliar	0.080 0.10	400 400	 59	35	0.39	CBK II-1202/88/ 9004
Mauguio, France (South) 1998 Arstar	45 g/kg WG	Foliar	0.082 0.083	411 418	51 65	47	1.8	9812901
Lansargues, France (south) 1998 Primadur	45 g/kg WG	Foliar	0.080 0.081	402 407	51 65	45	1.8	9812902
Mauguio, France (south) 1998	80 g/L EC	Foliar	0.073 0.084	367 418	32 65	37 47	1.2 1.6	9813103
Lansargues, France (South) 1998 Primadur	80 g/L EC	Foliar	0.082 0.084	409 422	32 65	36 45	0.80 1.7	9813104
Marsanne, France (South) 2001	80 g/L SC	Foliar	0.080 0.080	300 300	33 65	42	0.48	0112902
Rodinghausen, Germany (North) 1996 Pegasos	100 g/L SL	Foliar	0.01 0.10	400 400	49 61	35 54	0.66 1.1	R96-007
Rodinghausen, Germany (North) 1996 Pegasos	240 g/L EC	Foliar	0.96 0.96	400 400	49 61	35 54	0.48 0.92	R96-007
Rodinghausen, Germany (North) 1996 Pegasos	160 k/kg WG	Foliar	0.96 0.96	400 400	49 61	35 54	0.53 0.76	R96-007
Sandelsbronn, Germany (North) 1996 Contra	100 g/L SL	Foliar	0.10 0.10	300 300	49 61	35 65	0.34 0.42	R96-007

Location/ Year/ Variety	Application					PHI (days)	Cypro-conazole (mg/kg)	Study/ Report
	Formulation	Method	Rate (kg ai/ha)	Rate (kg ai/hL) or Volume (L/ha)	Growth Stage (BBCH)			
Sandelsbronn, Germany (North) 1996 Contra	240 g/L EC	Foliar	0.096 0.096	300 300	49 61	35 65	0.23 0.37	R96-007
Sandelsbronn, Germany (North) 1996 Contra	160 g/kg WG	Foliar	0.10 0.10	300 300	49 61	35 65	0.18 0.24	R96-007
Axien, Germany (North) 2001 Aristos	80 g/L SC	Foliar	0.80 0.80	300 300	32-33 69	35 50	0.12 0.23	gwh 32401
Gross-Niendorf, Germany (North) 2001	80 g/L SC	Foliar	0.80 0.80	300 300	31-32 69-73	42	0.57	gwh92401
Mehlbek, Germany (North) 1991 Kraka	100 g/L SL	Foliar	0.082 0.11 0.10	307 106 103	31 49 61	37	0.77	R10176/ BS 2781
Hilgermissen, Germany (North) 1991 Orestis	100 g/L SL	Foliar	0.078 0.093 0.10	300 280 320	31-32 52 61	35 43	2.1 1.3	R10177/ BS 2158
Upstedt, Germany (North) 1991 Ibis	100 g/L SL	Foliar	0.089 0.088 0.091	330 270 290	31 49 61	35 38	1.2 1.4	R10178/ BS 2767
Aarbergen-Panrod, Germany (North) 1991 Orestis	100 g/L SL	Foliar	0.079 0.10 0.10	400 100 100	31 49 61	35 42 50	2.4 2.3 2.3	R10178/ BS 2767
Riedstadt-Crumstadt, Germany (North) 1991 Bert	100 g/L SL	Foliar	0.082 0.10 0.11	410 410 430	31 49 61	35 42 53	0.67 0.69 0.92	R10180/ BS 2787
Niddatal-Ilbenstadt, Germany (North) 1991 Kanzler	100 g/L SL	Foliar	0.075 0.10 0.10	380 410 400	31 49 61	35 42 (BBCH85) 48 (BBCH91)	0.92 0.74 0.50	R10181/ BS 2772
Hilgermissen, Germany (North) 1992 Kontrast	100 g/L SL	Foliar	0.088 0.11 0.11	440 440 440	37 52 69	35 42	2.8 3.6	R10249/ BS 3641
Hilgermissen, Germany (North)	40 g/L SC	Foliar	0.081 0.087 0.080	410 430 400	37 52 69	35 42	< 0.01 0.78	R10249/ BS 3641

Location/ Year/ Variety	Application					PHI (days)	Cypro-conazole (mg/kg)	Study/ Report
	Formulation	Method	Rate (kg ai/ha)	Rate (kg ai/hL) or Volume (L/ha)	Growth Stage (BBCH)			
1992 Kontrast								
Jemstorf-Lute, Germany (North) 1992 Orestis	100 g/L SL	Foliar	0.088 0.11 0.097	440 430 390	37 55 61	35 42	1.5 1.7	R10249/ BS 3641
Jemstorf-Lute, Germany (North) 1992 Orestis	40 g/L SC	Foliar	0.081 0.080 0.080	400 400 400	37 55 61	35 42	0.79 0.56	R10249/ BS 3641
Bockenem 13, Germany (North) 1992 Astron	100 g/L SL	Foliar	0.084 0.093 0.088	420 370 350	36-37 51 61	36 42	0.78 0.54	R10249/ BS 3641
Bockenem 13, Germany (North) 1992 Astron	40 g/L SC	Foliar	0.084 0.078 0.076	420 390 380	36-37 51 61	36 42	0.22 0.20	R10249/ BS 3641
Goch-Nierswalde, Germany (North) 1992 Orestis	100 g/L SL	Foliar	0.078 0.097 0.10	390 390 410	37 49-51 61	35 42	0.62 0.85	R10249/ BS 3641
Nierswalde, Germany (North) 1992 Orestis	40 g/L SC	Foliar	0.074 0.081 0.083	370 400 410	37 49-51 61	35 42	0.62 0.85	R10249/ BS 3641
Dettelbach-Schernau, Germany (North) 1992 Agronom	100 g/L SL	Foliar	0.080 0.10 0.10	300 300 300	38 49 62	35 42	0.43 0.35	R10249/ BS 3641
Dettelbach-Schernau, Germany (North) 1992 Agronom	40 g/L SC	Foliar	0.080 0.080 0.080	300 300 300	38 49 62	35 42	0.12 0.15	R10249/ BS 3641
Hirblingen, Germany (North) 1992 Ares	100 g/L SL	Foliar	0.080 0.10 0.10	300 300 300	39 53 61	34 (BBCH79) 41 (BBCH85-89)	1.3 0.96	R10249/ BS 3641
Hirblingen, Germany (North) 1992 Ares	40 g/L SC	Foliar	0.080 0.080 0.080	300 300 300	39 53 61	34 41	0.36 0.30	R10249/ BS 3641
Bex/VD, Switzerland (North) 2001	80 g/L SC	Foliar	0.080 0.080	400 400	37 69	35 47	0.09 0.08	2075/01

Location/ Year/ Variety	Application					PHI (days)	Cypro-conazole (mg/kg)	Study/ Report
	Formulation	Method	Rate (kg ai/ha)	Rate (kg ai/hL) or Volume (L/ha)	Growth Stage (BBCH)			
Albis								
	80 g/L SC	Foliar	0.080 0.080	400 400	37 69	35 47	0.08 0.07	2075/01
Vouvry/VS, Switzerland (North) 2001 Galaxy	80 g/L SC	Foliar	0.080 0.080	400 400	33 69	35 47	0.07 < 0.05	2076/01
	80 g/L SC	Foliar	0.080 0.080	400 400	33 69	35 47	0.11 0.06	2076/01

Table 87 Rye straw field trials

Location/ Year/ Variety	Application					PHI (days)	Cypro-conazole (mg/kg)	Study/ Report
	Formulation	Method	Rate (kg ai/ha)	Rate (kg ai/hL) or Volume (L/ha)	Growth Stage (BBCH)			
Hilgermissen, Germany (North) 1992 Rapid	100 g/L SL	Foliar	0.11 0.11	460 420	54 64	35 41	0.64 0.64	R10250/ BS 4112
Braunschweig-Hondelage, Germany (North) 1992 Ammando Hybrid	100 g/L SL	Foliar	0.10 0.089	400 360	54 61	35 42	0.80 1.2	R10250/ BS 4112
Goch-Kessel, Germany (North) 1992 Halo	100 g/L SL	Foliar	0.11 0.098	440 390	37-51 61	40	0.68	R10250/ BS 4112
Ivenrode, Germany (North) 1998 Hacada	23 g/kg WG	Foliar	0.080 0.080	400 400	32 69-71	49 57	0.22 0.36	gr 39298
Wallersdorf, Germany (North) 1998 Borellus	53 g/kg WG	Foliar	0.080 0.080	400 400	31-32 71	49 56	0.19 0.16	gr 40498

Table 88 Barley straw field trials

Location/ Year/ Variety	Application					PHI (days)	Cypro-conazole (mg/kg)	Study/ Report
	Formulation	Method	Rate (kg ai/ha)	Rate (kg ai/hL) or Volume (L/ha)	Growth Stage (BBCH)			
Pinson, France (North) 1986 Illia	100 g/L SL	Foliar	0.10 0.10	100 100	-	44	0.16	R8730

Location/ Year/ Variety	Application					PHI (days)	Cypro-conazole (mg/kg)	Study/ Report
	Formulation	Method	Rate (kg ai/ha)	Rate (kg ai/hL) or Volume (L/ha)	Growth Stage (BBCH)			
	100 g/L SL	Foliar	0.10 0.10	100 100	-	44	0.16	R8730
Montalzat, France (South) 1997 Sonja	45.3 g/kg WG	Foliar	0.080 0.080	400 400	32 71	43	0.06	2186/97
Realville, France (South) 1997 Baraka	45.3 g/kg WG	Foliar	0.080 0.080	400 400	31-32 71	45	0.33	2187/97
	45.3 g/kg WG	Foliar	0.080 0.080	400 400	32 69	45	0.20	2187/97
Lansargues, France (South) 1997 Alpha	45.3 g/kg WG	Foliar	0.080 0.080	400 400	37 69	42	0.17	2189/97
Montalzat, France (South) 1997 Sonja	80 g/L EC	Foliar	0.080 0.080	400 400	32 71	43	0.07	2228/97
Realville, France (South) 1997 Baraka	80 g/L EC	Foliar	0.080 0.080	400 400	31-32 71	45	0.22	2228/97
Divajeu, France (South) 1997 Orelie	80 g/L EC	Folira	0.080 0.080	400 400	43 71	45	0.23	2229/97
Lasargues, France (South) 1997 Alpha	80 g/L EC	Foliar	0.080 0.080	400 400	37 69	45	0.20	2231/97
Dange, France (South) 2001 Ludine	160 g/L EC	Foliar	0.079 0.078	390 390	31 69	41	0.87	0110901
Ingrandes, France (South) 2001	160 g/L EC	Foliar	0.073 0.083	370 410	32 69	41	0.25	0110902
Cayrac, France (South) 2001 Sonja	80 g/L SC	Foliar	0.080 0.080	400 400	33 69-71	42	0.29	0112701
	80 g/L SC	Foliar	0.080 0.080	400 400	33 69-71	42	0.19	0113201
Marsillargues, France (South) 2001 Baraka	80 g/L SC	Foliar	0.080 0.080	400 400	34 71-73	42	1.1	0112702
	80 g/L SC	Foliar	0.080 0.080	400 400	32-33 69-71	42	0.68	0113202
Denges/VS, Switzerland (North) 1986	100 g/l SL	Foliar	0.10 0.10	1000 1000	61	42	0.42	R8811
Hugelshofen/TG, Switzerland (North)	100 g/L SL	Foliar	0.10 0.10	500 500	59	42	0.34	R8812

Location/ Year/ Variety	Application					PHI (days)	Cypro-conazole (mg/kg)	Study/ Report
	Formulation	Method	Rate (kg ai/ha)	Rate (kg ai/hL) or Volume (L/ha)	Growth Stage (BBCH)			
1986 Gerbel								
Hugelshofen/TG, Switzerland (North) 1986 Gerbel	400 g/L SL	Foliar	0.10 0.10	500 500	59	42	0.24	R8814
Bottens/VS, Switzerland (North) 1987 Gerbel	80 g/L EC	Foliar	0.060 0.060 0.080	500 500 500	61	35 42	0.17 0.13	R9136
Ottoberg/TG, Switzerland (North) 1987 Mammut	80 g/L EC	Foliar	0.060 0.060 0.060	500 500 500	-	34 41	0.18 0.27	R9137
Burgwedel-Thonse, Germany (North) 1992 Catinka	100 g/L SL	Foliar	0.11 0.10	460 410	58-59 63	35 42	0.40 0.56	R10250/ TDS BS4112
Dettelbach-Schernau, Germany (North) 1992 Alraune	100 g/L SL	Foliar	0.10 0.10	300 300	54 66	34 42	0.01 0.01	R10250/ TDS BS4112
Dettelbach-Schernau, Germany (North) 1992 Alraune	100 g/L SL	Foliar	0.10 0.10	300 300	54 66	34 42	0.20 0.12	R10250/ TDS BS4112
Anhausen (Augsburg), Germany (North) 1992	100 g/L SL	Foliar	0.10 0.10	300 300	55 62	35 42	0.22 0.17	R10250/ TDS BS4112
Ostkilver, Germany (North) 1996 Loreley	100 g/L SL	Foliar	0.095 0.095	400 400	59 61-63	35 48	0.53 0.53	R96-007
Ostkilver, Germany (North) 1996 Loreley	240 g/L EC	Foliar	0.097 0.097	400 400	59 61-63	35 48	0.63 0.53	R96-007
Ostkilver, Germany (North) 1996 Loreley	160 g/kg WG	Foliar	0.097 0.097	400 400	59 61-63	35 48	0.52 0.48	R96-007
Sandelsbronn, Germany (North) 1996 Astrid	100 g/L SL	Foliar	0.10 0.096	400 400	55-59 61	35 54	0.67 0.21	R96-007
Sandelsbronn, Germany (North) 1996 Astrid	240 g/L EC	Foliar	0.094 0.091	400 400	55-59 61	35 54	0.52 0.21	R96-007

Location/ Year/ Variety	Application					PHI (days)	Cypro-conazole (mg/kg)	Study/ Report
	Formulation	Method	Rate (kg ai/ha)	Rate (kg ai/hL) or Volume (L/ha)	Growth Stage (BBCH)			
Sandelsbronn, Germany (North) 1996 Astrid	160 g/kg WG	Foliar	0.090 0.090	400 400	55-59 61	35 54	0.52 0.21	R96-007
Sandelsbronn, Baden-Wurttemberg, Germany (North) 1996 Astrid	2 X 80 g/L SC 1 X 100 g/L SL	Foliar	0.098 0.10 0.075	300 300 280	32 49 61	35 54	0.14 (< 0.01 duplicate) 0.01	R96-002
Sandelsbronn, Baden-Wurttemberg, Germany (North) 1996 Astrid	2 X 80 g/L SC 1 X 100 g/L SL	Foliar	0.098 0.10 0.075	300 300 280	32 49 61	35 54	0.15 0.11	R96-002
Ostkilver, Germany (North) 1996 Loreley	2 X 0.080 g/L SC 1 X 100 g/L SL	Foliar	0.10 0.097 0.077	410 390 390	32 49 61	35 49	0.28 0.27	R96-022
Leiston, UK (North) 1987 Pipkin	100 g/L SL	Foliar	0.080 0.080 0.080	200 200 200	 60	69	0.15	R9237
Sibton, UK (North) 1987 Igri	100 g/L SL	Foliar	0.080 0.080 0.080	200 200 200	 61	70	0.37	R9240
Duns Tew, Oxon., UK (North) 1991 Igri	100 g/L SL	Foliar	0.080 0.080 0.080	200 200 200	 31 61	?	0.09	BS 5553
Bucknell, Oxon. , UK (North) 1991 Plaisant	100 g/L SL	Foliar	0.080 0.080 0.080	200 200 200	13-14 32 61-65	51	0.09	SDZ 0292/2/ BS 5553
Dalderby, Hornsastle, Lincs., UK (North) 1991 Puffin	100 g/L SL	Foliar	0.080 0.080 0.080	200 200 200	12-13 31 61	59	0.14	SDA 0292/2/ BS 5553
Chudleigh, Devon., UK (north) 1992 Frolic	100 g/L SL	Foliar	0.080 0.080 0.080	200 200 200	Z12 Z31 Z59	53	0.35	SDZ 0692/ BS 5573
Aynho, Oxon., UK (North) 1992 Gypsy	100 g/L SL	Foliar	0.080 0.080 0.080	200 200 200	Z23 Z31 Z55-59	54	0.12	SDZ 0692/ BS 5573
Newton St. Cyres, Devon., UK (North) 1992 Fighter	100 g/L SL	Foliar	0.080 0.080 0.080	200 200 200	Z13 Z31 Z59	52	0.07	SDZ 0692/ BS 5573
Haltham, Lincs., UK (North) 1992 Pipkin	100 g/L SL	Foliar	0.080 0.080 0.080	200 200 200	Z13-21 Z31 Z59-60	42	0.21	SDZ 0692/ BS 5573
St. Madoes,	100 g/L SL	Foliar	0.080	200	Z24	52	0.08	SDZ 0692/

Location/ Year/ Variety	Application					PHI (days)	Cypro-conazole (mg/kg)	Study/ Report
	Formulation	Method	Rate (kg ai/ha)	Rate (kg ai/hL) or Volume (L/ha)	Growth Stage (BBCH)			
Scotland, UK (North) 1992 Plaisant			0.080 0.080	200 200	Z31 Z59-65			BS 5573

Table 89 Maize fodder/straw

Location/ Year/ Variety	Application					PHI (days)	Cypro-conazole (mg/kg)	Study/ Report
	Formulation	Method	Rate (kg ai/ha)	Rate (kg ai/hL) or Volume (L/ha)	Growth Stage (BBCH)			
Hudson, NY, USA (Region 1) 2004 DKC53-54RR	100 g/L SC	foliar	0.040 0.041	20 20	75 89-93	30	0.24	T002814-03
Rose Hill, NC, USA (Region 2) 2004 Pioneer 34B97	100 g/l SC	Foliar	0.040 0.040	230 150	R2 R5-R6	30	0.28	T002814-03
Richlans, IA, USA (Region 5) 2004 Golden Harvest H-9247 BT	100 g/L SC	Foliar	0.040 0.041	180 160	R3-R4 R5	7 14 21 30 37	0.48 0.39 0.38 0.13 0.28	T002814-03
	100 g/L SC	Foliar	0.040 0.040	160 160	R3 R5	30	0.74	T002814-03
Chatsworth, IA, USA (Region 5) 2004 Midwest 7X088	100 g/L SC	Foliar	0.040 0.040	140 140	R3 R5	30	< 0.01	T002814-03
Chatsworth, IA, USA (Region 5) 2004 Midwest 7X088	100 g/L SC	Foliar	0.040 0.040	140 140	R3 R5	30	0.33	T002814-03
Searsville, IA, USA (Region 5) 2004 DKC 5145	100 g/L SC	Foliar	0.040 0.041	150 150	R4 R5	30	0.45	T002814-03
Champaign, Il, USA (Region 5) 2004 Pioneer 34H31	100 g/L SC	Foliar	0.41 0.43	100 140	- 91	7 14 21 30 37	0.13 0.29 0.59 0.21 0.15	T002814-03
Carlyle, Il, USA (Region 5) 2004	100 g/L SC	Foliar	0.041 0.040	170 91	75 79	30	0.12	T002814-03
Chemung Township/ Harvard, WI, USA (region 5) 2004 Hughes 5172RR	100 g/L SC	Foliar	0.041 0.039	170 160	75 79	30	0.27	T002814-03
York, NE, USA (Region 5)	100 g/L, SC	Foliar	0.040 0.040	190 150	71 86	30	0.23	T002814-03

Location/ Year/ Variety	Application					PHI (days)	Cypro-conazole (mg/kg)	Study/ Report
	Formulation	Method	Rate (kg ai/ha)	Rate (kg ai/hL) or Volume (L/ha)	Growth Stage (BBCH)			
2004 DKC60-19								
Osceola, NE, USA (Region 5) 2004 DKC60-19	100 g/L SC	Foliar	0.040 0.040	190 190	69 88	30	0.34	T002814-03
Geneva, MN, USA (Region 5) 2004 Pioneer 36N18	100 g/L SC	Foliar	0.040 0.040	150 150	R4 R5	30	0.12	T002814-03
Paynesville, MN, USA (Region 5) 2004 Pioneer 36B08	100 g/L SC	Foliar	0.041 0.040	190 190	85 85	30	< 0.01	T002814-03
Noblesville, IN, USA (region 5) 2004 5322	100 g/L SC	Foliar	0.041 0.041	130 130	R3 R5-R6	30	0.35	T002814-03
New Holland, OH, USA (Region 5) 2004 8627R	100 g/L SC	Foliar	0.041 0.042	200 150	Early dough R6	30	0.80	T002814-03
Centerville, SD, USA (Region 5) 2004 Dairyland Stealth 1608	100 g/L SC	Foliar	0.041 0.040	130 140	75 99	30	0.08	T002814-03
Fitchburg, WI, USA (Region 5) 2004 Pioneer 38H67	100 g/L SC	Foliar	0.041 0.040	280 280	86 86	30	0.35	T002814-03
LaPlata, MO, USA (Region 5) 2004 LG 2540	100 g/L SC	Foliar	0.041 0.040	160 160	77 R4-R5	30	0.46	T002814-03
Conklin, MI USA (Region 5) 2004 36B92	100 g/L SC	Foliar	0.040 0.040	200 190	R2 R5	7 14 21 30 37	0.44 0.41 0.31 1.5 0.60	T002814-03
Highland, KS, USA (Region 5) 2004 Pioneer 32P75	100 g/L SC	Foliar	0.040 0.040	130 94	R2 R5	30	0.22	T002814-03
Clay, TX, USA (Region 6) 2004 DKC-66-80	100 g/L EC	Foliar	0.040 0.040	19 19	- 85	30	0.35	T002814-03
Madera, CA, USA (Region 10) 2004	100 g/L SC	Foliar	0.04 0.041	280 280	Formed ears Ears with silk	30	0.08	T002814-03

Table 90 Maize forage field trials

Location/ Year/ Variety	Application					PHI (days)	Cypro-conazole (mg/kg)	Study/ Report
	Formulation	Method	Rate (kg ai/ha)	Rate (kg ai/hL) or Volume (L/ha)	Growth Stage (BBCH)			
Hudson, NY, USA (Region 1) 2004 DKC53-54RR	100 g/L SC	foliar	0.040	20	75	21	0.14	T002814-03
Rose Hill, NC, USA (Region 2) 2004 Pioneer 34B97	100 g/l SC	Foliar	0.040	230	R2	21	0.10	T002814-03
Richlans, IA, USA (Region 5) 2004 Golden Harvest H-9247 BT	100 g/L SC	Foliar	0.040	180	R3-R4	0 7 14 21 28	0.24 0.14 0.07 0.06 0.07	T002814-03
	100 g/L SC	Foliar	0.040	160	R3	21	0.08	T002814-03
Chatsworth, IA, USA (Region 5) 2004 Midwest 7X088	100 g/L SC	Foliar	0.040	140	R3	21	0.06	T002814-03
Searsville, IA, USA (Region 5) 2004 DKC 5145	100 g/L SC	Foliar	0.040	150	R4	21	0.23	T002814-03
Champaign, Il, USA (Region 5) 2004 Pioneer 34H31	100 g/L SC	Foliar	0.41	100	-	0 7 14 21 28	0.41 0.14 0.13 0.08 0.16	T002814-03
Carlyle, Il, USA (Region 5) 2004	100 g/L SC	Foliar	0.041	170	75	21	0.03	T002814-03
Chemung Township/ Harvard, WI, USA (Region 5) 2004 Hughes 5172RR	100 g/L SC	Foliar	0.041	170	75	21	0.44	T002814-03
York, NE, USA (Region 5) 2004 DKC60-19	100 g/L, SC	Foliar	0.040	190	71	21	0.24	T002814-03
Osceola, NE, USA (Region 5) 2004 DKC60-19	100 g/L SC	Foliar	0.040	190	69	21	0.09	T002814-03
Geneva, MN, USA (Region 5) 2004 Pioneer 36N18	100 g/L SC	Foliar	0.040	150	R4	21	0.29	T002814-03
Paynesville, MN, USA (Region 5) 2004 Pioneer 36B08	100 g/L SC	Foliar	0.041	190	85	21	< 0.01	T002814-03
Noblesville, IN, USA (region 5)	100 g/L SC	Foliar	0.041	130	R3	21	0.20	T002814-03

Location/ Year/ Variety	Application					PHI (days)	Cypro-conazole (mg/kg)	Study/ Report
	Formulation	Method	Rate (kg ai/ha)	Rate (kg ai/hL) or Volume (L/ha)	Growth Stage (BBCH)			
2004 5322								
New Holland, OH, USA (Region 5) 2004 8627R	100 g/L SC	Foliar	0.041	200	Early dough	21	0.10	T002814-03
Centerville, SD, USA (Region 5) 2004 Dairyland Stealth 1608	100 g/L SC	Foliar	0.041	130	75	21	0.06	T002814-03
Fitchburg, WI, USA (Region 5) 2004 Pioneer 38H67	100 g/L SC	Foliar	0.041	280	86	21	0.31	T002814-03
LaPlata, MO, USA (Region 5) 2004 LG 2540	100 g/L SC	Foliar	0.041	160	77	21	0.12	T002814-03
Conklin, MI USA (Region 5) 2004 36B92	100 g/L SC	Foliar	0.040	200	R2	0 7 14 21 28	0.27 0.07 0.06 0.09 0.13	T002814-03
Highland, KS, USA (Region 5) 2004 Pioneer 32P75	100 g/L SC	Foliar	0.040	130	R2	21	0.05	T002814-03
Clay, TX, USA (Region 6) 2004 DKC-66-80	100 g/L EC	Foliar	0.040	19	-	21	0.19	T002814-03
Madera, CA, USA (Region 10) 2004	100 g/L SC	Foliar	0.04	280	Formed ears	21	0.08	T002814-03

Miscellaneous fodder and forage crops

Table 91 Rape seed forage

Location/ Year/ Variety	Application					PHI (days)	Cypro-conazole (mg/kg)	Study/ Report
	Formulation	Method	Rate (kg ai/ha)	Rate (kg ai/hL) or Volume (L/ha)	Growth Stage (BBCH)			
Prunay, France (North) 2005 Aviso	80 g/L SC	Foliar	0.077 0.081	300 300	67 80	0 7 15 21 (BBCH 88) 30	0.95 0.43 0.54 0.21 0.28	05-0409

Location/ Year/ Variety	Application					PHI (days)	Cypro-conazole (mg/kg)	Study/ Report
	Formulation	Method	Rate (kg ai/ha)	Rate (kg ai/hL) or Volume (L/ha)	Growth Stage (BBCH)			
						(BBCH 89)		
La Chapelle de Guinchay, France (North) 2005 Hearty	80 g/L SC	Foliar	0.074 0.081	280 310	61-63 78-80	0 7 14 20 (BBCH 84) 33 (BBCH 89)	0.94 0.24 0.15 0.48 0.48	05-0410
Beine Nauroy, France (North) 2005 Aviso	80 g/L SC	Foliar	0.083 0.083	300 300	69 80	0 7 14 BBCH 88-89: 20 30	0.93 0.54 0.42 0.50 0.52	05-0409
La Sence Septoutre, France (North) 2005 Savana	30 g/L SC	Foliar	0.082 0.080	300 300	69 78	0 7 14 BBCH 87-89: 20 30	0.98 0.47 0.47 0.59 0.24	05-0409
Monferran-Saves, France (South) 2005 Standing	80 g/L SC	Foliar	0.080 0.085	300 320	67 80	0 7 14 BBCH 89-90 21 30	1.8 1.4 1.2 2.5 1.9	05-0310
Monferran-Saves, France (South) 2005 Oliris	80 g/L SC	Foliar	0.081 0.074	310 280	67 80	0 7 14 BBCH 88-89: 21 30	1.2 0.76 0.58 1.9 1.2	05-0310

Table 92 Field trials for soya bean forage

Location/ Year/ Variety	Application					PHI (days)	Cypro-conazole (mg/kg)	Study/ Report
	Formulation	Method	Rate (kg ai/ha)	Rate (kg ai/hL) or Volume (L/ha)	Growth Stage (BBCH)			
Kingston, NC, USA (Region 2) 2004 S57-A4	100 g/L SC	Foliar	0.042	150	Pod fill	0 5 10 14	0.76 0.21 0.54 0.31	T002037-03
Elko, SC, USA	100 g/L SC	Foliar	0.040	26	77	14	0.22	T002037-03

Location/ Year/ Variety	Application					PHI (days)	Cypro-conazole (mg/kg)	Study/ Report
	Formulation	Method	Rate (kg ai/ha)	Rate (kg ai/hL) or Volume (L/ha)	Growth Stage (BBCH)			
(Region 2) 2004 S73-Z5								
Proctor, AR, USA (Region 4) 2004 Pioneer 94B73RR	100 g/L SC	Foliar	0.040	140	69	14	0.40	T002037-03
Richland, IA, USA (Region 5) 2004 Pioneer 93M80/ Pioneer 93M87	100 g/L SC	0.040	Foliar	130	R5	0 5 10 14	1.4 0.85 0.76 0.37	T002037-03
	100 g/L SC	0.039	Foliar	130	R4	14	0.06	T002037-03
Carlyle, IL, USA (Region 5) 2004 B-T 383CR	100 /L SC	Foliar	0.042	130	69	14	0.82	T002037-03
Geneva, MN, USA (region 5) 2004 Pioneer 91M50	100 g/L SC	Foliar	0.040	150	R5	14	0.80	T002037-03
Noblesville, IN, USA (Region 5) 2004 387NRR	100 g/L SC	Foliar	0.041	130	R5-R6	14	0.11	T002037-03
Kirksville, MO, USA (Region 5) 2004 Asgrow 3302	100 g/L SC	Foliar	0.040	18160	71	14	0.31	T002037-03
York, NE, USA (Region 5) 2004 NC+2A44RR	100 g/L SC	Foliar	0.040	190	77	0 5 10 14	1.3 0.78 0.56 0.35	T002037-03
New Holland, OH, USA (Region 5) 2004 SC 9373RR	100 g/L SC	Foliar	0.041	150	71	14	0.33	T002037-03
Lesterville, SD, USA (Region 5) 2004 Mustang M-222RR	100 g/L SC	Foliar	0.040 0.040	140 130	71 75	14	0.48	T002037-03
Highland, KS, USA (Region 5) 2004 Pioneeer 93M80	100 g/L SC	Foliar	0.042	120	R5	14	0.52	T002037-03
Conklin, MI, USA (Region 5) 2004 Pioneeer 92B38	100 g/L SC	Foliar	0.040	19	R5	14	0.21	T002037-03
Gardner, ND, USA (Region 5) 2004 0332132	100 g/L SC	Foliar	0.042	160	75	14	0.50	T002037-03
Fitchburg, WI, USA (Region 5) 91M50	100 g/L SC	Foliar	0.041 0.040	220 210	65 71	14	0.41	T002037-03

Table 93 Field Trials for Soyabean Hay

Location/ Year/ Variety	Application					PHI (days)	Cypro-conazole (mg/kg)	Study/ Report
	Formulation	Method	Rate (kg ai/ha)	Rate (kg ai/hL) or Volume (L/ha)	Growth Stage (BBCH)			
Kingston, NC, USA (Region 2) 2004 S57-A4	100 g/L SC	Foliar	0.042	150	Pod fill	0 5 10 14	1.8 0.90 0.63 0.41	T002037-03
Elko, SC, USA (Region 2) 2004 S73-Z5	100 g/L SC	Foliar	0.040	26	77	14	0.33	T002037-03
Proctor, AR, USA (Region 4) 2004 Pioneer 94B73RR	100 g/L SC	Foliar	0.040 0.040	140	69	14	0.71	T002037-03
Richland, IA, USA (Region 5) 2004 Pioneer 93M80/ Pioneer 93M87	100 g/L SC	0.040	Foliar	130	R5	0 5 10 14	1.4 1.8 1.0 0.66	T002037-03
	100 g/L SC	0.039	Foliar	130	R4	14	0.09	T002037-03
Carlyle, IL, USA (Region 5) 2004 B-T 383CR	100 /L SC	Foliar	0.042	130	69	14	1.3	T002037-03
Geneva, MN, USA (region 5) 2004 Pioneer 91M50	100 g/L SC	Foliar	0.040	150	R5	14	0.43	T002037-03
Noblesville, IN, USA (Region 5) 2004 387NRR	100 g/L SC	Foliar	0.041	130	R5-R6	14	0.17	T002037-03
Kirksville, MO, USA (Region 5) 2004 Asgrow 3302	100 g/L SC	Foliar	0.040	18160	71	14	0.44	T002037-03
York, NE, USA (Region 5) 2004 NC+2A44RR	100 g/L SC	Foliar	0.040	190	77	0 5 10 14	1.8 1.3 1.0 0.75	T002037-03
New Holland, OH, USA (Region 5) 2004 SC 9373RR	100 g/L SC	Foliar	0.041	150	71	14	0.33	T002037-03
Lesterville, SD, USA (Region 5) 2004 Mustang M-222RR	100 g/L SC	Foliar	0.040	140	71	14	1.5	T002037-03
Highland, KS, USA (Region 5) 2004 Pioneeer 93M80	100 g/L SC	Foliar	0.042	120	R5	14	0.32	T002037-03

Location/ Year/ Variety	Application					PHI (days)	Cypro-conazole (mg/kg)	Study/ Report
	Formulation	Method	Rate (kg ai/ha)	Rate (kg ai/hL) or Volume (L/ha)	Growth Stage (BBCH)			
Conklin, MI, USA (Region 5) 2004 Pioneeer 92B38	100 g/L SC	Foliar	0.040	19	R5	14	0.67	T002037-03
Gardner, ND, USA (Region 5) 2004 0332132	100 g/L SC	Foliar	0.042	160	75	14	0.75	T002037-03
Fitchburg, WI, USA (Region 5) 91M50	100 g/L SC	Foliar	0.041 0.040	220 210	65 71	14	1.9	T002037-03

Table 94 Peanut Field Trials (Hulls or Shells)

Location/ Year/ Variety	Application					PHI (days)	Cypro-conazole (mg/kg)	Study/ Report
	Formulation	Method	Rate (kg ai/ha)	Rate (kg ai/hL) or Volume (L/ha)	Growth Stage (BBCH)			
Queensland, Australia 1990 Menzies	80 g/L SC	Foliar	0.060 0.060 0.060 0.060	250 250 250 250	10 day interval	2 6 13	0.32 0.11 0.07	AUS 03/08
	80 g/L SC	Foliar	0.12 0.12 0.12 0.12	250 250 250 250	10 day interval	2 6 13	0.33 0.33 0.11	AUS 03/08
	80 g/L SC + BS1000 (0.15%)	Foliar	0.060 0.060 0.060 0.060	250 250 250 250	10 day interval	6	0.13	AUS 03/08
Queensland, Site 2, Australia 1990 SO95	80 g/L SC	foliar	0.060 0.060 0.060 0.060	250 250 250 250	8 – 13 day interval	3 7 14	0.16 0.22 < 0.02	AUS 03/08
	80 g/L SC	Foliar	0.12 0.12 0.12 0.12	250 250 250 250	8 – 13 day interval	3 7 14	0.23 0.13 30	AUS 03/08
	80 g/L SC + BS1000 (0.15%)	Foliar	0.060 0.060 0.060 0.060	250 250 250 250	7 – 8 day interval	3	0.23	AUS 03/08
Queensland, Australia 1987 Virginia Bunch	100 g/L SL	Foliar	0.20 0.20 0.20 0.20 0.20 0.20	370 370 370 370 370 370	18	0.20	18	
Eakley, OK, USA 1987 Spanco	100 g/L SL	Foliar	0.062 0.062 0.062 0.062 0.062 0.062 0.062	140 140 140 140 140 140 140	13 day interval	21	0.24	433018-9

Location/ Year/ Variety	Application					PHI (days)	Cypro-conazole (mg/kg)	Study/ Report
	Formulation	Method	Rate (kg ai/ha)	Rate (kg ai/hL) or Volume (L/ha)	Growth Stage (BBCH)			
			0.062	140	Begin maturity			
Hartford, Geneva County, AL, USA 1988 Florunner	100 g/L SL	Foliar	0.086 0.086 0.086 0.086 0.086 0.086 0.086 0.086	140 130 140 140 140 140 140 140	13 day interval Begin maturity	19	0.32	433018-9
Suffolk, Suffolk County, VA, USA 1988 NC-7	100 g/L SL	Foliar	0.086 0.086 0.086 0.086 0.086 0.086 0.086 0.086	86 86 86 86 86 86 86 86	-	16	0.17	433018-9
Enfield, Halifax County, NC, USA 1988	100 g/L SL	Foliar	0.086 0.086 0.086 0.086 0.086 0.086 0.086 0.086	200 200 200 200 200 200 200 200	12-17 interval Mature	0	0.37	433018-9

Table 95 Peanut Forage/Vine (green)

Location/ Year/ Variety	Application					PHI (days)	Cypro-conazole (mg/kg)	Study/ Report
	Formulation	Method	Rate (kg ai/ha)	Rate (kg ai/hL) or Volume (L/ha)	Growth Stage (BBCH)			
Queensland, Australia 1990 Menzies	80 g/L SC	Foliar	0.060 0.060 0.060 0.060	250 250 250 250	10 day interval	0 3 6 13 20	5.9 3.2 1.8 1.3 2.6	AUS 03/08
	80 g/L SC	Foliar	0.12 0.12 0.12 0.12	250 250 250 250	10 day interval	0 3 6 13 20	14 5.3 6.2 5.2 5.9	AUS 03/08
	80 g/L SC + BS1000 (0.15%)	Foliar	0.060 0.060 0.060 0.060	250 250 250 250	10 day interval	6	6.0	AUS 03/08
Queensland, Site 2, Australia 1990 SO95	80 g/L SC	Foliar	0.060 0.060 0.060 0.060	250 250 250 250	8 – 13 day interval	0 8 15 19	2.3 3.2 5.3 3.6	AUS 03/08
	80 g/L SC	Foliar	0.12 0.12 0.12	250 250 250	8 – 13 day interval	0 8 15	7.9 12 0.06	AUS 03/08

Location/ Year/ Variety	Application					PHI (days)	Cypro-conazole (mg/kg)	Study/ Report
	Formulation	Method	Rate (kg ai/ha)	Rate (kg ai/hL) or Volume (L/ha)	Growth Stage (BBCH)			
			0.12	250		19	9.9	
	80 g/L SC + BS1000 (0.15%)	Foliar	0.060 0.060 0.060 0.060	250 250 250 250	7 – 8 day interval	8	5.8	AUS 03/08
Eakley, OK, USA 1987 Spanco	100 g/L SL	Foliar	0.062 0.062 0.062 0.062 0.062 0.062 0.062 0.062	140 140 140 140 140 140 140 140	13 day interval Begin maturity	15	5.4	433018-9
Hartford, Geneva County, AL, USA 1988 Florunner	100 g/L SL	Foliar	0.086 0.086 0.086 0.086 0.086 0.086 0.086 0.086	140 130 140 140 140 140 140 140	13 day interval Begin maturity	12	7.0	433018-9
Suffolk, Suffolk County, VA, USA 1988 NC-7	100 g/L SL	Foliar	0.086 0.086 0.086 0.086 0.086 0.086 0.086 0.086	86 86 86 86 86 86 86 86	-	16	3.6	433018-9
Enfield, Halifax County, NC, USA 1988	100 g/L SL	Foliar	0.086 0.086 0.086 0.086 0.086 0.086 0.086 0.086	200 200 200 200 200 200 200 200	12-17 interval Mature	0	8.3	433018-9
Unesp,Jaboticabal SP, Brazil 1991	100 g/L SL	Foliar	0.10 0.10 0.10	300 300 300	R3 R5 R6	15	< 0.01	RF 105505
	100 g/L SL	Foliar	0.20 0.20 0.20	300 300 300	R3 R5 R6	15	< 0.01	RF 105505

Table 96 Peanut Fodder/Hay

Location/ Year/ Variety	Application					PHI (days)	Cypro-conazole (mg/kg)	Study/ Report
	Formulation	Method	Rate (kg ai/ha)	Rate (kg ai/hL) or Volume (L/ha)	Growth Stage (BBCH)			
Queensland, Site 1, Australia 1990 Menzies	80 g/L SC	Foliar	0.060 0.060 0.060 0.060	250 250 250 250	10 day interval	0 3 6 13 20	28. 15. 6.7 5.3 10	AUS 03/08
	80 g/L SC	Foliar	0.12 0.12 0.12 0.12	250 250 250 250	10 day interval	0 3 6 13 20	68 30 24 22 26	AUS 03/08
	80 g/L SC + BS1000 (0.15%)	Foliar	0.060 0.060 0.060 0.060	250 250 250 250	10 day interval	6	23.	AUS 03/08
Queensland, Site 2, Australia 1990 SO95	80 g/L SC	Foliar	0.060 0.060 0.060 0.060	250 250 250 250	8 – 13 day interval	0 8 15 19	7.1 11 14 9.7	AUS 03/08
	80 g/L SC	Foliar	0.12 0.12 0.12 0.12	250 250 250 250	8 – 13 day interval	0 8 15 19	24 35 < 0.02 (ND) 26	AUS 03/08
	80 g/L SC + BS1000 (0.15%)	Foliar	0.060 0.060 0.060 0.060	250 250 250 250	7 – 8 day interval	8	15	AUS 03/08
Eakley, OK, USA 1987 Spanco	100 g/L SL	Foliar	0.062 0.062 0.062 0.062 0.062 0.062 0.062 0.062	140 140 140 140 140 140 140 140	13 day interval Begin maturity	21	3.1	433018-9
Hartford, Geneva County, AL, USA 1988 Florunner	100 g/L SL	Foliar	0.086 0.086 0.086 0.086 0.086 0.086 0.086 0.086	140 130 140 140 140 140 140 140	13 day interval Begin maturity	19	5.4	433018-9
Suffolk, Suffolk County, VA, USA 1988 NC-7	100 g/L SL	Foliar	0.086 0.086 0.086 0.086 0.086 0.086 0.086 0.086	86 86 86 86 86 86 86 86	-	20	11.	433018-9
Enfield, Halifax County, NC, USA 1988	100 g/L SL	Foliar	0.086 0.086 0.086 0.086	200 200 200 200	12-17 interval	0	14.	433018-9

Location/ Year/ Variety	Application					PHI (days)	Cypro-conazole (mg/kg)	Study/ Report
	Formulation	Method	Rate (kg ai/ha)	Rate (kg ai/hL) or Volume (L/ha)	Growth Stage (BBCH)			
			0.086 0.086 0.086 0.086	200 200 200 200	 Mature			

Farm animal feeding studies

A ruminant feeding study (Oakes, 1994, T021566-04; Blanz, 1995, TDS BS5217; Ali, 1995, TDS DP-391816) and a poultry feeding study (Oakes, 2006, T021566-04) were provided to the Meeting.

Three Friesian dairy cows per treatment group were administered cyproconazole orally for a five-week period at levels of 20, 60, 200 and 600 mg/cow per day, (equivalent to 1, 3, 10 and 30 ppm per day in the diet). The cows were fed twice daily at milking. The dosing period was 35 to 37 days, with the animals sacrificed on the day (16 to 24 hours) after the final dose. Three animals served as controls (no dose) and two additional animals were maintained at the high dose and sacrificed 7 and 14 days after cessation of dosing (day 35) to study depuration. All feed rations were consumed except one cow (10-ppm dose group) which refused about 15% of the feed at two separate feedings (Day 1 p.m. and Day 28 a.m.).

Samples of milk collected in the morning were stored at 4 C until milk was collected in the evening. The morning and evening milk collections were combined and a sample was collected and stored frozen at -20 °C until analysis. In addition, on days 14 and 35 of the study, the composited milk collections were stored overnight at 4 C then separated into cream and skim milk. Cream and skim milk samples were then stored frozen at -20 C until analysis.

The animals were sacrificed after 35-38 days of dosing; sacrifice occurred within 16-24 hours of the final dose. Two cows from the 30-ppm dose group were not sacrificed after the final dose but were instead fed feed ration without added cyproconazole for 7 or 14 days prior to sacrifice to provide withdrawal information. Samples of fat (subcutaneous and perirenal/omental), muscle (composite of pectoralis/adductor thigh muscle) as well as the entire liver and kidneys were collected and stored frozen (-20 C) prior to analysis.

The milk and tissue samples were analysed for residues of cyproconazole using a GC/NPD method. In addition, samples of milk were analysed for metabolites M21a and M36 using an HPLC/UV method, and tissue samples were analysed for metabolite M14 using an HPLC/MS method. Procedural recoveries for M36 at 0.05 and 0.2 mg/kg fortifications in milk averaged 71.2% + 14.8%. See the Analytical Methods section above for a description of the methods.

The results of the feeding study are presented in Table 97. Apparent residues of cyproconazole were non-detectable (< 0.003 ppm) in 15 samples of milk, 1 sample each of skimmed milk and cream, 2 samples of subcutaneous fat, and 3 samples each of peritoneal fat, kidney, liver, and muscle collected from the control cattle. One sample of subcutaneous fat from a control cow bore detectable residues of cyproconazole at 0.003 ppm; no explanation for these detectable residues was provided. Apparent residues of M21a and M36 were non-detectable (< 0.003 ppm each) in 14 samples of milk and 1 sample each of skimmed milk and cream from control cattle. Apparent residues of M14 were non-detectable (< 0.01 ppm) in 3 samples each of kidney and liver from control cattle.

To determine the amount of bound residues of metabolite M14 in liver, samples of liver that had been extracted for analysis for M14 were hydrolysed with 1 N trifluoroacetic acid for 1 hour, cleaned up using the same procedures as for the unhydrolysed samples, and analysed for residues of M14.

Table 97 Residues of cyproconazole and metabolites M14, M21a, and M36 found in dairy cattle matrices following oral administration of the test substance at 1 ppm, 3 ppm, 10 ppm, and 30 ppm for 35-38 consecutive days.

Dosing or Sampling Day	Residues (mg/kg)[a]			
	1 ppm	3 ppm	10 ppm	30 ppm
Cyproconazole in Milk				
7	< 0.003 (3)	< 0.003-0.003 (3)	< 0.003-0.006 (3) Avg 0.004	0.004-0.014 (5) Avg 0.007
14	< 0.003-0.025 (3) Avg 0.009	< 0.003-0.005 (3) Avg 0.004	< 0.003 (3)	< 0.003-0.010 (5) Avg 0.006
21	< 0.003 (3)	< 0.003 (3)	< 0.003 (3)	< 0.003-0.008 (5) Avg 0.005
28	< 0.003 (3)	< 0.003 (3)	< 0.003 (3)	< 0.003-0.007 (5) 0.004
35	< 0.003 (3)	< 0.003 (3)	< 0.003-0.003 (3)	< 0.003-0.007 (5)
42 [b]	--	--	--	< 0.003 (2)
44 [b]	--	--	--	< 0.003
49 [b]	--	--	--	< 0.003
Cyproconazole in Cream				
14	--	--	--	< 0.003-0.008 (5) Avg 0.003
Cyproconazole in Skimmed Milk				
14	--	--	--	< 0.003 (5)
M21a (NOA405870) in Milk				
1	--	--	--	< 0.003-0.006 (5)
2	--	--	--	0.005-0.014 (5)
3	--	--	--	0.006-0.020 (5)
4	--	--	--	0.007-0.020 (4)
5	--	--	--	0.005-0.051 (5)
6	--	--	--	0.006-0.039 (4)
7	< 0.003 (3)	< 0.003-0.003 (3)	0.006-0.008 (3avh 0.007	< 0.003-0.015 (5) Avg 0.010
8	--	--	--	0.005-0.013 (5)
9	--	--	--	0.006-0.016 (4)
10	--	--	--	0.003-0.020 (5)
12	--	--	--	0.004-0.015 (5)
14	< 0.003 (3)	< 0.003 (3)	< 0.003-0.005 (3) Avg 0.003	0.005-0.010 (5) Avg 0.008
18	--	--	--	0.005-0.018 (5)
21	< 0.003-0.003 (3)	< 0.003-0.003 (3)	0.005-0.008 (3) Avg 0.006	0.006-0.020 (5) Avg 0.010
23	--	--	--	0.010-0.018 (5)
25	--	--	--	0.007-0.016 (5)
26	--	--	--	0.006-0.016 (5)
27	--	--	--	0.006-0.020 (5)
28	< 0.003 (3)	< 0.003-0.003 (3)	0.006-0.012 (3) Avg 0.008	0.004-0.014 (5) Avg 0.009
30	--	--	--	0.007-0.019 (5)
32	--	--	--	0.008-0.020 (5)
35	< 0.003-0.004 (3)	< 0.003 (2)	< 0.003-0.005 (3) Avg 0.004	< 0.003-0.023 (5) Avg 0.011
37	--	--	--	0.005, 0.008, [c], 0.005, [b] 0.006 [b],
39 b	--	--	--	< 0.003 (2)
40 b	--	--	--	< 0.003 (2)
42 b	--	--	--	< 0.003 (2)
44 b	--	--	--	< 0.003
45 b	--	--	--	< 0.003
47 b	--	--	--	< 0.003
49 b	--	--	--	< 0.003

Dosing or Sampling Day	Residues (mg/kg)[a]			
	1 ppm	3 ppm	10 ppm	30 ppm
M21a (NOA405870) in Cream				
14	--	--	--	< 0.003 (5)
M21a (NOA405870) Skimmed Milk				
14	--	--	--	0.006-0.028 (5)
M36 (NOA405872) in Milk				
1	--	--	--	< 0.003-0.031 (5)
2	--	--	--	0.073-0.221 (5)
3	--	--	--	0.137-0.334 (5)
4	--	--	--	0.136-0.296 (4)
5	--	--	--	0.138-0.290 (5)
6	--	--	--	0.114-0.321 (4)
7	0.007-0.017 (3) Avg 0.009	0.016-0.025 (3) Avg 0.022	0.079-0.107 (3) Avg 0.093	0.057-0.314 (5) Avg 0.187
8	--	--	--	0.104-0.242 (5)
9	--	--	--	0.133-0.322 (4)
10	--	--	--	0.086-0.254 (5)
12	--	--	--	0.094-0.233 (5)
14	0.006-0.009 (3) Avg 0.007	0.014-0.021 (3) 0.018	0.077-0.095 (3) Avg 0.085	0.118-0.244 (5) Avg 0.177
18	--	--	--	0.124-0.290 (5)
21	0.005-0.011 (3) Avg 0.009	0.007-0.021 (3) 0.015	0.081-0.105 (3) Avg 0.095	0.117-0.278 (5) Avg 0.194
23	--	--	--	0.136-0.265 (5)
25	--	--	--	0.131-0.310 (5)
26	--	--	--	0.120-0.261 (5)
27	--	--	--	0.105-0.380 (5)
28	< 0.003-0.007 (3) Avg 0.004	0.019-0.022 (3) 0.021	0.062-0.123 (3) Avg 0.090	0.104-0.244 (5) Avg 0.191
30	--	--	--	0.109-0.196 (5)
32	--	--	--	0.139-0.336 (5)
35	0.006-0.009 (3) Avg 0.007	0.018, 0.021 Avg 0.020	0.062-0.092 (3) Avg 0.079	0.090-0.309 (5) Avg 0.215
37	--	--	--	0.092, 0.121, [c], 0.035, [b] 0.065, [b],
39 [b]	--	--	--	< 0.003 (2)
40 [b]	--	--	--	< 0.003 (2)
42 [b]	--	--	--	< 0.003 (2)
44 [b]	--	--	--	< 0.003
45 [b]	--	--	--	< 0.003
47 [b]	--	--	--	< 0.003
49 [b]	--	--	--	< 0.003
M36 (NOA405872) in Cream				
14	--	--	--	< 0.003-0.011 (5) Avg 0.004
M36 (NOA405872) in Skimmed Milk				
14	--	--	--	0.061-0.343 (5) Avg 0.230
Cyproconazole in Liver				
35-38	0.066-0.090 (3) Avg 0.082	0.211-0.218 (3) Avg 0.214	0.425-0.604 (3) Avg 0.514	0.563-0.930 (3) Avg 0.748
42 b	--	--	--	0.008
49 b	--	--	--	< 0.003
M14 in Liver [d]				
35-38	0.08-0.16 (3)	0.21-0.55 (3)	0.74-1.06 (3)	1.27-1.68 (3) [0.03-0.04] (3)
42 b	--	--	--	0.06 [< 0.01]
49 b	--	--	--	0.02 [< 0.01]

Dosing or Sampling Day	Residues (mg/kg)[a]			
	1 ppm	3 ppm	10 ppm	30 ppm
Cyproconazole in Kidney				
35-38	< 0.003 (3)	0.005-0.009 (3) Avg 0.007	0.009-0.031 (3) Avg 0.016	0.016-0.038 (3) Avg 0.028
42 [b]	--	--	--	0.009
49 [b]	--	--	--	0.012
M14 in Kidney				
35-38	< 0.01 (3)	< 0.01 (3)	< 0.01-0.02 (3)	0.01-0.03 (3)
42 [b]	--	--	--	< 0.01
49 [b]	--	--	--	< 0.01
Cyproconazole in Muscle				
35-38	< 0.003 (3)	< 0.003 (3)	< 0.003-0.003 (3)	< 0.003-0.005 (3)
42 [b]	--	--	--	< 0.003
49 [b]	--	--	--	< 0.003
Cyproconazole in Subcutaneous Fat				
35-38	< 0.003 (3)	< 0.003-0.003 (3)	0.006-0.012 (3) Avg 0.007	0.004-0.052 (3) Avg 0.022
42 [b]	--	--	--	< 0.003
49 [b]	--	--	--	< 0.003
Cyproconazole in Peritoneal Fat				
35-38	< 0.003 (3)	< 0.003-0.003 (3)	0.010-0.024 (3) Avg 0.017	0.007-0.017 (3) Avg 0.012
42 [b]	--	--	--	< 0.003
49 [b]	--	--	--	< 0.003

[a] Residues of cyproconazole were not corrected for procedural recoveries. Each residue value represents one sample unless otherwise indicated in parentheses.

[b] Dosing ceased on Day 35.

[c] Dosing ceased on Day 36.

[d] Residue values in brackets represent the residues of metabolite M14 found after hydrolysis of extracted samples.

In a newer study (Oakes, 2006, T021565-04), lactating Holstein dairy cows (3 cows/dose group) were dosed orally via capsule once a day for 29-30 days with cyproconazole at 40, 120, and 400 mg ai/cow/day. Based on the average feed consumption, these dose levels were equivalent to dietary concentrations of cyproconazole at 2.4, 6.9 and 22.3 ppm. Milk samples were collected for analysis on Study Days 0, 2, 5, 8, 12, 15, 19, 22, 26 and 29, and samples of liver, kidneys, fat (perirenal, omental and subcutaneous) and muscle (round, tenderloin and diaphragm) were collected from each animal within 24 hours of the final dose. Samples were stored at ≤-10 ºC for up to 8–9 months prior to analysis for cyproconazole, and 6 months prior to analysis of triazole-related residues.

Milk and tissue samples were analysed for residues of cyproconazole (free and conjugated) using a LC-MS/MS method (Syngenta Method RAM 499/01), and samples were analysed for possible residues of triazole, TA, and TAA using another LC-MS/MS method (Morse Method-160). Both methods were adequately validated in conjunction with the analysis of samples from treated livestock. The validated LOQ for cyproconazole is 0.01 ppm in milk and cattle tissues, and the estimated LOD was 0.002 mg/kg. The validated LOQ for each of the triazole-related analytes was 0.01 mg/kg in milk and tissues.

A summary of cyproconazole residues in milk and tissues is presented in Table 98. Note that metabolites (except triazole, see below) were not determined. Cyproconazole residues were < LOQ in all muscle samples from the high-dose group; therefore, residues were not determined in muscle samples from the mid- and low-dose groups.

Table 98 Residues in Milk, Liver, Kidney, Fat, and Muscle from Cattle Feeding Study

Matrix	Feeding Level (ppm)[a]	Cyproconazole Residues (mg/kg)[a]					
		n	Min.	Max.	Median	Mean	Std. Dev.
Milk (5-28 days)	22.3	24	< 0.01	0.02	0.01	0.011	0.003
	6.9	24	< 0.01	< 0.01	0.01	0.010	NA
Liver	22.3	3	0.46	0.46	0.46	0.46	0.0
	6.9	3	0.13	0.22	0.16	0.17	0.046
	2.4	3	0.02	0.08	0.03	0.043	0.032
Kidney	22.3	3	0.03	0.04	0.03	0.037	0.006
	6.9	3	< 0.01	0.01	0.01	0.01	0.0
	2.4	3	< 0.01	< 0.01	0.01	0.01	NA
Fat [b]	22.3	9	0.02	0.05	0.03	0.03	0.011
	6.9	9	< 0.01	0.01	0.01	0.01	0.0
	2.4	6	< 0.01	< 0.01	0.01	0.01	NA
Muscle [b]	22.3	9	< 0.01	< 0.01	0.01	0.01	NA

[a] The LOQ is 0.01 ppm for cyproconazole in each matrix. For calculating the median, mean and standard deviation, the LOQ was used for residue values of <LOQ.

[b] Fat includes separate samples of perirenal, omental, and subcutaneous fat, and muscle includes separate samples of round, tenderloin, and diaphragm muscle.

Triazole-related residues (triazole, TA and TAA) were generally low in milk and tissues from all three dose groups (Table 99).

Table 99 Summary of Triazole, TA (triazole alanine) and TAA (triazole acetic acid) Residue Data from Cattle Study

Matrix	Feeding level (ppm)[a]	n	Triazole (ppm)[b]		TA (ppm)[a]		TAA (ppm)[a]	
			range	mean	range	mean	range	mean
Milk (5-28 days)	22.3	24	< 0.01-0.02	0.014	< 0.01	< 0.01	< 0.01	< 0.01
	6.9	24	< 0.01-0.01	0.01	NA	NA	NA	NA
	2.4	24	< 0.01	0.01	NA	NA	NA	NA
Liver	22.3	3	< 0.01-0.01	0.01	0.03-0.04	0.033	< 0.01	< 0.01
	6.9	3	< 0.01	< 0.01	0.02-0.04	0.030	NA	NA
Kidney	22.3	3	< 0.01-0.01	0.01	< 0.01-0.01	0.010	< 0.01	< 0.01
	6.9	3	< 0.01	< 0.01	< 0.01-0.01	0.010	NA	NA
Fat[b]	22.3	9	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
Muscle[b]	22.3	9	< 0.01	< 0.01	< 0.01-0.02	0.011	< 0.01	< 0.01

[a] The LOQ is 0.01 ppm for each analyte in each matrix. For calculating the mean, the LOQ was used for residue values of <LOQ.

[b] Fat includes separate samples of perirenal, omental, and subcutaneous fat, and muscle includes separate samples of round, tenderloin, and diaphragm muscle.

White Leghorn hens (15 hens/dose group) were dosed orally for 29 days with cyproconazole in the feed at actual concentrations of 0.12, 0.45 and 1.87 ppm (Oakes, 2006, SAN619/8077). Based on the average feed consumption per hen in each group, these nominal feed concentrations were equivalent to doses of 0.02, 0.05, and 0.16 mg of cyproconazole/kg body weight/day. Whole eggs were collected for analysis on Days 0, 1, 3, 6, 9, 13, 16, 20, 23 and 28, and samples of skin with attached fat, peritoneal fat, liver, and muscle (breast + thigh) were collected from each hen within 24 hours of removal from the treated feed. All samples were composited by replicate (5 hens/subgroup) within each dose group and stored at ≤-10 °C for up to 7–8 months prior to analysis for cyproconazole, and 5 months prior to analysis of triazole-related residues.

Egg and tissue samples were analysed for residues of cyproconazole using a LC-MS/MS method (Method RAM 499/01), and samples were analysed for residues of triazole, TA, and TAA using another LC/MS/MS method (Morse Method-160). Both methods were adequately validated in conjunction with the analysis for samples from treated livestock. The validated LOQ is 0.01 mg/kg for cyproconazole in all poultry commodities, and the validated LOQ for each triazole analyte is also 0.01 mg/kg.

Residues of cyproconazole and its possible triazole metabolites (triazole, TA, and TAA) were each < 0.01 mg/kg in all samples of eggs (0-28 days) and tissues from hens dosed at 1.87 ppm in the feed. As residues of each analyte were < LOQ in the high-dose group, egg and tissue samples from the mid- and low-dose groups were not analysed.

Results are summarised in Table 99 and Table 100.

Table 100 Cyproconazole Residue Data from Poultry Feeding Study with Cyproconazole

Nominal Dose Level (ppm)	Matrix	Collection (Study day)	Cyproconazole Residues (mg/kg)[a]
2.0	Eggs (yolk + whites)	0	< 0.01, < 0.01, < 0.01
		1	< 0.01, < 0.01, < 0.01
		3	< 0.01, < 0.01, < 0.01
		6	< 0.01, < 0.01, < 0.01
		9	< 0.01, < 0.01, < 0.01
		13	< 0.01, < 0.01, < 0.01
		16	< 0.01, < 0.01, < 0.01
		20	< 0.01, < 0.01, < 0.01
		23	< 0.01, < 0.01, < 0.01
		28	< 0.01, < 0.01, < 0.01
	Skin + Attached fat	30	< 0.01, < 0.01, < 0.01
	Liver	30	< 0.01, < 0.01, < 0.01
	Peritoneal Fat	30	< 0.01, < 0.01, < 0.01
	Breast+ Thigh Muscle	30	< 0.01, < 0.01, < 0.01

[a]The LOQ is 0.01 ppm for cyproconazole in each matrix.

Table 101 Triazole Metabolite Residue Data from Poultry Feeding Study with Cyproconazole

Feeding Level (ppm)	Matrix	Collection (Study day)	Residues (mg/kg)[a]		
			T	TA	TAA
2.0	Eggs (yolk + whites)	0	< 0.01, < 0.01, < 0.01	< 0.01, < 0.01, < 0.01	< 0.01, < 0.01, < 0.01
		1	< 0.01, < 0.01, < 0.01	< 0.01, < 0.01, < 0.01	< 0.01, < 0.01, < 0.01
		3	< 0.01, < 0.01, < 0.01	< 0.01, < 0.01, < 0.01	< 0.01, < 0.01, < 0.01
		6	< 0.01, < 0.01, < 0.01	< 0.01, < 0.01, < 0.01	< 0.01, < 0.01, < 0.01
		9	< 0.01, < 0.01, < 0.01	< 0.01, < 0.01, < 0.01	< 0.01, < 0.01, < 0.01
		13	< 0.01, < 0.01, < 0.01	< 0.01, < 0.01, < 0.01	< 0.01, < 0.01, < 0.01
		16	< 0.01, < 0.01, < 0.01	< 0.01, < 0.01, < 0.01	< 0.01, < 0.01, < 0.01
		20	< 0.01, < 0.01, < 0.01	< 0.01, < 0.01, < 0.01	< 0.01, < 0.01, < 0.01
		23	< 0.01, < 0.01, < 0.01	< 0.01, < 0.01, < 0.01	< 0.01, < 0.01, < 0.01
		28	< 0.01, < 0.01, < 0.01	< 0.01, < 0.01, < 0.01	< 0.01, < 0.01, < 0.01
	Skin + attached fat	30	< 0.01, < 0.01, < 0.01	< 0.01, < 0.01, < 0.01	< 0.01, < 0.01, < 0.01
	Liver	30	< 0.01, < 0.01, < 0.01	< 0.01, < 0.01, < 0.01	< 0.01, < 0.01, < 0.01
	Peritoneal fat	30	< 0.01, < 0.01, < 0.01	< 0.01, < 0.01, < 0.01	< 0.01, < 0.01, < 0.01
	Breast + Thigh Muscle	30	< 0.01, < 0.01, < 0.01	< 0.01, < 0.01, < 0.01	< 0.01, < 0.01, < 0.01

[a] The LOQ is 0.01 ppm for each triazole metabolite in each matrix.

FATE OF RESIDUES IN STORAGE AND PROCESSING

Nature of Residue During Processing

The hydrolytic stability of cyproconazole was reported (Phaff, 2000, 99RP03) with [triazole-^{14}C] cyproconazole (batch number ILS-243.1B, radiochemical purity 99.1%, specific activity 2.54 MBq/mg). Duplicate sterilised aqueous solutions of the test substance in buffer (3.0 mg/L) at pH 4 (phthalate buffer), pH 5 (acetate buffer) and pH 6 (phosphate buffer) were heated to 90, 100 or 120°C for 20, 60 and 20 minutes, respectively. Samples were taken after incubation and analysed for radioactivity by LSC. Quantification of cyproconazole and degradation products was by HPLC and two-dimensional TLC.

Findings

The mean total recoveries of applied radioactivity ranged from 96.6 to 101.0% at the three pH values. No significant hydrolytic degradation of cyproconazole occurred under any of the conditions tested.

Radioactivity recovered for the heated samples is presented in Table 102

Table 102 Radioactivity recovered following incubation under processing conditions

Simulated	Incubation			Radioactivity recovered (% of applied)[a]		
Process	pH	Time (minutes)	Temp. (°C)	Total	Cyproconazole	Unresolved radioactivity
Pasteurisation	4	20	90	96.6	96.6	0.0
Baking/Brewing/Boiling	5	60	100	101.0	101.0	0.0
Sterilisation	6	20	120	98.5	97.0	1.4[b]

[a] After incubation and neutralisation. Mean of two samples

[b] Consisted of two fractions

Magnitude of the Residue during Processing

The Meeting received reports on processing studies for apples, maize, rape seed (canola), soya bean, and peanuts.

For apples (Ko, 1986, CBK 11037/86016, TDS No. DP304731), two residue field trials were conducted in Elyria, Ohio, USA (trial OH-15-004-85), and in State College, Pennsylvania, USA (trial PA-01-063-85). Cyproconazole formulated as a water dispersible granular formulation containing 100 g ai/kg, was applied to apples during 1984. Seven broadcast foliar applications at 74.1 g ai/ha were made with a 2 weeks interval, the first application made at the pre-blossom stage, which resulted in an exaggerated total application rate of 519g ai/ha.

Mature apples were harvested from control and treated plots about 35 days after the last application. Samples were collected from each of three trees per plot and combined. In the Pennsylvania trial the raw fruit was not collected for analysis.

The composite fruit samples of the replicates were pressed (without washing) by a commercial-like process, and juice and wet pomace were separated. Wet pomace was then dried in a dehydrator at 48.9 °C for 24 hours to produce dry pomace.

All samples were analysed for residues of cyproconazole using method no. CBK 11032/86001 (GC-NPD). The results are summarised in Table 103.

Table 103 Residues of cyproconazole and processing (concentration) factors in apple fruits and processed fractions following foliar application of a WG formulation

Trial	Matrix	Treatment	Cyproconazole residues (mg/kg)	Concentration Factor
OH-15-004-85 (OH)	Fruit	Control	< 0.01	–
	Fruit	7 × 74 g ai/ha	0.092	-

Trial	Matrix	Treatment	Cyproconazole residues (mg/kg)	Concentration Factor
USA	Juice	Control	< 0.01	–
	Juice	7 × 74 g ai/ha	0.031	0.34X
	Wet pomace	Control	0.02	–
	Wet pomace	7 × 74 g ai/ha	0.121	1.32X
	Dry pomace	Control	< 0.01	–
	Dry pomace	7 × 74 g ai/ha	0.237	2.58X
PA-01-063-85 (PA) USA	Fruit	Control	– [a]	–
	Fruit	7 × 74 g ai/ha	– [a]	–
	Juice	Control	< 0.01	–
	Juice	7 × 74 g ai/ha	< 0.01	–
	Wet pomace	Control	< 0.01	–
	Wet pomace	7 × 74 g ai/ha	0.061	–
	Dry pomace	Control	< 0.01	–
	Dry pomace	7 × 74 g ai/ha	0.244	–

[a] Raw fruit samples were not collected for analysis in the Pennsylvania trial.

In two field trials (Oakes, 2006, T002814-03) conducted in IA and WI, USA, during 2004, cyproconazole (100 g/L SC) was applied to maize (field corn) as a single broadcast foliar application at Growth Stage R5 at rates of 0.04 kg ai/ha and 0.20 kg ai/ha (ca 1× and 5× rates). Applications were made using ground equipment at 160 L/ha, and did not include any spray adjuvants. Single control and treated bulk samples of corn grain were harvested from each trial at the proposed PHI (30–31 DAT). The bulk grain samples were cleaned to yield aspirated grain fractions (AGF) and then processed using simulated wet- and dry-milling procedures into starch, grits, meal, flour and refined oil (wet- and dry-milled). Because of compliance monitoring requirements and sample size, the samples were processed by batch rather than continuous, as in commercial operation.

Samples of corn grain, AGF and processed fractions were stored at -20 ºC for up to 9–10 months prior to analysis.

Residues of cyproconazole (free and conjugated) in/on corn grain commodities were determined using a GC/MSD method (Method AM-0842-0790-0), and samples were analysed for possible residues of triazole, TA, and TAA using a LC-MS/MS method (Method Meth-160). The LOQ is 0.01 ppm for each analyte in corn grain, AGF and processed fractions.

Cyproconazole residues in/on corn grain harvested at 30-31 DAT were < 0.01 ppm from both the 1× and 5× rate trials. Cyproconazole residues were also < 0.01 ppm in grits, meal, flour, starch and refined oil; therefore, processing factors could not be calculated for these commodities. Residues were quantifiable in AGF (0.02–0.10 ppm) indicating that cyproconazole concentrated by at least 2×–10× in AGF (average 5.8×).

At both the 1× and 5× application rates, triazole and TAA residues were also < LOQ in corn grain, AGF and all processed fractions; therefore, processing factors could not be calculated for triazole and TAA residues. However, TA residues were quantifiable in corn grain from the 1× rate trials (< 0.01–0.03 ppm) and the 5× rate trials (0.04 ppm). Based on residue data from the trials in which TA residues were >LOQ in grain, TA residues were reduced on average by 0.7× in grits and meal and by < 0.3× in AGF, flour, starch and refined oil.

Results are summarised in Table 104 and Table 105.

Table 104 Cyproconazole Residue Data from Maize Processing Study (USA)

Location (County, State; Year) Trial ID	Commodity	Total Rate (kg ai/ha)	PHI (days)	Cyproconazole Residues (ppm)	Processing Factor
Richland, IA; 2004 5148	Grain (RAC)	0.040	30	< 0.01	NA
		0.20		< 0.01	
	AGF	0.040		0.02	> 2×
		0.20		0.08	> 8×
	Meal	0.040		< 0.01	-
		0.20		< 0.01	-
	Grits	0.040		< 0.01	-
		0.20		< 0.01	-
	Flour	0.040		< 0.01	-
		0.20		< 0.01	-
	Starch	0.040		< 0.01	-
		0.20		< 0.01	-
	Refined Oil (wet- and dry-milled)	0.040		< 0.01, < 0.01	-
		0.20		< 0.01, < 0.01	-
Harvard, WI; 2004 5153	Grain (RAC)	0.039	31	< 0.01	NA
		0.19		< 0.01	
	AGF	0.039		0.03	> 3×
		0.19		0.10	> 10×
	Meal	0.039		< 0.01	-
		0.19		< 0.01	-
	Grits	0.039		< 0.01	-
		0.19		< 0.01	-
	Flour	0.039		< 0.01	-
		0.19		< 0.01	-
	Starch	0.039		< 0.01	-
		0.19		< 0.01	-
	Refined Oil (wet- and dry-milled)	0.039		< 0.01, < 0.01	-
		0.19		< 0.01, < 0.01	-

Table 105 Triazole Alanine Residue Data from Maize Processing Study (USA)

Location (County, State; Year) Trial ID	Commodity	Total Rate (kg ai/ha)	PHI (days)	TA Residues (ppm)	Processing Factor
Richland, IA; 2004 5148	Grain (RAC)	control	30	0.02	
		0.040		0.03	
		0.20		0.04	
	AGF	control		< 0.01	< 0.5×
		0.040		< 0.01	< 0.3×
		0.20		0.01	0.3×

Location (County, State; Year) Trial ID	Commodity	Total Rate (kg ai/ha)	PHI (days)	TA Residues (ppm)	Processing Factor
	Meal	control		0.02	1×
		0.040		0.02	0.7×
		0.20		0.03	0.8×
	Grits	control		0.02	1×
		0.040		0.02	0.7×
		0.20		0.03	0.8×
	Flour	control		< 0.01	< 0.5×
		0.036		< 0.01	< 0.3×
		0.20		0.01	0.3×
	Starch	control		< 0.01	< 0.5×
		0.040		< 0.01	< 0.3×
		0.20		< 0.01	< 0.3×
	Refined Oil (wet- or dry-milled)	control		< 0.01, < 0.1	< 0.5×
		0.040		< 0.01, < 0.1	< 0.3×
		0.20		< 0.01, < 0.1	< 0.3×
Harvard, WI; 2004 5153	Grain	control		0.01	N/A
		0.049		< 0.01	
		0.19		0.04	
	AGF	control		< 0.01	< 1×
		0.049		< 0.01	-
		0.19		< 0.01	< 0.3×
	Meal	control		< 0.01	< 1×
		0.039		< 0.01	-
		0.19		0.02	0.5×
	Grits	control		< 0.01	< 1×
		0.039	30	< 0.01	-
		0.19		0.03	0.8×
	Flour	control		< 0.01	< 1×
		0.039		< 0.01	-
		0.19		< 0.01	< 0.3×
	Starch	control		< 0.01	< 1×
		0.039		< 0.01	-
		0.19		< 0.01	< 0.3×
	Refined Oil (wet- or dry-milled)	control		< 0.01, < 0.1	< 1×
		0.039		< 0.01, < 0.1	-
		0.19		< 0.01, < 0.1	< 0.3×
Richland, IA; 2004 5148	Grain (RAC)	control		0.02	N/A
		0.040		0.03	
		0.20		0.04	
	AGF	control		< 0.01	< 0.5×
		0.040		< 0.01	< 0.3×
		0.20		0.01	0.3×
	Meal	control	30	0.02	1×
		0.040		0.02	0.7×
		0.20		0.03	0.8×
	Grits	control		0.02	1×
		0.040		0.02	0.7×
		0.20		0.03	0.8×

Location (County, State; Year) Trial ID	Commodity	Total Rate (kg ai/ha)	PHI (days)	TA Residues (ppm)	Processing Factor
	Flour	control		< 0.01	< 0.5×
		0.040		< 0.01	< 0.3×
		0.20		0.01	0.3×
	Starch	control		< 0.01	< 0.5×
		0.040		< 0.01	< 0.3×
		0.20		< 0.01	< 0.3×
	Refined Oil (wet- or dry-milled)	control		< 0.01, < 0.1	< 0.5×
		0.040		< 0.01, < 0.1	< 0.3×
		0.20		< 0.01, < 0.1	< 0.3×
Harvard, WI; 2004 5153	Grain	control		0.01	N/A
		0.039		< 0.01	
		0.19		0.04	
	AGF	control		< 0.01	< 1×
		0.039		< 0.01	-
		0.19		< 0.01	< 0.3×
	Meal	control		< 0.01	< 1×
		0.039		< 0.01	-
		0.19		0.02	0.5×
	Grits	control		< 0.01	< 1×
		0.039	30	< 0.01	-
		0.19		0.03	0.8×
	Flour	control		< 0.01	< 1×
		0.039		< 0.01	-
		0.19		< 0.01	< 0.3×
	Starch	control		< 0.01	< 1×
		0.039		< 0.01	-
		0.19		< 0.01	< 0.3×
	Refined Oil (wet- or dry-milled)	control		< 0.01, < 0.1	< 1×
		0.039		< 0.01, < 0.1	-
		0.19		< 0.01, < 0.1	< 0.3×

One residue harvest field trial (Heillaut, 2007, T000677-06-REG) was conducted in Switzerland during 2006, in which a suspension concentrate (SC) containing 200 g/L azoxystrobin and 80 g/L cyproconazole was applied to oilseed rape (canola). Two applications were made to the treated plot, at a rate of 240 g ai/ha cyproconazole each (3 × GAP rate), separated by an interval of 38 days and with the final application at BBCH growth stage 85–87. At normal commercial harvest (BBCH growth stage 89), seven days after the second application, samples of treated and untreated oilseed rape seeds were harvested and transported at ambient temperature to the processing facility. Further control and treated subsamples were shipped by freezer truck at or below -18 °C to the analytical laboratory. One untreated (40 kg) and one treated (150 kg) sample were received by the processing facility. Immediately after receipt, the specimens were stored at or below -18 °C until processing.

Before the start of processing, the deep-frozen field specimens were defrosted at room temperature. Immediately after defrosting, and prior to their subsequent separation into the various processing specimens, two control and four treated oilseed rape seed sub-samples were taken. The treated sub-samples were used for the one balance and three follow-up processes.

Following separation, the seeds had to be conditioned by drying, as the moisture content was outside the optimal range of 6–10% for pressing. Following conditioning, the seeds were manually

cleaned to remove any fragments of coarse stalks and weed seeds. Two cleaned seed sub-specimens were taken for the balance and the three follow-up processes.

Relevant industrial practices and standardized procedures were applied, to simulate commercial practice.

During the pressing of the oilseed rape, the seed was separated into a liquid phase (crude oil) and a solid phase (press cake) using a heated press head. Two press cake and two crude oil sub-specimens were taken from the balance processing. Two crude oil sub-specimens were taken from the three follow-up processes.

The first extraction step started with addition of n-hexane (solvent) to the press cake (2 hours at approximately 60 °C). After circulating, the solvent-oil mixture (miscella) was pumped into a distillation vessel (distillation conditions 39–63 °C at a pressure of 440–450 mbar). After the first distillation, the extracted oil remained in the distillation vessel and the distilled n-hexane was transferred back to the press cake for the second extraction step, with addition of fresh n-hexane. The remainder of the solvent from the extracted oil (after the end of the miscella distillation) was removed using a rotary evaporator (70–74 °C).

Two extracted oil sub-specimens and two solvent-extracted meal sub-specimens were taken from the balance processing. Two extracted oil sub-specimens were taken from each of the three follow-up processes.

After the relevant oil specimens for analysis had been taken from the pressed crude oil and the solvent extracted oil, the remainder of the pressed crude oil and solvent extracted oil were mixed together to give a "combined oil" sample for refining. The "combined oil" specimens had the same ratio of pressed oil to extracted oil based on the initial weights obtained for these fractions prior to the relevant specimens being taken from each of these individual fractions.

Two "combined oil" sub-specimens were taken from the balance and each of the three follow-up processes. Before hydration, the combined oil was filtered using a vacuum pump and a suction filter.

Refining consisted of hydration, desliming, neutralization, washing, drying, bleaching, filtration and deodorization..

All samples were analysed for residues of cyproconazole using analytical method RAM 397/02. Samples were extracted using an acetone/water mixture (95:5, v/v), diluted with acetone/water mixture (20:80, v/v) and filtered on Acrodisc GHP. Cyproconazole was determined by HPLC-MS/MS. The limit of quantification for the method was 0.01 mg/kg. Results are summarised in Table 106.

Table 106 Cyproconazole residue data from rape seed processing study (Switzerland)

Trial No CH-FR-06-0126 Sample No CH-FR-06-0126-	Event	Commodity	Residue of Cyproconazole (mg/kg; uncorrected)
Field samples			
A-0002	7 DALA (NCH)	Seeds	0.12
Seeds after defrosting before separation to processing samples			
A-0003	After defrosting	Seeds	0.15
A-0004	After defrosting	Seeds	0.13
A-0005	After defrosting	Seeds	0.14
A-0006	After defrosting	Seeds	0.14
Processed samples - Balance study			
B-0016	After defrosting	Seeds	0.12
B-0018	After cleaning	Seeds	0.07
B-0020	After pressing	Press cake	0.10
B-0022	After pressing	Crude oil	0.10
B-0024	After extraction	Extracted oil	0.30
B-0057	After combining oil	Oil-combined	0.21
B-0026	After extraction	Solvent-extracted meal	0.03

Trial No CH-FR-06-0126 Sample No CH-FR-06-0126-	Event	Commodity	Residue of Cyproconazole (mg/kg; uncorrected)
B-0028	During refining - after neutralisation	Soap stock	0.05
B-0029	After refining	Refined oil	< 0.01
Follow-up F1			
F1-0031	After defrosting	Seeds	0.12
F1-0033	After cleaning	Seeds	0.08
F1-0035	After pressing	Crude oil	0.23
F1-0059	After extraction	Extracted oil	0.48
F1-0061	After combining oil	Oil-combined	0.27
F1-0037	After refining	Refined oil	< 0.01
Follow-up F2			
F2-0039	After defrosting	Seeds	0.13
F2-0041	After cleaning	Seeds	0.08
F2-0043	After pressing	Crude oil	0.09
F2-0063	After extraction	Extracted oil	0.51
F2-0065	After combining oil	Oil-combined	0.19
F2-0045	After refining	Refined oil	< 0.01
Follow-up F3			
F3-0047	2x240	After defrosting	Seeds
F3-0049	2x240	After cleaning	Seeds
F3-0051	2x240	After pressing	Crude oil
F3-0067	2x240	After extraction	Extracted oil
F3-0069	2x240	After combining oil	Oil-combined
F3-0053	2x240	After refining	Refined oil

DALA: Days After Last Application

NCH: Normal Commercial Harvest

The transfer (processing) factors for the one balance and three follow-up studies were calculated separately for each of the processing trials, as indicated in Table 107.

Table 107 Summary of processing factors for rape seed

Processed fraction	Transfer factor (single values)	Mean/Median transfer factor
Press cake	0.83	0.83/0.83
Crude oil	0.83, 1.92, 0.69, 0.90	1.09/0.86
Extracted oil	2.50, 4.00, 3.92, 4.40	3.71/3.96
Oil-combined	1.75, 2.25, 1.46, 1.70	1.79/1.72
Solvent-extracted meal	0.25	0.25/0..25
Soap stock	0.42	0.42/0.25
Refined oil	0.08, 0.08, 0.08, 0.10	0.08/0.08

Two residue field trials were conducted in Iowa and Kansas, USA, during 2004, in which a soluble concentrate (SL) containing 100 g cyproconazole per litre, was applied to soya beans (Oakes, 2006, Report Number T002037-03, Syngenta File No. SAN619/8079). Two applications were made with an interval of 14 days at rates of 40 g ai/ha (1× treatment) and 200 g ai/ha (5× treatment). Thirty days after the last application dried seed samples of treated and untreated soya beans were harvested and shipped at ambient temperature to the processing facility.

Approximately 227 kg of mature dried seed from both replicates of each treatment were composited to produce a sample weighing approximately 454 kg per composite sample. The dried seed samples were processed into meal, hulls, and refined oil. Aspirated grain fractions (AGFs) were also generated during the processing phase.

After determining the moisture content of the incoming maize (RAC), the samples were dried in a Proctor Schwartz oven at 43.3–57.2 °C until the moisture content was 10–13%.

After drying, the samples were placed in a dust generation room containing a holding bin, drag conveyors, and a bucket conveyor. As the sample was moved in the system, aspiration was used to remove light impurities (grain dust). The sample was moved for 120 minutes. Run times varied based on the type of sample and amount used. The light impurities were classified by sieving. After classification of each sample, all the material through the 2360 micron sieve was recombined to produce one aspirated grain fraction. A representative sample was removed and the ash content was determined (according to AOCS Method Ba 5a-49).

Because of compliance monitoring requirements and sample size, the samples were processed by batch rather than continuous, as in commercial operation.

For processing into oil, the whole soya bean sample was dried in a Proctor Schwartz oven between 54.4–71.1 °C. The final moisture content after drying was 7–10%. The light impurities were separated using a Kice aspirator. After aspiration, the sample was screened in a Vac-Away two screen cleaner. Large and small foreign particles (screenings) were separated from the soya bean.

The whole soya bean was fed into a Bauer disc mill to crack the hull and liberate the kernel. After hulling, the material was passed through the Kice aspirator to separate the hull and kernel material.

The kernel material was heated to 71.1–79.4 °C. The heated kernel material was flaked in a Ferrell-Ross flaking roll with a gap setting of 0.0203–0.0305 cm. The flaked kernel material was fed into an Read Corporation expander/extruder. As the material moved through the expander, steam was injected directly onto the product. Exiting temperature range of the material (collets) was 76.7–112.8 °C. After expansion, the collets were dried in a Proctor Schwartz oven for 30–40 minutes at 54.4–71.1 °C and promptly taken to solvent extraction.

The collets were placed in stainless steel batch extractors and submerged in 48.9–60 °C solvent (hexane). After 30 minutes, the hexane was drained and fresh hexane is added to repeat the cycle two more times. The final two washings were for 15-30 minutes each. Following the final draining, warm air was forced through the spent collets to remove residual hexane. At the request of the sponsor, the collets were toasted as opposed to forcing warm air through them.

The miscella (crude oil and hexane) was passed through a Precision Scientific Recovery unit to separate the crude oil and hexane. Crude oil was then heated to 72.8–90 °C for hexane removal. The crude oil was refined (AOCS Method Ca 9b-52), and the refined oil and soapstock were separated.

The soya beans were processed in a way that simulates industrial practice as closely as possible, but the samples were processed by batch rather than continuous, as in commercial operation.

All samples were analysed for residues of cyproconazole using method no. AM-0842-0790-0.

Results of the aspirated grain dust generation are provided in Table 108. Results of the processing are given in Table 109.

Table 108 Aspirated grain dust from soya beans treated with cyproconazole (USA)

Trial	Matrix	Treatment	Cyproconazole residues [mg/kg]	Concentration Factor
NE-FR-04-5104	Seed	Control	< 0.01	–
	Seed	1×	0.02	–
	Seed	5×	0.12	–
	AGF	Control	< 0.01	–
	AGF	1×	0.46	23×
	AGF	5×	2.2	18×
ND-FR-04-5112	Seed	Control	< 0.01	–

Trial	Matrix	Treatment	Cyproconazole residues [mg/kg]	Concentration Factor
	Seed	1×	0.03	–
	Seed	5×	0.12	–
	AGF	Control	< 0.01	–
	AGF	1×	0.22	7×
	AGF	5×	0.89	7×

Table 109 Processing of soya beans (USA)

Trial	Matrix	Treatment	Cyproconazole residues [mg/kg]	Transfer (Processing) Factor
NE-FR-04-5104	Seed	Control	< 0.01	–
	Seed	1×	0.02	–
	Seed	5×	0.12	–
	Meal	Control	< 0.01	–
	Meal	1×	0.02	1×
	Meal	5×	0.06	0. 5×
	Hulls	Control	< 0.01	–
	Hulls	1×	0.02	1×
	Hulls	5×	0.09	0.75×
	Refined oil	Control	< 0.01	–
	Refined oil	1×	0.05	2. 5×
	Refined oil	5×	0.20	1.7×
ND-FR-04-5112	Seed	Control	< 0.01	–
	Seed	1×	0.03	–
	Seed	5×	0.12	–
	Meal	Control	< 0.01	–
	Meal	1×	0.02	0.67×
	Meal	5×	0.07	0.60×
	Hulls	Control	< 0.01	–
	Hulls	1×	0.02	0.67×
	Hulls	5×	0.09	0.75×
	Refined oil	Control	< 0.01	–
	Refined oil	1×	0.01	0.3×
	Refined oil	5×	0.22	1.8×
Summary				
	Meal			1, 0.5, 0.67, 0.60 Avg 0.69 Med 0.64
	Hulls			1, 0.75, 0.67, 0.75 Avg 0.79 Med 0.75
	Refined Oil			2.5, 1.7, 0.3, 1.8 Avg 1.6 Med 1.8

One residue field trial was conducted in Vienna Dooly County, Georgia 31092, USA, in which cyproconazole formulated as 100 g/L SL (soluble concentrate) and 25 g/kg G (granular) was applied to peanuts during 2004 (Ali, 1991, SAN619/10061). Eight broadcast foliar applications at 259 g ai/ha were made with a 2 weeks interval using the SL-formulation, with an additional single soil application at 1480 g ai/ha at pegging stage using the G-formulation, which resulted in a total application rate of 3552 g ai/ha. This represents a 5× exaggeration of the GAP proposed at that time, but never finalized.

Vines, hay and nuts were harvested from control and treated plots 30 days after the last application and shipped frozen to the processing facility.

The peanuts were dried in a Proctor-Schwartz forced air oven at 60–71.1 °C until the moisture of the kernel was less than 10%. The samples were cleaned by aspiration and/or screening. The peanuts were dehulled with a peanut sheller, and the hull and kernel fractions were separated by aspiration.

The moisture content of the kernels was adjusted to 10%. The kernels were cooked until the temperature reached 93.3–104.4 °C. The oil was then mechanically removed with an Anderson expeller.

The residual oil in the presscake was extracted with hexane in a steam-jacketed stainless steel batch extractor at 48.9–60 °C. The solvent was drained and warm air forced through the presscake for de-solventisation.

The miscella (crude oil and hexane) was passed through a Precision Laboratory Evaporator at 72.8–85 °C. The reclaimed hexane was disposed of.

After determining the percent free fatty acids in the crude oil (AOCS Method Ca5a-40), a weighed sample of crude oil was placed in an Oil Refining Machine and 16 degree Baume NaOH was added. The solution was mixed for 30 minutes at 250 RPM at 20–23.9 °C, and then for an additional 12 minutes at 70 RPM at 62.8–67.2 °C. The neutralised oil was allowed to settle at 60–95 °C for one hour and the oil solution was then refrigerated overnight. The neutralised oil was decanted and filtered. The fraction settling to the bottom of the refrigerated container was the soapstock.

All samples were received frozen at the analytical laboratory and were analysed for residues of cyproconazole using method no. AM-0842-0790-0. Results are summarised in Table 110.

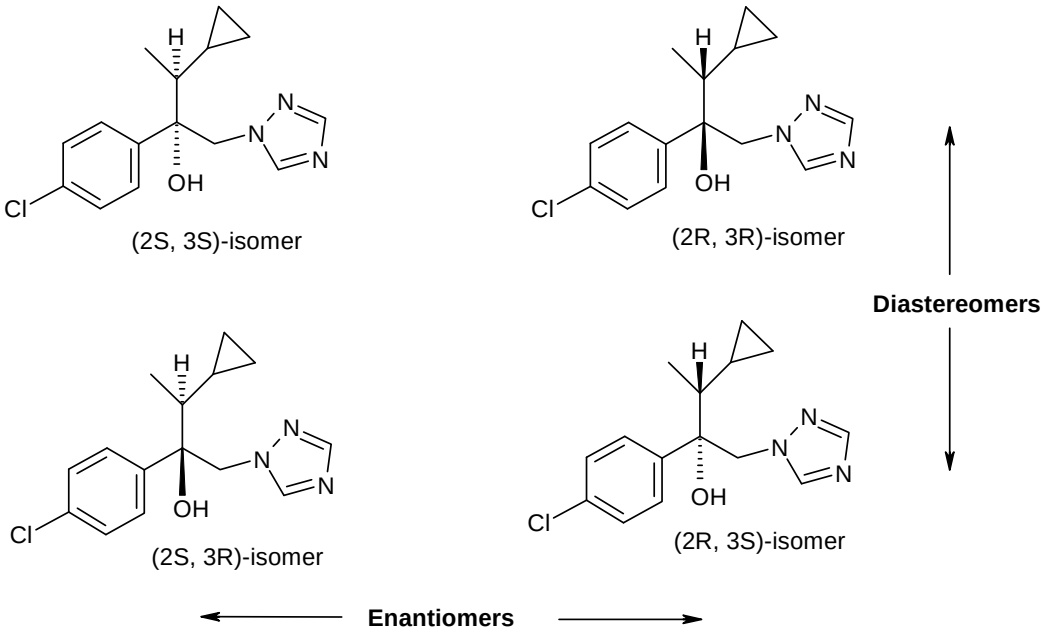
Table 110 Transfer (Processing) Factors from the processing of peanuts treated with cyproconazole (US)

Matrix	Treatment	Cyproconazole residues [mg/kg]	Concentration Factor
Hulls	Control	< 0.01	–
Hulls	Treated (5×)	1.97[a]	–
Kernels	Control	< 0.01	–
Kernels	Treated (5×)	0.28	–
Crude oil	Control	< 0.01	–
Crude oil	Treated (5×)	0.50	1.79×
Solvent extracted crude oil	Control	0.085[a]	–
Solvent extracted crude oil	Treated (5×)	0.50	1.79×
Press cake (meal)	Control	< 0.01	–
Press cake (meal)	Treated (5×)	0.14	0.50×
Solvent extracted press cake	Control	< 0.01	–
Solvent extracted press cake	Treated (5×)	0.08	0.29×
Refined oil	Control	< 0.01	–
Refined oil	Treated (5×)	0.47	1.68×
Soapstock	Control	< 0.01	–
Soapstock	Treated (5×)	0.76	2.71×

[a] Whole peanuts (hull + kernel) were not analysed. Therefore, a transfer factor for hulls cannot be determined.

APPRAISAL

Cyproconazole is an azole fungicide used to control a wide range of fungi on cereal crops, coffee, sugar beet, fruit trees, grapes, including rust on cereal crops, powdery mildew on cereal crops, fruit tree and grapes, and scab on apple. It was considered for the first time by the 2010 JMPR. Cyproconazole is an approximately 1:1 mixture of two diastereomers, each of which is exactly a 1:1 mixture of the enantiomers. All four stereoisomers are present in similar amounts.



The manufacturer submitted studies on physical and chemical properties, animal and plant metabolism, environmental fate in soil, rotational crops, analytical methods, freezer storage stability, use patterns, supervised field trials on plants, processing, and residues in animal commodities (livestock feeding). Japan and the Netherlands also submitted use pattern information.

List of metabolites and degradation products

CODE OR COMMON NAME	CHEMICAL NAME
Cyproconazole (CGA 221949) M1/M2	α-(4-chlorophenyl)-α-(1-cyclopropylethyl)-1*H*-1,2,4-triazole-1-ethanol
M9/M14 NOA 421153	2-(4-chlorophenyl)-3-cyclopropyl-1-[1,2,4]triazol-1-yl-butane-2,3-diol
M11/M18 NOA 421154	3-(4-chlorophenyl)-2-cyclopropyl-4-[1,2,4]triazol-1-yl-butane-1,3-diol
M10/M10a NOA 452154	3-(4-chlorophenyl)-2-cyclopropyl-3-hydroxy-4-[1,2,4]triazol-1-yl-butyric acid
M15 NOA 408616	1-(4-chlorophenyl)-2-[1,2,4]triazol-1-yl-ethanol
M16 CGA 123420	1-(4-chlorophenyl)-2-[1,2,4]triazol-1-yl-ethanone
M21/M21a NOA 405870	5-(4-chlorophenyl)-5-hydroxy-4-methyl-6-[1,2,4]triazol-1-yl-hex-2-enoic acid
M36 NOA 405872	δ-(4-chlorophenyl)-β,δ-dihydroxy-γ-methyl-1H-1,2,4-triazole-1-hexenoic acid

CODE OR COMMON NAME	CHEMICAL NAME
M31/M48 NOA 410714	2-chloro-5-(2-cyclopropyl-1-hydroxy-1-[1,2,4]triazol-1-ylmethyl-propyl)-phenol
M38 NOA 421155	1-[2-(4-chlorophenyl)-3-cyclopropyl-but-1-enyl]-1H-[1,2,4]triazole
M39 CGA 131013	3-(1H-1,2,4-triazol-1-yl)-alanine
M41 (C3/C5)	glucoside of 3-(4-chlorophenyl)-2-cyclopropyl)-4-(1H-1,2,4-triazol-1-yl)-1,3-butanediol
M42	glucoside of 2-(4-chlorophenyl)-3-cyclopropyl-1-(1H-1,2,4-triazol-1-yl)-2,3-butanediol
M43	glucoside of α-(4-chlorophenyl)-α-[1-(2-hydroxycyclopropyl)ethyl]-1H-1,2,4-triazole-1-ethanol
M44/M45 (C4)	glucosides of α-(4-chloro3-hydroxyphenyl)-α-(1-cyclopropylethyl)-1H-1,2,4-triazole-1-ethanol
M46	malonic acid conjugate of M42
M47	malonic acid conjugate of M41
M50	Sulfuric acid mono-[1-(4-chlorophenyl)-2-[1,2,4]triazol-1-yl-ethyl] ester
M51	Sulfuric acid mono-[3-(4-chlorophenyl)-2-cyclopropyl-2,3-dihydroxy-4-[1,2,4]triazol-1-yl-butyl] ester
M52 (M54) [stereoisomer of either M52 or M53]	Sulfuric acid mono-[3-(4-chlorophenyl)-2-cyclopropyl-3-hydroxy-4-[1,2,4]triazol-1-yl-butyl] ester
M53 (M54) [stereoisomer of either M52 or M53]	Sulfuric acid mono-[3-(4-chlorophenyl)-2-cyclopropyl-3-hydroxy-4-[1,2,4]triazol-1-yl-butyl] ester
M55 SYN 533911/SYN 533912	5-chloro-2-(1-hydroxy-2-4-[1,2,4]triazol-1-yl-ethyl) phenol or 2-chloro-5-(1-hydroxy-2-4-[1,2,4]triazol-1-yl-ethyl) phenol
M56 SYN 533921	5-[1-(4-chlorophenyl)-1-hydroxy-2-[1,2,4]triazol-1-yl-ethyl]-4-hydroxy-5-methyl-dihydro-furan-2-one
M57 NOA 405870	(E)-5-(4-chlorophenyl)-4,5-dihydroxy-4-methyl-6-[1,2,4]triazol-1-yl-hex-2-enoic acid
M58 CGA 155705	4-chlorobenozic acid
M59	2-(4-chlorophenyl)-3-methyl-1-[1,2,4]triazol-1-yl-pentane-2,4-diol

CODE OR COMMON NAME	CHEMICAL NAME
CGA71019 1,2,4-Triazole	1H-1,2,4-triazole
CGA142856 Triazole acetic acid	1,2,4-triazol-1-yl-acetic acid

Animal metabolism

The Meeting received animal metabolism studies with cyproconazole in rats, lactating goats and laying hens. The metabolism and distribution of cyproconazole in animals was investigated using the [α-carbon^{14}C]-cyproconazole in goats, hens, and rats and the [U-^{14}C-phenyl]-cyproconazole in hens. The rat studies are addressed in the Toxicology section of the Report.

Three lactating goat metabolism studies were provided in which goats were dosed with [α-carbon^{14}C]-Cyproconazole for 12 consecutive days at 1 ppm in the diet, for three consecutive days at 30 ppm in the diet, or for four consecutive days at 10 ppm in the diet. Most (> 85% TRR) of the radioactivity was extractable in milk and tissues. The TRR levels were low in muscle (about 0.01 mg/kg). Cyproconazole was a major component of the residue in liver (20% TRR), fat (27–47% TRR), kidney (24–32% TRR, of which up to 24% conjugated), and muscle (11% TRR), but minor in milk (0–9% TRR). The major metabolites in milk were NOA405872 (M36) (47–68% TRR) and NOA405870 (M21) (17–30% TRR), both of which are carboxylic acid derivatives. NOA452154 was a minor metabolite (8% TRR) in milk. Significant metabolites in liver were NOA421153/M9/M14 (27–27% TRR), NOA421155/M38 (4–16% TRR), and NOA421154/M10 (9–12% TRR). A significant metabolite in fat was NOA421155/M38 (11–36% TRR). In kidney, NOA405872/M36 was significant (12% TRR) in one study. Trace amounts of NOA408616/M15 (about 1% TRR) were found in liver, kidney, fat, and muscle, and slightly higher levels of the corresponding ketone CGA123420/M16 (1–4% TRR) were found in the same tissues.

Taken together, the studies show that cyproconazole is metabolized in goats via:

- Oxidation of the carbon bearing the methyl and cyclopropyl rings to form M14 (NOA421153);
- Oxidation of the methyl group to form M18/M11 (NOA421154) and to M10 (NOA452154);
- Elimination-reduction or removal of the cyclopropyl side chain to form M15 (NOA408616) and subsequent oxidation to the corresponding ketone M16C (GA123420) (minor);
- Elimination of water to form M38 (NOA421155);
- Oxidative opening of the cyclopropyl ring to form M36 (NOA405872) and subsequent dehydration to form M21 (NOA405870) and elimination into milk;
- Glucuronide and/or sulfate conjugation of cyproconazole (minor, except kidney, where it is 5× cyproconazole concentration).

The metabolic fate of cyproconazole was investigated in laying hens using [α-^{14}C]-cyproconazole (1 ppm for 3 days) and [U-^{14}C-phenyl]-cyproconazole (114 ppm for 4 days). Cyproconazole was a major part of the TRR in all matrices: 4% (TRR 0.07 mg/kg)–40% (TRR 3 mg/kg), muscle, 41%–67% fat, 4%–38% liver, 10–30% egg whites, 22–50% egg yolks. Conjugated cyproconazole was about 12% of the free cyproconazole concentration in eggs. NOA421153 (M9/M14) was a major metabolite in muscle (20–31% TRR), fat (15–37% TRR), liver (20–38% TRR), egg whites (35–44% TRR), and egg yolks (14–28% TRR). NOA408616 (M15) was significant

in muscle (14–46% TRR), liver (10–22% TRR), egg whites (18–36% TRR), and egg yolks (4–10% TRR).

The metabolism of cyproconazole in poultry proceeds predominantly via hydroxylation, oxidation and elimination reactions. Parent compound was a major component in eggs and all tissues. The major metabolites in eggs and tissues resulted from either (i) hydroxylation of the carbon bearing the cyclopropyl group (M9 and M14) or (ii) elimination of the methyl-cyclopropyl side chain (M16) followed by reduction (M15). Hydroxylation of the methyl group (M11 and M18) was also a route of metabolism.

The metabolism of cyproconazole is qualitatively similar in ruminants and poultry. The major routes of metabolism involved either hydroxylation of the carbon bearing the cyclopropyl group to form M9 /M14 or elimination of the methyl-cyclopropyl side chain (M16) followed by reduction (M15). Hydroxylation of the methyl group (M11 and M18) was also a major route of metabolism, as was opening and modification of the cyclopropyl ring (M21, M36, M56, M57, and M59). The data (ruminant faces with NOA 421152 or M3/M4)) indicate that there is only limited cleavage of the triazole ring and that the majority of residues retain the intact phenyl and triazole rings.

Metabolites found in the ruminant and poultry metabolism studies in edible tissues, eggs, and milk were also found in the rat metabolism study. Among the more prominent fractions in urine were NOA421152 (M3 & M4), NOA408616, NOA421154 (M18) and NOA452669 (M30/33). In faeces, NOA421152 (M3 & M4) and NOA421153 (M14) were the major metabolites beside parent. Further metabolites at significant amounts were NOA421152 (M4), NOA421153 (M9), NOA452154, NOA451353, NOA421154 (M18), and NOA452668.

Cyproconazole plant metabolism studies were considered for peanut, grape, apples, sugar beet, and wheat. The peanut study does not meet the needs of a metabolism study. Peanut vines in a glasshouse were painted with an EC formulation of [α-carbon^{14}C]-Cyproconazole and harvested 3 to 6 weeks later. The foliage contained cyproconazole (30–40% applied radioactivity) and very small amounts (1–2%) of M9/M14 and M18.

Grapes vines were treated with [α-carbon^{14}C]-Cyproconazole, and grape fruits were harvested 29 days after the last application. A portion (28% TRR) of the residue was removed by surface wash, and an additional 56% TRR was solvent extractable. The major component of the residue was cyproconazole (63% TRR). Identified metabolites were all < 2% TRR, e.g., M9/M14 and M13.

Apple trees were foliarly treated 4 times at 2 week intervals with [α-carbon^{14}C]-Cyproconazole. Apples were harvested 28 days after the last treatment. Surface residue was 17% TRR. About 60% TRR was associated with the washed fruit. Cyproconazole made up 76% of the whole fruit TRR. No metabolite exceeded 3% TRR, e.g., M9/M14 and M13 at 2.8% TRR.

[Phenyl(U)-^{14}C]-cyproconazole, and [U-triazole^{14}C]-cyproconazole were applied in separate studies in two applications at rates of 160 – 200 g ai/ha/application to wheat plants. Cyproconazole was the major component of the TRR for both labels for livestock commodities: 44% straw; 60–72% forage. Cyproconazole composed 5–45% TRR in grain. M39 (triazole alanine) was a significant grain metabolite for the [U-triazole^{14}C]cyproconazole (63% TRR), as was M9/M14 for the [Phenyl(U)-^{14}C]-cyproconazole (14% TRR).

The metabolism of [U-triazole^{14}C]-cyproconazole in sugar beets revealed that cyproconazole is the major portion of the TRR (80% roots; 76% leaves). M9/M14 comprised 2.5% TRR in leaves and 4% TRR in roots.

In all studies, levels of cyproconazole conjugates, as released by acid hydrolysis, were generally ≤ 5% TRR.

The metabolism of cyproconazole in the various plants studied is qualitatively similar. Generally cyproconazole is the major portion of the residue. The metabolism of cyproconazole in plants involves (i) hydroxylation of the methyl- and cyclopropyl-substituted carbon to form Metabolites M9/M14; (ii) oxidation of the methyl group to form Metabolites M11/M18; (iii) elimination of the cyclopropyl-substituted carbon to form the benzylic alcohol (M15) and further

oxidation to the ketone (M16); (iv) hydroxylation of the cyclopropyl ring and the phenyl ring; (v) conjugation of parent and hydroxylated metabolites to form various glycosides; and (vi) oxidative elimination of the triazole ring and its subsequent conversion to triazole alanine.

Plant metabolites were also metabolites of the rat metabolism, with the exception of M39/CGA 131013 (triazolyl-alanine).

Environmental fate

Under aerobic conditions in soil cyproconazole is moderately stable. Cyproconazole ([U-triazole^{14}C]-cyproconazole) has a half-life of about 150 days, with 1,2,4-triazole and 1,2,4-tgriazol-1-yl acetic acid at about 25% of the applied radioactivity at 140 days. The half-life (first order kinetics) varied from 100 days to > 1 year with ^{14}C-benzyl cyproconazole and is about 100 days with [Phenyl(U)-^{14}C]-cyproconazole. Mineralization to carbon dioxide varied from 2% to 33% over 112 days. No other degradates were identified.

Cyproconazole is photolytically stable in soil, with no loss after 30 days of irradiation.

Cyproconazole is hydrolytically stable at pHs of 4, 5, 7, and 9 for 5 days at 50 ºC.

Residues in succeeding crops

Residues of cyproconazole are found in succeeding crops in confined rotational crop situations. Studies were reported for the application of [α-^{14}C]cyproconazole to soil followed by the planting of representative crops. [U-triazole^{14}C]cyproconazole was not studied. Cyproconazole is the major component of the residue, and TRRs typically range from < 0.01 mg/kg to 0.44 mg/kg with a 30 day plantback interval (PBI). Following a soil application of [α-^{14}C]cyproconazole at 010 kg ai/ha, rotational crops were planted at intervals (PBI) of 30 and 90 days. Cyproconazole ranged from 33% TRR (0.003 mg/kg) in wheat grain and 39% TRR (0.036 mg/kg) in sugar beet tops to 72% TRR (0.029 mg/kg) in lettuce leaves at a 30 day PBI. Metabolites detected (2–12% TRR) included M9/M14 and M18.

In field crop rotational studies following 7 applications of cyproconazole, each about 0.10 kg ai/ha (0.7 kg ai/ha total), cyproconazole residues at a 30 day PBI were < 0.01 mg/kg in wheat grain and carrot tops, 0.034 in collard greens, 0.081mg/kg in wheat straw, 0.021 mg/kg in radish root and carrot root, 0.062 mg/kg in radish tops, and 0.13 mg/kg in mustard greens. All residues were < 0.01 mg/kg at 1 year PBI. In trials following a single application at 0.082 kg ai/ha (typical 1× for primary crops), cyproconazole was < 0.01 mg/kg in spinach, radish (root and top), and wheat at a 60 day PBI.

The Meeting concluded that residues of cyproconazole in rotational crops with a minimum plantback interval of 30 days may be possible, but residues would be at or near the LOQ of the analytical method, 0.01 mg/kg. This is based on primary crop use patterns under consideration.

Methods of analysis

Adequate analytical methods exist for the determination of cyproconazole residues for data collection and enforcement purposes in both plant and livestock matrices. Early methods for crop matrices involved an optional acid hydrolysis (1 N HCl) to release cyproconazole conjugates, extraction, clean-up, and analysis by GC with NPD, ECD, or MSD. The methods determined cyproconazole only with an LOQ of 0.01–0.04 mg/kg. These methods have been validated via the analysis of spiked samples and include an independent laboratory validation for the MSD variation.

An HPLC/UV method was described for data collection for wheat commodities. Samples are extracted with aqueous methanol, cleaned-up with SPE, and analysed by HPLC with UV detection. The limit of quantitation was 0.01–0.02 mg/kg.

More recently an HPLC-MS/MS method has been developed for plant matrices. Homogenized samples are extracted with aqueous acetonitrile, filtered, and monitored for m/z 292 (Q1) and m/z 70 (Q3) for cyproconazole. The demonstrated LOQ is 0.01 mg/kg.

Analytical residue enforcement method DFG S19 has been developed (HPLC- MS/MS) and validated for a dry crop, a high-fat crop, a high-water crop, and an acidic crop. The LOQ is 0.01 mg/kg.

An HPLC-MS/MS method (RAM 499/01) has been developed and validated for the determination of cyproconazole only in livestock commodities. Major metabolites such as M36 in milk and M14 in liver are not determined currently by the method. For this method, free and conjugated cyproconazole residues are extracted with acetonitrile (ACN):water and hydrolysed using either concentrated ammonia (eggs and tissues) or 2M HCl (milk). Cyproconazole residues are then determined by LC-MS/MS using external standards. The method LOQ is 0.01 ppm for cyproconazole in each livestock commodity. The method has also undergone a successful independent laboratory validation (ILV) trial and was radiovalidated using samples from a goat dosed with [^{14}C]cyproconazole.

Analytical residue enforcement method DFG S19 has been developed (HPLC-MS/MS) for cyproconazole in livestock matrices and validated by an independent laboratory.

Stability of residues in stored analytical samples

Cyproconazole has been shown to be stable in numerous plant commodities stored frozen at ≤-12 ºC. Cyproconazole is stable (≥ 80% recovery) for at least 40–42 months in grapes, raisins, nectarines, peaches, peanut nutmeat, peanut hay, and wheat hay. It is stable (≥ 80% recovery) for at least 36–39 months in wheat grain, wheat forage, and peanut hulls. Likewise, cyproconazole was stable in most livestock commodities fortified with cyproconazole at 0.01–10 mg/kg and stored frozen at -20 ºC. Cyproconazole was stable in milk for at least 12 months and in kidney and liver for at least 9 months. However, the percent cyproconazole remaining in fat at all fortification levels and storage intervals was variable and may be more a reflection of analytical method difficulties than actual storage stability. Some stability was indicated for up to one month in fat (60–90% remaining).

Definition of the residue

The livestock commodity analytical methods used for data collection (livestock feeding) determine cyproconazole and the metabolites M36 and M21. No hydrolyses were used to free potential conjugates. The analytical method validated for enforcement purposes determines only cyproconazole. This method uses an acid hydrolysis step (milk) or ammonia hydrolysis (eggs and tissues) to free conjugates.

Cyproconazole was the major component of the residue in all poultry commodities and all ruminant commodities except milk. Conjugated cyproconazole was found in eggs and ruminant kidney. Cyproconazole was a minor component in milk (10% TRR), whereas the major metabolites were M36 (NOA405872) and M21 (NOA405890). M36 and M21 comprised up to 80% of the TRR in milk. These two metabolites are carboxylic acids resulting from transformation of the cyclopropyl ring. Various toxicity studies with M36 reveal that this metabolite is very significantly less toxic than cyproconazole. Moreover, a feeding study with ruminants shows that M36/M21 residues are near the limit of quantitation of the analytical method at current livestock dietary burden levels. Therefore, M36/M21 need not be considered in the dietary risk assessment for milk.

While cyproconazole was a major component of the TRR in hen and goat liver, there were significant amounts of the hydroxylated cyproconazole metabolite M14, 30% TRR goat liver and 20 – 38% TRR hen liver. Cyproconazole was 27% TRR and 27%, respectively. In the feeding studies, M14 was 0.2–0.5 ppm in cow liver at a 3 ppm dietary burden and not determined in poultry liver. Cyproconazole was about 0.2 ppm in cow liver and < 0.01 mg/kg in poultry liver at a 3 ppm feeding level. M14 is considered to be less toxic than cyproconazole. Given the significant percentage of cyproconazole in the liver residue, the lower relative toxicity of M14, and the small contribution of liver to the diet, metabolite M14 need not be included in the residue definition for dietary intake.

Triazolyl-alanine was 63% TRR (0.13–0.20 mg/kg) on wheat grain in the wheat metabolism study. In livestock feeding studies, concentrations of triazole, triazolyl-alanine, and triazole acetic acid were < 0.01 mg/kg, except cattle liver (triazolyl-alanine 0.04 mg/kg).

The 2007 JMPR addressed the issue of triazole metabolites (JMPR 2007 Report, General Consideration 2.3). It was noted that 1,2,4-triazole, triazolyl-acetic acid and triazolyl-alanine may be derived from several sources. In a situation in which the metabolites arise from multiple triazole fungicides, they cannot be included in the residue definition. Since the metabolites cannot be linked to a specific triazole fungicide, they would have to be evaluated on their own. The 2007 Meeting further concluded that they did not have sufficient information to judge levels that would be without potential effect in consumers.

Cyproconazole was the major component of the residue in plant metabolism studies conducted with grape, apples, sugar beet, and wheat (except grain, 15% TRR). Concentrations of cyproconazole conjugates generally were < 5% TRR. No metabolite exceeded 10% TRR, except for M39 (triazole alanine) in wheat grain (63% TRR) and M9/M14 in wheat grain (14% TRR). Anaerobic soil metabolism studies showed that cyproconazole is relatively stable and does not form metabolites in significant concentrations. Confined rotational crop studies revealed that cyproconazole is the major quantifiable residue in follow-on crops; metabolites were < 12% TRR. A limited rotational crop field trial (conduced at 1× for the primary crop) indicated that cyproconazole residues in follow-on crops would most likely be near the LOQ (0.01 mg/kg).

The plant commodity analytical methods used for data collection and the methods validated for enforcement of MRLs determine only cyproconazole.

The Meeting recommended that the residue definition for plant and animal commodities for compliance with MRLs should be cyproconazole. While cyproconazole is a minor component of the residue in milk, there is sufficient cyproconazole present to monitor compliance.

The Meeting recommended that the residue definition for dietary risk assessment for plant commodities should be cyproconazole.

The Meeting recommended that the residue definition for dietary risk assessment for animal commodities should be cyproconazole, free and conjugated.

The log Kow of cyproconazole (log K_{ow} 3.1) suggests that cyproconazole will show no clear preference for distribution in fat versus water. The ratio of cyproconazole residues (TRR) in muscle and fat observed in the livestock metabolism studies (lactating cow 1 muscle: 4–6 fat) indicates a slight preference for fat solubility. In the cow feeding study, cyproconazole had a slight preference for cream over skim milk (0.008 ppm vs 0.003 ppm) and a more indicative preference for fat over meat (0.052 mg/kg fat versus 0.005 mg/kg meat, or about 10 to 1).

Proposed definition of the residue (for compliance with MRL for plant commodities): *cyproconazole*.

Proposed definition of the residue (for compliance with MRL for animal commodities): *cyproconazole*.

The residue is considered fat-soluble.

Proposed definition of the residue (for estimation of dietary intake for plant commodities): *cyproconazole*.

Proposed definition of the residue (for estimation of dietary intake for animal commodities): *cyproconazole, free and conjugated*.

Results of supervised trials on crops

The NAFTA calculator was used as a tool in the estimation of the maximum residue levels from the selected residue data sets obtained from trials conducted according to GAP. 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 with the calculator using expert judgment. Then, the NAFTA calculator was employed. If the statistical calculation spreadsheet suggested a different value than that recommended by the JMPR, a brief explanation of the deviation was supplied. Some common factors that may lead to rejection of the statistical estimate include when the number of data points in a data set is < 15 or when there are a large number of values < LOQ.

Pome fruits

Supervised residue trials on apples were provided from Spain and Brazil, but no GAP (label) was available for Brazil. Using the GAP of Italy (0.02 kg ai/hL, 7 day PHI), the trial results from Spain in ranked order are: 0.03 (2), 0.05 mg/kg.

The Meeting considered three trials insufficient for the estimation of an MRL and STMR.

Legume vegetables

Supervised field trials on succulent peas were provided from France (North and South) and the UK. A GAP was provided for France (0.06 kg ai/ha, 2 applications, 21 day PHI). The trial results for Europe in ranked order are: < 0.01 (7), 0.01 mg/kg.

The Meeting estimated a maximum residue level of 0.01 mg/kg an HR of 0.01 mg/kg, and an STMR of 0.01 mg/kg. Statistical calculation is not useful for cases with highly censored data.

Pulses

Field trials for dried pea and dried bean were reported from France (19) and the UK (10). A GAP was provided for pea and bean in the UK (0.08 kg as/ha or 0.04 kg ai/hL, 2 applications, 42 day PHI). Using this GAP, the ranked order of residues on peas (dry) in Europe for dry pea is: < 0.01 (14), 0.01 (2), < 0.02 (5). The ranked order of residues on beans (dry) in Europe were: < 0.01 (4) mg/kg. Additional bean trials at shorter PHIs (29–30) had residues of 0.01–0.05 mg/kg.

The Meeting used the dry pea and dry bean data for mutual support. The Meeting estimates a maximum residue level of 0.02 (*) mg/kg, an HR of 0.02 mg/kg, and an STMR estimate of 0.02 mg/kg for peas (dry) and beans (dry). Statistical calculation is not useful for cases with highly censored data.

Field trials on soya beans were reported from the USA. The GAP is 0.04 kg ai/ha, 2 applications, and a 30 day PHI. Nineteen trials were conducted at this GAP, and the residue results (n = 19) in ranked order are: < 0.01 (4), 0.01 (3), < 0.02 (2), 0.02 (4), 0.03 (4), 0.04, 0.05 mg/kg.

The Meeting estimated an STMR of 0.02 mg/kg, and HR of 0.05 mg/kg, and a maximum residue level of 0.07 mg/kg.

The NAFTA statistical method estimated a maximum residue level of 0.06 mg/kg, based on the mean plus three standard deviations. The statistical method is unreliable with multiple LOQ values (2) and 13 non-censored data points.

Root and tuber vegetables

Field trials for sugar beets were reported from Europe. A GAP was provided for Italy (0.08 kg ai/ha, 2 applications, 21 day PHI) and for the Netherlands (0.06 kg ai/ha, 2 applications, 45 day PHI). One trial in Switzerland, four trials in France North, and four trials in the UK meet the GAP of the Netherlands, and the results (n = 9) in ranked order are: < 0.01 (4), < 0.02 (4), and 0.02 mg/kg. Six trials in Italy, two trials in Spain, and one trial in France South meet the GAP of Italy, and the results of the trials (n = 9) in ranked order were: < 0.01, < 0.02 (5), 0.02, 0.03, 0.04 mg/kg. Using the trials matching the GAP of Italy, the Meeting estimated an STMR of 0.02 mg/kg, an HR of 0.04 mg/kg, and a maximum residue level of 0.05 mg/kg.

Statistical calculation is not useful for cases with highly censored data.

Cereal grains

Field trials for wheat were reported from Europe (12 France, 26 Germany, 2 Switzerland). A GAP for wheat was provided for Germany (0.096 kg ai/ha or 0.047 kg ai/hl, 2 applications, 35 day PHI or until BBCH 61). Three trials in France, 23 trials in Germany, and two trials in Switzerland meet the GAP of Germany, and the results of the trials (n = 28) in ranked order are: < 0.01 (8), 0.01 (4), < 0.02 (4), 0.02 (6), 0.04 (2), 0.05 (4) mg/kg.

Field trials for rye were reported from Europe (Germany 5). A GAP for rye was provided for Germany (0.096 kg ai/ha, 2 applications, 35 day PHI or application until BBCH 61) and for the Netherlands (0.08 kg ai/ha, 1 application, PHI 42 days). Note that the GAPs for wheat and rye are identical in Germany. Using the GAP of Germany, the residue results from the German trials in ranked order are: 0.01 (2) and 0.03 mg/kg.

Field trials for barley were reported from Europe (France (12), Switzerland (5), Germany (9) and the UK (10)). A GAP for barley was provided for Germany (0.096 kg ai/ha or 0.048 kg ai/hL, 2 applications, 35 day PHI or until BBCH stage 61). Note that this GAP is identical to the GAP for wheat and rye in Germany. One trial in France (North), three trials in Switzerland, and nine trials in Germany were at the GAP of Germany. The residue results in ranked order are: 0.01, 0.02 (4), 0.03 (4), 0.04 (3), 0.07 mg/kg.

The Meeting determined that the data sets for wheat, rye, and barley are from similar populations and combined the sets. The residues (n = 44) in ranked order are < 0.01 (8), 0.01 (7), < 0.02 (4), 0.02 (10), 0.03 (5), 0.04 (5), 0.05 (4), 0.07 mg/kg. The GAPs for the various grains were identical. No cereal grain group GAP was provided, but GAPs were provided for wheat, rye, triticale, barley, and oats, which represent all major small cereal grains except rice and which justify the extension. The Meeting estimated for cereal grains except rice and maize an STMR of 0.02, an HR of 0.07 mg/kg, and a maximum residue level of 0.08 mg/kg.

The maximum residue level estimate derived from use of the NAFTA statistical calculator was 0.07 m/kg based on the mean plus 3 standard deviations, but the Meeting considered that the estimate should be above the highest residue.

Field trials for maize (field corn) were reported from the USA, where the GAP is 0.04 kg ai/ha, 2 applications, and 30 day PHI. Twenty-two trials were conducted at this GAP, and the residue results in ranked order were: < 0.01 (22) mg/kg. In three trials, samples were collected at a PHI of 7 days, and residues were < 0.01 mg/kg.

The Meeting estimated an STMR of 0.01 mg/kg, an HR of 0.01 mg/kg, and a maximum residue level of 0.01 (*) mg/kg for maize.

Statistical method for maximum residue level estimation are not applicable where all data are < LOQ.

Oilseeds

Field trials on rape (canola) were reported from Europe. A GAP was provided for the UK (0.08 kg ai/ha, 2 applications, and a PHI of 30 days or BBCH 79, whichever occurs first). One trial from Switzerland, ten trials from France, and one trial from Germany complied with the UK GAP. The residue results (n = 12) in ranked order are: 0.01, 0.03 (3), 0.04, 0.05, 0.08 (2), 0.09, 0.10, 0.21, 0.23 mg/kg.

The Meeting estimated an STMR of 0.065, an HR of 0.23, and a maximum residue level of 0.4 mg/kg for rape seed.

The maximum residue level estimate derived from use of the NAFTA statistical calculator was 0.4 mg/kg, based on the UCL median 95th, which was in agreement with the Meeting's estimation.

Field trials on peanut were reported from Australia (3), Brazil (1), and the USA (4). GAP was provided for Australia (0.06 kg ai/ha, 5 applications, 14 day PHI). There is no registered use on

peanuts in the USA. No GAP was provided for Brazil. The ranked orders of residues from Australian trials, corresponding to the Australian GAP, were: < 0.02 (3) mg/kg.

The Meeting considered three trials insufficient for the estimation of an STMR, HR, and maximum residue level for peanuts.

Primary animal feed commodities

Legume animal feed

Field trials on soya beans were reported from the USA. The GAP is 0.04 kg ai/ha, 2 applications, and a 30 day PHI. The PHI for forage is 14 days. Fifteen trials were at GAP for soya bean forage, and the results in ranked order were: 0.11, 0.21, 0.22, 0.31 (2), 0.33, 0.35, 0.37, 0.40, 0.41, 0.48, 0.50, 0.52, 0.80, 0.82 mg/kg.

The Meeting estimated an STMR of 0.37 mg/kg and a highest residue of 0.82 mg/kg for soya bean forage.

Fifteen trials were at GAP for soya bean fodder (hay), and the results in ranked order were: 0.17, 0.32, 0.33 (2), 0.41, 0.43, 0.44, 0.66, 0.67, 0.71, 0.75 (2), 1.3, 1.5, 1.9 mg/kg.

The Meeting estimated an STMR of 0.66 mg/kg and a highest residue of 1.9 mg/kg. The Meeting also estimated a maximum residue level of 3 mg/kg for soya bean hay.

The maximum residue level estimate derived from use of the NAFTA statistical calculator was 3.0 mg/kg, based on the 99th percentile of a log normal distribution, which was in agreement with the Meeting's estimation.

Pea Vines (Green)

Supervised field trials on succulent peas were provided from France. A GAP was provided for France (0.07 kg ai/ha, 2 applications, 21 day PHI). All trials were conducted at about 125% of the GAP maximum application rate. The residue results in ranked order for pea vines (n = 6) were: 0.02, 0.07, 0.34, 0.35, 0.43, 0.83 mg/kg.

The Meeting estimated an STMR of 0.345 and a highest residue of 0.83 mg/kg.

Pea Hay or Fodder (dry)

Field trials for dried pea fodder were reported from France and the UK. A GAP was provided for pea in the UK (0.08 kg as/ha or 0.04 kg ai/hL, 2 applications, 42 day PHI). Only two of the 17 trials were conducted at GAP, and residues in ranked order for pea fodder are: 0.12, 0.24 mg/kg.

Bean Fodder

Field trials for dried bean fodder were reported from France and the UK. A GAP was provided for bean in the UK (0.08 kg as/ha or 0.04 kg ai/hL, 2 applications, 42 day PHI). Only 1 of 8 trials was at GAP, and residues in ranked order for bean fodder are: 0.09 mg/kg.

The Meeting combined pea fodder and bean fodder results in mutual support, but decided that 3 trials were not sufficient to estimate a highest residue, STMR, and/or maximum residue level.

Sugar Beet Tops (Leaves)

Field trials for sugar beets were reported from Europe. A GAP was provided for Italy (0.08 kg ai/ha, 2 applications, 21 day PHI) and for the Netherlands (0.06 kg ai/ha, 2 applications, 45 day PHI). Using the GAP of the Netherlands to evaluate the trials of the UK, France, Germany, and Switzerland residue values (n = 5; Switzerland and France) for sugar beet tops in ranked order are: 0.07, 0.15, 0.17, 0.23, 0.35 mg/kg. Using the GAP of Italy to evaluate the trials in Spain and Italy, residue values (n = 4; Italy) for sugar beet tops in ranked order were: 0.06, 0.29, 0.34, 0.54 mg/kg.

Using the trials evaluated against the GAP of Italy, the Meeting estimated an STMR of 0.315 mg/kg and a highest residue of 0.54 mg/kg for sugar beet tops.

Cereal Grain Straw, Forage, Fodder, Silage

Field trials for wheat were reported from Europe (12 France, 26 Germany, 2 Switzerland). A GAP was provided for Germany (0.096 kg ai/ha or 0.047 kg ai/hL, 2 applications, 35 day PHI or until BBCH 61). The residue results for wheat straw (n = 30; France – (n = 3) 0.39, 1.6, 1.7; Switzerland (n = 2) – 0.09, 0.11; Germany (n = 25) – 0.15, 0.22, 0.23, 0.24, 0.36, 0.37, 0.42, 0.43, 0.76, 0.77, 0.78 (2), 0.79, 0.85 (2), 0.92 (3), 1.1, 1.3, 1.4, 1.7, 2.1, 2.4, 3.6, in ranked order were: 0.09, 0.11. 0.15, 0.22, 0.23, 0.24, 0.36, 0.37, 0.39, 0.42, 0.43, 0.76, 0.77, 0.78 (2), 0.79, 0.85 (2), 0.92 (3), 1.1, 1.3, 1.4, 1.6, 1.7 (2), 2.1, 2.4. 3.6 mg/kg.

Field trials in rye were reported from Europe (Germany n = 5). A GAP was provided for Germany (0.096 kg ai/ha, 2 applications, 35 day PHI or application until BBCH 61) and for the Netherlands (0.08 kg ai/ha, 1 application, PHI 42 days). Note that the GAPs for wheat and rye are identical in Germany. Using the GAP of Germany, trial results at GAP for rye straw in ranked order were: 0.64, 0.68, 1.2 mg/kg.

Field trials for barley were reported from Europe (France 12, Switzerland 5, Germany 9, and the UK 10). A GAP was provided for Germany (0.096 kg ai/ha or 0.048 kg ai/hL, 2 applications, 35 day PHI or until BBCH stage 61). Note that this GAP is identical to the GAP for wheat and rye in Germany. For barley straw, One trial in France North (0.16), three trials in Switzerland (0.24, 0.34, 0.42), and 13 trials in Germany were at the GAP. The results (n = 17) in ranked order were: 0.01, 0.14, 0.15, 0.16, 0.20, 0.22, 0.24, 0.28, 0.34, 0.42, 0.52 (3), 0.53, 0.56, 0.63, 0.67 mg/kg.

Noting that the GAPs for wheat, rye, and barley were identical and that the residue values were similar, the Meeting decided to estimate values for the cereal grain straws group (except rice and maize). The residue values for wheat straw were used, as this set is the largest and contains the highest high residue.

The Meeting estimated an STMR of 0.785 mg/kg, and a highest residue of 3.6 mg/kg. The Meeting also estimated a maximum residue level of 5 mg/kg for cereal grain straws (except rice and maize).

The NAFTA statistical spreadsheet provided a maximum residue level estimate of 5.1 mg/kg, based on the 95th percentile at the 99th UCL.

Field trials for maize (field corn) were reported from the USA, where the GAP is 0.04 kg ai/ha, 2 applications, and 30 day PHI for grain and fodder/straw and a 21 day PHI for forage/silage. Twenty-three trials for maize fodder were at the GAP, and the results in ranked order were: < 0.01 (2), 0.08 (2), 0.12 (2), 0.21, 0.22, 0.23, 0.24, 0.27, 0.28 (2), 0.33, 0.34, 0.35 (3), 0.45, 0.46, 0.74, 0.80, 1.5 mg/kg.

The Meeting estimated an STMR of 0.28 and a highest residue of 1.5 mg/kg. The Meeting also estimates a maximum residue level of 2 mg/kg for maize fodder.

The NAFTA statistical calculation estimates a maximum residue level of 1.4 mg/kg. However, JMPR (FAO) guidance specifies that an estimate shall not be below the highest result (1.5 mg/kg).

Twenty-two trials for maize forage are at the GAP, and the results in ranked order were: < 0.01, 0.03, 0.05, 0.06 (2), 0.07, 0.08 (2), 0.09, 0.10 (2), 0.12, 0.13, 0.14, 0.16, 0.19, 0.20, 0.23, 0.24, 0.29, 0.31, 0.44 mg/kg.

The Meeting estimated an STMR of 0.11 and a highest residue of 0.44 mg/kg.

Oilseed forages and fodders

Field trials on rape (canola) were reported from Europe. A GAP was provided for the UK (0.08 kg ai/ha, 2 applications, and a PHI of 30 days). Six trials for rape forage from France were at GAP, and the results in ranked order were: 0.24, 0.28, 0.48, 0.52, 1.2, 1.9 mg/kg.

The Meeting estimated an STMR of 0.50 and a highest residue of 1.9 mg/kg for rape forage.

Field trials on peanut were reported from Australia (3), Brazil (1), and the USA (4). GAP was provided for Australia (0.06 kg ai/ha, 5 applications, 14 day PHI). There is no registered use on peanuts in the USA. For two trials at GAP in Australia, residues on peanut fodder were 5.3 and 14 mg/kg. For two trials at GAP in Australia, residues on peanut forage (green) were 1.3 and 5.3 mg/kg.

The Meeting considered two trials insufficient for the estimation of an STMR or highest residue for peanut fodder or peanut forage.

Fate of residue during processing

The effects of processing on the nature of residues in processed commodities were investigated in buffer solutions under conditions simulating pasteurization, boiling, and sterilization. Radio-labelled cyproconazole was demonstrated to be stable under these conditions.

The fate of cyproconazole residues has been studied in processing studies for apples, maize, rape seed (canola), soya bean, and peanuts. Estimated relevant processing factors and STMR-Ps are summarised below.

Commodity	Number of Studies (n)	Median Cyproconazole Transfer Factors	Cyproconazole RAC-STMR (mg/kg)	Cyproconazole STMR-P (mg/kg)
Oilseed rape – press cake	1	0.83	0.065	0.054
Oilseed rape – crude oil	4	0.86	0.065	0.056
Oilseed rape – solvent extracted meal	1	0.25	0.065	0.016
Oilseed rape – refined oil	4	0.08	0.065	0.0052
Soya bean – meal	4	0.64	0.02	0.013
Soya bean – hulls	4	0.75	0.02	0.015
Soya bean – refined oil	4	1.8	0.02	0.036

The Meeting decided to estimate a maximum residue level of 0.1 mg/kg for refined soya bean oil based on a highest residue of 0.05 mg/kg for soya beans and a processing factor of 1.8 (0.05 mg/kg × 1.8 = 0.09 mg/kg).

Residues in animal commodities

Farm animal dietary burden

The Meeting estimated the dietary burden of cyproconazole in farm animals on the basis of the diets listed in Appendix IX of the FAO Manual (2009 Edition). Calculation from highest residues, STMR (some bulk blended commodities), and STMR-P values provides the levels in feed suitable for estimating MRLs, while calculation from STMR and STMR-P values for feed is suitable foe estimating STMR values for animal commodities. The percentage dry matter is assumed to be 100% when the highest residue levels and STMRs are expressed on a dry weight basis.

Estimated maximum and mean dietary burdens of farm animals

Dietary burden calculations for beef cattle, dairy cattle, chicken broilers, and laying poultry are provided in Annex 6 of the 2010 JMPR Report. The calculations were made according to the animal diets from the US/CAN, EU, and Australia in Appendix IX of the FAO Manual (2009 Edition).

Commodity	Level	Animal Dietary Burden, Cyproconazole, ppm of dry matter diet.			
		US/CAN	EU	Australia	Japan
Beef cattle	Max	0.644	3.08	6.33[a]	0.022
	Mean	0.153	0.929	1.67[c]	0.022
Dairy cattle	Max	2.88	3.21	5.05[b]	0.711
	Mean	0.764	1.04	1.40[d]	0.180
Poultry – broiler	Max	0.022	0.022	0.022	0.014
	Mean	0.022	0.022	0.022	0.014
Poultry – layer	Max	0.022	1.40 [e,g]	0.022	0.013
	Mean	0.022	0.413 [f,h]	0.022	0.013

[a] Highest maximum beef or dairy cattle dietary burden suitable for MRL estimates for mammalian tissues

[b] Highest maximum dairy cattle dietary burden suitable for MRL estimates for mammalian milk

[c] Highest mean beef or dairy cattle dietary burden suitable for STMR estimates for mammalian tissues.

[d] Highest mean dairy cattle dietary burden suitable for STMR estimates for milk.

[e] Highest maximum poultry dietary burden suitable for MRL estimates for poultry tissues.

[f] Highest mean poultry dietary burden suitable for STMR estimates for poultry tissues.

[g] Highest maximum poultry dietary burden suitable for MRL estimates for poultry eggs.

[h] Highest mean poultry dietary burden suitable for STMR estimates for poultry eggs.

Farm animal feeding studies

A cow feeding study involved Friesian dairy cows dosed orally with cyproconazole for 35 days at levels equivalent to 1, 3, 10, and 30 ppm in the diet. Aside for one usually high value (0.025 ppm) from the 1-ppm dose group on day 14, cyproconazole residues in milk were ≤ 0.006 ppm for the 1, 3 and 10 ppm dose groups and were < 0.003–0.014 ppm for the 30 ppm dose group, with the maximum values occurring on days 7 or 14. At intervals in excess of 14 days, cyproconazole was found (> 0.003 ppm) in the 30 ppm dosing level only.

In tissues, cyproconazole was found at the following levels at dosing levels of 1, 2, 10, and 30 ppm, respectively: liver 0.090 (avg 0.082), 0.218 (avg 0.214), 0.604 (avg 0.514), 0.930 (avg 0.748); fat < 0.003; 0.003; 0.024 (avg 0.017), 0.052 (avg 0.022); kidney < 0.003, 0.009 (avg 0.007), 0.031 (avg 0.016, 0.038 (avg 0.028); muscle < 0.003, < 0.003, 0.003, 0.005 mg/kg.

In the table below, dietary burdens are shown in round brackets (), feeding levels and residue concentrations from the feeding study are shown in square brackets [], and estimated concentrations related to the dietary burdens are shown without brackets.

Cattle Dietary Burden [a] (ppm)						
Feeding Level [ppm]	Cream	Milk	Muscle	Liver	Kidney	Fat
MAXIMUM RESIDUE LEVEL	Mean	Mean	Highest	Highest	Highest	Highest
MAXIMUM RESIDUE LEVEL beef cattle (6.33) [3/10]			0.003 [< 0.003/0.003]	0.46 [0.218/0.604]	0.115 [0.054/0.1861]	0.015 [0.003/0.024]
MAXIMUM RESIDUE LEVEL dairy cattle (5.05) [3/10]	- -	0.006 [0.005/0.006]	0.003 [< 0.003/0.003]	0.36 [0.218/0.604]	0.092 [0.054/0.186]	0.012 [0.003/0.024]
STMR	Mean	Mean	Mean	Mean	Mean	Mean
STMR beef cattle (1.67) [1/3]			0.003 [< 0.003/< 0.003]	0.14 [0.082/0.214]	0.026 [< 0.018/0.042]	0.003 [< 0.003/ 0.003]
STMR dairy	-	0.009	0.003	0.11	0.023	0.003

Cattle Dietary Burden [a] (ppm)						
Feeding Level [ppm]	Cream	Milk	Muscle	Liver	Kidney	Fat
Cattle (1.40) [1/3]	-	[0.009/0.004]	[< 0.003/< 0.003]	[0.082/0.214]]	[< 0.018/0.042]	[< 0.003/0.003]

[a] Data from the first cattle feeding study (Oakes, 1994, T021566-04).

The data from the lactating dairy cow feeding study was used to support mammalian (except marine) milk and meat maximum residue levels. In this study only free cyproconazole was determined. The ruminant metabolism study showed that conjugated cyproconazole was about 5× the free cyproconazole concentration in kidney. Therefore, the measured cyproconazole concentration in kidney was multiplied by 6 for the dietary intake calculation.

Insufficient data were provided in the dairy cow feeding study to allow estimation of milk fat levels.

The Meeting estimated the following STMR values: milk 0.009 mg/kg; muscle 0.003 mg/kg; edible offal 0.14 mg/kg; fat 0.003 mg/kg. The HR values are: muscle 0.003 mg/kg, edible offal 0.46 mg/kg, fat 0.020 mg/kg.

The Meeting estimated the following maximum residue levels for mammalian commodities (except marine): milk at 0.01 mg/kg; meat (fat) at 0.02 mg/kg and edible offal at 0.5 mg/kg.

A poultry feeding study was also available, in which 15 hens/treatment group were dosed for 29 days with cyproconazole at feed concentrations of 0.12, 0.45, and 1.87 ppm. Cyproconazole was < 0.01 mg/kg in eggs and all tissues at all feeding levels. Noting that the mean and maximum dietary burden for poultry for meat and eggs are 0.44 ppm and 1.6 ppm, respectively, the Meeting concluded that cyproconazole residues are unlikely in poultry commodities.

The Meeting estimated the following STMR values: eggs, 0.01 mg/kg; muscle, 0.01 mg/kg fat, 0.01 mg/kg; edible offal, 0.01 mg/kg. The Meeting estimated the following maximum residue levels: eggs, 0.01 mg/kg meat, 0.01 (*) mg/kg; edible offal, 0.01 (*) mg/kg. The HR values are eggs, 0.01 mg/kg; muscle, 0.01 mg/kg; fat, 0.01 mg/kg; edible offal, 0.01 mg/kg.

RECOMMENDATIONS

The Meeting estimated the maximum residue levels and STMR values shown below. The maximum residue levels are recommended for use as maximum residue limits.

Definition of the residue

For plants (for compliance with maximum residue level and estimation of dietary intake):

Cyproconazole

For animals (for compliance with maximum residue level): *Cyproconazole*.

For animals (for estimation of dietary intake): *Cyproconazole, free and conjugated.*

Cyproconazole is fat-soluble.

CCN	Commodity	Maximum residue level, mg/kg	STMR or STMR-P, mg/kg	HR or HR-P, mg/kg
VD 0071	Beans (dry)	0.02 *	0.02	0.02
GC 0080	Cereal grains (except maize, except rice, except sorghum)	0.08	0.02	0.07
MO 0105	Edible offal (mammalian)	0.5	0.14	0.46
PE 0112	Eggs	0.01 *	0.01	0.01

CCN	Commodity	Maximum residue level, mg/kg	STMR or STMR-P, mg/kg	HR or HR-P, mg/kg
GC 0645	Maize	0.01 *	0.01	0.01
AS 0645	Maize fodder	2	0.28	1.5
MM 0095	Meat (from mammals other than marine mammals)	0.02(fat)	0.003 muscle 0.003 fat	0.003 muscle 0.020 fat
ML 0106	Milks	0.01	0.009	
VD 0072	Peas (dry)	0.02 *	0.02	0.02
VP 0064	Peas, shelled (succulent seeds)	0.01	0.01	0.01
PO 0111	Poultry, edible offal of	0.01 *	0.01	0.01
PM 0110	Poultry meat	0.01 *	0.01 muscle 0.01 fat	0.01 muscle 0.01 fat
SO 0495	Rape seed	0.4	0.065	0.23
OR 0495	Rape seed oil, refined		0.0052	
VD 0541	Soya bean (dry)	0.07	0.02	0.05
AL 0541	Soya bean fodder	3	0.66	1.9
OR 0541	Soya bean oil, refined	0.1	0.036	
AB 1265	Soya bean meal		0.013	
AS 0081	Straw and fodder (dry) of cereal grains (except maize, except rice, except sorghum)	5	0.785	3.6
VR 0596	Sugar beet	0.05	0.02	0.04

DIETARY RISK ASSESSMENT

Long-term intake

The International Estimated Daily Intakes (IEDIs) of cyproconazole were calculated for the 13 GEMS/Food Consumption Cluster Diets using STMRs and STMR-Ps estimated by the current Meeting (Annex 3 of the 2010 JMPR Report). The ADI is 0–0.02 mg/kg bw and the calculated IEDIs were 0.5–2% of the maximum ADI. The Meeting concluded that the long-term intake of residues of cyproconazole resulting from the uses considered by the current JMPR is unlikely to present a public health concern.

Short-term intake

The International Estimated Short-Term Intakes (IESTI) of cyproconazole were calculated for food commodities and their processed commodities using HRs/HR-Ps or STMRs/STMR-Ps estimated by the current Meeting (see Annex 4 of the 2010 JMPR Report). The ARfD is 0.06 mg/kg and the calculated IESTIs were 0–5% of the ARfD for the general population and 0–4% of the ARfD for children. The Meeting concluded that the short-term intake of residues of cyproconazole, when used in ways that have been considered by the JMPR, is unlikely to present a public health concern.

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Hertl P.	1992	Determination of the Residues of Cyproconazole at various Timings in Wheat, Winter, after Application with ALTO 100 SL under Field Conditions in Germany, Federal Republic of, 1991 (DC). Novartis Crop Protection AG, Basel, Switzerland Sandoz AG, Basel, Switzerland, R 10178 GLP, not published Syngenta File No SAN619/5388
Hertl P.	1992	Dissipation of Residues of Cyproconazole from Field Soil after Application of ALTO 100 SL under Field Conditions in France, 1989 (Field Soil Dissipation/Leaching Study). Novartis Crop Protection AG, Basel, Switzerland Sandoz AG, Basel, Switzerland, R 9661 GLP, not published Syngenta File No SAN619/5378
Hertl P.	1996	Dissipation of residues of Cyproconazole from Field Soil after repeated Applications of Alto 100 SL to Bare Soil in Switzerland, 1989-1993. (Field Soil Dissipation and Accumulation Study). Novartis Crop Protection AG, Basel, Switzerland Sandoz AG, Basel, Switzerland, R 9776 GLP, not published Syngenta File No SAN619/5126

Author(s)	Year	Study Title
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Hertl P.	1992	Determination of Residues of Cyproconazole at various Timings in Wheat, Winter after application with ALTO 100 SL under Field Conditions in Germany, Federal Republic of, 1991 (DC). Novartis Crop Protection AG, Basel, Switzerland Sandoz AG, Basel, Switzerland, R 10177 GLP, not published Syngenta File No SAN619/5431
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Hertl P.	1992	Determination of Residues of Cyproconazole at various Timings in Winter Wheat after application with ALTO 100 SL under Field Conditions in the Federal Republic of Germany, 1991 (DC). Novartis Crop Protection AG, Basel, Switzerland Sandoz AG, Basel, Switzerland, R 10180 GLP, not published Syngenta File No SAN619/5384
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Hertl P.	1990	Determination of Cyproconazole Residues in weathered Field Residue Samples after Storage Periods of 0, 3, 6, 12 and 36 Months at Temperatures below -18 °C. Novartis Crop Protection AG, Basel, Switzerland Sandoz AG, Basel, Switzerland, CBK 12995/90 GLP, not published Syngenta File No SAN619/6524
Hertl P.	1991	Determination of Cyproconazole Residues in weathered Field Residue Samples after Storage Periods of 0 and 12 months at Temperatures below -18 °C. Novartis Crop Protection AG, Basel, Switzerland Sandoz AG, Basel, Switzerland, CBK 13510/90 GLP, not published Syngenta File No SAN619/5478
Hertl P., Fricker P.	1995	Determination of Residues of Cyproconazole in Sugar Beet (Beta vulgaris) after application of Alto 100 SL under Field Conditions in the Federal Republic, 1993. Novartis Crop Protection AG, Basel, Switzerland Institut Fresenius, Taunusstein, Germany, TDS BS-5177 GLP, not published Syngenta File No SAN619/6675
Hertl P., Gasser A.	1993	Dissipation of Residues of Cyproconazole from Field Soil after Application of ALTO 100 SL under Field Conditions in France, 1989-1991 (Field Soil Dissipation/Leaching Study). FINAL REPORT. Novartis Crop Protection AG, Basel, Switzerland Sandoz AG, Basel, Switzerland, R 9662 GLP, not published Syngenta File No SAN619/5355

Author(s)	Year	Study Title
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Hertl P., Vogler F.	1993	Dissipation of Cyproconazole from Field Soil after repeated Applications of SAN 619 F 100 SL to Bare Soil in Switzerland, 1989-1993 (Field Soil Dissipation and Accumulation Study). Novartis Crop Protection AG, Basel, Switzerland Sandoz AG, Basel, Switzerland, R 9777 GLP, not published Syngenta File No SAN619/5346
Kaethner M.	1997	SAN 619, ALTO MARATHON, Peas (green; pods and grains), France, 1994 Novartis Crop Protection AG, Basel, Switzerland Novartis Agro Europe, ACES, Huningue, France, R10295 GLP, not published Syngenta File No SAN619/0065
Kaethner M.	1996	SAN 619, SAN 619 F 240 SL, Peas, France, 1993 Novartis Crop Protection AG, Basel, Switzerland Sandoz Agro Ltd., Huningue, France, R93003F GLP, not published Syngenta File No SAN619/0076
Karapally J.C., Vollmin S., Spielmann M.	1987	SAN 619 F - Metabolism in the rat. Novartis Crop Protection AG, Basel, Switzerland Sandoz AG, Basel, Switzerland, 31302, CBK 11816/87 GLP, not published Syngenta File No SAN619/6085
Karapally J.C., Vollmin S., Spielmann M.	1987	SAN 619 F - Metabolism of the Diastereomer A and B in the rat. Novartis Crop Protection AG, Basel, Switzerland Sandoz AG, Basel, Switzerland, 31303, CBK 11730/87 GLP, not published Syngenta File No SAN619/6087
Kennedy E.	1994	The Analysis of Cyproconazole in Winter Barley, Grain and Straw Novartis Crop Protection AG, Basel, Switzerland Restec Laboratories Ltd., Birlingham, United Kingdom, SDZ 0692 GLP, not published Syngenta File No SAN619/5223
Kissling M.	1998	Residue Study with CGA 279202, Cyproconazole (SAN 619) In or On Wheat in France (North) Novartis Crop Protection AG, Basel, Switzerland Novartis Crop Protection AG, Basel, Switzerland, 2238/97 GLP, not published Syngenta File No SAN619/0339
Kissling M.	1998	Residue Study With CGA 279202, Cyproconazole (SAN 619) In or On Barley in France (South) Novartis Crop Protection AG, Basel, Switzerland Novartis Crop Protection AG, Basel, Switzerland, 2228/97 GLP, not published Syngenta File No SAN619/0329
Kissling M.	1998	Residue Study With CGA 279202, Cyproconazole (SAN 619) In or On Barley in France (South) Novartis Crop Protection AG, Basel, Switzerland Novartis Crop Protection AG, Basel, Switzerland, 2229/97 GLP, not published Syngenta File No SAN619/0330

Author(s)	Year	Study Title
Kissling M.	1998	Residue Study With CGA 279202, Cyproconazole (SAN 619) In or On Barley in France (South) Novartis Crop Protection AG, Basel, Switzerland Novartis Crop Protection AG, Basel, Switzerland, 2230/97 GLP, not published Syngenta File No SAN619/0331
Kissling M.	1998	Residue Study With CGA 279202, Cyproconazole (SAN 619) In or On Barley in France (South) Novartis Crop Protection AG, Basel, Switzerland Novartis Crop Protection AG, Basel, Switzerland, 2231/97 GLP, not published Syngenta File No SAN619/0332
Klimmek S.	2004	Analytical Method Development and Validation of the DFG Method S19 for the Determination of Residues of Cyproconazole in Animal Matrices Syngenta Crop Protection AG, Basel, Switzerland Institut Fresenius, Taunusstein, Germany, IF-04/00202232 GLP, not published Syngenta File No SAN619/7504
Ko J.	1986	Method for Determining SAN 619 Residues in Mixed Crop Substrates Novartis Crop Protection AG, Basel, Switzerland Zoecon Corp., Dallas, USA, CBK 11032/86011 Not GLP, not published Syngenta File No SAN619/5034
Ko J.	1986	SAN 619 Residues in 1984 and 1985 - Apple Field and Processed SampIes Syngenta , CBK 11037/86016 Not GLP, not published Syngenta File No SAN619_10060
Konig P.	1996	Determination of Residues of Cyproconazole in Apples (whole fruits)after Application of ATEMI 10 WG under Field Conditions in Spain 1994 Novartis Crop Protection AG, Basel, Switzerland Sandoz AG, Basel, Switzerland, R10301 GLP, not published Syngenta File No SAN619/5082
Konig P.	1996	Determination of Residues of Cyproconazole at harvest in Wheat and Barley treated with ALTO 240 SL under Field Conditions in France, 1991 (Residue at Harvest) Novartis Crop Protection AG, Basel, Switzerland Sandoz AG, Basel, Switzerland, BS9358 91034M/5_7D GLP, not published Syngenta File No SAN619/6401
Krennhuber K., Pfarl Ch.	1996	Validation of an Analytical Method for Determination of Residues of Cyproconazole in Human Food, Animal Feed and Environmental Matrices Novartis Crop Protection AG, Basel, Switzerland Agrolinz Agrarchemikalien Gmbh, Leonding, Austria, R 96-98, 1283 GLP, not published Syngenta File No SAN619/0063
Krennhuber K., Pfarl Ch.	1996	Validation of an Analytical Method for Determination of Residues of Cyproconazole in Groundwater Novartis Crop Protection AG, Basel, Switzerland Agrolinz Agrarchemikalien Gmbh, Leonding, Austria, 1282 GLP, not published Syngenta File No SAN619/0051
Krips H.J.	1996	Determination of the explosive properties of cyproconazole techn. Novartis Crop Protection AG, Basel, Switzerland NOTOX B.V., Hertogenbosch, Netherlands, 166286 GLP, not published Syngenta File No SAN619/5160

Author(s)	Year	Study Title
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Lakaschus S.	2004	Independent Laboratory Validation of the DFG Method S19 (Extended Revision) for the Determination of Residues of Cyproconazole in Wheat Grain and Melon Syngenta Crop Protection AG, Basel, Switzerland Dr. Specht & Partner Chem. Laboratorien GmbH, Hamburg, Germany, SYN-0421V GLP, not published Syngenta File No SAN619/7559
Lakaschus S.	2005	Independent Laboratory Validation of Multi-Residue Method DFG S19 (L00.00-34) For the Determination of Residues of Cyproconzole in Different Animal Tissues With LC-MS/MS Detection Syngenta Crop Protection AG, Basel, Switzerland Dr. Specht & Partner Chem. Laboratorien GmbH, Hamburg, Germany, SYN-0504V GLP, not published Syngenta File No SAN619/7870
Lin K.	2004	Propiconazole, Cyproconazole and Chlorothalonil - Magnitude of the Residues in Soya bean Seed Syngenta Crop Protection AG, Basel, Switzerland Syngenta Crop Protection, Inc., Greensboro, USA, T001901-03 GLP, not published Syngenta File No CGA64250/4702
Lin K.	2005	Amendment - Propiconazole, Cyproconazole and Chlorothalonil - Magnitude of the Residues in Soya bean Seed Not Known Syngenta Crop Protection, Inc., Greensboro, USA, T001901-03 Not GLP, not published Syngenta File No SAN619/7686
Loisel S.	1994	Determination of the Residues of Cyproconazole in Leek following treatment with ATEMI 10 Pepite under Field Conditions in France, 1992 (Decline curve) Novartis Crop Protection AG, Basel, Switzerland Sandoz AG, Basel, Switzerland, 92013/ 333022 GLP, not published Syngenta File No SAN619/5279
Maffezzoni M.	1999	Residue Study with CGA 219417 + Cyproconazole in or on Wheat in South of France Novartis Crop Protection AG, Basel, Switzerland ADME - Bioanalyses, Aigues-Vives, France, 9812901 GLP, not published Syngenta File No SAN619/6764
Maffezzoni M.	1999	Residue Study with CGA 219417 + Cyproconazole in or on Wheat in South of France Novartis Crop Protection AG, Basel, Switzerland ADME - Bioanalyses, Aigues-Vives, France, 9812902 GLP, not published Syngenta File No SAN619/6763
Maffezzoni M.	1999	Residue Study with CGA 279202 + Cyproconazole in or on Wheat in South of France Novartis Crop Protection AG, Basel, Switzerland ADME - Bioanalyses, Aigues-Vives, France, 9813103 GLP, not published Syngenta File No SAN619/6828

Author(s)	Year	Study Title
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Mamouni A	2003	[^{14}C]-CGA71019: Anaerobic soil degradation Syngenta Crop Protection AG, Basel, Switzerland RCC Ltd., Itingen, Switzerland, 798660 GLP, not published Syngenta File No CGA71019/0062
Martin N.	1999	Final report on surface tension Novartis Crop Protection AG, Basel, Switzerland Novartis Services AG, Basel, Switzerland, PP-99/01T.SUR GLP, not published Syngenta File No SAN619/6767
McKenzie J.	1993	The determination of concentrations of cyproconazole in Winter and Spring Barley (grains, straw and whole plant) Novartis Crop Protection AG, Basel, Switzerland Restec Laboratories Ltd., Birlingham, United Kingdom, SDZ 0292/2 GLP, not published Syngenta File No SAN619/5227
Mellet M., Puy E., Wasser Ch.	1993	Determination of Residues of Cyproconazole at Harvest in Peanuts treated with ALTO 100 SL applied under Field Conditions in Brazil, 1991 (Residues at Harvest). Novartis Crop Protection AG, Basel, Switzerland Anadiag SA, Haguenau, France, R 10091 GLP, not published Syngenta File No SAN619/5340
Morrow P.	1991	Residue Report on Evaluation of SAN 619 F (Cyproconazole) for control of Leaf Spot and Rust in Peanuts, Queensland, 1987 Novartis Crop Protection AG, Basel, Switzerland Sandoz Australia Pty. Ltd., Agro Division, Queensland, Australia, CBK 13130/90 GLP, not published Syngenta File No SAN619/5444
Oakes, T	2006	Cyproconazole - Field Accumulation in Rotational Crops (60-, 120-, 180-, and 270-Day PBI): Interim Report. Project Number: ML05/1265/SYN, T003259/03. Unpublished study prepared by Morse Laboratories and Agvise Inc.
Oakes T.	2006	Cyproconazole - Magnitude of the Residues in or on Soya beans Syngenta Crop Protection AG, Basel, Switzerland Morse Laboratories, Inc., Sacramento, USA, ML05-1214-SYN, T002037-03 GLP, not published Syngenta File No SAN619/8079
Oakes T.	2006	Cyproconazole - Magnitude of the Residues in or on Field Corn Syngenta Crop Protection AG, Basel, Switzerland Morse Laboratories, Inc., Sacramento, USA, ML05-1250-SYN, T002814-03 GLP, not published Syngenta File No SAN619/8080
Oakes T.	2006	Cyproconazole - Magnitude of the Residues in Meat and Eggs Resulting from the Feeding at Three Levels to Laying Hens Syngenta Crop Protection AG, Basel, Switzerland Syngenta Crop Protection, Inc., Greensboro, USA, ML05-1275-SYN, T021566-04 GLP, not published Syngenta File No SAN619/8077
Oakes, T.	2006	Cyproconazole – Cyproconazole Magnitude of Residues in Meat and Milk Resulting form the Feeding at Three Levels to Dairy Cattle. Final Report. Syngenta Crop Protection T021565-04 GLP, not published

Author(s)	Year	Study Title
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Oggenfuss P.	2000	Spectra of SAN 619 (molar extinction coefficient >= 290 nm) Novartis Crop Protection AG, Basel, Switzerland Novartis Crop Protection Munchwilen AG, Munchwilen, Switzerland, 102936 GLP, not published Syngenta File No SAN619/7018
Oliver S, Hurt A D	2002	Aqueous Photolysis of 14 C-Triazole labelled SAN619 Syngenta Crop Protection AG, Basel, Switzerland Syngenta Crop Protection AG, Basel, Switzerland, RJ3322B GLP, not published Syngenta File No SAN619/7282
Phaff R.	2000	Hydrolysis of ^{14}C-triazole labelled CGA 221949 under processing conditions Novartis Crop Protection AG, Basel, Switzerland Novartis Crop Protection AG, Basel, Switzerland, 99RP03 GLP, not published Syngenta File No SAN619/6871
Pointurier R.	2000	Residue study with CGA 279202 and cyproconazole (SAN 619) in or on sugarbeets in Spain Novartis Crop Protection AG, Basel, Switzerland ADME - Bioanalyses, Aigues-Vives, France, 2060/99 GLP, not published Syngenta File No SAN619/6905
Pointurier R.	2000	Residue study with CGA 279202 and cyproconazole (SAN 619) in or on sugarbeets in Spain Novartis Crop Protection AG, Basel, Switzerland ADME - Bioanalyses, Aigues-Vives, France, 2061/99 GLP, not published Syngenta File No SAN619/6906
Pointurier R.	2000	Residue study with CGA 279202 and cyproconazole (SAN 619) in or on sugarbeets in Italy Novartis Crop Protection AG, Basel, Switzerland ADME - Bioanalyses, Aigues-Vives, France, 2074/99 GLP, not published Syngenta File No SAN619/6904
Pointurier R.	2000	Residue study with CGA 279202 and cyproconazole (SAN 619) in or on sugarbeets in Italy Novartis Crop Protection AG, Basel, Switzerland ADME - Bioanalyses, Aigues-Vives, France, 2075/99 GLP, not published Syngenta File No SAN619/6903
Pointurier R.	2000d	Residue study with CGA 279202 and cyproconazole (SAN 619) in or on sugarbeets in France (south) Novartis Crop Protection AG, Basel, Switzerland ADME - Bioanalyses, Aigues-Vives, France, 9911001 GLP, not published Syngenta File No SAN619/6983
Pointurier R.	2000	Residue study with CGA 279202 and cyproconazole (SAN 619) in or on sugarbeets in France (south) Novartis Crop Protection AG, Basel, Switzerland ADME - Bioanalyses, Aigues-Vives, France, 9911002 GLP, not published Syngenta File No SAN619/6984

Author(s)	Year	Study Title
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Pointurier R.	2001	Residue Study with CGA 279202 + Cyproconazole (SAN 619) in or on Sugarbeets in France (south) Syngenta Crop Protection AG, Basel, Switzerland ADME - Bioanalyses, Vergeze, France, NOV/RES/00062 GLP, not published Syngenta File No CGA279202/4476
Pointurier R.	2002	Residue Study with Azoxystrobin (ICI 5504) and Cyproconazole (SAN 619) in or on Wheat in Switzerland Syngenta Crop Protection AG, Basel, Switzerland ADME - Bioanalyses, Vergeze, France, 2075/01 GLP, not published Syngenta File No SAN619/7186
Pointurier R.	2002	Residue Study with Azoxystrobin (ICI 5504) and Cyproconazole (SAN 619) in or on Wheat in Switzerland Syngenta Crop Protection AG, Basel, Switzerland ADME - Bioanalyses, Vergeze, France, 2076/01 GLP, not published Syngenta File No SAN619/7185
Pointurier R.	2002	Residue Study with Azoxystrobin (ICI 5504) and Cyproconazole (SAN 619) in or on Wheat in Switzerland Syngenta Crop Protection AG, Basel, Switzerland ADME - Bioanalyses, Vergeze, France, 2077/01 GLP, not published Syngenta File No SAN619/7184
Pointurier R.	2002	Residue Study with Azoxystrobin (ICI 5504) and Cyproconazole (SAN 619) in or on Wheat in Switzerland Syngenta Crop Protection AG, Basel, Switzerland ADME - Bioanalyses, Vergeze, France, 2078/01 GLP, not published Syngenta File No SAN619/7183
Pointurier R.	2002	Residue Study with Azoxystrobin (ICIA 5504) and Cyproconazole (SAN 619) in or on Wheat in France (South) Syngenta Crop Protection AG, Basel, Switzerland ADME - Bioanalyses, Vergeze, France, 0112902 GLP, not published Syngenta File No SAN619/7232
Pointurier R.	2002	Residue Study with Cyproconazole (SAN 619) and Propiconazole (CGA 64250) in or on Barley in France (South) Syngenta Crop Protection AG, Basel, Switzerland ADME - Bioanalyses, Vergeze, France, 0110901 GLP, not published Syngenta File No SAN619/7202
Pointurier R.	2002	Residue Study with Cyproconazole (SAN 619) and Propiconazole (CGA 64250) in or on Barley in France (South) Syngenta Crop Protection AG, Basel, Switzerland ADME - Bioanalyses, Vergeze, France, 0110902 GLP, not published Syngenta File No SAN619/7203
Pointurier R.	2002	Residue Study with Azoxystrobin (ICIA 5504) and Cyproconazole (SAN 619) in or on Barley in France (South) Syngenta Crop Protection AG, Basel, Switzerland ADME - Bioanalyses, Vergeze, France, 0112701 GLP, not published Syngenta File No SAN619/7229

Author(s)	Year	Study Title
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Pointurier R.	2002	Residue Study with Azoxystrobin (ICIA 5504) and Cyproconazole (SAN 619) in or on Barley in France (South) Syngenta Crop Protection AG, Basel, Switzerland ADME - Bioanalyses, Vergeze, France, 0113201 GLP, not published Syngenta File No SAN619/7256
Pointurier R.	2002	Residue Study with Azoxystrobin (ICIA 5504) and Cyproconazole (SAN 619) in or on Barley in France (South) Syngenta Crop Protection AG, Basel, Switzerland ADME - Bioanalyses, Vergeze, France, 0113202 GLP, not published Syngenta File No SAN619/7255
Rixon C	2005	Residues of Azoxystrobin and Cyproconazole in Peanuts Syngenta - Jealott's Hill, Bracknell, United Kingdom , AUS 03/08 GLP, not published Syngenta File No A12910C_10008
Royer A.	2007	Azoxystrobin (ICI5504) and cyproconazole (SAN619) - Residue study on dried beans in the United Kingdom in 2006 Syngenta Crop Protection AG, Basel, Switzerland ADME - Bioanalyses, Vergeze, France, T014143-05-REG GLP, not published Syngenta File No SAN619/8492
Ryan J.	2006	Propiconazole (CGA64250): Summary of Validation Data for Analytical Methods REM 130.02 and REM 130.08 on Cereal Crops with Final Determination by GC-NPD Syngenta Crop Protection AG, Basel, Switzerland Syngenta - Jealott's Hill International, Bracknell, Berkshire, United Kingdom, T005934-05-TEC1 Not GLP, not published Syngenta File No CGA64250/5088
Ryan J.	2006	Cyproconazole (SAN619): Validation of Residue Analytical Method RAM 499/01 for the Determination of Free and Conjugated Residues in Animal Matrices. Final Determination by LC-MS/MS Syngenta Crop Protection AG, Basel, Switzerland Syngenta - Jealott's Hill International, Bracknell, Berkshire, United Kingdom, T022776-04-REG GLP, not published Syngenta File No SAN619/7957
Ryan J., Clark T.	2006	Summary and Validation Data for Analytical Methods REM 130.02 and REM 130.08 on Cereal Crops with Final Determination by GC-NPD. Syngenta, Jealot's Hill, Berks, UK. Method No. REM 130.02. Report No. T005934-05-TEC1. Syngenta File No. CGA64250/5088 GLP. Not published.
Sack S.	1997	Magnitude of Residues after Application of Propiconazole (CGA 64250) and Cyproconazole (SAN 619) as Formulation EC 410 (A-9856A) in Winter Wheat Novartis Crop Protection AG, Basel, Switzerland Novartis Crop Protection AG, Basel, Switzerland, 2301/97 GLP, not published Syngenta File No SAN619/0199
Scacchi A, Pizzingrilli G	2003	Aerobic degradation of [^{14}C-U-ring] triazolyl acetic acid (TAA) in three soils ISAGRO RICERCA, Novara, Italy ISAGRO RICERCA, Novara, Italy, MEF.02.02 GLP, not published Syngenta File No CGA142856/0012

Author(s)	Year	Study Title
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Schachtele M., Karapally J.C.	1987	SAN 619 F - Metabolism in Grapevine Seedlings Novartis Crop Protection AG, Basel, Switzerland Sandoz AG, Basel, Switzerland, CBK 11989/87 GLP, not published Syngenta File No SAN619/6078
Schachtele M., Karapally J.C.	1988	SAN 619 F - Metabolism in Grape Berries Novartis Crop Protection AG, Basel, Switzerland Sandoz AG, Basel, Switzerland, CBK 11995/87 GLP, not published Syngenta File No SAN619/6079
Schachtele M., Karapally J.C.	1988	SAN 619 F - Metabolism in apples Novartis Crop Protection AG, Basel, Switzerland Sandoz AG, Basel, Switzerland, CBK 11861/87 GLP, not published Syngenta File No SAN619/6081
Schachtele M., Karapally J.C.	1987	^{14}C-Cyproconazole. Metabolism in wheat. Analysis of wheat leaves and stems. (Attachment to Report CBK 11950/87) Novartis Crop Protection AG, Basel, Switzerland Sandoz AG, Basel, Switzerland, CBK 11950/87 GLP, not published Syngenta File No SAN619/5523
Scholtz R.	1996	Cyproconazole / Testing of Biological Degradability with Fungal and Bacterial Cultures Novartis Crop Protection AG, Basel, Switzerland Not Known, BMG569-95 Not GLP, not published Syngenta File No SAN619/5081
Schulz H.	2004	Analytic Method Development and Validation of the DFG method S19 for the Determination of Residues of Cyproconazole in Plant Matrices Syngenta Crop Protection AG, Basel, Switzerland Institut Fresenius, Taunusstein, Germany, IF-04/00192720 GLP, not published Syngenta File No SAN619/7489
Schulze C.	1987	SAN 619 F Residues in Barley Novartis Crop Protection AG, Basel, Switzerland Sandoz AG, Basel, Switzerland, 8812 GLP, not published Syngenta File No SAN619/6339
Schweitzer A.	1987	SAN 619 F - Absorption, distribution and excretion in rats after single and multiple doses of [^{14}C] SAN 619 F. Novartis Crop Protection AG, Basel, Switzerland Sandoz AG, Basel, Switzerland, CBK 11738/87 GLP, not published Syngenta File No SAN619/6086
Schweitzer A.	1987	SAN 619 F - Quantitative whole-body autoradiography in rats after single oral doses of [^{14}C] SAN 619 F. Novartis Crop Protection AG, Basel, Switzerland Sandoz AG, Basel, Switzerland, CBK 11870/87 Not GLP, not published Syngenta File No SAN619/5421

Author(s)	Year	Study Title
Serra G., Pupin A.	1994	Determination of Cyproconazole (Alto 100) Residue in Apples Syngenta Crop Protection AG, Basel, Switzerland Unicamp Universidade Estadual de Campinas, Campinas, Brazil, SAN948 12/93-R305 181 Not GLP, not published Syngenta File No SAN619/8097
Serra G., Pupin A.	1994	Determination of Cyproconazole (Alto 100) Residue in Apples Syngenta Crop Protection AG, Basel, Switzerland Unicamp Universidade Estadual de Campinas, Campinas, Brazil, SAN944 12/93-R137A 160 Not GLP, not published Syngenta File No SAN619/8099
Serra G., Pupin A.	1994	Determination of Cyproconazole (Alto 100) Residue in Apples (Degradation Curve) Syngenta Crop Protection AG, Basel, Switzerland Unicamp Universidade Estadual de Campinas, Campinas, Brazil, SAN945 12/93-R137B 161 Not GLP, not published Syngenta File No SAN619/8098
Shadrick BA, Bloomberg AM, Helfrich KK	1999	Freezer Storage Stability of 1H-1,2,4-Triazole[3,5-^{14}C] in Soil Syngenta Crop Protection AG, Basel, Switzerland Bayer Corporation, Kansas City, USA, 108303 GLP, not published Syngenta File No CGA71019/0068
Simon P.	2002	Determination of Residues of Azoxystrobin and Cyproconazole in Winter Wheat in Germany Syngenta Crop Protection AG, Basel, Switzerland Syngenta Agro GmbH, Maintal, Germany, gwh32401 (amended) GLP, not published Syngenta File No SAN619/7260
Simon P.	2002	Determination of Residues of Azoxystrobin and Cyproconazole in Winter Wheat in Germany Syngenta Crop Protection AG, Basel, Switzerland Syngenta Agro GmbH, Maintal, Germany, gwh92401 (amended) GLP, not published Syngenta File No SAN619/7259
Simon P.	2005	Azoxystrobin and cyproconazole: Residue study in or on oilseed rape in Germany 2004 (Test product: A12910C) Syngenta Crop Protection AG, Basel, Switzerland Syngenta Agro GmbH, Maintal, Germany, gra230004 GLP, not published Syngenta File No SAN619/7926
Skinner W.S.	1987	Confined Accumulation of ^{14}C-SAN 619 F in Rotational Crops. Novartis Crop Protection AG, Basel, Switzerland Zoecon Corp., Palo Alto, USA, PA-B86-06 GLP, not published Syngenta File No SAN619/6030
Skinner W.S., et al.	1985	Adsorption, Desorption and Mobility of SAN 619 F in Soil Novartis Crop Protection AG, Basel, Switzerland Zoecon Corp., Palo Alto, USA, 3760-24-11-85 Not GLP, not published Syngenta File No SAN619/6102
Skinner W.S., Sakai D.H., Collier K.D., Quistad G.B.	1987	Metabolism of [^{14}C]SAN 619F by a Lactating Goat Novartis Crop Protection AG, Basel, Switzerland Zoecon Corp., Palo Alto, USA, PA-B86-03 GLP, not published Syngenta File No SAN619/0533

Author(s)	Year	Study Title
Skinner W.S., Sakai D.H., Collier K.D., Quistad G.B., Reuter C.C.	1987	Metabolism of SAN 619 F by Laying Hens Novartis Crop Protection AG, Basel, Switzerland Zoecon Corp., Palo Alto, USA, PA-B86-04 GLP, not published Syngenta File No SAN619/6091
Skinner, W.S. et al.	1987	Metabolism of SAN 619 F by Peanuts Novartis Crop Protection AG, Basel, Switzerland Zoecon Corp., Palo Alto, USA, PA-B86-05 GLP, not published Syngenta File No SAN619/6080
Slangen P.J.	2000	Degradation of 1,2,4-triazole in Three Soils under Aerobic Conditions Novartis Crop Protection AG, Basel, Switzerland NOTOX B.V., Hertogenbosch, Netherlands, NOTOX 278336 GLP, not published Syngenta File No CGA64250/4345
Smith J.	1999	Determination of Residues of Cyprodinil + Cyproconazole in Winter Rye Novartis Crop Protection AG, Basel, Switzerland Novartis Agro GmbH, Frankfurt, Germany, GR 39298 GLP, not published Syngenta File No SAN619/0576
Smith J.	1999	Determination of Residues of Cyprodinil + Cyproconazole in Winter Rye Novartis Crop Protection AG, Basel, Switzerland Novartis Agro GmbH, Frankfurt, Germany, GR 40498 GLP, not published Syngenta File No SAN619/0571
Smith K.L., Wisson M.	1995	[U-^{14}C-Phenyl] Cyproconazole - Metabolism in Wheat Novartis Crop Protection AG, Basel, Switzerland Sandoz AG, Basel, Switzerland, TDS BS5239 GLP, not published Syngenta File No SAN619/5239
Spare W.C.	1983	Determination of the hydrolysis rate constants of 1,2,4-H-Triazole (CGA 71019) Novartis Crop Protection AG, Basel, Switzerland Ciba-Geigy Corp., Greensboro, USA, 83-E-074 Not GLP, not published Syngenta File No CGA71019/0033
Stulz J.	1998	Report on solubility in organic solvents Novartis Crop Protection AG, Basel, Switzerland Novartis Crop Protection Munchwilen AG, Munchwilen, Switzerland, 69561 GLP, not published Syngenta File No SAN619/0522
Stulz J.	1998	Report on octanol / water partition coefficient Novartis Crop Protection AG, Basel, Switzerland Novartis Crop Protection Munchwilen AG, Munchwilen, Switzerland, 66839 GLP, not published Syngenta File No SAN619/0518
Toussaint G.	1989	Determination of Cyproconazole residues in sugarbeets (leaves and roots). Novartis Crop Protection AG, Basel, Switzerland Hazleton France, Les Oncins, France, 811072 / R 9335 GLP, not published Syngenta File No SAN619/6172

Author(s)	Year	Study Title
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Tribolet R.	1999	Validation of Analytical method BS 3786 for the Determination of Cyproconazole (SAN 619) in Air by analysis of fortified Air sampling tubes and Evaluation of recoveries Novartis Crop Protection AG, Basel, Switzerland Novartis Crop Protection AG, Basel, Switzerland, 211/99 GLP, not published Syngenta File No SAN619/6768
Tummon O.	2005	Cyproconazole: Validation of an Analytical Method for the Determination of Residues of Cyproconazole in Water Syngenta Crop Protection AG, Basel, Switzerland Syngenta - Jealott's Hill, Bracknell, United Kingdom, RJ3561B 04-S621 GLP, not published Syngenta File No SAN619/7588
Tummon O. J.	2004	Cyproconazole: Validation of an Analytical Method for the Determination of Residues of Cyproconazole in Air Syngenta Crop Protection AG, Basel, Switzerland Syngenta, Jealott's Hill, United Kingdom, RJ3497B GLP, not published Syngenta File No SAN619/7478
Unknown	1987	SAN 619 F Residues in Barley. Novartis Crop Protection AG, Basel, Switzerland Sandoz AG, Basel, Switzerland, 8814 Not GLP, published Syngenta File No SAN619/5575
Valli F.	2003	Residue Study with A12910C in or on Sugar beet in Italy, degradation curve determination Syngenta Crop Protection AG, Basel, Switzerland AGRI 2000, Bologna, Italy, 0224R/25R GLP, not published Syngenta File No SAN619/7305
van Helvoirt J.A.	1994	Determination of the flammability of cyproconazole techn. Novartis Crop Protection AG, Basel, Switzerland NOTOX B.V., Hertogenbosch, Netherlands, 128868 GLP, not published Syngenta File No SAN619/6239
van Helvoirt J.A.	1994	Determination of the relative self-ignition temperature of cyproconazole techn. Novartis Crop Protection AG, Basel, Switzerland NOTOX B.V., Hertogenbosch, Netherlands, 128879 GLP, not published Syngenta File No SAN619/6240
Volkel W.	1997	U-^{14}C-Triazol-Cyproconazole: route and rate of degradation in aerobic aquatic systems Novartis Crop Protection AG, Basel, Switzerland RCC Ltd., Itingen, Switzerland, 613001 GLP, not published Syngenta File No SAN619/0186

Author(s)	Year	Study Title
Vollmin S.	1991	^{14}C-Cyproconazole: Transfer of Residues into the Milk Following Oral Administration of Cyproconazole to Lactating Goat Novartis Crop Protection AG, Basel, Switzerland Sandoz AG, Basel, Switzerland, BS-2050 GLP, not published Syngenta File No SAN619/5445
Vollmin S.	1994	^{14}C-Cyproconazole : Metabolism in Wheat Novartis Crop Protection AG, Basel, Switzerland Sandoz AG, Basel, Switzerland, BS-4603 GLP, not published Syngenta File No SAN619/5282
Vollmin S.	1997	[U-^{14}C-Triazolyl] Cyproconazole Metabolism in Sugarbeet (Beta Vulgaris L.) Novartis Crop Protection AG, Basel, Switzerland Novartis Crop Protection AG, Basel, Switzerland, E96-07 GLP, not published Syngenta File No SAN619/0396
Vollmin S., Karapally J.C.	1992	Supplementary Cyproconazole metabolism in the rat. Novartis Crop Protection AG, Basel, Switzerland Sandoz AG, Basel, Switzerland, 433015/BS-2754 GLP, not published Syngenta File No SAN619/5386
Walser M.	1997	Magnitude of Residues in Straw and Grains of Wheat after Application of Cyprodinil (CGA 219417) and Cyproconazole (SAN 619) as Formulation WG 45.3 (A-9849 A) Novartis Crop Protection AG, Basel, Switzerland Novartis Crop Protection AG, Basel, Switzerland, 2210/97 GLP, not published Syngenta File No SAN619/0213
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Walser M.	1997	Magnitude of Residues in Straw and Grains of Barley After application of Cyprodinil (CGA 219417) and Cyproconazole (SAN 619) as Formulation WG 45.3 (A-9849 A) Novartis Crop Protection AG, Basel, Switzerland Novartis Crop Protection AG, Basel, Switzerland, 2189/97 GLP, not published Syngenta File No SAN619/0210
Wassell W.D., Gilles Ch.	1991	Independent Laboratory Confirmation of Method AM-0842-0790-0 Novartis Crop Protection AG, Basel, Switzerland Biospherics Inc., Beltsville, USA, B9007-CN1 GLP, not published Syngenta File No SAN619/6406

Author(s)	Year	Study Title
Widmer H.	1998	Vapour pressure of SAN 619 Novartis Crop Protection AG, Basel, Switzerland Novartis Crop Protection AG, Basel, Switzerland, 98WI32 GLP, not published Syngenta File No SAN619/0532
Wisson M.	1989	Solubility of the pure active ingredient in redistilled water at pH 3-5, 7 and 9-11 (Final report) Novartis Crop Protection AG, Basel, Switzerland Sandoz AG, Basel, Switzerland, 41311 GLP, not published Syngenta File No SAN619/6125
Wisson M.	1992	[^{14}C-Benzyl]-Cyproconazole / Aerobic Degradation in Three Types of Soil (Balance Study) Novartis Crop Protection AG, Basel, Switzerland Sandoz AG, Basel, Switzerland, 41321 GLP, not published Syngenta File No SAN619/5362
Wisson M.	1990	Cyproconazole / Degradation in Three Types of Soil under Various Conditions (Laboratory Study with Field Soils) Novartis Crop Protection AG, Basel, Switzerland Sandoz AG, Basel, Switzerland, 41'313 GLP, not published Syngenta File No SAN619/6143