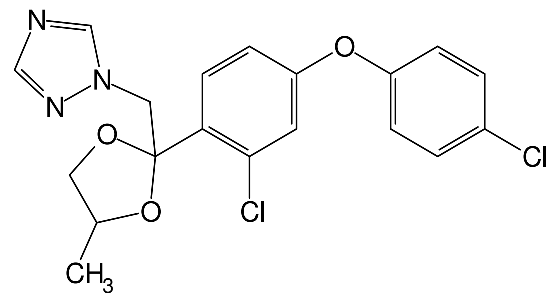
DIFENOCONAZOLE (224)

The first draft was prepared by Mr. Denis J. Hamilton Department of Primary Industries and Fisheries Brisbane, Australia

EXPLANATION

Difenoconazole, a broad-spectrum fungicide, was considered for the first time by the present meeting. It is a broad-spectrum fungicide used for disease control in many fruits, vegetables, cereals and other field crops. It has preventive and curative action. Difenoconazole acts by inhibition of demethylation during ergosterol synthesis.

IDENTITY

ISO common name	difenoconazole
Synonyms:	CGA 169374
IUPAC name	1-[2-[2-chloro-4-(4-chloro-phenoxy)-phenyl]-4-methyl[1,3]dioxolan-2-ylmethyl]-1H-1,2,4-triazole
Chemical Abstracts name	1-[[2-[2-chloro-4-(4-chlorophenoxy)phenyl]-4-methyl-1,3-dioxolan-2-yl]methyl]-1H-1,2,4-triazole
CAS Number	119446-68-3
CIPAC Number	687
Molecular formula	$C_{19}H_{17}Cl_2N_3O_3$
Molecular mass	406.3
Structural formula (note that the 2 chiral carbons in difenoconazole result in a cis-trans pair of diastereoisomers)	

PHYSICAL AND CHEMICAL PROPERTIES

Pure active ingredient

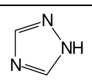
Property	Description or result	Reference
Physical state	White, odourless, fine crystalline powder	70160
Melting point (purity 99.3 %)	82 – 83 °C	70157
Relative density (purity 99.3 %)	1.39 at 22 °C	PP-98/137P.DES
Vapour pressure (purity 99 %)	3.32×10^{-8} Pa at 25 °C OECD Guideline 104 (measurements between 78.1 °C and 181.1 °C)	AG 88/11 P
Solubility in water (purity 99 %,)	15 mg/L at 25 °C OECD Guideline 105	23321
Octanol/water partition coefficient (purity 99.3 %)	log P_{OW} = 4.4 at 25 °C OECD Guideline 107	76303

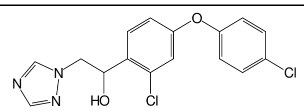
Property	Description or result	Reference
Hydrolysis rate (purity 98.8 %	Essentially no hydrolysis in 2 mg/L buffered sterile solutions at pH 5, 7 and 9 in the dark for 30 days at 25 °C	494
Photolysis rate (purity 99.3 %)	Less than 10% photo-degradation by simulated sunlight of 1.5 mg/L buffered sterile solutions at pH 7 for 15 days continuous irradiation at 25 °C.	815635
Dissociation constant in water (purity 99.3 %)	pK_a = 1.1 at 20 °C OECD Guideline 112	70159

Technical material

			Reference
Description	Off-white, slightly sweet odour, powder		8804
Solubility in organic solvents at 25 °C (purity 94.6 %)	Acetone Dichloromethane Ethyl acetate Hexane Methanol Octanol Toluene	> 500 g/L > 500 g/L > 500 g/L 3.0 g/L > 500 g/L 110 g/L > 500 g/L	76994

Metabolites

		Reference
1,2,4-triazole		
Vapour pressure	0.34 Pa at 25 °C OECD Guideline 104 (measurements between -14 °C and 46 °C)	100415
Water solubility	730 g/L at 25 °C interpolated value, measurements at 20 °C and 25 °C	MO-01-005554 Vlasov and Sukhova, 1988
Octanol/water partition coefficient	log P_{OW} = -1.0 at 22 °C, pH 6.2	M8274
Hydrolysis rate (purity 98.6 %)	Essentially no hydrolysis in 10 mg/L buffered sterile solutions at pH 5, 7 and 9 for 30 days at 25 °C in the dark.	83-E-074

CGA 205375		
Vapour pressure	< 5 × 10^{-6} Pa at 25 °C OECD Guideline 104 (measurements at 25 °C)	2001WI07

		Reference
Water solubility	12 mg/L at 25 °C OECD Guideline 105	107459
Octanol/water partition coefficient	log P_{OW} = 3.8 at 25 °C OECD Guideline 107	107458
Hydrolysis rate (purity 99 %)	Essentially no hydrolysis in 0.23 mg/L buffered sterile solutions at pH 4, 7 and 9 for 5 days at 50 °C in the dark.	798658
Photolysis rate (purity 99 %)	Less than 10% photo-degradation by simulated sunlight of 1.0 mg/L buffered sterile solutions at pH 7 for 15 days continuous irradiation at 24.8 °C	815657

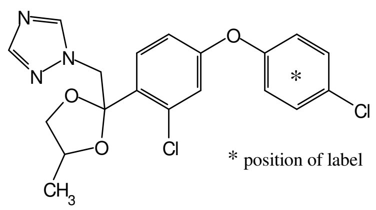
FORMULATIONS

Difenoconazole is available as EC, SC, WG and WP commercial formulations. Some products are mixtures with other fungicides.

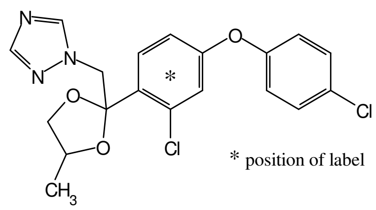
Code	Description	Concentration	Examples
EC	emulsifiable concentrate	100 g/L + fenpropidin	Spyrale
EC	emulsifiable concentrate	150 g/L + propiconazole	Armure 300 EC, Rias 300 EC
EC	emulsifiable concentrate	250 g/L	Score, Bogard
EC	emulsifiable concentrate	250 g/L + propiconazole	Taspa 500 EC, Gem
SC	suspension concentrate	62.5 g/L + carbendazim	Eria, Tenor
SC	suspension concentrate	125 g/L + azoxystrobin	Amistar Top
WG	water dispersible granules	12.5 g/kg + folpet + cymoxanil	Covax WG
WG	water dispersible granules	62.5 g/kg + cyprodinil	Play 31.25 WG
WG	water dispersible granules	100 g/kg	Score, Bogard
WP	wettable powder	100 g/kg	Score 10 WP, Purugen

METABOLISM

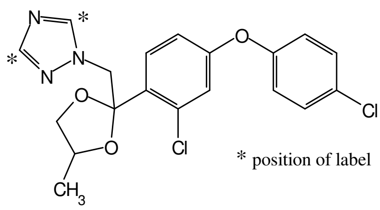
Animal and plant metabolism and environmental fate studies used difenoconazole ^{14}C labelled in the chlorophenoxy ring, the chlorophenyl ring or the triazole ring.



chlorophenoxy label



phenyl label



triazole label

Structures, names and codes for metabolites are summarised below. Five possibilities for describing each metabolite are:

1) A simple name, which could be a common name, a simplified systematic name (e.g. triazole) or a pseudo-common name (e.g. hydroxy-difenoconazole).

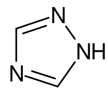
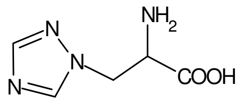
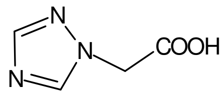
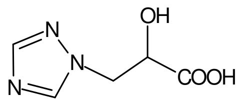
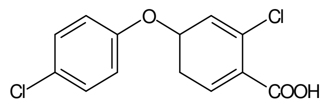
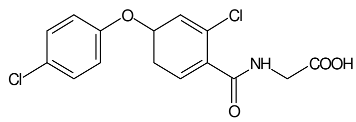
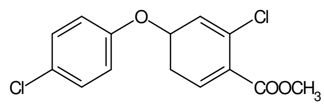
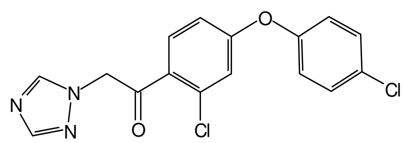
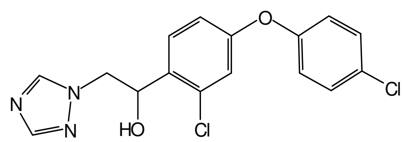
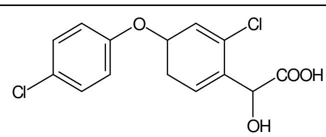
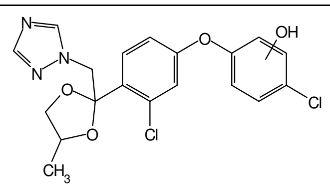
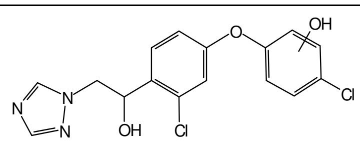
2) The systematic chemical name – it may be too cumbersome for use in discussion and tables.

3) The CAS number – CAS numbers are not available for many metabolites.

4) The company code number, e.g. CGA 205375.

5) Serial numbers, e.g. metab 1, metab 2, etc – not generally used here.

In this evaluation, metabolites are described by a simple name where available or by a company code number.

Simple: triazole Systematic: 1,2,4-triazole CAS number: 288-88-0 Code: CGA 71019	
Simple: triazolyl alanine Systematic: 2-amino-3-(1,2,4]triazol)-1-yl-propionic acid CAS number: 86362-20-1 Code: CGA 131013	
Simple: triazolyl acetic acid Systematic: 1,2,4-triazol-1-yl-acetic acid Code: CGA 142856	
Simple: triazolyl lactic acid Systematic: 1,2,4-triazol-1-yl-lactic acid Code: CGA 205369	
Systematic: 2-chloro-4-(4-chlorophenoxy)-benzoic acid Code: CGA 189138	
Simple: glycine-CGA-189138	
Systematic: 2-chloro-4-(4-chloro-phenoxy)-benzoic acid methyl ester Code: CGA 190978	
Systematic: 1-(2-chloro-4-(4-chloro-phenoxy)-phenyl)-2-(1,2,4-triazol)-1-yl-ethanone Code: CGA 205374	
Systematic: 1-[2-chloro-4-(4-chloro-phenoxy)-phenyl]-2-(1,2,4-triazol)-1-yl-ethanol Code: CGA 205375, CGA 211391	
Systematic: 2-chloro-4-(4-chlorophenoxy)-phenyl-hydroxyacetic acid Code: NOA 448731	
Simple: hydroxy-difenoconazole	
Simple: hydroxy-CGA-205375	

Animal metabolism

The Meeting received animal metabolism studies with difenoconazole in rats, lactating goats and laying hens.

Difenoconazole is rapidly metabolized, initially to CGA 205375 and then with cleavage of the triazole moiety from the chlorophenoxyphenyl moiety. TRR levels are higher in the liver than in other tissues. Most of the TRR is rapidly excreted.

Rats

Capps *et al*. 1990, (ABR-90019) identified the metabolites of difenoconazole in rats after oral dosing with [^{14}C-triazole]difenoconazole and [^{14}C-phenyl]difenoconazole. The following metabolites were identified in excreta: CGA 205375, 1,2,4-triazole, CGA 189138, Metabolites A1 and A2 and Metabolites B (diastereomers). A subsequent report (Capps and Anderson, 1993, ABR-90019) identified NOA 448731, sulphate conjugates of CGA 205375 and sulphate conjugates of Metabolites A in urine. See also the toxicology evaluation.

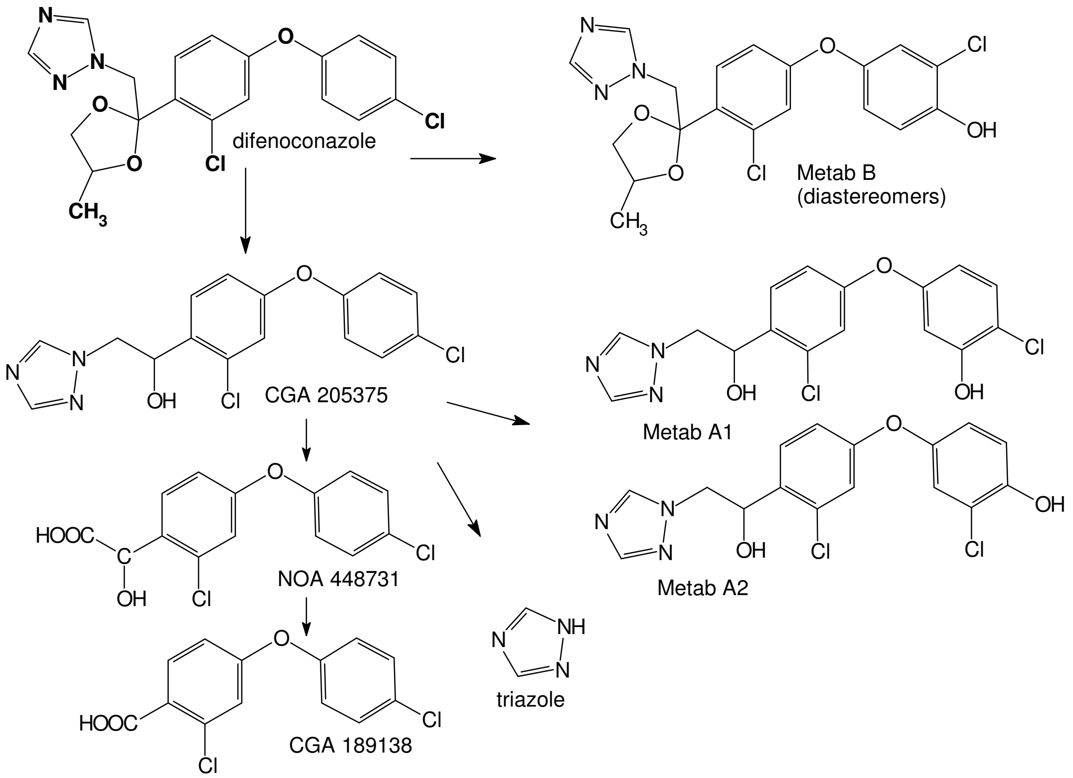


Figure 1. Proposed pathway for the metabolism of difenoconazole in the rat

Lactating goats

A lactating goat weighing 31.5 kg was dosed orally once daily for 10 consecutive days by gelatin capsule with 7.5 mg/animal/day of [^{14}C-triazole]difenoconazole, equivalent to 5.6 ppm in the feed (Madrid, 1988, ABR-88087) for a 1.35 kg/day feed consumption. A second lactating goat weighing 32 kg was dosed orally once daily for 10 consecutive days by gelatin capsule with 7.5 mg/animal/day of [^{14}C-phenyl]difenoconazole, equivalent to 4.7 ppm in the feed for a 1.80 kg/day feed consumption.

Milk and excreta were collected daily. The animals were slaughtered approximately 22 and 23 hours after the final dose for tissue collection. Recoveries of administered ^{14}C were 107% and 89% for the [^{14}C]triazole and [^{14}C]phenyl labels respectively.

The majority of the administered ^{14}C was present in the excreta (31% in urine, 75% in faeces for [^{14}C]triazole label; 21% in urine, 67% in faeces for [^{14}C]phenyl label). Milk accounted for 0.50% and 0.18% and tissues for 0.90% and 0.44% of the administered ^{14}C. The distribution of the radiolabel and identified metabolites in tissues and milk are summarised in Table 1. Residues of ^{14}C were higher in liver (0.28 and 0.26 mg/kg) than in other tissues.

Residues in milk reached a plateau by day 2 (0.007 mg/kg) for the [^{14}C]phenyl label and by days 4-7 (0.032-0.043 mg/kg) for the [^{14}C]triazole label. The concentration of ^{14}C appearing in milk and milk fat was higher for the [^{14}C]triazole label. Of the ^{14}C in milk, 19% and 32% were distributed into the fat portion for the [^{14}C]triazole and [^{14}C]phenyl labels respectively.

Table 1. Distribution of ^{14}C residue and metabolites in tissues and milk of lactating goats dosed orally for 10 days with 7.5 mg/animal/day of [^{14}C-triazole] difenoconazole, equivalent to 5.6 ppm in the feed or [^{14}C-phenyl]difenoconazole, equivalent to 4.7 ppm in the feed

Residue component	Concentration, mg/kg, expressed as parent							
	Loin muscle	Leg muscle	Liver	Kidney	Omental fat	Perirenal fat	Milk, day 7	Milk day 8
[^{14}C-triazole] label								
Total ^{14}C residue (TRR)	0.026	0.028	0.28	0.094	0.064	0.035	0.043	
Extracted residue			0.25	0.075				
Unextractable			0.042	0.018				
Difenoconazole			0.002					
CGA 205375			0.16				0.0014	
1,2,4-triazole			0.009				0.020	
Metab B			0.005					
Metab C			0.003					
Metab D			0.002				0.001	
Metab G			0.031					
[^{14}C-phenyl] label								
Total ^{14}C residue (TRR)	0.008	0.007	0.26	0.064	0.025	0.022		0.008
Extracted residue			0.21					
Unextractable			0.079					
Difenoconazole			0.003					
CGA 205375			0.15					
CGA 205374			0.002					
CGA 189138			0.004					
Metab B			0.004					
Metab C			0.005					
Metab D			0.002					
Metab G			0.013					

Four lactating goats were dosed orally once daily for 3 consecutive days by gelatin capsule with 150 mg/animal/day of [^{14}C-triazole]difenoconazole (2 goats) and [^{14}C-phenyl]difenoconazole (2 goats), equivalent to 100 ppm in the feed (Maynard, 1990. ABR-89100).

Milk was collected twice daily and excreta were collected daily. The animals were slaughtered approximately 4 – 6 h after the final dose for tissue collection. Recoveries of administered ^{14}C were 64 and 52% for the [^{14}C]phenyl label and 40 and 63% for the [^{14}C]triazole label.

Tissues were extracted with organic and aqueous solvents. Milk was extracted with organic solvents and separated into aqueous, organic and solid phases. Components of the TRR were separated by LC and TLC and were identified by comparison with standard compounds (Table 2).

Table 2. Distribution of ^{14}C residue and metabolites in tissues and milk of lactating goats dosed orally for 3 days with 150 mg/animal/day of [^{14}C]difenoconazole, equivalent to 100 ppm in the feed

Residue component	Concentration, mg/kg, expressed as parent (mean of samples from 2 animals)					
	Muscle	Liver	Kidney	Omental fat	Milk, day 1	Milk day 2
[^{14}C-triazole] label						
Total ^{14}C residue (TRR)	0.57	7.5	1.8	1.14	0.18	0.38
Extracted residue %	92%	105%	107%	94%		83%
Unextractable %	14%	4%	8%	6%		6%
Difenoconazole	0.021	0.62	0.095	0.074		0.023
CGA 205375	0.24	3.7	0.93	0.86		0.13
CGA 205374	0.008					
1,2,4-triazole	0.010					0.022
Hydroxy-difenoconazole				0.021		
Hydroxy- CGA-205375	0.013			0.014		0.011
% of TRR identified	52%	58%	56%	85%		49%
[^{14}C-phenyl] label						
Total ^{14}C residue (TRR)	0.20	6.0	1.55	0.56	0.105	0.14
Extracted residue %	86%	95%	110%	96%		100%
Unextractable %	14%	3%	12%	4%		18%
Difenoconazole	0.007	0.40	0.023	0.018		0.012
CGA 205375	0.14	3.2	0.48	0.41		0.029
CGA 205374		0.14				
CGA 189138						0.009
Hydroxy-difenoconazole				0.033		0.021
Hydroxy- CGA-205375		0.37	0.031	0.019		0.006
% of TRR identified	72%	75%	34%	86%		56%

Two lactating goats weighing 46 and 51 kg were dosed orally once daily for 4 consecutive days by gelatin capsule with 150 mg/animal/day of [^{14}C-phenyl]difenoconazole, equivalent to 100 ppm in the feed (Ray, 1996, ABR-95099) for a 1.5 kg/day feed consumption.

Milk and excreta were collected daily. The animals were slaughtered approximately 6 hours after the final dose for tissue collection. Subsamples of hind leg and tenderloin muscle were combined to produce the muscle sample. Subsamples of omental fat and perirenal fat were combined for the fat sample. Recoveries of administered ^{14}C were approximately 71% (37% in faeces, 29% in urine, 0.18% in bile, 0.42% in blood, 3.3% in tissues and 0.28% in milk).

Tissues were extracted with acetonitrile and water. Milk was extracted with acetonitrile, water and acetone. Components of the TRR were separated by LC and TLC and were identified by comparison with standard compounds (Table 3).

CGA 205375 was the major component of the residue in all tissues and milk. Parent difenoconazole was present in all tissues and milk, but never exceeding 10% of the TRR. A number of metabolites resulted from hydroxylation and conjugation with glucuronic acid, sulphate and glycine. The concentration of the main component, CGA 205375, in fat was 2.3 times its concentration in muscle, but much below its concentration in liver and similar to that in kidney, suggesting borderline fat solubility.

Table 3. Distribution of ^{14}C residue and metabolites in tissues and milk of a lactating goat dosed orally for 4 consecutive days with 150 mg/animal/day of [^{14}C-phenyl]difenoconazole, equivalent to 100 ppm in the feed

Residue component	Concentration, mg/kg, expressed as parent				
	Muscle	Liver	Kidney	Fat	Milk, day 3
Total ^{14}C residue (TRR)	0.45	9.8	2.5	1.1	0.32
Extracted residue %	101%	95%	96%	106%	108%
Unextractable %	0.75%	4.9%	2.0%	2.4%	4.9%
Difenoconazole	0.020	0.89	0.014	0.095	0.028
Glucuronide-hydroxy-difenoconazole	nd	0.16	0.019	nd	nd
Sulphate-hydroxy-difenoconazole	nd	nd	nd	nd	0.009
CGA 205375	0.42	7.1	1.2	0.95	0.12
Hydroxy-CGA-205375	0.009	0.098	0.016	0.020	0.001
Glucuronide-CGA-205375	0.006	0.73	0.46	nd	0.002
Sulphate-CGA-205375	0.002	0.068	0.27	nd	0.034
Sulphate-hydroxy-CGA-205375	nd	0.18	0.082	nd	0.013
Glucuronide-hydroxy-CGA-205375	nd	nd	0.027	nd	nd
CGA 205374	nd	nd	nd	0.033	nd
CGA-189138	0.002	0.020	0.057	nd	0.003
Glycine-CGA-189138	0.004	0.039	0.30	nd	0.11
Glucuronide-hydroxide-CGA-189138	nd	nd	0.087	nd	nd
% of TRR identified	101%	95%	96%	106%	108%

nd: not detected

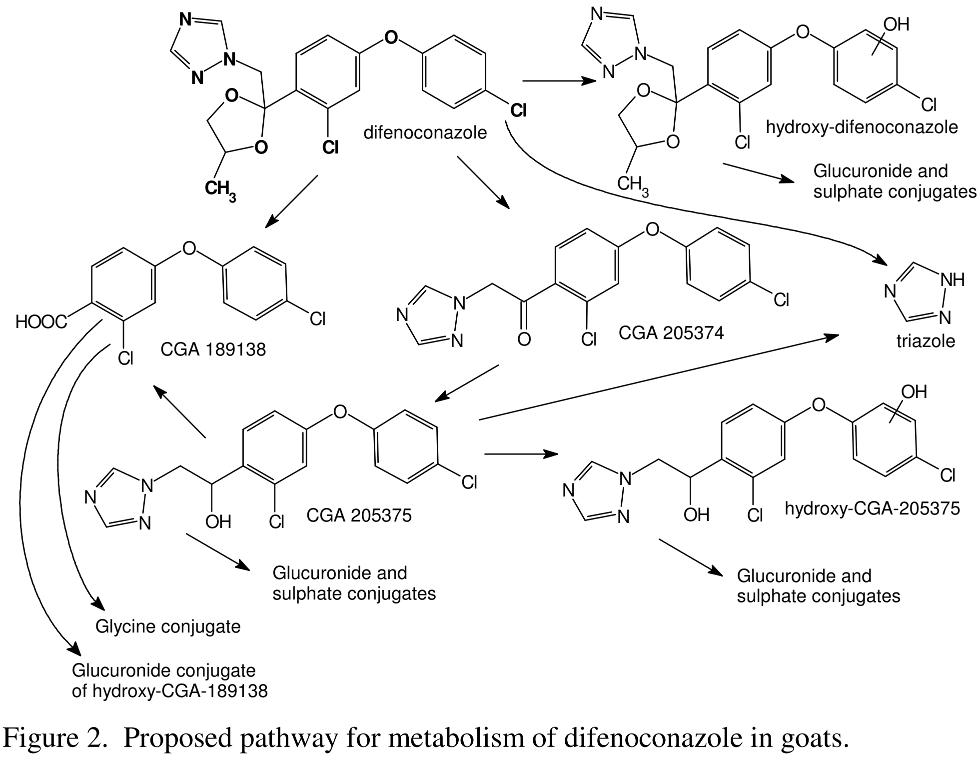


Figure 2. Proposed pathway for metabolism of difenoconazole in goats.

Laying hens

A group of laying white leghorn hens (4 birds), mean body weight 1.5 kg at study initiation, were dosed orally once daily via gelatin capsule for 14 consecutive days with 0.55 mg/bird/day of

[14C]difenoconazole (2 birds with [14C]phenyl label and 2 birds with [14C]triazole label), equivalent to 5 ppm in the feed for a 108 g/day mean feed consumption (Madrid, 1989, ABR-89051).

Eggs were collected daily. The birds were slaughtered approximately 22 hours after the final dose for tissue collection (lean meat, liver, kidney, skin and attached fat and peritoneal fat). Recovery of administered ^{14}C ranged from 91.5% to 97.5%. Most of the ^{14}C (over 89% of administered dose) was eliminated via the excreta.

Tissues, egg whites and egg yolks were subjected to biphasic extraction, producing organic, aqueous and nonextractable fractions.

Apparent plateaus for TRR in egg whites and yolks were reached after approximately 4 and 7 days of dosing respectively. The plateau TRR values in egg whites were quite different for the two labels: 0.14 mg/kg for [14C]triazole label and 0.011 mg/kg for [14C]phenyl label, whereas the plateau levels in the yolks were essentially the same: 0.28 and 0.29 mg/kg for the [14C]triazole and [14C]phenyl labels respectively (Figure 3). Levels of ^{14}C in tissues are summarised in Table 4.

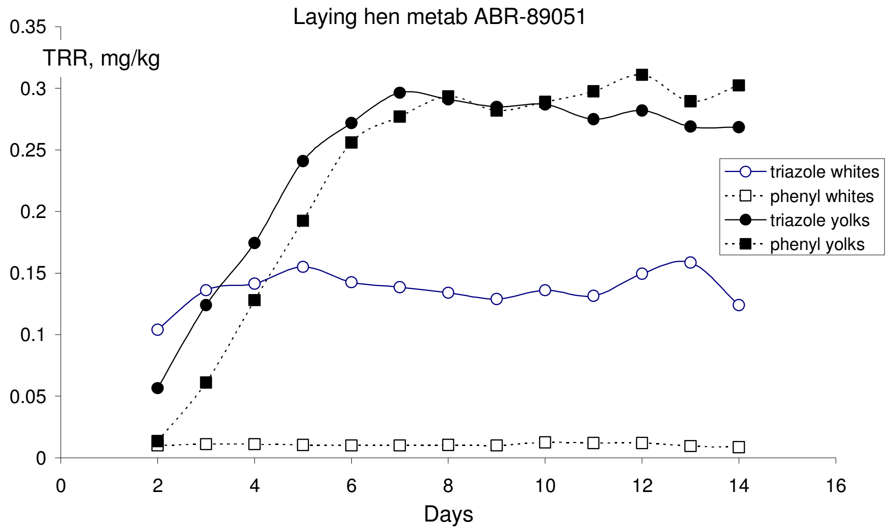


Figure 3. TRR in egg whites and yolks from a laying hen metabolism study.

Table 4. Distribution of ^{14}C residue in tissues and eggs of laying hens dosed orally daily for 14 consecutive days with 0.55 mg/bird/day of [14C]difenoconazole, equivalent to 5 ppm in the feed

Residue component	Concentration, mg/kg, expressed as parent (mean of samples from 2 birds)				
	Skin + fat	Lean meat	Peritoneal fat	Liver	Kidney
[14C-triazole] label					
Total ^{14}C residue (TRR)	0.031	0.083	0.019	0.13	0.43
Extracted residue %		93%		85%	85%
Unextractable %		8%		12%	11%
[14C-phenyl] label					
Total ^{14}C residue (TRR)	0.016	0.008	0.040	0.13	0.49
Extracted residue %				63%	76%
Unextractable %				27%	29%

Two groups of laying hens were dosed orally once daily via gelatin capsule for 3 consecutive days with 7.5 mg/bird/day of [14C]difenoconazole (10 birds with [14C]phenyl label and 10 birds with [14C]triazole label), equivalent to 68 ppm in the feed (Maynard, 1990, ABR-89101).

Eggs were collected daily. The birds were slaughtered approximately 4 – 6 hours after the final dose for tissue collection (liver, kidney, muscle and fat). Recoveries of administered ^{14}C were 78% and 80% for the [^{14}C]phenyl and [^{14}C]triazole labels respectively. Most of the ^{14}C (76% of the administered dose for both labels) was eliminated via the excreta.

Egg whites and egg yolks were extracted with acetonitrile. Tissues were extracted with aqueous and solvent phases. After cleanup steps, metabolites were identified by TLC and LC. Metabolite distributions are summarised in Table 5.

Table 5. Distribution of 14C residue and metabolites in tissues and eggs of laying hens dosed orally daily for 3 consecutive days with 7.5 mg/bird/day of [14C]difenoconazole, equivalent to 68 ppm in the feed

Residue component	Concentration, mg/kg, expressed as parent					
	Liver	Kidney	Muscle	Fat	Egg white, day 2	Egg yolk
[^{14}C-triazole] label						
Total ^{14}C residue (TRR)	4.3	1.9	0.51	0.46	0.27	0.13
Extracted residue %	90%	86%	108%	63%		
Unextractable %	31%	9%	25%	37%		
Difenoconazole		0.032	0.005	0.007	0.013	0.001
Hydroxy-difenoconazole		0.061			0.003	0.006
1,2,4-triazole	0.23	0.13	0.025	0.004	0.18	0.043
CGA-205375	1.3	0.37	0.045	0.212	0.021	0.047
Hydroxy-CGA-205375	0.35	0.092	0.007	0.026	0.002	0.003
CGA-205374	0.060	0.040		0.004		
% of TRR identified	45%	39%	16%	55%	82%	75%
[^{14}C-phenyl] label						
Total ^{14}C residue (TRR)	4.7	2.2	0.10	0.45	0.023	0.037
Extracted residue %	86%	74%		85%		
Unextractable %	35%	30%		15%		
Difenoconazole	0.20			0.007	0.001	< 0.001
Hydroxy-difenoconazole	0.14	0.11			0.003	0.005
CGA-205375	1.6	0.49	0.035	0.29	0.019	0.027
Hydroxy-CGA-205375	0.46	0.072	0.004	0.037	0.001	< 0.001
CGA-189138	0.13					
CGA-205374	0.084	0.12		0.008		
% of TRR identified	57%	35%	39%	75%	103%	87%

Five laying white leghorn hens, mean body weight 1.54 – 1.68 kg at study initiation, were dosed orally once daily via gelatin capsule for 4 consecutive days with 12.5 mg/bird/day of [^{14}C-triazole]difenoconazole, equivalent to a nominal 100 ppm (87 – 215 ppm, mean 121 ppm) in the feed for a 64 – 144 g/day feed consumption (Ray, 2004, 786-02).

Eggs were collected daily. The birds were slaughtered approximately 6 hours after the final dose for tissue collection (liver, muscle and peritoneal fat). The recovery of administered ^{14}C was 92% with most of the ^{14}C (66% of the administered dose) in the excreta. Eggs contained 1.2% and tissues 6.5% of the administered ^{14}C.

The tissue and egg samples from each bird were combined to produce composite samples for analysis. Samples were extracted with acetonitrile + water. After cleanup steps, metabolites were identified by TLC and LC. Metabolite distributions are summarised in Table 6.

Table 6. Distribution of ^{14}C residue and metabolites in tissues and eggs of laying white leghorn hens dosed orally daily for 4 consecutive days with 12.5 mg/bird/day of [^{14}C-triazole]difenoconazole, equivalent to a nominal 100 ppm in the feed.

Residue component	Concentration, mg/kg, expressed as parent				
	Liver	Muscle	Peritoneal fat	Egg white, day 4	Egg yolk, day 3
Total ^{14}C residue (TRR)	13	4.9	10.4	4.0	4.5
Extracted residue %	97%	92%	101%	99.9%	99.9%
Unextractable %	2.7%	4.8%	0.74%	0.38%	1.7%
Difenoconazole	0.78	0.11	1.9	nd	0.24
1,2,4-triazole	2.4	2.7	0.48	3.0	1.4
CGA-205375	7.3	1.2	6.3	0.10	2.4
CGA-205374	0.24	nd	nd	nd	nd
% of TRR identified	79%	82%	83%	78%	91%

nd: not detectable

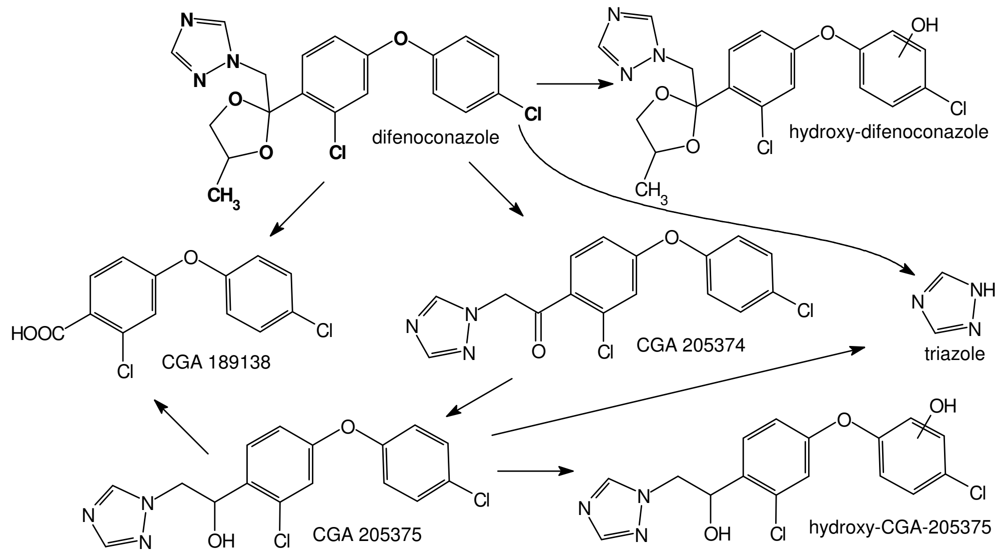


Figure 4. Proposed pathway for the metabolism of difenoconazole in the laying hen

Plant metabolism

The Meeting received plant metabolism studies with difenoconazole in tomatoes, wheat, potatoes, grapes and oilseed rape.

Difenoconazole is generally slowly absorbed and metabolized. In most cases, particularly for parts of the plant directly exposed to the treatment, the parent difenoconazole is the dominant part of the residue. The residue in parts of the plant not directly exposed are more likely to contain a residue dominated by a mobile water-soluble metabolite such as triazolylalanine.

The following plant metabolites apparently do not occur as animal metabolites of difenoconazole: triazolylalanine (2-amino-3-(1,2,4]triazol)-1-yl-propionic acid), triazolyl acetic acid (1,2,4-triazol-1-yl-acetic acid) and triazolyl-lactic acid (1,2,4-triazol-1-yl-lactic acid).

Tomatoes

In a tomato metabolism study in USA, (Madrid and Huber, 1987, ABR-87025) foliar sprayed tomato plants (variety Sunny) in pots in a greenhouse 6 times at 7-day intervals with [^{14}C]phenyl and [^{14}C]triazole labelled difenoconazole at the equivalent of 0.12 kg ai/ha. Samples of tomato plants were taken at various stages of treatment and mature fruit were sampled 7 days after the final treatment. TLC was used to identify components of the residue (Table 7). The results were consistent between the label positions, with parent difenoconazole constituting a major or substantial component of the residue on the foliage.

Table 7. Distribution of ^{14}C residue and metabolites in cuttings, foliage and fruit of greenhouse tomato plants sprayed 6 times with [^{14}C]phenyl and [^{14}C]triazole labelled difenoconazole at the equivalent of 0.12 kg ai/ha

TOMATO METABOLISM Residue component	Concentration, mg/kg, expressed as parent or% TRR						
	Cuttings after spray 1	Cuttings before spray 3	Foliage before spray 5	Fruit before spray 5	Mature foliage	Mature fruit (green)	Mature fruit (ripe)
[^{14}C-phenyl] label							
Total ^{14}C residue (TRR)	4.0	4.0	3.3	0.079	2.8	0.016	0.037
Extracted residue % TRR	87%	91%	96%	87%	82%	84%	86%
Unextractable % TRR	1.4%	9.3%	12%	5.0%	13%	12 %	10%
Difenoconazole % TRR		58%	55%		37%		
CGA 205375 + CGA 205374 %TRR		1.2%	1.3%		0.8%		
CGA 189138 %TRR		2.4 %	3.6%		5.6%		
[^{14}C-triazole] label							
Total ^{14}C residue (TRR)	2.6	2.0	2.4	0.23	2.8	0.13	0.12
Extracted residue %TRR	94%	100%	90%	96%	79%	93%	89%
Unextractable %TRR	2.6%	9.6%	8.2%	6.9%	12%	0.6%	1.3%
Difenoconazole %TRR		58%	51%		36%		
CGA 205375 + CGA 205374 %TRR		1.9%	1.1%		0.9%		

In a field-grown tomato metabolism study in California, USA, (Madrid and Huber, 1987, ABR-87033) foliar sprayed tomato plants (variety UC-82) in field plots 3 times at 14-day intervals with [^{14}C]phenyl and [^{14}C]triazole labelled difenoconazole at the equivalent of 0.25 kg ai/ha. Samples of tomato plants were taken at various stages of treatment and mature fruit were sampled 40 days after the final treatment. Components of the residue were identified by TLC (Table 8). The results were consistent between the two label positions, with parent difenoconazole constituting a major or substantial component of the residue on the foliage. The results were also consistent with those of the previous greenhouse tomato metabolism study.

Table 8. Distribution of ^{14}C residue and metabolites in cuttings, foliage and fruit of field-grown tomato plants sprayed with [^{14}C]phenyl and [^{14}C]triazole labelled difenoconazole at the equivalent of 0.25 kg ai/ha

TOMATO METABOLISM Residue component	Concentration, mg/kg, expressed as parent or% TRR						
	Cuttings after spray 1	Cuttings before spray 2	Foliage before spray 3	Fruit before spray 3	Mature foliage	Mature fruit (green)	Mature fruit (ripe)
[^{14}C-phenyl] label							
Total ^{14}C residue (TRR)	9.4	1.0	2.1	0.012	3.5	0.029	0.026
Extracted residue% TRR	93%	90%	91%		77%		
Unextractable % TRR	3.2%	9.0 %	13%		16%		
Difenoconazole% TRR			59%		31%		
CGA 205375 + CGA 205374 %TRR			3.8%		3.4%		
CGA 189138 %TRR			4.3%		5.2%		
[^{14}C-triazole] label							
Total ^{14}C residue (TRR)	6.7	0.98	2.9	0.11	7.4	0.24	0.27
Extracted residue %TRR	102%	89%	75%	107%	87%	100%	89%
Unextractable %TRR	4.4%	8.9%	8.0%	1.3%	21%	0.6%	1.0%
Difenoconazole %TRR			52%		28%		
CGA 205375 + CGA 205374 %TRR			3.5%		4.3%		

In a tomato metabolism study in USA, (Velagaleti, 1990, N-0964-0600) foliar sprayed tomato plants (variety Sunny) in pots in a greenhouse 6 times at 7 day intervals with [^{14}C-triazole]difenoconazole at the equivalent of 0.12 kg ai/ha. Samples of tomato plants were taken at various stages of treatment and mature fruit were sampled 33 days after the final treatment. Components of the residue were identified by TLC (Table 9).

A high percentage of the residue was extractable from cuttings, foliage and fruit. In fruit, parent difenoconazole and metabolite triazolylalanine were major components of the residue. Metabolites CGA 205374 and CGA 203575 were identified as very minor parts (< 2%) of the residue in fruits and foliage. Hydroxy-difenoconazole, hydroxy-CGA 205374 and hydroxy-CGA 205375 were also identified as very minor components of the residue in foliage after cellulase digestion.

Table 9. Distribution of ^{14}C residue and metabolites in cuttings, foliage and fruit of greenhouse tomato plants sprayed 6 times with [^{14}C]triazole labelled difenoconazole at the equivalent of 0.12 kg ai/ha

Residue component	Concentration, mg/kg, expressed as parent or% TRR					
Cuttings and foliage	Cuttings after dose 1	Cuttings before dose 3	Cuttings before dose 5	Cuttings before dose 6	Foliage, final harvest	
Total ^{14}C residue (TRR)	3.8 mg/kg	3.5 mg/kg	6.4 mg/kg	9.7 mg/kg	7.7 mg/kg	
Extracted residue % TRR	91%	110%	93%	86%	101%	
Unextractable % TRR	2%	3%	4%	14%	7%	
Difenoconazole % TRR conc					68% 5.2 mg/kg	
CGA 205374 %TRR conc					1.6% 0.13 mg/kg	
CGA 205375 %TRR conc					1.2% 0.096 mg/kg	
Fruit	Fruit green before dose 5	Fruit green before dose 6	Fruit green 7 days after dose 6	Fruit green final harvest	Fruit coloured final harvest	Fruit ripe final harvest
Total ^{14}C residue (TRR)	0.17 mg/kg	0.15 mg/kg	0.16 mg/kg	0.14 mg/kg	0.13 mg/kg	0.20 mg/kg
Extracted residue %TRR	95%	93%	93%	92%	88%	93%
Unextractable %TRR	3.0%	1.7%	1.9%	2.4%	1.6%	3%
Difenoconazole %TRR conc			47% 0.074 mg/kg	12% 0.017 mg/kg	13% 0.016 mg/kg	51% 0.10 mg/kg
CGA 205374 %TRR conc			0.73% 0.001 mg/kg	0.35% 0.0005 mg/kg	0.21% 0.0003 mg/kg	0.52% 0.001 mg/kg
CGA 205375 %TRR conc			0.63% 0.001 mg/kg	0.33% 0.0005 mg/kg	0.46% 0.0006 mg/kg	0.74% 0.0015 mg/kg
Triazolylalanine %TRR conc			22% 0.034 mg/kg	42% 0.059 mg/kg	39% 0.050 mg/kg	19% 0.039 mg/kg

In a tomato metabolism study in USA, (Schweitzer, 1990, N-0964-0700) foliar sprayed tomato plants (variety Sunny) in pots in a greenhouse 6 times at 7 day intervals with [^{14}C]chlorophenoxy[1] labelled difenoconazole at the equivalent of 0.12 kg ai/ha. Samples of tomato plants were taken at various stages of treatment and mature fruit were sampled 33 days after the final treatment. TLC was used to identify components of the residue (Table 10).

A high percentage of the residue was extractable. Parent difenoconazole was the major component of the residue in both foliage and fruit. Metabolites CGA 205374, CGA 205375 were identified as minor residue components in the fruit (< 2% of TRR). Cellulase treatment of tomato foliage released small amounts of hydroxy-difenoconazole and hydroxy-CGA 205375, demonstrating glycoside conjugation of some metabolites. Low concentrations of glycosides were also observed in the fruit.

1 Although the report title uses the words "phenyl-14C-" the 14C label was in the chlorophenoxy ring.

Table 10. Distribution of ^{14}C residue and metabolites in foliage and fruit of greenhouse tomato plants sprayed with [^{14}C]chlorophenoxy labelled difenoconazole at the equivalent of 0.12 kg ai/ha.

Residue component	Concentration, mg/kg, expressed as parent or %TRR				
Cuttings and foliage	Foliage after spray 1	Foliage before spray 3	Foliage before spray 5	Foliage before spray 6	Foliage, final harvest
Total ^{14}C residue (TRR)	2.6 mg/kg	4.0 mg/kg	5.3 mg/kg	6.8 mg/kg	8.3 mg/kg
Extracted residue %TRR	100%	91%	84%	84%	89%
Unextractable %TRR	1.1%	4.1%	10%	12%	5%
Difenoconazole %TRR conc					65% 5.4 mg/kg
CGA 205374 %TRR conc					3.9% 0.32 mg/kg
CGA 205375 %TRR conc					1.3% 0.11 mg/kg
CGA 189138 %TRR conc					0.9% 0.08 mg/kg
Fruit	Fruit green before spray 5	Fruit green before spray 6	Fruit green 7 days after spray 6	Fruit green final harvest	Fruit ripe final harvest
Total ^{14}C residue (TRR)	0.20 mg/kg	0.19 mg/kg	0.22 mg/kg	0.04 mg/kg	0.17 mg/kg
Extracted residue %TRR	85%	102%	82%	94%	98%
Unextractable %TRR	12%	5%	14%	12%	5%
Difenoconazole %TRR conc					66% 0.11 mg/kg
CGA 205374 %TRR conc					1.4% 0.002 mg/kg
CGA 205375 %TRR conc					1.7% 0.003 mg/kg

Wheat

In a wheat metabolism study in USA, (Hubbard 1991, ABR-90009) treated spring wheat seeds (variety Marshall FL-890836) with [^{14}C]phenyl and [^{14}C]triazole labelled difenoconazole formulated as an FS (flowable concentrate for seed treatment) at a target rate of 0.24 g ai/kg seed (measured 0.32 and 0.23 g ai/kg for phenyl and triazole labels respectively) and sowed them in experimental field plots of 1 – 2 m^2 at locations in New York and Illinois. The sowing rate was 79 kg seed per hectare. The [^{14}C]phenyl-difenoconazole treated seed failed to germinate at the New York site possibly because of solvent contamination. Plant samples were taken at 25% maturity (31 – 34 days post sowing), 50% maturity (48 – 62 days post sowing) and stalks, hull and grain samples were taken 59 – 83 days post sowing. The distribution of ^{14}C in the crops is summarised in Table 11.

When TRR exceeded 0.05 mg/kg, extractability of the residue with methanol+water (8+2) was measured and the extracts were examined by TLC. Generally, the TRR was higher for the [^{14}C]triazole label than for the [^{14}C]phenyl label, suggesting metabolic changes of the residue (Table 11). A high percentage of [^{14}C]triazole-labelled residues was extractable (mostly partition to aqueous phase), also suggesting that the triazole moiety was readily translocated throughout the plant.

TLC analysis of extracts of mature wheat stalks and wheat grain from the [^{14}C]triazole label difenoconazole treated seed demonstrated the presence of 1,2,4-triazole and 1,2,4-triazolylacetic acid. Metabolite CGA 205375 was present in cellulase-treated extracts of immature wheat tops from the [^{14}C]phenyl label treatment, as shown by TLC.

Table 11. Distribution of ^{14}C residue in field-plot wheat plants and grain produced from seed treated with [^{14}C]phenyl and [^{14}C]triazole labelled difenoconazole at 0.32 and 0.23 g ai/kg seed respectively

WHEAT METABOLISM Residue component	Concentration, mg/kg expressed as parent, or %TRR Tops, 25% mature	Tops 50% mature	Stalks, mature	Hulls, mature	Grain, mature
[^{14}C]triazole label (NY)					
Total ^{14}C residue (TRR)	0.049 mg/kg	0.053 mg/kg	0.059 mg/kg	0.075 mg/kg	0.14 mg/kg
Extracted residue % TRR		89%	97%		90%
Unextractable % TRR		6.4%	16%		10%
1,2,4-triazole			identified		identified
triazolylacetic acid			identified		identified
[^{14}C-triazole] label (IL)					
Total ^{14}C residue (TRR)	0.007 mg/kg	0.010 mg/kg	0.011 mg/kg	0.016 mg/kg	0.024 mg/kg
[^{14}C]phenyl label (IL)					
Total ^{14}C residue (TRR)	0.095 mg/kg	0.008 mg/kg	0.013 mg/kg	0.004 mg/kg	0.004 mg/kg
Extracted residue %TRR	42%				
Unextractable %TRR	23%				
CGA 205375	identified				

In a greenhouse wheat metabolism study in USA, (Hubbard, 1991, ABR-90010) treated spring wheat seeds (variety Hill 81) with [^{14}C]phenyl and [^{14}C]triazole labelled difenoconazole formulated as an FS (flowable concentrate for seed treatment) at a target rate of 0.24 g ai/kg seed (measured 0.25 and 0.30 g ai/kg for phenyl and triazole labels respectively) and sowed them in pails of loamy sand. Plant samples were taken at 25% maturity (40 days post sowing), 50% maturity (72 days post sowing) and stalks, hull and grain samples were taken at maturity (236 days post sowing) for measurement of ^{14}C content (Table 12). When TRR exceeded 0.05 mg/kg, extractability of the residue with methanol+water (8+2) was measured and the extracts were examined by TLC.

The TRR content in the mature crop was much higher for the triazole label than for the phenyl label, suggesting cleavage of the difenoconazole and translocation mobility of the triazole containing moiety. Extracts were cleaned up and examined by TLC. Parent difenoconazole and metabolite CGA 205375 were detected in the 25% maturity sampling. The presence of triazolylalanine and triazolylacetic acid was suggested but not confirmed for extracts of the mature grain (triazole label).

Table 12. Distribution of ^{14}C residue in greenhouse wheat plants and grain produced from seed treated with [^{14}C]phenyl and [^{14}C]triazole labelled difenoconazole at 0.25 and 0.30 g ai/kg seed respectively

WHEAT METABOLISM Residue component	Concentration, mg/kg expressed as parent, or %TRR Tops, 25% mature	Tops 50% mature	Stalks, mature	Hulls, mature	Grain, mature
[^{14}C]triazole label					
Total ^{14}C residue (TRR)	0.15 mg/kg	0.010 mg/kg	0.069 mg/kg	0.14 mg/kg	0.18 mg/kg
Extracted residue %TRR	77%		88%	96%	80%
Unextractable %TRR	15%		18%	10%	25%
Difenoconazole %TRR	7%				
[^{14}C]phenyl label					
Total ^{14}C residue (TRR)	0.075 mg/kg	0.016 mg/kg	0.016 mg/kg	0.005 mg/kg	0.003 mg/kg
Extracted residue %TRR	91%				
Unextractable %TRR	15%				
Difenoconazole %TRR	8%				
CGA 205375 %TRR	23%				

In a greenhouse wheat metabolism study in USA, (Hubbard 1991, ABR-90011) foliar sprayed spring wheat (variety James) 4 times with [^{14}C]phenyl and [^{14}C]triazole labelled difenoconazole formulated as an EC at a rate equivalent to 0.25 kg ai/ha. Wheat was grown in pails of loamy sand, at 15 – 20 plants per pail. The first difenoconazole application was 43 days post sowing at the early boot stage. Three further applications followed at 7 or 8 day intervals. Mature samples were harvested 29 days after the final application. The distribution of ^{14}C in wheat plants and grain is summarised in Table 13.

When TRR exceeded 0.05 mg/kg, extractability of the residue with methanol+water (8+2) was measured and the extracts were cleaned up, subjected to enzyme hydrolysis for release of conjugates, and the constituents identified and characterized by TLC.

Exposed parts of the plant, i.e. tops and stalks, produced quite similar TRR levels for the two different labels. In this situation difenoconazole was the major part of the residue. In the unexposed part, i.e., the grain, the TRR levels are quite different (more than 20×) because only the triazole moiety metabolites are mobile within the plant and can translocate to any part. Parent difenoconazole was not identified in the grain.

Table 13. Distribution of ^{14}C residue in greenhouse wheat plants and grain subjected to 4 foliar applications of [^{14}C]phenyl and [^{14}C]triazole labelled difenoconazole formulated as an EC at a rate equivalent to 0.25 kg ai/ha

WHEAT METABOLISM	Concentration, mg/kg expressed as parent, or %TRR				
Residue component	Tops, 25% mature, 0 days after applic 1	Tops 50% mature, 8 days after applic 2	Stalks, mature, 29 days after applic 4	Hulls, mature, 29 days after applic 4	Grain, mature, 29 days after applic 4
[^{14}C]triazole label					
Total ^{14}C residue (TRR)	6.3 mg/kg	8.7 mg/kg	54 mg/kg	4.1 mg/kg	1.4 mg/kg
Extracted residue %TRR	86%	88%	78%	58%	70%
Unextractable % TRR	7.0%	10%	13%	31 %	23%
Difenoconazole %TRR conc	90% 5.6 mg/kg		50% 27 mg/kg		
Hydroxy-difenoconazole %TRR conc			1% 0.54 mg/kg		
CGA 205375 %TRR conc			5% 2.7 mg/kg		
Hydroxy-CGA 205375 %TRR conc			1% 0.54 mg/kg		
Triazolylacetic acid %TRR conc					20% 0.28 mg/kg
1,2,4-triazole %TRR conc					10% 0.14 mg/kg
[^{14}C]phenyl label					
Total ^{14}C residue (TRR)	6.9	8.3	47	5.2	0.064
Extracted residue %TRR	92%	91%	82%	53%	0.0%
Unextractable %TRR	5.8%	10%	14%	41%	82%
Difenoconazole %TRR conc	85% 5.8 mg/kg		50% 23 mg/kg		

Potatoes

In a greenhouse potato metabolism study in USA, (Schweitzer, 1990, N-0964-0400) foliar sprayed potato plants (variety Red Pontiac) in pots of a sandy loam 6 times at 7 day intervals with [^{14}C]chlorophenoxy labelled difenoconazole[2] at the equivalent of 0.12 kg ai/ha per application. Samples of potato plants were taken at various stages of treatment and the mature crop was harvested 7 days after the final application. The distribution of the ^{14}C and the identification of components of the residue are summarised in Table 14.

[2] Although the report title uses the words "phenyl-^{14}C-" the ^{14}C label was in the chlorophenoxy ring.

Very little of the ^{14}C translocated to the tubers, but parent difenoconazole was identified, together with two primary metabolites, as a component of the residue at a low level (0.001 mg/kg). Parent difenoconazole was the major component of the foliage residue.

Table 14. Distribution of ^{14}C residue and metabolites in foliage and tubers of greenhouse potato plants sprayed with [^{14}C]chlorophenoxy labelled difenoconazole at the equivalent of 0.12 kg ai/ha per application

Residue component	Concentration, mg/kg, expressed as parent or %TRR					
	Foliage after spray 1	Foliage before spray 3	Foliage before spray 5	Foliage, final harvest	Tubers before spray 5	Tubers, final harvest
Total ^{14}C residue (TRR)	3.5 mg/kg	6.0 mg/kg	9.9 mg/kg	12 mg/kg	0.006 mg/kg	0.012 mg/kg
Extracted residue % TRR	96%	100%	91%	94%	51%	50%
Unextractable % TRR	2%	6%	6%	10%	58%	51%
Difenoconazole % TRR conc				76% 9.5 mg/kg		8.7% 0.0010 mg/kg
CGA 205374 %TRR conc				1.1% 0.14 mg/kg		3.1% 0.0004 mg/kg
CGA 205375 %TRR conc				2.2% 0.27 mg/kg		3.0% 0.0004 mg/kg
CGA 189138 %TRR conc				0.5% 0.07 mg/kg		

In a greenhouse potato metabolism study in USA, Velagaleti (1990, N-0964-0500) foliar sprayed potato plants (variety Red Pontiac) in pots of a sandy loam 6 times at 7 day intervals with [^{14}C]triazole labelled difenoconazole at the equivalent of 0.12 kg ai/ha per application. Samples of potato plants were taken at various stages of treatment and the mature crop was harvested 11 days after the final application. The distribution of the ^{14}C and the identification of components of the residue are summarised in Table 15.

A small part of the ^{14}C translocated to the tubers, and parent difenoconazole was identified, together with two primary metabolites, as a component of the residue at a low level (0.0016 mg/kg). Triazolylalanine was the major part (79%) of the residue in tubers (0.069 mg/kg). Parent difenoconazole was the major component of the foliage residue.

Table 15. Distribution of ^{14}C residue and metabolites in foliage and tubers of greenhouse potato plants sprayed with [^{14}C]triazole labelled difenoconazole at the equivalent of 0.12 kg ai/ha per application

Residue component	Concentration, mg/kg, expressed as parent or % TRR					
	Foliage after spray 1	Foliage before spray 3	Foliage before spray 5	Foliage, final harvest	Tubers before spray 5	Tubers, final harvest
Total ^{14}C residue (TRR)	2.2 mg/kg	3.1 mg/kg	5.5 mg/kg	9.1 mg/kg	0.052 mg/kg	0.087 mg/kg
Extracted residue % TRR	103%	100%	100%	102%	93%	92%
Unextractable %TRR	1.5%	4.0%	4.5%	4.6%	1.8%	1.9%
Difenoconazole % TRR conc				71% 6.7 mg/kg		1.8% 0.0016 mg/kg
CGA 205374 %TRR conc				0.78% 0.073 mg/kg		0.14% 0.0001 mg/kg
CGA 205375 %TRR conc				1.9% 0.17 mg/kg		
Triazolylalanine %TRR conc						79% 0.069 mg/kg

Grapes

In a field plot grape metabolism study in USA, (Capps, 1992, ABR-92003) foliar sprayed grape vines (variety Chenin Blanc) with [^{14}C]phenyl and [^{14}C]triazole labelled difenoconazole in an EC formulation. One vine (∇) was treated 5 times with 4.5-14.2 mg, total 45 mg [^{14}C]triazole labelled difenoconazole at 14 to 28 day intervals. A second vine (Φ1) was treated twice with 4.5 and 4.9 mg [^{14}C]phenyl labelled difenoconazole with a 14-day interval. A third vine (Φ2) was treated 3 times with 8.7 – 15.6 mg [^{14}C]phenyl labelled difenoconazole with 14 to 15 day intervals. Samples of grape foliage and grapes were taken at various stages. The distribution of the ^{14}C and the identification of components of the residue are summarised in Table 16.

Foliage and grapes were extracted with methanol+water (8+2) and the extracts were cleaned up, subjected to enzyme hydrolysis for release of conjugates, and the constituents identified and characterized by TLC.

Parent difenoconazole was the major component of the residue. None of the identified metabolites exceeded 10% of the TRR in grapes.

Table 16. Distribution of ^{14}C residue and metabolites in foliage and fruit of 3 field-plot grape vines (Φ1, Φ2 and ∇) sprayed with [^{14}C]phenyl and [^{14}C]triazole labelled difenoconazole

Residue component	Concentration, mg/kg, expressed as parent or% TRR					
[^{14}C-phenyl] label	Foliage Φ1, 35 days after spray 2	Foliage Φ1, mature, 77 days after spray 2	Grapes Φ1, 77 days after spray 2	Foliage Φ2, 7 days after spray 1	Foliage Φ2, mature, 20 days after spray 3	Grapes Φ2, 20 days after spray 3
Total ^{14}C residue (TRR)	6.6 mg/kg	1.6 mg/kg	0.047 mg/kg	8.6 mg/kg	9.2 mg/kg	0.13 mg/kg
Extracted residue %TRR	62%	67%	64%	88%	87%	81%
Unextractable % TRR	38%	33%	36%	12%	13%	19%
Difenoconazole %TRR	20%	16%	17%	36%	46%	51%
CGA 205374 %TRR	5.2%	3.7%	2.5%	4.2%	8.3%	4.1%
CGA 205375 %TRR	2.3%	7.8%	4.6%	2.6%	4.3%	6.6%
CGA 189138 %TRR	6.3%	2.0%	2.1%	3.8%	5.3%	4.0%
[^{14}C-triazole] label	Foliage ∇, 7 days after spray 3	Foliage ∇, mature, 20 days after spray 5	Grapes ∇, 20 days after spray 5			
Total ^{14}C residue (TRR)	8.7	5.8	0.12			
Extracted residue %TRR	77%	73%	83%			
Unextractable %TRR	24%	27%	17%			
Difenoconazole %TRR	31%	27%	45%			
CGA 205374 %TRR	11%	3.0%	1.7%			
CGA 205375 %TRR	5.7%	3.9%	3.5%			
1,2,4-triazole	2.8%	3.2%	7.3%			

Oilseed rape

In a field plot oilseed rape metabolism study in Switzerland, (Neumann, 1993, 11/93) foliar sprayed spring rape (variety Golda) twice, 14 day interval, with [^{14}C]chlorophenoxy labelled difenoconazole[3] in an EC formulation at the equivalent of 0.13 kg ai/ha. Plant samples were taken at various intervals through the treatment and stalks, pods and seeds were taken at mature harvest 39 days after the final application. The distribution of the ^{14}C and the identification of components of the residue are summarised in Table 17.

Foliage, stalks and pods were homogenized and extracted with methanol+water (8+2), then hot methanol and the extracts were cleaned up, subjected to enzyme hydrolysis for release of conjugates, and the constituents identified and characterized by TLC. Homogenized seeds were extracted with hexane to provide the oil and then the meal was extracted with methanol+water (8+2) and then hot methanol. A ^{14}C concentration of 0.15 mg/kg in the seeds produced 0.10 mg/kg in the oil and 0.17 mg/kg in the meal.

Parent difenoconazole was the major identified component of the residue. Metabolite CGA 205375 exceeded 10% of TRR in the stalks (14%) and pods (11%).

[3] Although the report title uses the words "phenyl-^{14}C-" the ^{14}C label was in the chlorophenoxy ring.

Table 17. Distribution of ^{14}C residue and metabolites in foliage, pods, seeds and oil of field-plot grown spring rape sprayed twice with [^{14}C]chlorophenoxy labelled difenoconazole at the equivalent of 0.13 kg ai/ha

OILSEED RAPE METABOLISM Residue component	Concentration, mg/kg, expressed as parent or % TRR						
	Foliage 0 days after spray 1	Foliage 15 days after spray 1	Foliage 0 days after spray 2	Stalks 39 days after spray 2	Seeds 39 days after spray 2	Pods 39 days after spray 2	Oil 39 days after spray 2
Total ^{14}C residue (TRR)	7.0 mg/kg	1.5 mg/kg	5.0 mg/kg	4.3 mg/kg	0.15 mg/kg	3.1 mg/kg	0.10 mg/kg
Difenoconazole % TRR conc	93% 6.5 mg/kg	31% 0.46 mg/kg	78% 3.9 mg/kg	17% 0.75 mg/kg	15% 0.022 mg/kg	17% 0.52 mg/kg	26% 0.026 mg/kg
CGA 205375 %TRR				14%	7.9%	11%	
CGA 205374 %TRR						0.3%	
CGA 189138 %TRR				1.6%	0.3%	1.7%	

In a second metabolism study on oilseed rape in Switzerland, (Neumann, 1993, 12/93) foliar sprayed spring rape (variety Golda) in field plots twice, 14 day interval, with [^{14}C]triazole labelled difenoconazole in an EC formulation at the equivalent of 0.13 kg ai/ha. Plant samples were taken at various intervals through the treatment and stalks, pods and seeds were taken at mature harvest 39 days after the final application. The distribution of the ^{14}C and the identification of components of the residue are summarised in Table 18.

Samples of foliage, stalks, pods and seeds were extracted and cleaned up by the same processes as previously described (Neumann, 1993, 11/93). A ^{14}C concentration of 2.3 mg/kg in the seeds produced 0.17 mg/kg in the oil and 2.5 mg/kg in the meal.

Parent difenoconazole was a major identified component of the residue except in the seed. Metabolite CGA 205375 exceeded 10% of TRR in the stalks (17%) and pods (13%). Triazolylalanine the major residue component in the seed (56% TRR) also exceeded 10% in pods (12%). Triazolylalanine was also the major residue component in the meal (56% TRR). Other identified components of the residue in the meal were triazolylacetic acid (2.8% TRR), CGA 205375 (0.6% TRR) and difenoconazole (2.3% TRR).

Table 18. Distribution of ^{14}C residue and metabolites in foliage, pods, seeds and oil of field-plot grown spring rape sprayed twice with [^{14}C]triazole labelled difenoconazole at the equivalent of 0.13 kg ai/ha

OILSEED RAPE METABOLISM Residue component	Concentration, mg/kg, expressed as parent or % TRR						
	Foliage 0 days after spray 1	Foliage 15 days after spray 1	Foliage 0 days after spray 2	Stalks 39 days after spray 2	Seeds 39 days after spray 2	Pods 39 days after spray 2	Oil 39 days after spray 2
Total ^{14}C residue (TRR)	5.2 mg/kg	1.2 mg/kg	4.8 mg/kg	4.8 mg/kg	2.3 mg/kg	4.7 mg/kg	0.17 mg/kg
Difenoconazole% TRR conc	90% 4.7 mg/kg	36% 0.43 mg/kg	82% 3.9 mg/kg	17% 0.83 mg/kg	4.1% 0.093 mg/kg	14% 0.65 mg/kg	84% 0.14 mg/kg
CGA 205375 %TRR				17%	0.6%	13%	
CGA 205374 %TRR				1.3%		0.8%	
Triazolylalanine %TRR				4.1%	56%	12%	
Triazolylacetic acid %TRR				3.3%	2.8%	6.7%	
1,2,4-triazole				1.6%			

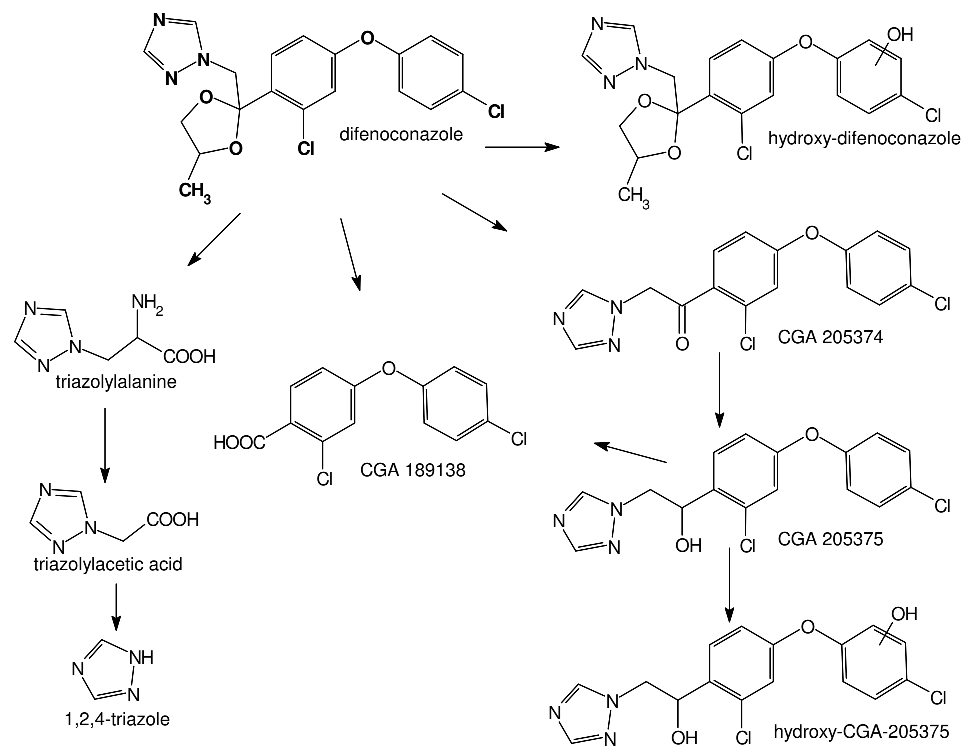


Figure 5. Proposed pathway for the metabolism of difenoconazole in plants

Environmental fate in soil

The 2003 JMPR (JMPR, 2003) explained the data requirements for studies of environmental fate. The focus should be on those aspects that are most relevant to MRL setting. For difenoconazole, supervised residue trials data are available for root and tuber vegetables, which means that aerobic degradation in soil is relevant, as well as the normal requirements for hydrolysis and rotational crop studies. The 2003 report does not mention soil photolysis studies; however, such studies should be relevant for the same reasons as for aerobic soil degradation – nature and magnitude of residues in soil.

The Meeting received information on soil aerobic metabolism and soil photolysis properties of difenoconazole as well as studies on the behaviour of difenoconazole residues in crop rotations.

Difenoconazole residues are reasonably persistent in soils and are expected to be present in the soil at harvest time for treated root and tuber crops. Difenoconazole residues are also expected to persist in the soil until the sowing of rotational crops. The confined rotational crops studies demonstrate that difenoconazole itself does not appear as a residue in the rotational crop. The water-soluble and mobile metabolites triazolylalanine, triazolylacetic acid and triazolyl-lactic acid have been identified in the rotational crops.

Soil metabolism

Aerobic soil metabolism studies are summarized below, showing the test conditions, the nature of the soils, estimated half-lives and the nature of identified soil metabolites. These are laboratory soil incubation studies with ^{14}C labelled compounds. Metabolism or degradation rates are influenced by the nature of the soil, temperature, moisture status of the soil and dose. Estimated aerobic soil metabolism half-lives for difenoconazole at 20 °C ranged from 63 to 700 days (n=12) with a median of 181 days. When difenoconazole is used on root and tuber crops its residues in soil are likely to persist until harvest.

After 220 – 300 days, mineralization and unextractable residues (20 – 54% of dose) were major sinks for the ^{14}C label. The degree of mineralization was different for the phenyl and triazole label positions, 0.8 – 4.6% of the dose for the triazole label and 3.4 – 33% for the phenyl label.

CGA 250375 and 1,2,4-triazole were identified as soil metabolites. Metabolite CGA 205375 consistently reached a maximum (as parent) of 5 – 10% of the dose and had begun to decline by the end of the observation period. Metabolite 1,2,4-triazole typically reached a maximum (expressed as parent) around 20% of the dose during the observation period. Further information was provided on the persistence of these two metabolites in soil.

Aerobic soil metabolism — Ref: Mamouni, 2000, 738606

Test material: [^{14}C-triazole]difenoconazole
Dose rate: 0.19 mg ai/kg
Duration: 293 days
Temp: 20 °C
Moisture: 40% max water-holding capacity
Soil: loam
pH: 7.2
Organic carbon: 2.1 %
Half-life (parent): 105 days
^{14}C accountability 88-101 %
% difenoconazole remaining, day 293 = 14% of dose
% mineralization, day 293 = 4.5% of dose
% unextractable, day 293 = 49% of dose

Metabolites	Max (% of dose)	Day
CGA 205375	4.6 %	56
1,2,4-triazole	21 %	190

Aerobic soil metabolism — Ref: Mamouni, 2000, 738617

Test material: [^{14}C-phenyl]difenoconazole
Dose rate: 0.19 mg ai/kg
Duration: 293 days
Temp: 20 °C
Moisture: 40% max water-holding capacity
Soil: loam
pH: 7.2
Organic carbon: 2.1 %
Half-life (parent): 120 days
^{14}C accountability 88-104 %
% difenoconazole remaining, day 293 = 18% of dose
% mineralization, day 293 = 23% of dose
% unextractable, day 293 = 38% of dose

Metabolites	Max (% of dose)	Day
CGA 205375	5.1 %	84

Aerobic soil metabolism — Ref: Gonzalez-Valero, 1992, 91GJ05

Test material: [^{14}C-phenyl]difenoconazole
Dose rate: 0.0.097 mg ai/kg
Duration: 106 days
Temp: 20 °C
Moisture: 40% max water-holding capacity
Soil: sand
pH: 5.0
Organic carbon: 1.8 %
Half-life (parent): approx 140 days
^{14}C accountability 101-112 %
% difenoconazole remaining, day 106 = 63% of dose
% mineralization, day 106 = 3.7% of dose
% unextractable, day 106 = 21% of dose

Metabolites not identifiable

Aerobic soil metabolism — Ref: Gonzalez-Valero, 1992, 91GJ01

Test material: [^{14}C-triazole]difenoconazole
Dose rate: 1 mg ai/kg
Duration: 273 days
Temp: 20 °C
Moisture: 60% field capacity
Soil: silt loam
pH: 7.2
Organic carbon: 2.0 %
Half-life (parent): approx 500 days
^{14}C accountability 96-106 %
% difenoconazole remaining, day 273 = 61% of dose
% mineralization, day 273 = 0.8% of dose
% unextractable, day 273 = 34% of dose

Metabolites not identifiable

Aerobic soil metabolism — Ref: Gonzalez-Valero, 1992, 91GJ01

Test material: [^{14}C-triazole]difenoconazole
Dose rate: 1 mg ai/kg
Duration: 178 days
Temp: 30 °C
Moisture: 60% field capacity
Soil: silt loam
pH: 7.2
Organic carbon: 2.0 %
Half-life (parent): 180 days
^{14}C accountability 88-100 %
% difenoconazole remaining, day 178 = 44% of dose
% mineralization, day 178 = 1.2% of dose
% unextractable, day 178 = 36% of dose

Metabolites not identifiable

Aerobic soil metabolism
Test material: [^{14}C-phenyl]difenoconazole
Duration: 281 days
Temp: 20 °C
Soil: silt loam
pH: 7.2
Half-life (parent): approx 700 days
% difenoconazole remaining, day 281 = 70% of dose

Metabolites not identifiable

Ref: Gonzalez-Valero, 1992, 91GJ01
Dose rate: 1 mg ai/kg
Moisture: 30% field capacity
Organic carbon: 2.0 %
^{14}C accountability 97-101 %
% mineralization, day 281 = 3.4% of dose
% unextractable, day 281 = 21% of dose

Aerobic soil metabolism
Test material: [^{14}C-phenyl]difenoconazole
Duration: 281 days
Temp: 10 °C
Soil: silt loam
pH: 7.2
Half-life (parent): approx 600 days
% difenoconazole remaining, day 281 = 71% of dose

Metabolites not identifiable

Ref: Gonzalez-Valero, 1992, 91GJ01
Dose rate: 1 mg ai/kg
Moisture: 60% field capacity
Organic carbon: 2.0 %
^{14}C accountability 98-105 %
% mineralization, day 281 = 5.8% of dose
% unextractable, day 281 = 15% of dose

Aerobic soil metabolism
Test material: [^{14}C-phenyl]difenoconazole
Duration: 281 days
Temp: 20 °C
Soil: silt loam
pH: 7.2
Half-life (parent): approx 350 days
% difenoconazole remaining, day 281 = 51% of dose

Metabolites not identifiable

Ref: Gonzalez-Valero, 1992, 91GJ01
Dose rate: 1 mg ai/kg
Moisture: 60% field capacity
Organic carbon: 2.0 %
^{14}C accountability 98-104 %
% mineralization, day 281 = 17% of dose
% unextractable, day 281 = 28% of dose

Aerobic soil metabolism
Test material: [^{14}C-phenyl]difenoconazole
Duration: 281 days
Temp: 20 °C
Soil: silt loam
pH: 7.2
Half-life (parent): 114 days
% difenoconazole remaining, day 281 = 17% of dose

Metabolites not identifiable

Ref: Gonzalez-Valero, 1992, 91GJ01
Dose rate: 0.1 mg ai/kg
Moisture: 60% field capacity
Organic carbon: 2.0 %
^{14}C accountability 95-126 %
% mineralization, day 281 = 33% of dose
% unextractable, day 281 = 35% of dose

Aerobic soil metabolism
Test material: [^{14}C-chlorophenoxy]difenoconazole [4]
Duration: 228 days
Temp: 20 °C
Soil 1: sandy loam
pH: 7.4
Half-life (parent): 169 days
% difenoconazole remaining, day 228 = 39 % of dose

Ref: Mamouni, 2002, 775438
Dose rate: 0.26 mg ai/kg dw
Moisture: 40% max water-holding capacity
Organic carbon: 1.2 %
^{14}C accountability 96-101 %
% mineralization, day 228 = 19% of dose
% unextractable, day 228 = 23% of dose

Metabolites	Max (% of dose)	Day
CGA 205375	9.7 %	84

Aerobic soil metabolism
Test material: [^{14}C-chlorophenoxy]difenoconazole
Duration: 228 days
Temp: 20 °C
Soil 2: sandy loam/loamy sand
pH: 7.5
Half-life (parent): approx 200 days
% difenoconazole remaining, day 228 = 51% of dose

Ref: Mamouni, 2002, 775438
Dose rate: 0.26 mg ai/kg dw
Moisture: 40% max water-holding capacity
Organic carbon: 1.2 %
^{14}C accountability 96-100 %
% mineralization, day 228 = 19% of dose
% unextractable, day 228 = 20% of dose

Metabolites	Max (% of dose)	Day
CGA 205375	8.5 %	120

[4] Although the report title uses the words " ^{14}C-chlorophenyl" the ^{14}C label was in the chlorophenoxy ring.

Aerobic soil metabolism Ref: Mamouni, 2002, 775438

Test material: [^{14}C-chlorophenoxy]difenoconazole
Dose rate: 0.26 mg ai/kg dw
Duration: 228 days
Temp: 20 °C
Moisture: 40% max water-holding capacity
Soil 3: silty clay loam
pH: 6.7
Organic carbon: < 0.3 %
Half-life (parent): 209 days
^{14}C accountability 95-102 %
% difenoconazole remaining, day 228 = 45% of dose
% mineralization, day 228 = 20% of dose
% unextractable, day 228 = 23% of dose

Metabolites	Max (% of dose)	Day
CGA 205375	4.4 %	120

Aerobic soil metabolism Ref: Völkel, 2000, 738628

Test material: [^{14}C-triazole]difenoconazole
Dose rate: 0.17 mg ai/kg dw
Duration: 271 days
Temp: 20 °C
Moisture: 30% field capacity
Soil: loam
pH: 7.2
Organic carbon: 2.2 %
Half-life (parent): 110 days
^{14}C accountability 86-102 %
% difenoconazole remaining, day 271 = 16% of dose
% mineralization, day 271 = 2.2% of dose
% unextractable, day 271 = 43% of dose

Metabolites	Max (% of dose)	Day
CGA 205375	6.2 %	125
1,2,4-triazole	18%	177

Aerobic soil metabolism Ref: Völkel, 2000, 738628

Test material: [^{14}C-triazole]difenoconazole
Dose rate: 0.17 mg ai/kg dw
Duration: 271 days
Temp: 10 °C
Moisture: 60% field capacity
Soil: loam
pH: 7.2
Organic carbon: 2.2 %
Half-life (parent): approx 340 days
^{14}C accountability 98-101 %
% difenoconazole remaining, day 271 = 56% of dose
% mineralization, day 271 = 3.2% of dose
% unextractable, day 271 = 21% of dose

Metabolites	Max (% of dose)	Day
CGA 205375	8.9 %	271
1,2,4-triazole	8.6 %	271

Aerobic soil metabolism Ref: Völkel, 2000, 738628

Test material: [^{14}C-triazole]difenoconazole
Dose rate: 0.017 mg ai/kg dw
Duration: 271 days
Temp: 20 °C
Moisture: 60% field capacity
Soil: loam
pH: 7.2
Organic carbon: 2.2 %
Half-life (parent): 63 days
^{14}C accountability 98-102 %
% difenoconazole remaining, day 271 = 4.6% of dose
% mineralization, day 271 = 4.6% of dose
% unextractable, day 271 = 54% of dose

Metabolites	Max (% of dose)	Day
CGA 205375	7.4 %	56
1,2,4-triazole	23 %	271

Aerobic soil degradation (sterile system) Ref: Völkel, 2000, 738628

Test material: [^{14}C-triazole]difenoconazole
Dose rate: 0.17 mg ai/kg dw
Duration: 271 days
Temp: 20 °C
Moisture: 60% field capacity
Soil: loam
pH: 7.2
Organic carbon: 2.2 %
Half-life (parent): approx 1000-1500 days
^{14}C accountability 98-102 %
% difenoconazole remaining, day 271 = 85% of dose
% mineralization, day 271 = 0.2% of dose
% unextractable, day 271 = 6.5% of dose

Degradation products	Max (% of dose)	Day
CGA 205375	3.4 %	271
1,2,4-triazole	1.5 %	271

Aerobic soil metabolism Ref: Völkel, 2002, 775451

Test material: [^{14}C-triazole]CGA 205375
Dose rate: 0.11 mg ai/kg dw
Duration: 228 days
Temp: 20 °C
Moisture: 40% max water-holding capacity
Soil: sandy loam
pH: 7.4
Organic carbon: 0.95 %
Half-life (parent): 129 days
^{14}C accountability 93-101 %
% CGA 205375 remaining, day 228 = 28% of dose
% mineralization, day 228 = 32% of dose
% unextractable, day 228 = 26% of dose

Metabolites	Max (% of dose)	Day
CGA 205374	1.2 %	30
1,2,4-triazole	16 %	30

Aerobic soil metabolism — Ref: Völkel, 2002, 775451

Test material: [^{14}C-triazole]CGA 205375 — Dose rate: 0.11 mg ai/kg dw

Duration: 210 days — Temp: 20 °C — Moisture: 40% max water-holding capacity

Soil: sandy loam /loamy sand — pH: 7.5 — Organic carbon: 1.0 %

Half-life (parent): 104 days — ^{14}C accountability 94-102 %

% CGA 205375 remaining, day 210 = 22% of dose — % mineralization, day 210 = 14% of dose

% unextractable, day 210 = 33% of dose

Metabolites	Max (% of dose)	Day
CGA 205374	2.7 %	14
1,2,4-triazole	32 %	150

Aerobic soil metabolism — Ref: Völkel, 2002, 775451

Test material: [^{14}C-triazole]CGA 205375 — Dose rate: 0.11 mg ai/kg dw

Duration: 228 days — Temp: 20 °C — Moisture: 40% max water-holding capacity

Soil: silt loam — pH: 5.8 — Organic carbon: 0.99 %

Half-life (parent): approx 190 days — ^{14}C accountability 94-100 %

% CGA 205375 remaining, day 228 = 43% of dose — % mineralization, day 228 = 0.8% of dose

% unextractable, day 228 = 34% of dose

Metabolites	Max (% of dose)	Day
CGA 205374	0.7 %	30
1,2,4-triazole	24 %	150

Slangen (2000, 278336) incubated [^{14}C]1,2,4-triazole dosed at 0.06 mg ai/kg dw in three soils (sandy loam, loamy sand and silt loam) under aerobic conditions for 120 days and measured evolved CO_2 and residue levels at 9 sampling times. By day 120, mineralization to CO_2 had occurred for 11%, 1.6% and 32% of the dose for the three soils respectively and the unextractable residue formed 66%, 65% and 42% of the applied dose. The initial disappearance half-life for 1,2,4-triazole was only a few days, but the rate declined substantially as the residue aged. By day 120, remaining 1,2,4-triazole constituted 12%, 30% and 2.4% of the dose for the three soils respectively. Triazolylacetic acid was detected as a minor metabolite.

In a soil surface photolysis study, Atkins (1994, 791) applied [^{14}Cphenyl]difenoconazole to a film of moist sandy loam in petri dishes for a concentration of 10 mg/kg. Irradiated samples were maintained at 25 ˚C during a light-dark cycle of 12 hours each per day. Irradiation was provided by two xenon arc lamps, each of 1500 W, filtered through borosilicate glass to cut off wavelengths shorter than 290 nm. The light intensity was measured as 4022-4023 µW/cm^2. Sufficient petri dishes were prepared to allow duplicate sampling at days 0, 1, 3, 5, 7, 11, 21 and 30 days. Extracts of the soil were examined by TLC and HPLC.

After 30 days, parent difenoconazole accounted for 91.4% of the dose (dark controls, 92.5% of dose), which demonstrated that difenoconazole is stable to photolysis on the soil surface. A degradation product, CGA 205374, was detected at 0.2% of the dose on day 30.

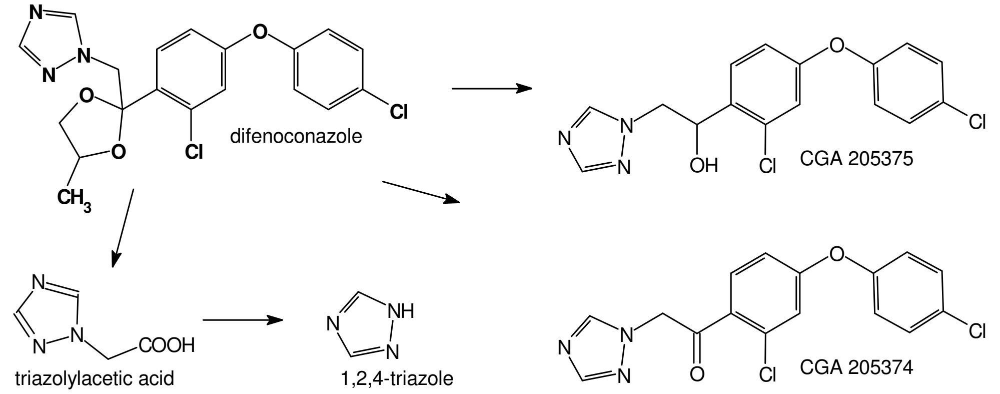


Figure 6. Proposed pathway for the aerobic metabolism of difenoconazole in soil

Crop rotation studies

Information on the fate of radiolabelled difenoconazole in confined and non-confined crop rotational studies were made available to the meeting.

With the ^{14}C label in the phenyl moiety, the level of carry-over residues in rotational crops was too low for characterization or identification. With the ^{14}C label in the triazole moiety, metabolites triazolylalanine, triazolylacetic acid and triazolyl-lactic acid were identified in rotational crops.

In an outdoor confined rotational crop study in Switzerland (Walser, 1994, 8/94) bare ground (sandy loam, 2.3% organic carbon, 25% sand, 47% silt, 27% clay, pH 7.3) was treated directly with [^{14}C-phenyl]difenoconazole, formulated as an EC, at a rate equivalent to 0.13 kg ai/ha. The bare ground plot of 2 m × 2 m was divided into 4 sections for the 4 rotational crops, which were sown at intervals after treatment: lettuce 98 days, winter wheat 126 days, maize 342 days and sugar beet 369 days.

Samples were taken at immature and mature stages for analysis. Data are summarised in Table 19. Levels of TRR expressed as parent difenoconazole in the plant material were too low, mostly in the range of 0.001 to 0.009 mg/kg, for further characterization or identification.

In an outdoor confined rotational crop study in Switzerland (Walser, 1994, 4/94) bare ground (sandy loam, 2.3% organic carbon, 25% sand, 47% silt, 27% clay, pH 7.3) was treated directly with [^{14}C-triazole]difenoconazole, formulated as an EC, at a rate equivalent to 0.13 kg ai/ha. The bare ground plot of 2 m × 2 m was divided into 4 sections for the 4 rotational crops, which were sown at intervals after treatment: lettuce 98 days, winter wheat 126 days, maize 342 days and sugar beet 369 days.

Three metabolites were identified in tissues from rotational crops grown on bare ground treated with [^{14}C-triazole]difenoconazole. The metabolites were triazolylalanine, triazolylacetic acid and triazolyl-lactic acid (Table20).

In an outdoor confined rotational crop study in USA (Close, 1995, ABR-95057) bare ground (sandy loam, 0.5% organic matter, 62% sand, 30% silt, 8% clay, pH 5.5 – 7.2) was treated directly with [^{14}C-chlorophenoxy]difenoconazole, at a rate equivalent to 0.031 kg ai/ha. This application rate simulates the rate resulting from a seed treatment use. The bare ground plot was approximately 2.4 m^2. Spring wheat, mustard and turnips were chosen as representative rotational crops. Residues arising from a 33 day TSI (treatment to sowing interval) were very low (Table 19), so samples from a 90 days TSI were not analysed.

Seed treatment uses of difenoconazole at 0.031 kg ai/ha are unlikely to produce residues in rotational crops sown 33 days after the sowing of difenoconazole-treated seed.

In an outdoor non-confined rotational crop study in Germany (Heyer, 1995, 488002), bare ground (loam, pH 6.9) was treated directly with difenoconazole formulated as an EC, at a rate equivalent to 0.75 kg ai/ha and the upper 10 cm soil layer was turned over to mix in the applied material. Carrots (variety Rote Riesen) were sown 30 days after the difenoconazole application and harvested for analysis 97, 114 and 136 days after the application. Residues of difenoconazole (LOQ 0.02 mg/kg) and triazolylalanine (LOQ 0.05 mg/kg) in the carrots did not exceed the LOQs. Difenoconazole residue levels in the soil were 0.19, 0.20 and 0.18 mg/kg for samples taken on days 97, 114 and 136 after treatment respectively.

In a parallel study to the one above on carrots, Heyer (1995, 488001) used spinach (variety Adele) as the rotational crop. Bare ground was treated directly with difenoconazole formulated as an EC, at a rate equivalent to 0.75 kg ai/ha and the upper 10 cm soil layer was turned over to mix in the applied material. Spinach was sown 31 days after the difenoconazole application and harvested for analysis 62, 70 and 77 days after the application. Residues of difenoconazole (LOQ 0.02 mg/kg) and triazolylalanine (LOQ 0.05 mg/kg) in the spinach did not exceed the LOQs. Difenoconazole residue levels in the soil were 0.16, 0.23 and 0.15 mg/kg for samples taken on days 62, 70 and 77 after treatment respectively.

Table 19. Confined rotational crop studies with [^{14}C]difenoconazole

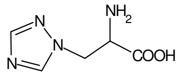
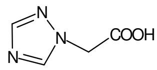
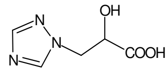
Application country, year, ref.	Rotational crop (variety)	TSI a/ days	THI b/ days	Sample	TRR as difenoconazole mg/kg	Extractable, residues % c/	Difenoconazole mg/kg
Bare soil, Switzerland, 1992-93, (Walser, 1994, 8/94). [^{14}C-phenyl]difenoconazole							
	lettuce (Soraya)	98	126	heads	0.003	na	na
			151	heads	0.002	na	na
	winter wheat (Sardona)	126	167	whole tops	0.003	na	na
			342	whole tops	0.002	na	na
			369	whole tops	0.002	na	na
			418	stalks	0.009	na	na
			418	husks	0.002	na	na
			418	grains	0.003	na	na
	maize (DK 250)	342	398	whole tops	0.001	na	na
			427	whole tops	0.001	na	na
			488	stalks	0.001	na	na
			488	cobs	0.001	na	na
			488	grains	0.001	na	na
	sugar beet (Regina)	369	427	tops	< 0.001	na	na
			427	roots	0.001	na	na
			473	tops	< 0.001	na	na
			473	roots	0.001	na	na
			488	tops	0.001	na	na
			488	roots	0.001	na	na
Bare soil, Switzerland, 1992-93, (Walser, 1994, 4/94). [^{14}C-triazole]difenoconazole							
	lettuce (Soraya)	98	126	heads	0.021	96 %	< 0.006
			151	heads	0.017	94 %	< 0.006
	winter wheat (Sardona)	126	167	whole tops	0.028	na	< 0.006
			342	whole tops	0.045	89 %	< 0.006
			369	whole tops	0.072	101 %	< 0.006
			418	stalks	0.11	82 %	< 0.006
			418	husks	0.15	88 %	< 0.006
			418	grains	0.34	88 %	< 0.006
	maize (DK 250)	342	398	whole tops	0.071	100 %	< 0.006
			427	whole tops	0.057	89 %	< 0.006
			488	stalks	0.027	77 %	< 0.006
			488	cobs	0.040	66 %	< 0.006
			488	grains	0.21	86 %	< 0.006
	sugar beet (Regina)	369	427	tops	0.019	90 %	< 0.006
			427	roots	0.011	83 %	< 0.006
			473	tops	0.034	95 %	< 0.006
			473	roots	0.007	na	< 0.006
			488	tops	0.029	96%	< 0.006
			488	roots	0.005	na	< 0.006
Bare soil, USA (CA), 1994-95, (Close, 1995, ABR-95057). [^{14}C-chlorophenoxy]difenoconazole							
	mustard (Florida Broadleaf)	33	129	mature plant	< 0.0001		
	spring wheat (Aldura)	33	109	forage	0.002		
			175	straw	0.004		
			175	grain	0.001		
	turnips (Purple Top White Globe)	33	129	tops	< 0.0001		
				roots	0.0001		

a - TSI: interval between treatment on soil and sowing of rotation crop, days.

b - THI: interval between treatment on soil and harvest of rotation crop (or sampling of soil), days.

c - na: not analysed.

Table 20. Identity of residue in plant tissues from confined rotational crop study

Plant tissue	TRR, mg/kg as difenoconazole	% of TRR triazolylalanine 	triazolylacetic acid 	triazolyl-lactic acid 
Maize grain	0.21	66%	0%	9.7%
Wheat grain	0.34	44%	26%	0%
Wheat stalks	0.11	10%	36%	21%
Wheat husks	0.15	19%	39%	12%
Lettuce heads	0.017	31%	3.3%	43%
Sugar beet tops	0.029	25%	2.7%	54%

METHODS OF RESIDUE ANALYSIS

Analytical methods

The Meeting received descriptions and validation data for analytical methods for residues of difenoconazole in raw agricultural commodities, processed commodities, feed commodities, animal tissues, milk and eggs. Methods were provided also for metabolite CGA 205375 in animal tissues, milk and eggs. Recovery data are summarised in Table 22.

Method AG-575A was tested for selectivity in the analysis of difenoconazole residues in cereal grains in the presence of other pesticides that may be present (Yokley, 1993, ABR-92084). The pesticides tested were those with US tolerances at the time in wheat, barley and rye and the fortification levels were equivalent to the tolerances. None of the compounds interfered with the analysis of difenoconazole at 0.01 mg/kg.

Method AG-575 was tested by an independent laboratory unfamiliar with the analysis (Yarko, 1990, 900201). No background interferences were observed and recoveries between 70 and 120% were achieved for wheat grain and straw at spiking levels of 0.01 – 0.25 mg/kg.

Method AG-544 for difenoconazole residues in animal commodities was tested by an independent laboratory unfamiliar with the analysis (Wurz, 1993, ABR-93022). No background interferences were observed and recoveries between 52 and 124% were achieved for beef liver, eggs and milk at spiking levels of 0.01 – 0.25 mg/kg.

Detector linearity, storage stability of sample extracts, matrix effects on detector sensitivity and interferences from control samples were investigated in the validation of method REM 147.07 (Ryan, 2004, RJ3478B). The detector was linear through zero over the dynamic range tested (×80). Residues were stable in liver and milk extracts held at < 7 °C for 7 – 8 days. Suppression or enhancement of response by sample matrix was not significant. Interferences from control samples were below 30% of the LOQ. The LOQs for difenoconazole and CGA 205375 were 0.01 mg/kg in liver, kidney, muscle, fat and eggs and 0.005 mg/kg for milk.

Detector linearity, storage stability of sample extracts, matrix effects on detector sensitivity and interferences from control samples were investigated in the validation of method REM 147.08 (Ely and Ryan, 2004, RJ3560B). The detector was linear through zero over the range tested, from below the LOQ (0.01 mg/kg) to above the highest validated recovery level (1 mg/kg). Residues were stable in sample extracts held at < 7 °C for 7 – 8 days. Suppression or enhancement of response by crop matrix was less than 10%. Interferences from control samples were below 30% of the LOQ.

Steinhauer (2002, SYN-0211V Az.G02-0092) showed that DFG Method S19, with a number of modifications, could be used for the satisfactory analysis of difenoconazole residues in asparagus with an LOQ of 0.02 mg/kg. Steinhauer (2004, SYN-0301V Az. G03-0012) showed that DFG Method S19 is also suitable for residues of difenoconazole in apple, lettuce, wheat grain and oilseed rape with LOQs of 0.02 mg/kg in apples and lettuce and 0.05 mg/kg for wheat grain and oilseed rape.

DFG Method S19 (revision) was subject to an independent laboratory validation for the analysis of difenoconazole residues in apples, oilseed rape, wheat grain and lettuce (Schulz, 2004, IF-04/00160619). No significant background interferences were observed and recoveries between 70 and 120% were generally achieved.

Method REM 147.07 was subject to an independent laboratory validation for the analysis of difenoconazole and CGA 205375 residues in eggs, milk and bovine muscle (Benazeraf, 2004, SYN/DIF/04031). Interferences from control samples were below 30% of the LOQ and recoveries between 70 and 110% were generally achieved.

Brown (2005, T008949-04) extracted poultry tissues and egg yolk from a [^{14}C-triazole]-difenoconazole labelled metabolism study (Ray, 2004, 786-02) with acetonitrile-water as described in Method REM 147.07. The extracted difenoconazole, CGA 205375 and 1,2,4-triazole concentrations were compared with the levels found by exhaustive extraction (Table 21). The acetonitrile-water procedure extracted a high percentage of each of the residues from the tissues and egg yolk.

Table 21. Extractability of residues by acetonitrile-water from poultry tissues and eggs, where residues were ^{14}C labelled from a metabolism study

Substrate	1,2,4-triazole conc, mg/kg			CGA 205375 conc, mg/kg			difenoconazole conc, mg/kg		
	exhaustive [a]	single ext [b]	% extracted	exhaustive	single ext	% extracted	exhaustive	single ext	% extracted
Liver	2.9	2.4	84.4%	8.0	8.0	100%	0.30	0.23	76%
Fat	0.086	0.094	109%	7.7	7.6	99%	2.3	2.0	88%
Egg yolk	1.5	1.5	102%	2.5	2.3	94%	0.20	0.18	88%
Muscle	2.1	2.1	99%	1.5	1.5	103%	0.097	0.11	111%

a - Exhaustive extraction: samples were extracted 3 times for approximately 10 minutes with acetonitrile:water (8:2) as in the metabolism study.

b - Single extraction: samples were extracted once for approximately 5 minutes with acetonitrile:water (8:2) adjusted for water content of sample.

Plant material (Kühne H, 1986, REM 7/86)

Analyte:	difenoconazole	GC-ECD	Method REM 7/86
LOQ:	0.04 mg/kg.		
Description	Homogenized sample is extracted with acetonitrile. An aliquot of the filtrate is evaporated and the residue is taken up in acetonitrile and washed with hexane. The acetonitrile solution is evaporated and the residue is dissolved in hexane for cleanup with a phenyl-solid phase extraction tube using hexane-ether and then methanol to recover the difenoconazole residue. The residue was partitioned into hexane-ether after the addition of water and saturated sodium chloride. After evaporation, the residue was dissolved in hexane-ethanol for GLC analysis.		

Potato and tomato (Williams and Shoffner, 1987, AG-514)

Analyte:	difenoconazole	GLC-NPD	Method AG-514
LOQ:	0.05 mg/kg.		
Description	Residues are extracted by refluxing the sample with methanol-ammonia for 2 hours and then filtering. An aliquot of the filtrate is diluted with water and saturated salt and extracted with hexane, which is then extracted with acetonitrile. The acetonitrile is evaporated and the residue is taken up in toluene for solid phase column cleanup. The eluate is evaporated and the residue dissolved in toluene for GLC analysis.		
Extractability	Extractability of total ^{14}C from tomatoes and potatoes from plants treated with [^{14}C-phenoxyphenyl]difenoconazole and grown in a greenhouse ranged from 86% to 113%. Concentrations were too low for analysis of difenoconazole.		

Wheat commodities (Williams, 1988, AG-537)

Analyte:	difenoconazole	GLC-NPD	Method AG-537
LOQ:	0.05 mg/kg.		
Description	See method AG-514. A cleanup step with charcoal, magnesium oxide and Celite is added. The method is suitable for wheat forage, hay, straw and grain.		
Extractability	Extractability of total ^{14}C from wheat treated with [^{14}C-phenoxyphenyl]difenoconazole and grown in a test plot was measured for forage (83 %, 105% and 86), stalks (80 %, 80% and 81 %) and grain (67 %, 74% and 74 %).		

*Dairy and poultry tissues, eggs and milk (*Ward, 1988, AG-544)

Analyte: difenoconazole GLC-NPD Method AG-544

LOQ: 0.05 mg/kg. Milk 0.01 mg/kg.

Description Animal tissues are chopped and mixed thoroughly before sampling. Eggs and milk are homogenized for a few seconds before the analytical sample is taken. Sample is homogenized with acetonitrile + concentrated ammonium hydroxide for 1 minute. An aliquot of the filtered extract is diluted with water and saturated sodium chloride and subjected to a partition cleanup with hexane and acetonitrile followed by a silica solid phase cleanup. The resulting toluene-acetone solution is evaporated and the residues dissolved in toluene for GLC analysis.

Version Method AG-544A is a slightly modified version of AG-544 (Wurz, 1994, AG-544A).

Wheat commodities (Darnow and Sayers, 1990, AG-575)

Analyte: difenoconazole GLC-NPD Method AG-575

LOQ: 0.05 mg/kg, wheat grain. 0.05 mg/kg, wheat forage.

Description See method AG-537. A larger aliquot is taken and a phenyl Bond-elut step is added to achieve a lower LOQ for wheat grain.

Version Methods AG-575A and AG-575B are later versions of Method AG-575 with amendments. Note that instead of GLC-NPD, sometimes GLC-ECD (Ryan, 2005, TMJ5014B) or GLC-MSD (Ryan, 2005, TMJ5031B) may be used.

Brassica vegetables (Brown, 1992, CGA 03291)

Analyte: difenoconazole GLC-ECD Method CGA 169374 - Brassicas/EK/91/2

LOQ: 0.05 mg/kg

Description Residues are extracted by macerating sample with methanol. An aliquot of centrifuged and filtered extract is diluted with water and saturated sodium chloride and extracted with dichloromethane. The dichloromethane extract is evaporated and the residue is taken up in hexane for a phenyl solid-phase cartridge cleanup. The residues are eluted with a methanol-water mixture which, after dilution with water and saturated sodium chloride is extracted with hexane + diethyl ether. The solvent is evaporated and the residue is dissolved in hexane-ethanol for GLC analysis.

Vegetable matter, grapes and wine (Bussy and Maffezzoni, 1993, RES 10/93)

Analyte: difenoconazole GC-ECD Method RES 10/93

LOQ: 0.02 mg/kg. Wine 0.01 mg/kg.

Description Residues are extracted from the sample with methanol and the filtrate is concentrated to approximately 15 ml. The residue is mixed with water, saturated sodium chloride and dichloromethane and extracted in a liquid-liquid extractor for 2 hours with dichloromethane. The dichloromethane is evaporated and the residue is taken up in hexane which is also then evaporated. The residue is taken up in benzene for column chromatography cleanup with basic alumina. The eluate is evaporated and the residue is taken up in hexane for GLC analysis.

Milk, liver, kidney, muscle, fat (Tribolet, 2000, 202/99)

Analytes: difenoconazole, CGA 205375 LC-MS-MS Method AG-544A

LOQ: 0.01 mg/kg tissues. 0.005 mg/L milk.

Description Procedure AG-544A with modifications.
Fat was melted and shaken with solvent in place of cold maceration.
Tert butyl ether replaced hexane in the partition cleanup steps to extract both analytes.
LC-MS-MS replaced GLC in the final determination step.

Celeriac (Pigeon, 2002, RE 20245 / 2001)

Analyte: difenoconazole GLC-NPD Method MR-046-02-01

LOQ: 0.02 mg/kg.

Description Residues are extracted from the sample with a mixture of acetonitrile and water. Difenoconazole residues are extracted into hexane from the aqueous phase and then into acetonitrile. Cleanup is effected by silica gel column chromatography. The eluate is evaporated and the residue is taken up in isooctane-acetone for GLC analysis.

Validation Method validation testing included: linearity of response, repeatability of injections, selectivity and repeatability.

Animal commodities (Crook, 2004, REM 147.07)

Analyte: difenoconazole, CGA 205375 LC-MS-MS Method REM 147.07

LOQ: 0.01 mg/kg for liver, kidney, muscle, fat, eggs. 0.005 mg/kg for milk.

Description Residues are extracted by homogenizing with acetonitrile-water. After centrifugation, an aliquot of the supernatant layer (e.g. 1 ml) is diluted with water and cleaned up on a solid-phase extraction cartridge. The cartridge is washed with hexane and then the residue is eluted with a dichloromethane - ethyl acetate mixture. After solvent evaporation, the residue is taken up in acetonitrile and diluted with water ready for LC-MS-MS analysis. Suppression or enhancement of response by substrate matrix was less than 10%, so non-matrix matched standards were suitable.

Crops and crop fractions (Crook, 2004, REM 147.08)

Analyte:	difenoconazole	LC-MS-MS	Method REM 147.08
LOQ:	0.01 mg/kg.		
Description	Residues are extracted by refluxing the sample with methanol-ammonia for 2 hours. An aliquot of the supernatant layer (e.g. 1 ml) is diluted with water (e.g. 10 ml) and cleaned up on a solid-phase extraction cartridge. The cartridge is washed with hexane and then the residue is eluted with a dichloromethane - ethyl acetate mixture. After solvent evaporation, the residue is taken up in acetonitrile and diluted with water ready for LC-MS-MS analysis. Suppression or enhancement of response by crop matrix was less than 10%, so non-matrix matched standards were suitable.		

Recovery data from the internal and independent laboratory validation (ILV) testing are summarised in Table 22.

Table 22. Analytical recoveries for spiked difenoconazole in various substrates

Commodity	Spiked analyte	Spike conc, mg/kg	n	Mean recov%	Range recov%	Method	Ref
apple	difenoconazole	0.01-0.1	10	88%	81-97%	DFG S19, revised	IF-04/00160619
apple	difenoconazole	0.01-0.3	10	88%	78-101%	REM 147.08	REM 147.08
asparagus	difenoconazole	0.02-0.2	10	104%	78-123%	DFG S19, revised	SYN-0211V Az.G02-0092
barley, wheat	difenoconazole	0.04-0.4	10	93	85-101%	RES 7/86	RES 7/86
beef liver	difenoconazole	0.05-0.25	6	96%	52-122%	AG-544 GC-NPD	ABR-93022
bovine fat	CGA 205375	0.01-0.1	10	95%	92-99%	REM 147.07	RJ3478B
bovine fat	difenoconazole	0.01-0.1	10	95%	89-99%	REM 147.07	RJ3478B
bovine kidney	CGA 205375	0.01-0.1	10	98%	91-106%	REM 147.07	RJ3478B
bovine kidney	difenoconazole	0.01-0.1	10	95%	90-107%	REM 147.07	RJ3478B
bovine liver	CGA 205375	0.01-0.1	10	98%	92-100%	REM 147.07	RJ3478B
bovine liver	difenoconazole	0.01-0.1	10	96%	93-100%	REM 147.07	RJ3478B
bovine milk	CGA 205375	0.005-0.05	10	93%	88-107%	REM 147.07	RJ3478B
bovine milk	difenoconazole	0.005-0.05	10	92%	85-101%	REM 147.07	RJ3478B
bovine muscle	CGA 205375	0.01-0.1	10	95%	89-100%	REM 147.07	RJ3478B
bovine muscle	CGA 205375	0.01-0.1	10	100%	96-110%	REM 147.07	SYN/DIF/04031
bovine muscle	difenoconazole	0.01-0.1	10	94%	92-96%	REM 147.07	RJ3478B
bovine muscle	difenoconazole	0.01-0.1	10	94%	72-101%	REM 147.07	SYN/DIF/04031
broccoli	difenoconazole	0.01-4	10	86%	74-99%	AG-575A GC-ECD	TMJ5014B
broccoli	difenoconazole	0.01-0.1	10	98%	80-119%	REM 147.08	REM 147.08
cabbage, head	difenoconazole	0.02-0.2	11	94%	76-108%	AG-575A GC-ECD	TMJ5014B
carrot	difenoconazole	0.04-0.2	6	110%	85-129%	RES 10/93	TMJ4940B
cauliflower	difenoconazole	0.04-0.2	4	97%	68-118%	RES 10/93	TMJ4940B
celeriac	difenoconazole	0.02-0.2	20	91%	75-110%	MR-046-02-01	RE 20245 / 2001
celery	difenoconazole	0.04-1	10	99%	88-131%	AG-575A GC-ECD	TMJ5014B
cherry	difenoconazole	0.01-0.5	11	87%	72-103%	AG-575A GC-MSD	TMJ5031B
cherry	difenoconazole	0.01-0.2	10	88%	81-95%	REM 147.08	REM 147.08
eggs	CGA 205375	0.01-0.1	10	87%	82-92%	REM 147.07	RJ3478B
eggs	CGA 205375	0.01-0.1	10	100%	94-109%	REM 147.07	SYN/DIF/04031
eggs	difenoconazole	0.05-0.5	4	80%	74-83%	AG-544 GC-NPD	AG-544
eggs	difenoconazole	0.05-0.25	6	103%	92-113%	AG-544 GC-NPD	ABR-93022
eggs	difenoconazole	0.01-0.1	10	84%	78-92%	REM 147.07	RJ3478B
eggs	difenoconazole	0.01-0.1	10	96%	84-110%	REM 147.07	SYN/DIF/04031
fat, cow	difenoconazole	0.05-0.5	8	95%	81-108%	AG-544 GC-NPD	AG-544
fennel	difenoconazole	0.01-0.1	10	84%	74-97%	AG-575A GC-MSD	TMJ5031B
grapes	difenoconazole	0.01-0.1	10	92%	80-102%	AG-575A GC-MSD	TMJ5031B
grapes	difenoconazole	0.01-0.1	10	104%	92-120%	REM 147.08	REM 147.08
kale	difenoconazole	0.02-10	5	81%	67-90%	AG-575A GC-ECD	TMJ5014B
kale	difenoconazole	0.01-0.5	10	104%	90-124%	AG-575A GC-MSD	TMJ5031B
kidney, cow	difenoconazole	0.05-0.5	4	94%	89-102%	AG-544 GC-NPD	AG-544
leeks	difenoconazole	0.01-0.2	10	87%	78-93%	REM 147.08	REM 147.08
lettuce	difenoconazole	0.04-0.2	12	96%	81-133%	AG-575A GC-ECD	TMJ5014B
lettuce	difenoconazole	0.01-0.5	10	98%	70-110%	AG-575A GC-MSD	TMJ5031B

Commodity	Spiked analyte	Spike conc, mg/kg	n	Mean recov%	Range recov%	Method	Ref
lettuce	difenoconazole	0.01-0.1	10	84%	71-96%	DFG S19, revised	IF-04/00160619
liver, cow	difenoconazole	0.05-0.5	4	115%	109-121%	AG-544 GC-NPD	AG-544
milk	CGA 205375	0.05-0.005	10	94%	88-110%	REM 147.07	SYN/DIF/04031
milk	difenoconazole	0.01-0.5	4	116%	102-134%	AG-544 GC-NPD	AG-544
milk	difenoconazole	0.01-0.05	6	118%	113-124%	AG-544 GC-NPD	ABR-93022
milk	difenoconazole	0.05-0.005	10	83%	62-92%	REM 147.07	SYN/DIF/04031
muscle, cow	difenoconazole	0.05-0.5	8	104%	96-110%	AG-544 GC-NPD	AG-544
olive fruit	difenoconazole	0.04-1	15	95%	75-104%	AG-575A GC-ECD	TMJ5014B
olive fruit	difenoconazole	0.01-1	10	104%	93-115%	REM 147.08	REM 147.08
olive oil	difenoconazole	0.01-1	10	93%	81-103%	REM 147.08	REM 147.08
olives	difenoconazole	0.01-5	10	85%	74-97%	AG-575A GC-MSD	TMJ5031B
peach	difenoconazole	0.01-0.2	10	93%	69-110%	AG-575A GC-ECD	TMJ5014B
peach	difenoconazole	0.01-0.5	10	93%	80-107%	AG-575A GC-MSD	TMJ5031B
peach, apricot	difenoconazole	0.04-0.2	12	102%	86-113%	RES 10/93	TMJ4940B
plum	difenoconazole	0.01-0.1	11	83%	70-109%	AG-575A GC-MSD	TMJ5031B
plum	difenoconazole	0.04-0.2	6	97%	76-110%	RES 10/93	TMJ4940B
pome fruit	difenoconazole	0.01-0.5	35	87%	72-131%	AG-575A GC-ECD	TMJ5014B
poultry fat	difenoconazole	0.05-0.5	4	103%	98-110%	AG-544 GC-NPD	AG-544
poultry liver	difenoconazole	0.05-0.5	4	94%	91-97%	AG-544 GC-NPD	AG-544
poultry meat	difenoconazole	0.05-0.5	4	89%	76-91%	AG-544 GC-NPD	AG-544
poultry skin	difenoconazole	0.05-0.5	4	96%	92-100%	AG-544 GC-NPD	AG-544
rape seed	difenoconazole	0.02-0.1	6	86%	69-95%	AG-575A GC-ECD	TMJ5014B
rape seed	difenoconazole	0.01-0.1	10	105%	95-124%	AG-575A GC-MSD	TMJ5031B
rape seed	difenoconazole	0.01-0.1	10	96%	79-108%	DFG S19, revised	IF-04/00160619
rape seed	difenoconazole	0.01-0.1	10	89%	81-94%	REM 147.08	REM 147.08
strawberry	difenoconazole	0.04-0.2	6	94%	90-99%	AG-575A GC-ECD	TMJ5014B
sugar beet leaves	difenoconazole	0.02-1	7	93%	65-109%	AG-575A GC-ECD	TMJ5014B
sugar beet leaves	difenoconazole	0.01-1	10	90%	75-102%	REM 147.08	REM 147.08
sugar beet root	difenoconazole	0.02-0.2	19	90%	72-110%	AG-575A GC-ECD	TMJ5014B
sugar beet root	difenoconazole	0.01-0.2	10	88%	83-93%	REM 147.08	REM 147.08
tomato	difenoconazole	0.05-0.5	6	103%	84-125%	AG-514	AG-514
tomato	difenoconazole	0.01-0.5	17	90%	70-107%	AG-575A GC-ECD	TMJ5014B
tomato	difenoconazole	0.01-0.4	11	95%	74-109%	AG-575A GC-MSD	TMJ5031B
tomato	difenoconazole	0.01-0.5	10	84%	76-89%	REM 147.08	REM 147.08
tomato puree	difenoconazole	0.01-1	10	92%	80-101%	REM 147.08	REM 147.08
wheat	difenoconazole	0.01-0.1	10	79%	68-88%	DFG S19, revised	IF-04/00160619
wheat grain	difenoconazole	0.01	4	86%	79-97%	AG-575	AG-575
wheat grain	difenoconazole	0.01-0.25	6	114%	106-120%	AG-575	900201
wheat grain	difenoconazole	0.01-1.0	16	88%	70-109%	AG-575A	AG-575A
wheat grain	difenoconazole	0.05-0.25	4	77%	73-80%	EMS9003.1	AG-537
wheat grain	difenoconazole	0.01-0.1	10	93%	77-107%	REM 147.08	REM 147.08
wheat grain, hay, forage, straw	difenoconazole	0.05-20	19	89%	70-109%	AG-537	AG-537
wheat straw	difenoconazole	0.01-0.25	6	106%	84-119%	AG-575	900201
wheat straw	difenoconazole	0.05-0.25	4	101%	90-112%	EMS9003.1	AG-537

Stability of residues in stored analytical samples

Information was received on the freezer storage stability of difenoconazole residues in plant and animal commodities, and of residues of CGA 205375 in animal commodities.

The Meeting received information on the stability of residues of difenoconazole in the following plant-based feed and food commodities: banana, cotton seed, cotton seed meal, cotton seed oil, lettuce, potatoes, soya beans, tomatoes, wheat forage, wheat grain and wheat straw. Residues were apparently stable in each case, with some commodities tested for 1 year, but most for 2 years. A summary of data is presented in Table 23.

Table 23. Freezer storage stability data for difenoconazole spiked into matrices of banana, cotton seed, cotton seed meal, cotton seed oil, lettuce, potatoes, soya beans, tomatoes, wheat forage, wheat grain and wheat straw.

Storage interval	Procedural recov %	Difenoconazole, mg/kg	Storage interval	Procedural recov %	Difenoconazole, mg/kg
TOMATOES, homogenized matrix, fortified with difenoconazole at 0.5 mg/kg (Beidler, 1991, ABR-90069), storage temperature approx -20 ˚C.			POTATOES, homogenized matrix, fortified with difenoconazole at 0.5 mg/kg (Beidler, 1991, ABR-90070), storage temperature approx -20 ˚C.		
0		0.45 0.46	0		0.50 0.54
28 days	118%	0.59 0.59	28 days	106%	0.56 0.54
88 days	116%	0.56 0.57	91 days	116%	0.72 0.56
182 days	104%	0.54 0.55	182 days	104%	0.54 0.57
419 days	120%	0.57 0.55	419 days	128%	0.57 0.55
530 days	108%	0.52 0.51	530 days	112%	0.50 0.52
735 days	120%	0.55 0.51	735 days	106%	0.50 0.51
residues apparently stable			residues apparently stable		
LETTUCE, homogenized matrix, fortified with difenoconazole at 0.2 mg/kg (Beidler, 1992, ABR-91024), storage temperature approx -20 ˚C.			SOYA BEANS, homogenized matrix, fortified with difenoconazole at 0.2 mg/kg (Beidler, 1992, ABR-91024), storage temperature approx -20 ˚C.		
0		0.15 0.15	0		0.19 0.16
35 days	100%	0.20 0.20	35 days	130%	0.23 0.25
91 days	115%	0.22 0.22	91 days	125%	0.21 0.23
199 days	95%	0.17 0.20	199 days	125%	0.18 0.19
371 days	105%	0.24 0.23	371 days	105%	0.21 0.29
residues apparently stable			residues apparently stable		
WHEAT FORAGE, homogenized matrix, fortified with difenoconazole at 0.5 mg/kg (Beidler, 1992, ABR-91024), storage temperature approx -20 ˚C.			BANANA WHOLE FRUIT, homogenized matrix, fortified with difenoconazole at 0.2 mg/kg (Kühne-Thu, 1994, 125/93), storage temperature approx -20 ˚C.		
0		0.46 0.47	0		0.16 0.19 0.16
35 days	98%	0.56 0.64	28 days	88% 91%	0.18 0.18 0.18
91 days	112%	0.54 0.56	84 days	95% 100%	0.16 0.17 0.17
199 days	102%	0.44 0.44	168 days	98% 100%	0.17 0.19 0.19
371 days	118%	0.47 0.46	364 days	93% 94%	0.17 0.19 0.18
residues apparently stable			residues apparently stable		
COTTON SEED, sample preparation" matrix (a/), fortified with difenoconazole at 0.4 mg/kg (Hayworth, 1998, ABR-98061), storage temperature approx -20 ˚C.			COTTON SEED OIL, fortified with difenoconazole at 0.4 mg/kg (Hayworth, 1998, ABR-98061), storage temperature approx -20 ˚C.		
0	106% 102% 97% 99% 90% 104% 113%		0	79% 78% 80% 79%	
2.9 months	101% 115%	0.42 0.46	2.3 months	82% 81%	0.31 0.33
7.0 months	103% 104%	0.46 0.41	6.9 months	80% 77%	0.30 0.31
16.0 months	117% 107%	0.42 0.46	14.8 months	98% 85%	0.36 0.33
24.2 months	116% 122%	0.64 0.67	23.5 months	84% 87%	0.36 0.35
residues apparently stable			residues apparently stable		
COTTON SEED MEAL, fortified with difenoconazole at 0.5 mg/kg (Hayworth, 1998, ABR-98061), storage temperature approx -20 ˚C.			WHEAT STRAW, sample preparation matrix (a/) fortified with difenoconazole at 1.0 mg/kg (Hayworth, 1998, ABR-98061), storage temperature approx -20 ˚C.		
0	86% 98% 73% 100% 68% 75% 96% 101%		0	108% 109% 97% 92%	
3.0 months	101% 104%	0.47 0.47	4.9 months	101% 107%	1.03 1.00
7.1 months	104% 98%	0.52 0.51	9.5 months	102% 102%	1.00 0.97
15.3 months	115% 115%	0.58 0.63	17.2 months	94% 101%	1.16 1.13
24.0 months	125% 119%	0.62 0.59	26.6 months	114% 124%	1.15 1.18
residues apparently stable			residues apparently stable		

Storage interval	Procedural recov %	Difenoconazole, mg/kg	Storage interval	Procedural recov %	Difenoconazole, mg/kg
WHEAT FORAGE, sample preparation matrix (a) fortified with difenoconazole at 1.0 mg/kg (Hayworth, 1998, ABR-98061), storage temperature approx -20 ˚C.			WHEAT GRAIN, sample preparation matrix (a) fortified with difenoconazole at 0.2 mg/kg (Hayworth, 1998, ABR-98061), storage temperature approx -20 ˚C.		
0	104% 104% 109% 105%		0	105% 107% 98% 101%	
4.6 months	96% 104%	1.01 1.09	4.6 months	109% 111%	0.22 0.22
8.7 months	81% 81%	0.68 0.74	8.5 months	91% 86%	0.16 0.17
17.0 months	108% 115%	1.13 1.06	18.0 months	113% 103%	0.23
25.5 months	130% 118%	1.22 1.09	25.6 months	114% 119%	0.24
residues apparently stable			residues apparently stable		

a - Sample preparation guidelines were based on FDA Pesticide Analytical Manual, Vol 1, Section 141 and 40CFR180.1 (j).

The Meeting received information on the stability of residues of difenoconazole and metabolite CGA 205375 in animal tissues, milk and eggs when stored at freezer temperatures for 1 year. Residues were generally stable.

Tribolet (2000, 202/99) tested the freezer storage stability of difenoconazole and metabolite CGA 205375 spiked into animal tissues and milk (Table 24). Milk (10 mL portions) and tissues (10 g portions) were spiked and thoroughly mixed in 25 mL or 100 mL jars prior to storage in a freezer at or below -18 ˚C. At the end of the storage period, freshly spiked samples were analysed as the procedural recoveries at the same time that the stored samples were analysed using procedure AG-544A. The storage testing for CGA 205375 in liver was examined in a second series because only 59% remained in the first. The analytical variability made interpretation of small losses difficult. Generally, difenoconazole and metabolite CGA 205375 appeared reasonably stable during storage.

Table 24. Freezer storage stability of difenoconazole and metabolite CGA 205375 spiked into animal tissues and milk and stored at or below -18 ˚C for approximately 10 months

Substrate	Analyte	Spike conc, mg/kg	Procedural recoveries %	Storage interval, days	Residues remaining, mg/kg	average% remaining,
Muscle	difenoconazole	0.2	120 86	312	0.17 0.17 0.19 0.17 0.17	87%
Muscle	CGA 205375	0.2	97 82	312	0.16 0.17 0.18 0.18 0.15	84%
Liver	difenoconazole	0.2	78 80	296	0.14 0.15 0.10 0.13 0.13	65%
Liver	CGA 205375	0.2	117 85	296	0.11 0.12 0.08 0.12 0.16	59%
Liver	CGA 205375	0.2	89	389	0.18 0.18 0.18 0.19 0.17	90%
Kidney	difenoconazole	0.2	76 81	301	0.15 0.15 0.09 0.11 0.13	63%
Kidney	CGA 205375	0.2	73 83	301	0.14 0.17 0.16 0.13 0.14	74%
Fat	difenoconazole	0.2	77 78	303	0.16 0.15 0.14 0.16 0.17	78%
Fat	CGA 205375	0.2	73 79	303	0.17 0.16 0.16 0.17 0.16	82%
Milk	difenoconazole	0.05	87	305	0.040 0.039 0.040 0.041 0.039	80%
Milk	CGA 205375	0.05	80	305	0.039 0.037 0.036 0.037 0.039	75%

Wurz and McCaskill (1993, ABR-93012) reported on the freezer storage stability testing of difenoconazole residues spiked into eggs, milk, poultry muscle and beef liver when stored for 12 months at -20 ˚C. Analysis relied on procedure AG-544. Residues were apparently stable, but no procedural recovery data were available to confirm the performance of the test method on each occasion.

USE PATTERN

Difenoconazole is a broad-spectrum fungicide used for disease control in many fruits, vegetables, cereals and other field crops. It has preventive and curative action. Difenoconazole acts by inhibition of demethylation during ergosterol synthesis; it is a DMI fungicide. Labels or translations of labels for the following uses (Table 25) were available to the Meeting.

Table 25. Registered uses of difenoconazole in Australia, Belgium, Belize, Brazil, Costa Rica, Dominican Republic, El Salvador, France, Germany, Guatemala, Honduras, Indonesia, Italy, Luxembourg, Nicaragua, Panama, Poland, Spain, Switzerland and UK

Crop	Country	Application						
		Form	Type	Rate kg ai/ha	Conc kg ai/hL	Spray vol, L/ha	Max number	PHI days
Apple	Australia	WG	foliar		0.0025-0.0035			28
Apple	Belgium	EC	foliar	0.0375				14
Apple	Brazil	EC	foliar		0.0035	800-1500	8	5
Apple	France	EC	foliar		0.00375	> 1000	3	30
Apple	France	EC	foliar	0.0375		< 1000	3	30
Apple	Italy	EC	foliar		0.00375		4	14
Apple	Poland	EC	foliar	0.05			3	14
Apple	Spain	EC	foliar		0.005		5	14
Apple	Spain	EC	foliar	0.075		< 1500	5	14
Apricot	France	EC	foliar		0.005	> 1000	3	14
Apricot	France	EC	foliar	0.05		< 1000	3	14
Asparagus	Belgium	EC	foliar [5]	0.125				
Asparagus	France	EC	foliar	0.125			3	a
Asparagus	Germany	EC	foliar	0.1			3	
Asparagus	Italy	EC	foliar	0.125			4	7
Asparagus	Spain	EC	foliar	0.125			3	
Banana	Australia	EC	foliar	0.1				1
Banana	Belize	EC	foliar, aerial	0.1			8	0
Banana	Brazil	EC	foliar	0.1		500-1000	5	7
Banana	Brazil	EC	foliar, aerial	0.1		15	5	7
Banana	Costa Rica	EC	foliar, aerial	0.1			8	0
Banana	Dominican Republic	EC	foliar, aerial	0.1			8	0
Banana	El Salvador	EC	foliar, aerial	0.1			8	0
Banana	Guatemala	EC	foliar, aerial	0.1			8	0
Banana	Honduras	EC	foliar, aerial	0.1			8	0
Banana	Nicaragua	EC	foliar, aerial	0.1			8	0
Banana	Panama	EC	foliar, aerial	0.1			8	0
Broccoli	Belgium	EC	foliar	0.125			2	14
Broccoli	UK	EC	foliar	0.075		400	3	21
Brussels sprouts	Belgium	EC	foliar	0.125			2	21
Brussels sprouts	France	EC	foliar	0.125			3	21
Brussels sprouts	Germany	EC	foliar	0.1			3	21
Brussels sprouts	UK	EC	foliar	0.075		400	3	21
Cabbage	Belgium	EC	foliar	0.125			2	21
Cabbage	France	EC	foliar	0.125			3	21
Cabbage	Germany	EC	foliar	0.1			3	21
Cabbage	UK	EC	foliar	0.075		400	3	21
Cabbage, Chinese	Belgium	EC	foliar	0.125			2	14

[5] Asparagus. Field spray after harvest is taken.

Crop	Country	Application						
		Form	Type	Rate kg ai/ha	Conc kg ai/hL	Spray vol, L/ha	Max number	PHI days
Cabbage, Chinese	Germany	EC	foliar	0.1			3	21
Carrot	Australia	EC	foliar	0.075-0.125				7
Carrot	Belgium	EC	foliar	0.125			3	14
Carrot	Brazil	EC	foliar	0.15		200-400	8	15
Carrot	France	EC	foliar	0.125			3	14
Carrot	Germany	EC	foliar	0.1			3	21
Carrot	Italy	EC	foliar	0.1-0.125			4	7
Cauliflower	Belgium	EC	foliar	0.125			2	14
Cauliflower	Brazil	EC	foliar		0.005	200-400	5	14
Cauliflower	France	EC	foliar	0.125			3	14
Cauliflower	Germany	EC	foliar	0.1			3	21
Cauliflower	Italy	EC	foliar	0.1-0.125			4	14
Cauliflower	UK	EC	foliar	0.075		400	3	21
Celeriac	Belgium	EC	foliar	0.125			4	14
Celeriac	France	EC	foliar	0.125			3	21
Celeriac	Germany	EC	foliar	0.1			3	21
Celery	Belgium	EC	foliar	0.125			3	14
Celery	France	EC	foliar	0.125			3	14
Celery	Germany	EC	foliar	0.1			3	21
Celery	Italy	EC	foliar	0.1-0.125			4	21
Celery	Spain	EC	foliar	0.075-0.125			4	14
Cherry	Poland	EC	foliar	0.05		500-750	3	14
Citrus fruit	Brazil	EC	foliar		0.005	500-1000	2	30
Cucumber	Italy	EC	foliar	0.125			4	7
Garlic	Brazil	EC	foliar	0.125		200-400	6	14
Garlic	Spain	EC	foliar	0.125			4	30
Grape	Brazil	EC	foliar		0.003	200-800	6	21
Grape	France	EC	foliar	0.03			3	-
Grape	Italy	EC	foliar		0.005		4	21
Grapes	Luxembourg	EC	foliar	0.03				
Leek	Germany	EC	foliar	0.1			3	21
Lettuce	Brazil	EC	foliar		0.005	200-400	5	14
Lettuce	Spain	EC	foliar	0.20[6]			3	14
Macadamia nuts	Australia	EC	foliar		0.0125			-
Mango	Brazil	EC	foliar		0.0125	500-1000	3	7
Olive	Spain	EC	foliar		0.015		2	30
Papaya	Brazil	EC	foliar		0.0075		4	14
Peach	France	EC	foliar		0.005	> 1000	3	14
Peach	France	EC	foliar	0.05		< 1000	3	14
Peach	Italy	EC	foliar		0.005-0.0075		3	7
Pear	Australia	WG	foliar		0.0025-0.0035			28
Pear	Belgium	EC	foliar	0.0375				14
Pear	France	EC	foliar		0.00375	> 1000	3	30
Pear	France	EC	foliar	0.0375		< 1000	3	30
Pear	Italy	EC	foliar		0.00375		4	14
Pear	Poland	EC	foliar	0.05			3	14
Pear	Spain	EC	foliar		0.005		5	14
Pear	Spain	EC	foliar	0.075		< 1500	5	14
Pepper, chili	Indonesia	EC	foliar		0.0063-0.013		7 days intervals	-

[6] Lettuce in Spain. The registration document states that difenoconazole is registered for use on lettuce at a rate of 0.125-0.20 kg ai/ha with a 14 days PHI. The maximum application rate on the available label was 0.125 g ai/ha.

Crop	Country	Application						
		Form	Type	Rate kg ai/ha	Conc kg ai/hL	Spray vol, L/ha	Max number	PHI days
Plum	France	EC	foliar		0.005	> 1000	3	14
Plum	France	EC	foliar	0.05		< 1000	3	14
Potato	Australia	EC	foliar	0.075-0.125				7
Potato	Brazil	EC	foliar	0.075		200-400	4	7
Potato	Italy	EC	foliar	0.1-0.125			4	14
Potato	Spain	EC	foliar	0.2			4	30
Rape	Switzerland	SC, includes carbendazim	foliar	0.125			1	growth stage
Rape	UK	EC	foliar	0.125		200	2	BBCH 69[7]
Rape, winter	Germany	EC	foliar	0.25			1	
Rice	Brazil	EC	foliar	0.075		100-200	1	45
Rice	Indonesia	EC	foliar	0.05 – 0.1			2	BBCH 63-67 [8]
Rye	Switzerland	EC, includes propiconazole	foliar	0.125			1	BBCH 61[9]
Soya	Brazil	EC	foliar	0.075		100-200	1	30
Soya	Brazil	EC	foliar, aerial	0.075		20-50	1	30
Sugar beet	Belgium	EC	foliar	0.125				21
Sugar beet	Germany	EC	foliar	0.1			2	28
Sugar beet	Italy	EC	foliar	0.05-0.075			3	21
Sugar beet	Spain	EC	foliar	0.075-0.125			3	30
Sugar beet	Switzerland	EC, includes propiconazole	foliar	0.125			1	
Sunflower	Switzerland	SC, includes carbendazim	foliar	0.125			1	BBCH 51[10]
Tomato	Australia	EC	foliar	0.075-0.125				3
Tomato	Brazil	EC	foliar		0.0125	200-800	3	14
Tomato	France	EC	foliar	0.125			3	20
Tomato	Indonesia	SC, includes azoxystrobin	foliar		0.00625-0.0125			
Tomato	Italy	EC	foliar	0.125			4	7
Tomato	Spain	EC	foliar		0.0125-0.016		4	7
Tomato	Spain	EC	foliar	0.125-0.2			4	7
Watermelon	Brazil	EC	foliar		0.0075		4	3
Wheat	Germany	EC, includes propiconazole	foliar	0.1			1	35
Wheat, winter	Switzerland	EC, includes propiconazole	foliar	0.125			1	BBCH 61[11]
Wheat, winter	UK	EC	foliar	0.075		200	1	Up to BBCH 71[12]

a - Asparagus, difenoconazole use in France. Treatment starts in April/May on young asparagus plants not yet in production and in June on asparagus plants in production. In asparagus crops protected by 6 to 8 applications of fungicide per year, use the difenoconazole product for the first three treatments and finish the season with a product that acts in a different way.

[7] Rape seed. Growth stage BBCH 69: end of flowering.
[8] Registered use on rice in Indonesia. Timing: 2 applications in season at mid booting stage (45 days after planting) and 75% of flowering (60 days after planting). Interpreted as BBCH 43-45 and BBCH 63-67.
[9] Rye. Growth stage BBCH 61: beginning of flowering, first anthers visible.
[10] Sunflower. Growth stage BBCH 51: inflorescence just visible between youngest leaves.
[11] Wheat. Growth stage BBCH 61: beginning of flowering, first anthers visible.
[12] Wheat. Growth stage BBCH 71: watery ripe, first grains have reached half their final size.

RESIDUES RESULTING FROM SUPERVISED TRIALS

The Meeting received information on supervised field trials for difenoconazole uses that produced residues on the following commodities.

Commodity	Crop	Table
Citrus fruits	Orange	Table 27
Pome fruits	Apple	Table 28
	Pear	Table 29
Stone fruits	Cherries	Table 30
	Peach	Table 31
	Plum	Table 32
Berry fruits	Grapes	Table 33
Tropical fruits, edible peel	Olive	Table 34
Tropical fruits, inedible peel	Banana	Table 35
	Mango	Table 36
	Papaya	Table 37
Bulb vegetables	Garlic	Table 38
	Leek	Table 39
Brassica vegetables	Broccoli	Table 40
	Brussels sprouts	Table 41
	Cabbages	Table 42
	Cauliflower	Table 43
Fruiting vegetables, cucurbits	Watermelon	Table 44
Fruiting vegetables, other than cucurbits	Chili peppers	Table 45
	Tomatoes	Table 46
Leafy vegetables	Lettuce	Table 47
Pulses	Soya bean	Table 48
Root and tuber vegetables	Carrot	Table 49
	Potato	Table 50
	Sugar beet	Table 51
Stalk and stem vegetables	Asparagus	Table 52
	Celeriac	Table 53
	Celery	Table 54
Cereal grains	Rice	Table 55
	Wheat	Table 56
Oilseed	Rape seed	Table 57
	Sunflower seed	Table 58
Feeds	Wheat straw and fodder	Table 59
	Rice straw and fodder	Table 60
	Sugar beet leaves and tops	Table 61
	Oilseed rape fodder	Table 62
	Sunflower plant and stubble	Table 63

Trials were generally well documented with laboratory and field reports. Laboratory reports included method validation with procedural recoveries from spiking at residue levels similar to those occurring in samples from the supervised trials. Dates of analyses or duration of residue sample storage were also provided. Although trials included control plots, no control data are recorded in the tables except where residues in control samples exceeded the LOQ. Residue data are recorded unadjusted for recovery.

In trials where duplicate field samples from an unreplicated plot were taken at each sampling time and analysed separately the mean of the two analytical results was taken as the best estimate of the residues in the plot and the means are recorded in the tables.

When residues were not detected they are shown as below the LOQ (e.g., < 0.01 mg/kg). Residues, application rates and spray concentrations have generally been rounded to two significant figures or, for residues near the LOQ, to one significant figure. Residue values from the trials conducted according to maximum GAP have been used for the estimation of maximum residue levels. Those results included in the evaluation are double underlined.

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

Table 26. Summary of sprayers, plot sizes and field sample sizes in the supervised trials

Crop	Place	Year	Sprayer	Plot size	Sample size
Apples	Europe	2000-2001	mist sprayer, motorised knapsack, tractor	6-15 trees, 40-100 m^2	2-3 kg
Asparagus	Europe	1988-91	motorised sprayer, knapsack, pneumatic sprayer	10-39 m^2	1-2 kg
Banana	Colombia	1997	knapsack (simulated aerial), aerial	1000-4800 m^2	6 bunches
Banana	Costa Rica	1993	knapsack	5100-6000 m^2	18 fruit
Banana	Ecuador	1997	knapsack (simulated aerial), aerial	5000-6250 m^2	6 bunches
Banana	Guatemala	1993	mist sprayer	2800 m^2	18 fruit
Banana	Honduras	1997	knapsack (simulated aerial), aerial	180-4800 m^2	6 bunches
Broccoli	Europe	2002, 2004	plot sprayer, knapsack, boom sprayer	60-120 m^2	0.5-12 kg
Brussels sprouts	Europe	1990, 1995, 1999	CO_2 powered knapsack, knapsack, plot sprayer	16-48 m^2	1 kg
Cabbage	Europe	1990, 1998-99, 2000 -03	CO_2 powered knapsack, boom sprayer, plot sprayer, knapsack	16-120 m^2	5-12 heads
Carrots	France	1991-96, 2000	knapsack, boom sprayer, plot sprayer	15-45 m^2	2-3.9 kg
Carrots	Switzerland	1987	?	?	?
Cauliflower	Europe	1999-2000, 2005	plot sprayer, knapsack	18-180 m^2	0.5-15 kg
Celeriac	Belgium	2001	CO_2 powered sprayer	15-20 m^2	2 kg (12 units)
Celery	Europe	1988, 1990, 2003-04	plot sprayer, knapsack, motorised sprayer, foliar hand sprayer	5-60 m^2	0.5-5 kg
Cherries	Europe	2003-2004	air blast sprayer, tractor powered blower	72-90 m^2	0.5-2 kg
Chili peppers	Indonesia, Malaysia	1990, 1991	CO_2 powered knapsack, motorised knapsack	11-12 m^2	
Garlic	Brazil	1995	CO_2 powered knapsack	10 m^2	1 kg
Grapes	Europe	2003-2005	air blast sprayer, mist sprayer, knapsack	15-144 m^2	1-20 kg
Leeks	Europe	1990, 1992, 1998, 2004	plot sprayer, knapsack, foliar hand sprayer	18-120 m^2	1-5 kg
Lettuce	Spain	1991, 2003	motorised knapsack, knapsack	10-50 m^2	12 units
Mango	Brazil	2003	motorised knapsack	210-600 m^2	12 fruits
Oilseed rape	France, Germany	1988, 1996-97	high volume sprayer	27-60 m^2	0.5-3 kg
Olives	Europe	2003-2005	air blast sprayer, knapsack mistblower	200-660 m^2	1-20 kg
Orange	Brazil	1995	motorised sprayer	3 trees	1 kg
Papaya	Brazil	2002	knapsack air blast sprayer	153-189 m^2	12 fruits
Peaches	Europe	2003-2004	knapsack, air blast sprayer	4-10 trees	2-5 kg (12-24 fruits)
Pears	Europe	2001-2002	motorised knapsack	24-100 m^2	2- 4 kg
Plums	Europe	2003-2004	knapsack, tractor powered blower	64-260 m^2	1-3 kg
Potato	Italy, Spain	2003, 2005	knapsack	20-180 m^2	2-7 kg
Rice	Indonesia, Malaysia	1994, 1998	knapsack, motorised knapsack	200-370 m^2	0.6-1 kg
Soya beans	Brazil	2000, 2003	CO_2 powered knapsack	30-100 m^2	1-1.6 kg
Sugarbeet	Europe	1985-91, 1995-96, 2004	plot sprayer, knapsack, high volume sprayer	30-100 m^2	1-10 kg
Sunflower	Europe	2004-2005	knapsack, plot sprayer	60-120 m^2	1-21 kg

Crop	Place	Year	Sprayer	Plot size	Sample size
Tomatoes	Europe	2001-2005	plot sprayer, knapsack, motorised knapsack	20-80 m^2	0.5-5 kg
Watermelons	Brazil	1995	motorised sprayer, CO_2 powered sprayer	90-180 m^2	8-12 fruits
Wheat	Europe	1985-90, 1993, 1998	knapsack, plot sprayer, motorised sprayers	20-600 m^2	0.5-2.8 kg

Table 27. Difenoconazole residues in oranges resulting from supervised trials in Brazil

ORANGES	Application					PHI	Commodity	Residues, mg/kg	Ref
country, year (variety)	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	days		difenoconazole	
Brazil (SP), 1995 (Valencia)	EC		0.005	2000	2	30	fruit	< 0.05	E-9079-94
Brazil (SP), 1995 (Pera Rio)	EC		0.005	2000	2	16 30	fruit	< 0.05 < 0.05	E-9081-94
Brazil (SP), 1995 (Pera Rio)	EC		0.01	2000	2	16 30	fruit	< 0.05 < 0.05	E-9082-94
Brazil (SP), 1995 (Valencia)	EC		0.01	2000	2	30	fruit	< 0.05	E-9080-94

Table 28. Difenoconazole residues in apples resulting from supervised trials in France, Greece, Italy and Spain

APPLES	Application					PHI	Commodity	Residues, mg/kg	Ref
country, year (variety)	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	days		difenoconazole	
France, 2000 (Royal Gala)	EC	0.077-0.066	0.0075	1025-875	4	14	fruit	0.11	0012201
France, 2001 (Royal Gala, Pajam)	EC	0.075	0.015	500	4	0 3 7 10 14	fruit	0.29 0.24 0.35 0.23 0.28	0110601
Greece, 2000 (Granny Smith)	EC	0.072	0.005	1350	5	0 7 14 21	fruit	0.12 0.13 0.13 0.08	2042/00
Greece, 2001 (Granny Smith)	EC	0.074	0.0063	1160	4	14	fruit	0.05	2019/01
Italy, 2000 (Golden Delicious Smoothee)	EC	0.075	0.0075	1000	4	0 14 21	fruit	0.15 0.04 0.06	2036/00
Italy, 2001 (Fuji)	EC	0.061	0.0052	1200-1170	4	0 7 14 21	fruit	0.19 0.12 0.08 0.05	2070/01
Spain, 2000 (Golden Delicious)	EC	0.075	0.005	1500	5	0 3 7 14 21	fruit	0.21 0.29 0.25 0.14 0.14	2025/00
Spain, 2000 (World Gala)	EC	0.075	0.005	1500	5	0 3 7 14 21	fruit	0.26 0.21 0.16 0.15 0.10	2026/00
Spain, 2001 (Reineta)	EC	0.075	0.0049	1490-1620	4	0 14	fruit	0.19 0.10	2096/01

Table 29. Difenoconazole residues in pears resulting from supervised trials in France and Greece

PEARS country, year (variety)	Application					PHI days	Commodity	Residues, mg/kg difenoconazole	Ref
	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.				
France, 2002 (Conference)	EC	0.075	0.005	1500	4	14	fruit	0.07	02-2085
Greece, 2001 (Highland)	EC	0.072	0.0068	1060	4	14	fruit	0.16	2020/01

Table 30. Difenoconazole residues in cherries resulting from supervised trials in France and Germany

CHERRIES country, year (variety)	Application					PHI days	Commodity	Residues, mg/kg difenoconazole	Ref
	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.				
France, 2004 (Alegria) sour cherries	EC	0.057 +0.054 +0.047	0.00375 +0.00375 +0.00375	1510 +1430 +1250	3	14	fruit	0.08	04-0309
Germany, 2003 (Burlat) sweet cherries	EC	0.059		1500	3	0 3 7 10 14	fruit	0.31 0.20 0.13 0.08 0.06	gch218403
Germany, 2003 (Schattenmorelle) sour cherries	EC	0.039	0.0039	1000	3	0 3 7 10 14	fruit	0.32 0.21 0.15 0.09 0.10	gch218103

Table 31. Difenoconazole residues in peaches resulting from supervised trials in France, Greece and Italy

PEACH country, year (variety)	Application					PHI days	Commodity	Residues, mg/kg difenoconazole	Ref
	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.				
France, 2004 (Promesse)	EC	0.11	0.0075	1500	3	7	fruit	0.18	04-0505
Greece, 2004 (Evert)	EC	0.11	0.0075	1460	3	7	fruit	0.26	04-0412 GR/FR/04-0093
Greece, 2004 (Red Haven)	EC	0.11	0.0075	1460	3	0 3 7	fruit	0.14 0.18 0.21 0.16	04-0412 GR/FR/04-0094
Italy, 2003 (Maria Marta)	EC	0.090	0.0075	1500	3	7	fruit	0.14	03-0443
Italy, 2003 (Padana)	EC	0.090	0.0075	1500	3	0 1 3 5 7	fruit	0.26 0.24 0.20 0.21 0.19	03-0441
Italy, 2003 (Stark Redgold)	EC	0.090	0.0075	1500	3	0 1 3 5 7	fruit	0.13 0.09 0.08 0.08 0.07	03-0440
Italy, 2003 (Sweet Lady)	EC	0.090	0.0075	1500	3	0 1 3 5 7	fruit	0.15 0.16 0.18 0.13 0.14	03-0442
Italy, 2004 (Sweet Lady)	EC	0.090	0.0075	1500	3	7	fruit	0.11	04-0307

Table 32. Difenoconazole residues in plums resulting from supervised trials in France, Germany and Spain

PLUM	Application					PHI	Commodity	Residues, mg/kg	Ref
country, year (variety)	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	days		difenoconazole	
France, 1998 (Quetsches d'Alsace)	EC	0.050	0.005	1000	3	14	fruit	0.07	2161/98
France, 1999 (Quetsches d'Alsace)	EC	0.053	0.005	1070	3	14	fruit	0.10	2108/99
France, 2004 (Reine Claude)	EC	0.056	0.00375	1500	2 3	10 0 3 7 10 14	fruit	0.04 0.05 0.04 0.03 0.03 0.03	04-0506 AF/7874/SY/1
France, 2004 (Stanley)	EC	0.056	0.00375	1500	2 3	10 0 3 7 10 14	fruit	0.02 0.06 0.06 0.03 0.06 0.02	04-0506 AF/7874/SY/2
Germany, 2003 (Cacaks Beste)	EC	0.040	0.0039	1010	3	0 3 7 10 14	fruit	0.03 0.03 0.03 0.03 0.04	gpl258303
Germany, 2003 (Hauszwetsche)	EC	0.079	0.0039	2000	3	0 14	fruit	0.05 0.02	gpl258203
Germany, 2003 (Hermann)	EC	0.059	0.0039	1500	3	0 3 7 10 14	fruit	0.03 0.02 0.01 < 0.01 < 0.01	gpl258103
Germany, 2003 (Opal)	EC	0.059	0.0039	1500	2 3	11 11	fruit	0.02 0.01	gpl258403
Spain, 2005 (Angelino)	EC	0.073 +0.080 +0.085	0.005 0.005 0.005	1460 +1600 +1690	2 3	7 0 3 7 10 13	fruit	0.05 0.11 0.05 0.11 0.08 0.08	05-0503 ES-FR-05-0429
Spain, 2005 (Black Gold)	EC	0.071 +0.073 +0.073	0.005 0.005 0.005	1410 +1460 +1460	2 3	7 0 3 7 10 14	fruit	0.03 0.09 0.07 0.06 0.04 0.03	05-0503 ES-FR-05-0430

Table 33. Difenoconazole residues in grapes resulting from supervised trials in France and Italy

GRAPES	Application					PHI	Commodity	Residues, mg/kg	Ref
country, year (variety)	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	days		difenoconazole	
France, 2004 (Abouriou)	EC	0.050	0.005	820	4	21 28	bunch	0.04 0.04	04-0601 AF/7875/SY/1
France, 2004 (Gamay)	EC	0.050	0.005	870	4	21 28	bunch	0.07 0.07	04-0601 AF/7875/SY/2
Italy, 2003 (Italia)	EC	0.050	0.005	1000	4	21 28	bunch	0.04 0.05	03-0426

GRAPES country, year (variety)	Application Form	 kg ai/ha	 kg ai/hL	 water (L/ha)	 no.	PHI days	Commodity	Residues, mg/kg difenoconazole	Ref
Italy, 2003 (Malvasia)	EC	0.050	0.005	1000	4	0 7 14 21 28	bunch	0.07 0.04 0.04 0.02 0.02	03-0427
Italy, 2003 (Riesling)	EC	0.050	0.005	1000	4	0 7 14 21 28	bunch	0.08 0.07 0.03 0.03 0.03	03-0428
Italy, 2004 (Merlot)	EC	0.050	0.005	1000	3 4	10 0 7 14 21 28	bunch	0.02 0.06 0.02 0.03 0.02 0.02	04-0501 IT-FR-04-0184
Italy, 2004 (Sangiovese)	EC	0.050	0.005	990	3 4	9 0 7 14 21 28	bunch	0.03 0.06 0.03 0.01 0.01 0.03	04-0501 IT-FR-04-0214
Italy, 2004 (Trebbiano)	EC	0.050	0.005	830	4	21 28	bunch	0.03 0.03	04-0601 AF/7875/SY/3

Table 34. Difenoconazole residues in olives resulting from supervised trials in France and Spain

OLIVE country, year (variety)	Application Form	 kg ai/ha	 kg ai/hL	 water (L/ha)	 no.	PHI days	Commodity	Residues, mg/kg difenoconazole a	Ref
France, 2005 (Verdale)	EC	0.13	0.016	800	2	30	fruit	0.76	05-0603 AF/8567/SY/1
Spain, 2003 (Arbequina)	EC	0.14	0.016	850	2	0 7 14 21 30 35	fruit	3.2 1.3 0.79 0.56 0.51 0.41	03-0522
Spain, 2003 (Marteña)	EC	0.13	0.018	790	2	0 7 14 21 30 35	fruit	0.81 0.42 0.48 0.29 0.42 0.38	03-0523
Spain, 2003 (Zorzaleña)	EC	0.12	0.016	760	2	30	fruit	0.90	03-0524
Spain, 2004 (Alberquina)	EC	0.12	0.015	800	2	30	fruit	0.40	04-6067 AF/7872/SY/1
Spain, 2004 (Alberquina)	EC	0.12	0.016	770	1 2	14 0 7 14 21 30 35	fruit	0.69 2.0 0.66 0.44 0.34 0.29 0.16	04-6067 AF/7872/SY/2

OLIVE country, year (variety)	Application Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	PHI days	Commodity	Residues, mg/kg difenoconazole a	Ref
Spain, 2004 (Marteña)	EC	0.12	0.015	800	1 2	14 0 7 14 21 30 35	fruit	0.24 0.69 0.58 0.44 0.52 0.22 0.14	04-6067 AF/7872/SY/3
Spain, 2005 (Arbequino)	EC	0.13	0.015	840	2	30	fruit	1.2	05-0603 AF/8567/SY/2

a - Analysis on fruit without stone, but calculated on whole fruit from weights of stone and fruit

In the banana trials in 1997 in Ecuador, Colombia and Honduras, unbagged fruit were chosen for study although these cropping conditions rarely occur in commercial banana production. Unbagged bananas represent the extreme case for consumer exposure. In these trials, difenoconazole residues were not present above the LOQ (< 0.02 mg/kg) in the edible portion.

Table 35. Difenoconazole residues in bananas resulting from supervised trials in Colombia, Costa Rica, Ecuador, Guatemala and Honduras

BANANA country, year (variety)	Application Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	PHI days	Commodity	Residues, mg/kg difenoconazole [a b]	Ref
Colombia, 1997 (Cavendish Robusta)	EC	0.093-0.10	0.025	19-21	8	0 [c] 0 0	fruit, unbagged fruit, unbagged fruit, unbagged	pu < 0.02 (6) pe < 0.02 (3) 0.25 0.02 0.24 wf < 0.02 (4) 0.13 0.12 (mean 0.04)	119/96 2118/96
Colombia, 1997 (Cavendish Robusta)	EC	0.091-0.11	0.025	18-22	8 aerial	0 [c] 0 0	fruit, unbagged fruit, unbagged fruit, unbagged	pu < 0.02 (6) pe < 0.02 (6) wf < 0.02 (6)	119/96 2119/96
Colombia, 1997 (Cavendish Robusta)	EC	0.095-0.11	0.025	14-17	8 aerial	0 [c] 0 0	fruit, unbagged fruit, unbagged fruit, unbagged	pu < 0.02 (6) pe < 0.02 (5) 0.03 wf < 0.02 (5) 0.02	119/96 2120/96
Costa Rica, 1993 (Grand Naine)	EC	0.10	0.44	24	8	0 1 2 0 1 2	fruit, bagged fruit, bagged fruit, bagged fruit, unbagged fruit, unbagged fruit, unbagged	pu < 0.02 wf < 0.02 pu < 0.02 wf < 0.02 pu < 0.02 wf < 0.02 pu < 0.02 wf 0.03 pu < 0.02 wf 0.04 pu < 0.02 wf 0.02	2127/92
Costa Rica, 1993 (Grand Naine)	EC	0.10	0.44	24	8	0 1 2 0 1 2	fruit, bagged fruit, bagged fruit, bagged fruit, unbagged fruit, unbagged fruit, unbagged	pu < 0.02 wf < 0.02 pu < 0.02 wf < 0.02 pu < 0.02 wf < 0.02 pu < 0.02 wf 0.03 pu < 0.02 wf 0.03 pu < 0.02 wf 0.03	2128/92
Ecuador, 1997 (Giant Cavendish)	EC	0.10	0.025	19-22	8	0 [c] 0 0	fruit, unbagged fruit, unbagged fruit, unbagged	pu < 0.02 (6) pe < 0.02 (5) 0.02 wf < 0.02 (6)	118/96 2115/96
Ecuador, 1997 (Giant Cavendish)	EC	0.10	0.025	19-22	8 aerial	0 [c] 0 0	fruit, unbagged fruit, unbagged fruit, unbagged	pu < 0.02 (6) pe < 0.02 (6) wf < 0.02 (6)	118/96 2116/96
Ecuador, 1997 (Giant Cavendish)	EC	0.075-0.11	0.025	11-16	8 aerial	0 [c] 0 0	fruit, unbagged fruit, unbagged fruit, unbagged	pu < 0.02 (6) pe < 0.02 (6) wf < 0.02 (6)	118/96 2117/96
Guatemala, 1993 (Grand Naine)	EC	0.10	0.025	40	8	0 1 2 0 1 2	fruit, bagged fruit, bagged fruit, bagged fruit, unbagged fruit, unbagged fruit, unbagged	pu < 0.02 wf < 0.02 pu < 0.02 wf < 0.02 pu < 0.02 wf < 0.02 pu < 0.02 wf 0.03 pu < 0.02 wf 0.07 pu < 0.02 wf 0.03	2091/93

BANANA country, year (variety)	Application					PHI days	Commodity	Residues, mg/kg difenoconazole [a b]	Ref
	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.				
Honduras, 1997 (Cavendish Robusta)	EC	0.10-0.11	0.025	20-23	8	0 [c] 0 0	fruit, unbagged fruit, unbagged fruit, unbagged	pu < 0.02 (6) pe < 0.02 (3) 0.02 0.03 0.12 wf < 0.02 (4) 0.02 0.06 (mean 0.03)	120/96 2121/96
Honduras, 1997 (Cavendish Robusta)	EC	0.10-0.12	0.025	20-26	8 aerial	0 [c] 0 0	fruit, unbagged fruit, unbagged fruit, unbagged	pu < 0.02 (6) pe < 0.02 (6) wf < 0.02 (6)	120/96 2122/96
Honduras, 1997 (Cavendish Robusta)	EC	0.09-0.14		10-15	8 aerial	0 [c] 0 0	fruit, unbagged fruit, unbagged fruit, unbagged	pu < 0.02 (6) pe < 0.02 (6) wf < 0.02 (6)	120/96 2123/96

a - pu: pulp. pe: peel. wf: whole fruit, residue level calculated from residues in peel and pulp and relative weights.

b - Residues measured on pulp and peel and calculated on the whole fruit.

c - Six replicate samples were taken. Each sample consisted of 6 bananas (2 from the top, middle and bottom of a bunch). Peel and pulp were analysed separately and the residue concentration in the whole fruit was calculated.

Table 36. Difenoconazole residues in mango resulting from supervised trials in Brazil

MANGO country, year (variety)	Application					PHI days	Commodity [a]	Residues, mg/kg difenoconazole	Ref
	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.				
Brazil, 2003 (Tommy Atkins)	EC	0.13	0.013		3	7	mango	0.025	M02017 LZF1
Brazil, 2003 (Tommy Atkins)	EC	0.13	0.013		3	0 3 5 7 9	mango	0.065 0.04 0.04 0.015 0.025	M02017 LZF2
Brazil, 2003 (Tommy Atkins)	EC	0.13	0.013		3	7	mango	0.04	M02017 LZF3
Brazil, 2003 (Tommy Atkins)	EC	0.13	0.013		3	0 3 5 7 9	mango	0.075 0.06 0.03 0.035 0.02	M02017 LZF4

a - Analysis on fruits without seed, but concentration calculated on whole fruit.

Table 37. Difenoconazole residues in papaya resulting from supervised trials in Brazil

PAPAYA country, year (variety)	Application					PHI days	Commodity [a]	Residues, mg/kg difenoconazole	Ref
	Form	kg ai/ha	kg ai/hL	water L/ha	no.				
Brazil, 2002 (Golden)	EC	0.063	0.0075	840	4	0 3 7 10 14	whole fruit pe: 0.35 pe: 0.20 pe: 0.16 pe: 0.19	0.17 pu: < 0.01 wf: 0.11 pu: < 0.01 wf: 0.06 pu: < 0.01 wf: 0.05 pu: < 0.01 wf: 0.07	02-2076
Brazil, 2002 (Golden)	EC	0.13	0.015	890	4	0 3 7 10 14	whole fruit pe: 0.88 pe: 0.51 pe: 0.43 pe: 0.29	0.34 pu: 0.01 wf: 0.25 pu: 0.01 wf: 0.16 pu: < 0.01 wf: 0.13 pu: < 0.01 wf: 0.09	02-2076
Brazil, 2002 (Golden)	EC	0.065	0.0076	860	4	0 3 7 10 14	whole fruit pe: 0.33 pe: 0.39 pe: 0.42 pe: 0.30	0.27 pu: 0.01 wf: 0.11 pu: < 0.01 wf: 0.11 pu: < 0.01 wf: 0.12 pu: < 0.01 wf: 0.10	02-2077

PAPAYA country, year (variety)	Application Form	 kg ai/ha	 kg ai/hL	 water L/ha	 no.	PHI days	Commodity [a]	Residues, mg/kg difenoconazole	Ref
Brazil, 2002 (Golden)	EC	0.13	0.015	850	4	0 3 7 10 14	whole fruit pe: 0.86 pe: 0.95 pe: 1.01 pe: 0.73	0.44 pu: 0.01 wf: 0.24 pu: 0.03 wf: 0.25 pu: 0.02 wf: 0.30 pu: 0.02 wf: 0.20	02-2077
Brazil, 2002 (Taiwan)	EC	0.062	0.0075	830	4	0 3 7 10 14	whole fruit pe: 0.34 pe: 0.19 pe: 0.16 pe: 0.11	0.18 pu: < 0.01 wf: 0.11 pu: < 0.01 wf: 0.05 pu: < 0.01 wf: 0.04 pu: < 0.01 wf: 0.03	02-2078
Brazil, 2002 (Taiwan)	EC	0.13	0.015	840	4	0 3 7 10 14	whole fruit pe: 0.60 pe: 0.48 pe: 0.27 pe: 0.43	0.34 pu: < 0.01 wf: 0.18 pu: 0.01 wf: 0.12 pu: < 0.01 wf: 0.07 pu: < 0.01 wf: 0.12	02-2078
Brazil, 2002 (Golden)	EC	0.062	0.0075	830	4	0 3 7 10 14	whole fruit pe: 0.27 pe: 0.17 pe: 0.15 pe: 0.07	0.16 pu: 0.02 wf: 0.09 pu: 0.01 wf: 0.06 pu: < 0.01 wf: 0.04 pu: < 0.01 wf: 0.02	02-2079
Brazil, 2002 (Golden)	EC	0.13	0.015	840	4	0 3 7 10 14	whole fruit pe: 0.77 pe: 0.33 pe: 0.15 pe: 0.34	0.25 pu: 0.02 wf: 0.23 pu: < 0.01 wf: 0.09 pu: < 0.01 wf: 0.05 pu: < 0.01 wf: 0.09	02-2079

a - pu: pulp. pe: peel. wf: whole fruit, residue level calculated from residues in peel and pulp and relative weights.

Table 38. Difenoconazole residues in garlic resulting from supervised trials in Brazil

GARLIC country, year (variety)	Application Form	 kg ai/ha	 kg ai/hL	 water (L/ha)	 no.	PHI days	Commodity	Residues, mg/kg difenoconazole	Ref
Brazil, 1995 (Caçador)	EC	0.19	0.038	500	6	0 8 15 21	bulb	< 0.02 < 0.02 < 0.02 < 0.02	FR001/95
Brazil, 1995 (Caçador)	EC	0.38	0.076	500	6	0 8 15 21	bulb	< 0.02 < 0.02 < 0.02 < 0.02	FR002/95
Brazil, 1995 (Chonam)	EC	0.19	0.038	500	6	0 7 14 22	bulb	< 0.02 < 0.02 < 0.02 < 0.02	FR003/95
Brazil, 1995 (Chonam)	EC	0.38	0.076	500	6	0 7 14 22	bulb	< 0.02 < 0.02 < 0.02 < 0.02	FR004/95

Table 39. Difenoconazole residues in leeks resulting from supervised trials in France, Germany, Italy and Switzerland

LEEK country, year (variety)	Application Form	 kg ai/ha	 kg ai/hL	 water (L/ha)	 no.	PHI days	Commodity	Residues, mg/kg difenoconazole	Ref
France, 2004 (Durina)	EC	0.13		800	3	21	whole plant	0.03	04-0404 AF/7894/SY/1

LEEK country, year (variety)	Application					PHI days	Commodity	Residues, mg/kg difenoconazole	Ref
	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.				
France, 2004 (Nunens)	EC	0.13		800	2 3	12 0 3 7 14 21	whole plant	0.16 1.2 1.2 0.33 0.09 0.05	04-0404 AF/7894/SY/2
France, 2004 (Porilux)	EC	0.12		790	2 3	12 0 3 7 14 21	whole plant	0.13 0.89 0.53 0.24 0.18 0.13	04-0602 AF/7893/SY1
France, 2004 (Porilux)	EC	0.13		820	2 3	12 0 3 7 14 21	whole plant	0.24 1.6 1.1 0.51 0.50 0.21	04-0602 AF/7893/SY2
Germany, 1998 (Amundo)	EC	0.10		600	3	0 21	whole plant [a]	0.37 0.02	gr 57898
Germany, 1998 (Amundo)	EC	0.10		600	3	0 21	whole plant [a]	1.4 0.12	gr 58898
Germany, 1998 (Preliner)	EC	0.10		600	3	0 21	whole plant [a]	0.96 0.09	gr 59998
Germany, 1998 (Rami)	EC	0.10		600	3	0 21	whole plant [a]	1.2 0.07	RU-NO-08 98 MZ 2221/98
Italy, 1990 (Puerro)	EC	0.13	0.021	600	3	0 7 14 21 28	whole plant	2.1 0.67 0.43 0.17 0.09	2060/90
Italy, 2004 (Armour)	EC	0.13		810	3	21	whole plant	0.14	04-0404 AF/7894/SY/3
Switzerland, 1992 (Armor)	EC	0.13	0.025	500	4	14 21	stems [b]	0.06 0.04	2011/92
Switzerland, 1992 (Dubouchet-Selma)	EC	0.13	0.025	500	4	14 21	stems [b]	0.04 0.02	2010/92

a - Leek samples for analysis: whole plants with roots removed.

b - Only edible parts.

Table 40. Difenoconazole residues in broccoli resulting from supervised trials in France, Netherlands and Spain

BROCCOLI country, year (variety)	Application					PHI days	Commodity	Residues, mg/kg difenoconazole	Ref
	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.				
France, 2002 (Chevalier)	EC	0.13		400	2 3	6 0 15	flower heads	0.09 0.76 0.10	02-2027
France, 2002 (Marathon)	EC	0.13		520	2 3	7 0 13	flower heads	0.07 1.1 0.02	02-2026
France, 2004 (Belstar)	EC	0.13		410	2 3	7 0 3 7 14 21	whole plant whole plant whole plant whole plant flower heads flower heads	0.23 2.0 0.39 0.34 0.08 0.03	02-2043 AF/7866/SY/1

BROCCOLI	Application					PHI	Commodity	Residues, mg/kg	Ref
country, year (variety)	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	days		difenoconazole	
France, 2004 (Marathon)	EC	0.13		410	2 3	7 0 3 7 14 21	whole plant whole plant whole plant whole plant flower heads flower heads	0.59 2.5 2.0 0.94 0.05 0.01	02-2043 AF/7866/SY/2
Netherlands, 2002 (Lord)	EC	0.12		490	2 3	7 0 14	flower heads	0.06 0.81 0.03	02-2042
Netherlands, 2002 (Lord)	EC	0.13 +0.12 +0.15		530 +490 +600	2 3	7 0 14	flower heads	0.07 0.47 < 0.02	02-2043
Spain, 2004 (Maraton)	EC	0.13		400	2 3	6 0 3 7 14 21	whole plant whole plant whole plant whole plant flower heads flower heads	3.5 4.4 2.1 2.2 0.41 0.33	04-0426 AF/7867/SY/2
Spain, 2005 (Monaco)	EC	0.13		400	2 3	7 0 3 7 14 21	whole plant whole plant whole plant whole plant flower heads flower heads	0.65 1.5 0.48 0.45 0.12 0.15	04-0426 AF/7867/SY/3

Table 41. Difenoconazole residues in Brussels sprouts resulting from supervised trials in Belgium and UK

BRUSSELS SPROUTS	Application					PHI	Commodity	Residues, mg/kg	Ref
country, year (variety)	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	days		difenoconazole	
Belgium, 1999 (Edmund)	EC	0.13	0.031	420	3	0 7 14 21 28	buttons	0.06 0.07 0.05 0.07 0.05	RE 12038/1999
Belgium, 1999 (Louis)	EC	0.13	0.031	400	3	0 6 13 20 27	buttons	0.04 0.04 0.06 0.05 0.04	RE 12038/1999
Belgium, 1999 (Maximus)	EC	0.12	0.031	380	3	0 7 14 21 28	buttons	0.04 0.03 0.02 < 0.02 0.02	RE 12038/1999
Belgium, 1999 (Philemon)	EC	0.13	0.031	410	3	0 7 14 21 28	buttons	0.03 0.03 0.04 0.07 0.09	RE 12038/1999
UK, 1990 (Cavalier)	EC	0.13	0.031	400	3	21	buttons	0.05	CGA 0391
UK, 1990 (Cavalier)	EC	0.25	0.062	400	3	21	buttons	0.09	CGA 0391
UK, 1990 (Gavin)	EC	0.13	0.031	400	3	21	buttons	0.06	CGA 0391
UK, 1990 (Gavin)	EC	0.25	0.062	400	3	21	buttons	0.13	CGA 0391
UK, 1990 (Rodger)	EC	0.25	0.062	400	3	21	buttons	0.11	CGA 0391
UK, 1990 (Rodger)	EC	0.13	0.031	400	3	21	buttons	0.07	CGA 0391
UK, 1990 (Tardis)	EC	0.25	0.062	400	3	22	buttons	0.24	CGA 0391
UK, 1990 (Tardis)	EC	0.13	0.031	400	3	22	buttons	0.14	CGA 0391

BRUSSELS SPROUTS	Application					PHI	Commodity	Residues, mg/kg	Ref
country, year (variety)	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	days		difenoconazole	
UK, 1995	EC	0.13	0.05	250	3 4	14 21	buttons	0.065 0.05	12845
UK, 1995 (Amerose RS)	EC	0.13	0.05	250	3 4	14 21	buttons	0.03 0.04	12847
UK, 1995 (Corrinth BS)	EC	0.13	0.05	250	3 4	14 21	buttons	0.045 0.08	12846
UK, 1995 (Stephen)	EC	0.13	0.05	250	3 4	14 21	buttons	0.15 0.10	12844

Table 42. Difenoconazole residues in cabbage resulting from supervised trials in Belgium, France, Germany, Netherlands and UK

CABBAGE	Application					PHI	Commodity	Residues, mg/kg	Ref
country, year (variety)	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	days		difenoconazole	
Belgium, 1999 (Castello) White cabbage	EC	0.13	0.031	400	3	0 7 14 21 28	head	0.04 < 0.02 < 0.02 < 0.02 < 0.02	12039/3
Belgium, 1999 (Indurp) Red cabbage	EC	0.13	0.031	410	3	0 7 14 21 28	head	0.04 < 0.02 < 0.02 < 0.02 < 0.02	12039/4
Belgium, 1999 (Melissa) Savoy cabbage	EC	0.13	0.031	400	3	20	head	< 0.02	12039/5
Belgium, 1999 (Tasmania) Savoy cabbage	EC	0.13	0.031	410	3	0 7 14 21 27	head	0.89 < 0.02 < 0.02 < 0.02 < 0.02	12039/1
Belgium, 1999 (Tasmania) Savoy cabbage	EC	0.12	0.031	390	3	21	head	< 0.02	12039/2
France, 2000 (Chou pointu de Chateaurenard)	EC	0.12		600	3	21	whole product	< 0.05	RLPM0690
France, 2000 (Wintessa)	EC	0.12		460	3	21	whole product	< 0.05	RLPM0690
France, 2002 (Clarisa)	EC	0.12		470	2 3	7 0 14 21	head	0.03 0.27 < 0.01 < 0.01	02-2046
France, 2002 (Gloster)	EC	0.12		500	2 3	7 0 14 21	head	0.24 1.2 0.05 < 0.02	02-2095
France, 2003 (Castello)	EC	0.13		600	3	0 7 14 21 28	head	0.39 0.02 0.01 < 0.01 < 0.01	03-0421
France, 2003 (Rigoleto)	EC	0.12		600	3	0 7 14 21 28	head	2.2 0.96 0.02 0.01 0.01	03-0422

CABBAGE country, year (variety)	Application					PHI days	Commodity	Residues, mg/kg difenoconazole	Ref
	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.				
Germany, 1998 (Julius) Savoy cabbage	EC	0.1		600	3	0 7 14 21 28	head	0.40 0.07 0.02 < 0.02 < 0.02	RU-L-19 98 MZ. 2222/98
Germany, 1998 (Julius) Savoy cabbage	EC	0.1		600	3	0 7 14 21 28	head	0.60 0.04 0.02 < 0.02 < 0.02	RU-L-20 98 MZ. 2223/98
Germany, 2003 (Castello) White head cabbage	EC	0.14		630	3	0 7 14 21 28	head	0.70 0.37 0.23 0.19 0.09	ghc228103
Germany, 2003 (Castello) White head cabbage	EC	0.13		590	3	0 7 14 21 28	head	0.01 < 0.01 < 0.01 < 0.01 < 0.01	ghc228203
Netherlands, 2002 (Duchy)	EC	0.13		510	3	0 14 21	head	0.16 < 0.02 < 0.02	02-2044
Netherlands, 2002 (Duchy)	EC	0.13		530	3	0 14 21	head	0.15 < 0.02 < 0.02	02-2045
UK, 1990 (Tarvoy)	EC	0.25	0.062	400	3	21	heart	0.22	CGA 0391
UK, 1990 (Tarvoy)	EC	0.13	0.031	400	3	21	heart	0.10	CGA 0391
UK, 1990 (Wivoy)	EC	0.13	0.031	400	3	21	heart	0.13	CGA 0391
UK, 1990 (Wivoy)	EC	0.25	0.062	400	3	21	heart	0.39	CGA 0391
UK, 1990 (Zorro)	EC	0.13	0.031	400	3	21	heart	0.06	CGA 0391
UK, 1990 (Zorro)	EC	0.25	0.062	400	3	21	heart	0.14	CGA 0391

Table 43. Difenoconazole residues in cauliflower resulting from supervised trials in France, Switzerland and UK

CAULIFLOWER country, year (variety)	Application					PHI days	Commodity	Residues, mg/kg difenoconazole	Ref
	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.				
France, 1999 (Aviso)	EC	0.13	0.025	500	3	13	flower head	0.03	2070/99
France, 1999 (Escale)	EC	0.13	0.025	500	2	0 3 7 14 21	flower head	< 0.02 < 0.02 < 0.02 < 0.02 ≤ 0.02	2064/99 plot 1
France, 1999 (Escale)	EC	0.13	0.025	500	3	0 3 7 14 21	flower head	< 0.02 < 0.02 < 0.02 < 0.02 ≤ 0.02	2064/99 plot 2
France, 1999 (Fremont)	EC	0.13	0.025	500	3	15	flower head	< 0.02	2069/99
France, 1999 (Nautilus)	EC	0.13	0.025	500	2	14	flower head	< 0.02	2065/99 plot 1
France, 1999 (Nautilus)	EC	0.13	0.025	500	3	14	flower head	< 0.02	2065/99 plot 2
France, 1999 (Notilus)	EC	0.13	0.025	500	3	0 3 7 14	flower head	0.08 0.14 0.06 0.10	2068/99

CAULIFLOWER country, year (variety)	Application Form	 kg ai/ha	 kg ai/hL	 water (L/ha)	 no.	PHI days	Commodity	Residues, mg/kg difenoconazole	Ref
France, 1999 (Sergent)	EC	0.13	0.025	500	2	14	flower head	< 0.02	2066/99 plot 1
France, 1999 (Sergent)	EC	0.13	0.025	500	3	14	flower head	< 0.02	2066/99 plot 2
France, 1999 (Sirente)	EC	0.13	0.025	500	4	0 3 7 14 21	flower head	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02	2067/99
France, 2000 (Vinson)	EC	0.12	0.031	390	3	0 7 14 21	flower head	0.20 < 0.02 < 0.02 < 0.02	2113/00
France, 2005 (Amsterdam)	EC	0.13		400	2 3	14 0 3 7 10 14	whole plant whole plant whole plant whole plant whole plant flower head	0.44 2.4 0.75 0.49 0.36 0.01	05-0514 AF/8564/SY/2
Switzerland, 2005 (Fremont F1)	EC	0.14		650	3	14	flower head	< 0.01	05-0530
UK, 1999 (Plana)	EC	0.13	0.032	400	3	14	flower head	< 0.02	2006/99
UK, 2005 (Avalon)	EC	0.13		500	2 3	14 0 3 7 10 14	whole plant whole plant whole plant whole plant whole plant flower head	0.03 1.4 0.22 0.24 0.05 0.02	05-0514 AF/8564/SY/1

Table 44. Difenoconazole residues in watermelons resulting from supervised trials in Brazil.

WATERMELONS country, year (variety)	Application Form	 kg ai/ha	 kg ai/hL	 water (L/ha)	 no.	PHI days	Commodity	Residues, mg/kg difenoconazole	Ref
Brazil (SP), 1995 (Hibrido Imperor)	EC	0.040	0.010	400	4	0 3 7 14 29	fruits, edible portions	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	Fr 037/95
Brazil (SP), 1995 (Hibrido Imperor)	EC	0.080	0.020	400	4	0 3 7 14 29	fruits, edible portions	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	Fr 038/95
Brazil (SP), 1995 (Taiti)	EC	0.060	0.010	600	4	0 3 7 14 21	fruits, edible portions	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	Fr 039/95 [a]
Brazil (SP), 1995 (Taiti)	EC	0.12	0.020	600	4	0 3 7 14 21	fruits, edible portions	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	Fr 040/95 [a]

a - Trials Fr 039/95 and Fr 040/95: analytical recoveries quite variable at 28 – 194%.

Table 45. Difenoconazole residues in chilli peppers resulting from supervised trials in Indonesia and Malaysia

CHILLI PEPPERS	Application					PHI	Commodity	Residues, mg/kg	Ref
country, year (variety)	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	days		difenoconazole [a]	
Indonesia, 1990 (Cipanas)	EC	3×0.038 +2×0.050 +2×0.075	3×0.013 +2×0.013 +2×0.013	3×300 +2×400 +2×600	7	6	fruits	1.2 u: 0.03	2005/89
Indonesia, 1990 (Cipanas)	EC	3×0.075 +2×0.10 +2×0.15	3×0.025 +2×0.025 +2×0.025	3×300 +2×400 +2×600	7	6	fruits	1.8 u: 0.03	2006/89
Malaysia, 1991 (MC 4)	EC	0.11	0.013	850	5	0 3 7 14	fruits	0.85 0.77 0.67 0.66	2164/91
Malaysia, 1991 (MC 4)	EC	0.23	0.025	900	5	0 3 7 14	fruits	1.8 1.7 1.9 1.2	2165/91

a - u: sample from control (untreated) plot.

Table 46. Difenoconazole residues in tomatoes resulting from supervised trials in France, Greece, Spain and UK

TOMATOES	Application					PHI	Commodity	Residues, mg/kg	Ref
country, year (variety)	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	days		Difenoconazole [a]	
France, 2005 (Belle) glasshouse	EC	0.13		1010	2 3	8 0 1 3 7	fruits	0.03 0.04 0.08 0.03 0.04	05-0414 AF/8577/SY/2
France, 2005 (Brenda) polytunnel	EC	0.12		980	2 3	10 0 1 3 7	fruits	0.05 0.04 0.06 0.10 0.05	05-0413 AF/8577/SY/1
Greece, 2001 (Noa)	EC	0.12	0.010	1240	3 4	7 0 1 3 7 14	fruits	0.20 0.51 0.24 0.16 0.36 0.23	2021/01
Greece, 2002 (Boss, hybrid)	EC	0.12	0.025	490	4	0 3 7 14	fruits	0.51 0.17 0.18 0.16	02-2087
Greece, 2002 (Senna, hybrid)	EC	0.12	0.025	490	4	0 3 7 14	fruits	0.73 u: < 0.01 0.36 0.28 u: 0.01 0.16 u: < 0.01	02-2086
Greece, 2003 (CV Indo)	EC	0.12		690	3	7	fruits	0.10	03-0613
Greece, 2003 (CV Rio Grande)	EC	0.12		780	3	0 1 3 7 10	fruits	0.22 0.15 0.15 0.11 0.13	03-0614

TOMATOES country, year (variety)	Application Form	 kg ai/ha	 kg ai/hL	 water (L/ha)	 no.	PHI days	Commodity	Residues, mg/kg Difenoconazole [a]	Ref
Spain, 2003 (Jaguar)	EC	0.13		1240	3	0 1 3 7 10	fruits	0.19 0.12 0.13 0.09 0.07	03-0520
Spain, 2003 (Jaguar)	EC	0.13		1210	3	7	fruits	0.03	03-0521
Spain, 2005 (Dici) polytunnel	EC	0.12		970	2 3	10 0 1 3 7	fruits	0.07 0.16 0.16 0.12 0.12	05-0413 AF/8577/SY/2
UK, 2005 (Espiro) glasshouse	EC	0.13		1040	2 3	9 0 1 3 7	fruits	0.11 0.11 0.13 0.09 0.10	05-0414 AF/8577/SY/1

a - u: sample from control (untreated) plot.

Table 47. Difenoconazole residues in lettuce resulting from supervised trials in Spain

LETTUCE country, year (variety)	Application Form	 kg ai/ha	 kg ai/hL	 water (L/ha)	 no.	PHI days	Commodity	Residues, mg/kg difenoconazole [a]	Ref
Spain, 1991 (Francesa)	EC	0.18	0.035	500	3	0 7 14 21	lettuce	8.6 1.6 0.56 0.18	2021/91
Spain, 1991 (Francesa)	EC	0.18	0.035	500	3	0 7 14 21	lettuce	5.5 1.5 1.0 0.04	2022/91
Spain, 1991 (Francesa)	EC	0.18	0.035	500	3	0 7 14 21	lettuce	7.0 1.2 0.65 0.41	2023/91
Spain, 1991 (Inverna)	EC	0.18	0.022	800	3	0 7 13 20	lettuce	2.5 1.0 0.31 0.11 [b]	2024/91
Spain, 1991 (Inverna)	EC	0.18	0.022	800	3	0 7 13 20	lettuce	2.0 u: 0.02 0.74 0.51 0.44	2027/91
Spain, 2003 (Little Gem) cos lettuce	EC	0.17		700	3	0 3 7 14	lettuce	2.7 0.41 0.55 0.07	03-0423
Spain, 2003 (Inverna) cos lettuce	EC	0.17		580-800	3	0 3 7 14 21	lettuce	3.9 2.0 1.1 0.29 0.13	03-0424
Spain, 2003 (Baby) cos lettuce	EC	0.18		610-860	3	0 3 7 14 21	lettuce	1.9 1.1 0.50 0.08 0.04	03-0425

a - u: sample from control (untreated) plot.

b - The analytical results (< 0.02 and 0.10 mg/kg for the replicate samples) and 0.11 mg/kg for the sample from the untreated plot, suggested an interchange of samples.

Table 48. Difenoconazole residues in soya beans resulting from supervised trials in Brazil

SOYA BEAN	Application					PHI	Commodity	Residues, mg/kg	Ref
country, year (variety)	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	days		difenoconazole	
Brazil (SP), 2003 (Foscarim)	SC includes azoxystrobin	0.075		200	2	30	dry bean	< 0.01	MO2065
Brazil (MG), 2003 (Esplendor)	SC includes azoxystrobin	0.075		200	2	30	dry bean	< 0.01	MO2065
Brazil (GO), 2003 (Monarca)	SC includes azoxystrobin	0.075		200	2	30	dry bean	< 0.01	MO2065
Brazil, 2000 (Monsoy)	EC	0.075		300	2	10 14 20 25 30	dry bean	0.20 0.04 < 0.02 < 0.02 < 0.02	FR018/2000-RK
Brazil, 2000 (RS-10)	EC	0.075		300	2	11 15 20 25 31	dry bean	0.07 0.04 < 0.02 < 0.02 < 0.02	FR018/2000-MF
Brazil, 2000 (1 AC 22)	EC	0.075		300	2	10 15 20 25 30	dry bean	0.42 0.29 < 0.02 < 0.02 < 0.02	FR018/2000-LZF

Table 49. Difenoconazole residues in carrots resulting from supervised trials in France and Switzerland

CARROT	Application					PHI	Commodity	Residues, mg/kg	Ref
country, year (variety)	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	days		Difenoconazole [a]	
France, 1991 (Anglia)	EC	0.13		400	3	13	root	0.05	OF91059
France, 1991 (Luxor)	EC	0.13		400	3	13	root	0.03	OF91089
France, 1992 (Nandrin)	EC	0.13		1000	3	14 28	root	0.07 0.02	OF92025
France, 1992 (Nantaise)	EC	0.13		1000	3	14 28	root	0.02 0.02	OF92025
France, 1993 (Nantaise)	EC	0.13		1000	3	14 33	root	0.02 u: 0.03 0.02	OF93153
France, 1993 (Valor)	EC	0.13		1000	3	14 29	root	0.11 u 0.04 0.04 u 0.06	OF93153
France, 1996 (Tourino)	EC	0.13	0.031	400	3	14	root	< 0.02 [b]	OF96134
France, 2000 (Carotan)	EC	0.12		390	3	0 3 7 10 14	root	0.02 0.03 0.01 0.03 0.04	0012001
France, 2000 (Colmar Coeur Rouge)	EC	0.13	0.031	400	3	14	root	0.13	0011902
France, 2000 (Nanda)	EC	0.13		410	3	14	root	0.11	0011901
France, 2000 (Presto)	EC	0.12	0.031	400	3	14	root	0.03	0011903
France, 2000 (Presto)	EC	0.13	0.031	400	3	0 3 7 10 15	root	0.01 0.01 0.03 0.02 0.02	0012002
Switzerland, 1987 (Nantaise Express)	EC	0.13		600	3 4	13 0 7 14 21	root	0.06 0.07 0.13 0.07 0.07	2005/87

CARROT	Application					PHI	Commodity	Residues, mg/kg	Ref
country, year (variety)	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	days		Difenoconazole [a]	
Switzerland, 1987 (Tip-Top)	EC	0.13		600	3 4	14 0 7 14 21	root	0.14 < 0.02 [c] u: 0.19 0.07 0.12 0.07	2006/87

a - u: sample from control (untreated) plot.

b - Sample stored for 34 months before analysis.

c - Noted in the study as an anomalous result, perhaps test and control samples switched.

Table 50. Difenoconazole residues in potatoes resulting from supervised trials in Italy and Spain

POTATO	Application					PHI	Commodity	Residues, mg/kg	Ref
country, year (variety)	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	days		Difenoconazole	
Italy, 2005 (Elvira)	EC	0.13 +0.20		1020 +1020	2	29	tuber	< 0.01	05-0505
Spain, 2003 (Carlita)	EC	0.22		660	2	28	tuber	< 0.01	03-0431
Spain, 2003 (Espunta)	EC	0.21		410	2	0 8 14 21 30 35	tuber	< 0.01 0.01 [a] < 0.01 < 0.01 0.01 [a] < 0.01	03-0430
Spain, 2003 (Fabula)	EC	0.21		630	2	0 7 13 19 27 34	tuber	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	03-0429
Spain, 2003 (Kennebec)	EC	0.22		500	2	30	tuber	< 0.01	03-0432
Spain, 2005 (Agata)	EC	0.20		410	2	0 7 14 22 31	tuber	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	05-0419 ES-FR-05-0413
Spain, 2005 (Hermes)	EC	0.21		410	2	29	tuber	< 0.01	05-0419 ES-FR-05-0414
Spain, 2005 (Kennebec)	EC	0.19		390	2	0 6 14 21 30	tuber	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	05-0419 ES-FR-05-0412

a - Confirmed by triplicate analysis (0.01, 0.01, 0.01 mg/kg).

Table 51. Difenoconazole residues in sugar beet resulting from supervised trials in Denmark, France, Germany and UK

SUGAR BEET	Application					PHI	Commodity	Residues, mg/kg	Ref
country, year (variety)	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	days		Difenoconazole	
Denmark, 1989 (Maribo Armada)	EC	0.13		400	2	24 37 59	root root root	0.08 0.08 0.06	2059/89
France, 1985 (Alto)	EC	0.13		500	2	24	root	≤ 0.02	48/87
France, 1991 (Allyx)	EC includes propiconazole	0.1		500	2	33	tuber	< 0.02	0210F91 [a]

SUGAR BEET country, year (variety)	Application					PHI days	Commodity	Residues, mg/kg Difenoconazole	Ref
	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.				
France, 1991 (Monos)	EC includes propiconazole	0.1		500	2	29	tuber	< 0.02	0200F91 [a]
Germany, 1987 (Britta)	EC	0.1		400	2 3	30 0 14 28 36 43	root	0.02 0.03 0.08 0.02 < 0.02 0.03	2150/87
Germany, 1987 (Diadem)	EC	0.1		400	2 3	30 0 14 28 36 43	root	< 0.02 < 0.02 0.04 0.02 < 0.02 0.02	2149/87
Germany, 1987 (Eva)	EC	0.1		400	2 3	20 0 14 28 35 42 50	root	0.05 0.22 0.07 0.04 0.08 0.06 0.06	2148/87
Germany, 1988 (Hilma)	EC	0.1		400	1 2	32 0 7 14 22 28 36 42	root	< 0.02 < 0.02 < 0.02 0.02 < 0.02 < 0.02 < 0.02 0.02	2053/88
Germany, 1988 (Kaweduca)	EC	0.1		400	1 2	32 0 7 14 22 28 36 42	root	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02	2052/88
Germany, 1988 (Primahill)	EC	0.1		400	1 2	20 0 7 14 21 28 35 42	root	0.03 0.04 0.03 0.04 0.03 0.08 0.02 0.02	2050/88
Germany, 1995 (Ribella)	EC, 100 g/L	0.1		400	2	30	tuber	0.02	gr 4995 gr 41595
Germany, 1995 (Ribella)	EC, 250 g/L	0.1		400	2	30	tuber	0.06	gr 4995 gr 41595
Germany, 1995 (Sonja)	EC, 100 g/L	0.1		400	2	28	tuber	0.10	gr 4995 gr 31595
Germany, 1995 (Sonja)	EC, 250 g/L	0.1		400	2	28	tuber	< 0.02	gr 4995 gr 31595
Germany, 1996 (Reka)	EC includes fenpropidin	0.098		300	2	23 27 39	root root root	< 0.02 < 0.02 < 0.02	96 10 61 009
Germany, 1996 (Ribella)	EC includes fenpropidin	0.1		400	2	21 28 35	root root root	0.06 < 0.02 0.02	gr 49496
Germany, 1996 (Ribella)	EC includes fenpropidin	0.10		400	2	21 28	roots roots	0.054 0.033	gr 50596

SUGAR BEET	Application					PHI	Commodity	Residues, mg/kg	Ref
country, year (variety)	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	days		Difenoconazole	
Germany, 1996 (Hilma)	EC includes fenpropidin	0.098		300	2	25 28 35	root root root	0.03 ≤ 0.02 < 0.02	96 10 62 010
UK, 2004 (Veronica)	EC includes fenpropidin	0.1		300	2	27	root	0.01	04-6047

a - Inadequate supporting field data and analytical method

Table 52. Difenoconazole residues in asparagus resulting from supervised trials in France, Italy and Switzerland

ASPARAGUS	Application					PHI	Commodity	Residues, mg/kg	Ref
country, year (variety)	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	days		Difenoconazole	
France, 1989 (Aneto)	EC	0.13	0.025	500	8	222	shoots	0.02	78/90
France, 1989 (Aneto)	EC	0.13	0.025	500	8	234	shoots	< 0.02	77/90
France, 1990 (Desto)	EC	0.13		300	7	203	shoots	< 0.02	0090F/91
France, 1990 (Larac)	EC	0.13	0.025	500	9	179	shoots	< 0.02	0070F/91
Italy, 1990 (Larac)	EC	0.13	0.013	1000	6	256	shoots	< 0.02	2056/90
Italy, 1991 (Larac)	EC	0.13	0.013	1000	6	239	shoots	< 0.02	2088/91
Switzerland, 1988	EC	0.13	0.021	600	4	248 260	shoots	< 0.02 < 0.02	2114/88
Switzerland, 1988 (Novalis Vallieres)	EC	0.13	0.021	600	4	290 298	shoots	< 0.02 < 0.02	2113/88

Table 53. Difenoconazole residues in celeriac resulting from supervised trials in Belgium

CELERIAC	Application					PHI	Commodity	Residues, mg/kg	Ref
country, year (variety)	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	days		Difenoconazole [a]	
Belgium, 2001 (Brilliant)	EC	0.13	0.031	410	4	15	roots	0.12	RE 20245 / 2001
Belgium, 2001 (Diamant)	EC	0.12	0.031	400	4	15	roots	0.08	RE 20245 / 2001
Belgium, 2001 (Monarch)	EC	0.13	0.031	430	4	15	roots	0.22 u: 0.08	RE 20245 / 2001
Belgium, 2001 (Monarch)	EC	0.13	0.031	420	4	15	roots	0.21 u: 0.18	RE 20245 / 2001

a - u: sample from control (untreated) plot.

Table 54. Difenoconazole residues in celery resulting from supervised trials in France, Italy, Spain and Switzerland

CELERY	Application					PHI	Commodity, Residues, mg/kg		Ref
country, year (variety)	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	days	Difenoconazole [a]		
France, 2003 (Elne)	EC	0.13		630	3	14	stem 0.14	leaf 0.95	RLCE21403
France, 2003 (Lino)	EC	0.14		420	3	14	stem 0.03	leaf 0.39	RLCE21403
France, 2004 (Golden Spartane)	EC	0.13		600	2 3	14 0 3 7 14 21	stem 0.03 stem 0.11 stem 0.12 stem 0.06 stem 0.04 stem 0.03	leaf 0.77 leaf 5.1 leaf 1.6 leaf 1.2 leaf 1.0 leaf 0.82	04-0427 AF/7868/SY/2

CELERY country, year (variety)	Application					PHI days	Commodity, Residues, mg/kg Difenoconazole [a]		Ref
	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.				
France, 2004 (Tall Utah)	EC	0.13		600	2	14	stem 0.08	leaf 0.40	04-0427
					3	0	stem 0.19	leaf 6.6	AF/7868/SY/1
						3	stem 0.54	leaf 3.5	
						7	stem 0.34	leaf 2.0	
						14	stem 0.26	leaf 1.3	
						21	stem 0.15	leaf 1.7	
Italy, 1990 (Elne)	EC	0.10	0.017	600	3	0	edible parts	3.7	2063/90
						7	edible parts	2.0	
						14	edible parts	1.2	
						21	edible parts	1.2	
						28	edible parts	0.61	
Italy, 1990 (Utah)	EC	0.10	0.017	600	3	0	stems	6.4	2064/90
						7	stems	3.6	
						14	stems	2.0	
						21	stems	0.81	
						28	stems	0.74	
Spain, 2004 (Hurta)	EC	0.13		620	3	7	stem 0.07	leaf 3.0	04-0306
						14	stem 0.04	leaf 1.3	
Spain, 2004 (Local Population Variety)	EC	0.13		600	3	7	stem 0.06	leaf 2.9	04-0306
						14	stem 0.05	leaf 2.0	
Switzerland, 1988 (Claret)	EC	0.13		600	4	0	stem 0.12	leaf 3.0	2117/88
						7	stem 0.087	leaf 1.3	
						14	stem 0.058 u: 0.02	leaf 1.1	
Switzerland, 1988 (Claret)	EC	0.13		600	4	0	stem 0.069	leaf 2.6	2118/88
						7	stem 0.16	leaf 1.6	
						14	stem 0.17	leaf 1.6	

a - u: sample from control (untreated) plot.

Table 55. Difenoconazole residues in rice resulting from supervised trials in Indonesia and Malaysia

RICE country, year (variety)	Application					PHI days	Commodity	Residues, mg/kg Difenoconazole [a]	Ref
	Form	kg ai/ha	kg ai/hL	water (L/ha)	no., growth stage final				
Indonesia, 1998 (Pelita)	EC includes propiconazole	0.063	0.013	500	2 BBCH 57	0	ears	4.2	2110/98
						7	ears	0.96	
						14	ears	1.3	
						21	ears	1.4	
						28	grain	1.3	
Indonesia, 1998 (Pelita)	EC includes propiconazole	0.063	0.013	500	2 BBCH 57	0	ears	3.5	2111/98
						7	ears	1.7	
						14	ears	0.78	
						21	ears	0.38	
						28	grain	0.75	
Malaysia, 1994 (MR 84)	EC	0.075		350	3 milky stage	30	grain	0.15	2184/94
Malaysia, 1998 (MR 185)	EC includes propiconazole	0.064	0.013	500	2 BBCH 69-75	0	ears	2.9	2113/98
						7	grain	0.30	
						14	grain	0.17	
						21	grain	0.10	
						28	grain	0.16	
Malaysia, 1998 (MR 84)	EC includes propiconazole	0.12	0.025	460	2 BBCH 69-73	0	ears	3.5	2112/98
						14	grain	0.97	
						28	grain	0.76	
Malaysia, 1998 (MR 84)	EC includes propiconazole	0.066	0.013	500	2 BBCH 69-73	0	ears	2.4	2112/98
						7	grain	0.99	
						14	grain	0.52	
						21	grain	0.50	
						28	grain	0.37	

a: u: sample from control (untreated) plot.

Table 56. Difenoconazole residues in wheat resulting from supervised trials in Denmark, France, Switzerland and UK

WHEAT	Application [a]					PHI	Commodity	Residues, mg/kg	Ref
country, year (variety)	Form	kg ai/ha	water (L/ha)	no.	BBCH stage	days		Difenoconazole	
Denmark, 1989 (Sleipner)	EC	0.13	300	1	BBCH 54	75	wheat grain	< 0.02	2060/89
Denmark, 1990 (Kraka)	EC	0.13	250	1	BBCH 60	58	wheat grain	< 0.02	2047/90
Denmark, 1990 (Kraka)	EC	0.13	250	1	BBCH 60	57	wheat grain	< 0.02	2048/90
France, 1988 (Festival)	SC includes CGA 18251	0.13	450	1	BBCH 66-76	51	wheat grain	< 0.02	53/88
France, 1989 (Garant)	SC includes CGA 18251	0.13	500	1	BBCH 61	57	wheat grain	< 0.02	08/90
France, 1989 (Garant)	SC includes CGA 18251	0.13	500	1	BBCH 55-57	63	wheat grain	< 0.02	09/90
France, 1989 (Goeland)	SC includes CGA 18251	0.13	500	1	BBCH 61	52	wheat grain	< 0.02	07/90
France, 1989 (Tango)	SC includes CGA 18251	0.13	500	1	ripen-ing	57	wheat grain	< 0.02	06/90
France, 1993 (Capitaine)	GL includes CGA 18251	0.13	400	1	BBCH 83	31	wheat grain	< 0.02	OF93148
France, 1993 (Recital)	GL includes CGA 18251	0.13	400	1	BBCH 87	39	wheat grain	< 0.02	OF93148
France, 1993 (Soisson)	GL includes CGA 18251	0.13	400	1	BBCH 77	42	wheat grain	< 0.02	OF93148
France, 1998 (Arstar)	GL includes carbendazim	0.13	400	1	BBCH 65	47	wheat grain	< 0.02	9813303
France, 1998 (Excalibur)	GL includes carbendazim	0.13	400	1	BBCH 69	49	wheat grain	< 0.02	9813302
France, 1998 (Primadur)	GL includes carbendazim	0.12	400	1	BBCH 65	45	wheat grain	< 0.02	9813304
France, 1998 (Rubbens)	GL includes carbendazim	0.14	430	1	BBCH 61-65	47	wheat grain	< 0.02	9813301
Switzerland, 1989 (Remia)	EC	0.13	500	1	BBCH 59	45	wheat grain	< 0.02 < 0.02 [b]	2031/89
UK (Cambs), 1989 (Brock)	EC	0.15	200	1	BBCH 64-65	49	wheat grain	< 0.02	R/0157/01
UK (Cambs), 1989 (Mercia)	EC	0.15	200	1	BBCH 61-63	65	wheat grain	< 0.02	R/0157/01
UK (Essex), 1989 (Galahad)	EC	0.15	200	1	BBCH 65	55	wheat grain	< 0.02	R/0157/01
UK (Gt Halingbury), 1989 (Hornet)	EC	0.15	200	1	BBCH 65	57	wheat grain	< 0.02	R/0157/01
UK(Bulbeck), 1989 (Mercia)	EC	0.15	200	1	BBCH 64-65	57	wheat grain	< 0.02	R/0157/01
UK, 1985 (Avalon)	?	0.13	200	1	BBCH 65	59	wheat grain	< 0.02	2229/85
UK, 1985 (Norman)	?	0.13	200	1	BBCH 65	67	wheat grain	< 0.02	2230/85

a - BBCH growth stages for wheat (Stauss, 1994)

55: middle of heading, half of inflorescences emerged. 57: 70% of inflorescences emerged.
59: end of heading, inflorescence fully emerged. 61: beginning of flowering, first anthers visible.
65: full flowering, 50% of anthers mature.
69: end of flowering, all spikelets have completed flowering but some dehydrated anthers may remain.
71: watery ripe, first grains have reached half their final size. 73: early milk.

75: medium milk, grain content milky, grains reached full size, still green. 77: late milk.
83: early dough. 85: soft dough, grain content soft but dry, fingernail impression not held.
87: hard dough, grain content solid, fingernail impression held.

b - Replicate data are from replicate plots.

Table 57. Difenoconazole residues in oilseed rape resulting from supervised trials in France and Germany

RAPE SEED	Application [a]					PHI	Commodity	Residues, mg/kg	Ref
country, year (variety)	Form	kg ai/ha	water (L/ha)	no.	BBCH stage, final ap	days		difenoconazole	
France, 1988 (Jet Neuf)	SC includes CGA 18251	0.13	500	1	F2-G1[13]	83	seed	0.04	48/89
France, 1988 (Jet Neuf)	EC	0.13	500	1	F2-G1	83	seed	0.04	48/89
Germany, 1996 (Evita)	SC includes carbendazim	0.13	400	2	BBCH 65	56	seed	< 0.02	gr 54696
Germany, 1996 (Lirajet)	SC includes carbendazim	0.13	400	2	BBCH 63-65	69	seed	< 0.02	gr 53496
Germany, 1996 (Synergy)	SC includes carbendazim	0.13	400	2	BBCH 63-65	80	seed	< 0.02	gr 51296
Germany, 1996 (Wotan)	SC includes carbendazim	0.13	400	2	BBCH 65	80	seed	< 0.02	gr 52396
Germany, 1997 (Capitol)	SC includes carbendazim	0.13	400	2	BBCH 69-71	56	seed	< 0.02	gr 52297
Germany, 1997 (Express)	SC includes carbendazim	0.13	400	2	BBCH 71-75	55	seed	< 0.02	gr 51197
Germany, 1997 (Express)	SC includes carbendazim	0.13	400	2	BBCH 71	55	seed	< 0.02	gr 53497

a - BBCH growth stages for oilseed rape (Stauss, 1994)
63: 30% of flowers on main raceme open
65: full flowering, 50% of flowers on main raceme open, older petals falling.
67: flowering declining, majority of petals fallen.
69: end of flowering.
71: 10% of pods have reached final size.
73: 30% of pods have reached final size.
75: 50% of pods have reached final size.

Table 58. Difenoconazole residues in sunflower seed resulting from supervised trials in France and Switzerland

SUNFLOWER SEED	Application [a]					PHI	Commodity	Residues, mg/kg	Ref
country, year (variety)	Form	kg ai/ha	water (L/ha)	no.	BBCH stage, final ap	days		difenoconazole	
France, 2004 (Alstars)	SC includes azoxystrobin	0.13	400	2	BBCH 51-55	59	seed	< 0.01	04-0416 FR-FR-04-0125
France, 2004 (DK 3792)	SC includes azoxystrobin	0.13	400	2	BBCH 51-55	101	seed	< 0.01	04-0415
France, 2004 (Galix)	SC includes azoxystrobin	0.13	300	2	BBCH 51-55	66	seed	0.01	04-0416 FR-FR-04-0123
France, 2004 (Kolda)	SC includes azoxystrobin	0.13	310	2	BBCH 59	73	seed	< 0.01	04-0416 FR-FR-04-0124
France, 2004 (LG5655)	SC includes azoxystrobin	0.13	210	2	BBCH 51-55	83	seed	< 0.01	04-0416 FR-FR-04-0126
France, 2005 (Cargisol)	SC includes azoxystrobin	0.13	300	2	BBCH 51-55	90	seed	< 0.01	05-0411 AF/8542/SY/2
France, 2005 (Orasol)	SC includes azoxystrobin	0.13	300	2	BBCH 51-55	74	seed	< 0.01	05-0411 AF/8542/SY/1

[13] Rapeseed 48/89. Growth stage F2-G1 is not clear. Assume from the 83 days PHI that it is similar to BBCH 63-65.

SUNFLOWER SEED country, year (variety)	Application [a] Form	kg ai/ha	water (L/ha)	no.	BBCH stage, final ap	PHI days	Commodity	Residues, mg/kg difenoconazole	Ref
Spain, 2005 (Circe)	SC includes azoxystrobin	0.13	200 +400	2	BBCH 51-55	87	seed	< 0.01	05-0411 AF/8542/SY/3
Spain, 2005 (Latino)	SC includes azoxystrobin	0.13	200 +410	2	BBCH 51-55	74	seed	< 0.01	05-0411 AF/8542/SY/4
Switzerland, 2004 (Prodisol)	SC includes azoxystrobin	0.13	400	2	BBCH 51-55	68	seed	< 0.01	04-0311
Switzerland, 2005 (Aurasol)	SC includes azoxystrobin	0.13	410	2	BBCH 51-55	73	seed	< 0.01	05-0401 CH-FR-05-0313
Switzerland, 2005 (Elansol)	SC includes azoxystrobin	0.12	390	2	BBCH 51-55	73	seed	< 0.01	05-0401 CH-FR-05-0314

a - BBCH growth stages for sunflower (Stauss, 1994)
51: inflorescence just visible between youngest leaves.
53: inflorescence separating from youngest leaves, bracts distinguishable from foliage leaves.
55: inflorescence separated from youngest foliage leaf.
57: inflorescence clearly separated from foliage leaves.
59: ray florets visible between the bracts, inflorescence still closed.

Table 59. Difenoconazole residues in wheat straw and fodder resulting from supervised trials in Denmark, France, Switzerland and UK

WHEAT STRAW AND FODDER country, year (variety)	Application [a] Form	kg ai/ha	water (L/ha)	no.	BBCH stage	PHI days	Commodity	Residues, mg/kg Difenoconazole [b]	Ref
Denmark, 1989 (Sleipner)	EC	0.13	300	1	BBCH 54	27 40 75	whole plant whole plant wheat straw	0.21 0.14 0.31	2060/89
Denmark, 1990 (Kraka)	EC	0.13	250	1	BBCH 60	0- 7 22 37 58	whole plant whole plant whole plant whole plant wheat straw	< 0.02 1.4 0.52 0.35 0.64	2047/90
Denmark, 1990 (Kraka)	EC	0.13	250	1	BBCH 60	0- 7 22 37 57	whole plant whole plant whole plant whole plant wheat straw	< 0.02 0.80 0.28 0.04 0.26 u: 0.03	2048/90
France, 1988 (Festival)	SC includes CGA 18251	0.13	450	1	BBCH 66-76	51	wheat straw	0.12 u: 0.15	53/88
France, 1989 (Garant)	SC includes CGA 18251	0.13	500	1	BBCH 61	57	wheat straw	0.73	08/90
France, 1989 (Garant)	SC includes CGA 18251	0.13	500	1	BBCH 55-57	63	wheat straw	0.82	09/90
France, 1989 (Goeland)	SC includes CGA 18251	0.13	500	1	BBCH 61	52	wheat straw	0.16 u: 0.16	07/90
France, 1989 (Tango)	SC includes CGA 18251	0.13	500	1	ripen-ing	57	wheat straw	0.28	06/90
France, 1993 (Capitaine)	GL includes CGA 18251	0.13	400	1	BBCH 83	31	wheat straw	0.95	OF93148
France, 1993 (Recital)	GL includes CGA 18251	0.13	400	1	BBCH 87	39	wheat straw	2.5	OF93148
France, 1993 (Soisson)	GL includes CGA 18251	0.13	400	1	BBCH 77	42	wheat straw	2.4	OF93148
France, 1998 (Arstar)	GL includes carbendazim	0.13	400	1	BBCH 65	47	wheat straw	2.0	9813303
France, 1998 (Excalibur)	GL includes carbendazim	0.13	400	1	BBCH 69	49	wheat straw	0.36	9813302

WHEAT STRAW AND FODDER	Application [a]					PHI	Commodity	Residues, mg/kg	Ref
country, year (variety)	Form	kg ai/ha	water (L/ha)	no.	BBCH stage	days		Difenoconazole [b]	
France, 1998 (Primadur)	GL includes carbendazim	0.12	400	1	BBCH 65	45	wheat straw	1.3	9813304
France, 1998 (Rubbens)	GL includes carbendazim	0.14	430	1	BBCH 61-65	47	wheat straw	0.46	9813301
Switzerland, 1989 (Remia)	EC	0.13	500	1	BBCH 59	45	wheat straw	1.2 0.9 [c]	2031/89
UK (Cambs), 1989 (Brock)	EC	0.15	200	1	BBCH 64-65	49	wheat straw	0.84 u: 0.02	R/0157/01
UK (Cambs), 1989 (Mercia)	EC	0.15	200	1	BBCH 61-63	65	wheat straw	1.4 u: 0.36	R/0157/01
UK (Essex), 1989 (Galahad)	EC	0.15	200	1	BBCH 65	55	wheat straw	3.7 u: 0.07	R/0157/01
UK (Gt Halingbury), 1989 (Hornet)	EC	0.15	200	1	BBCH 65	57	wheat straw	0.71 u: 0.16	R/0157/01
UK(Bulbeck), 1989 (Mercia)	EC	0.15	200	1	BBCH 64-65	57	wheat straw	0.32 u: 0.40	R/0157/01

a - BBCH growth stages for wheat (Stauss, 1994)
55: middle of heading, half of inflorescences emerged.
57: 70% of inflorescences emerged.
59: end of heading, inflorescence fully emerged.
61: beginning of flowering, first anthers visible.
65: full flowering, 50% of anthers mature.
69: end of flowering, all spikelets have completed flowering but some dehydrated anthers may remain.
71: watery ripe, first grains have reached half their final size.
73: early milk.
75: medium milk, grain content milky, grains reached full size, still green.
77: late milk.
83: early dough.
85: soft dough, grain content soft but dry, fingernail impression not held.
87: hard dough, grain content solid, fingernail impression held.

b - u: sample from control (untreated) plot.

c - Replicate data are from replicate plots.

Table 60. Difenoconazole residues in rice straw and fodder resulting from supervised trials in Indonesia and Malaysia

RICE STRAW AND FODDER	Application					PHI	Commodity	Residues, mg/kg	Ref
country, year (variety)	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	days		Difenoconazole [a]	
Indonesia, 1998 (Pelita)	EC includes propiconazole	0.063	0.013	500	2	0 7 14 21 28	stalks stalks stalks stalks stalks	3.7 1.9 1.1 0.68 0.55	2110/98
Indonesia, 1998 (Pelita)	EC includes propiconazole	0.063	0.013	500	2	0 7 14 21 28	stalks stalks stalks stalks stalks	1.1 1.3 0.45 0.30 1.1	2111/98
Malaysia, 1998 (MR 185)	EC includes propiconazole	0.064	0.013	500	2	0 7 14 21 28	stalks stalks stalks stalks straw	1.8 0.22 0.14 0.10 0.08	2113/98

RICE STRAW AND FODDER	Application					PHI	Commodity	Residues, mg/kg	Ref
country, year (variety)	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	days		Difenoconazole [a]	
Malaysia, 1998 (MR 84)	EC includes propiconazole	0.066	0.013	500	2	0 7 14 21 28	stalks stalks stalks stalks straw	1.3 0.62 0.34 0.40 0.19 u: 0.05	2112/98
Malaysia, 1998 (MR 84)	EC includes propiconazole	0.12	0.025	460	2	0 14 28	stalks stalks straw	2.1 0.75 0.44	2112/98

a - u: sample from control (untreated) plot.

Table 61. Difenoconazole residues in sugar beet leaves and tops resulting from supervised trials in Denmark, France, Germany and UK

SUGAR BEET LEAVES & TOPS	Application					PHI	Commodity	Residues, mg/kg	Ref
country, year (variety)	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	days		Difenoconazole	
Denmark, 1989 (Maribo Armada)	EC	0.13		400	2	24 37 59	leaf leaf leaf	0.74 0.45 0.24	2059/89
France, 1985 (Alto)	EC	0.13		500	2	24	leaves	0.17	48/87
France, 1991 (Allyx)	EC includes propiconazole	0.1		500	2	33	leaf	0.07	0210F91 [a]
France, 1991 (Monos)	EC includes propiconazole	0.1		500	2	29	leaf	0.32	0200F91 [a]
Germany, 1987 (Britta)	EC	0.1		400	2 3	30 0 14 28 36 43	leaf	0.07 2.2 0.29 0.11 0.10 0.05	2150/87
Germany, 1987 (Diadem)	EC	0.1		400	2 3	30 0 14 28 36 43	leaf	0.06 1.9 0.22 0.09 0.08 0.06	2149/87
Germany, 1987 (Eva)	EC	0.1		400	2 3	20 0 14 28 35 42 50	leaf	0.29 4.6 1.2 0.39 0.95 0.37 0.61	2148/87
Germany, 1988 (Hilma)	EC	0.1		400	1 2	32 0 7 14 22 28 36 42	leaf	0.15 4.4 1.2 0.72 0.49 0.53 0.21 0.28	2053/88
Germany, 1988 (Kaweduca)	EC	0.1		400	1 2	32 0 7 14 22 28 36 42	leaf	0.17 4.3 1.0 0.64 0.38 0.43 0.31 0.38	2052/88

SUGAR BEET LEAVES & TOPS	Application					PHI	Commodity	Residues, mg/kg	Ref
country, year (variety)	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	days		Difenoconazole	
Germany, 1988 (Primahill)	EC	0.1		400	1 2	20 0 7 14 21 28 35 42	leaf	0.13 1.8 1.4 1.1 0.55 0.47 0.33 0.39	2050/88
Germany, 1995 (Ribella)	EC, 100 g/L	0.1		400	2	30	tops	0.62	gr 4995 gr 41595
Germany, 1995 (Ribella)	EC, 250 g/L	0.1		400	2	30	tops	0.26	gr 4995 gr 41595
Germany, 1995 (Sonja)	EC, 100 g/L	0.1		400	2	28	tops	0.25	gr 4995 gr 31595
Germany, 1995 (Sonja)	EC, 250 g/L	0.1		400	2	28	tops	0.43	gr 4995 gr 31595
Germany, 1996 (Hilma)	EC includes fenpropidin	0.098		300	2	0 14 25 28 35	whole plant whole plant leaves+tops leaves+tops leaves+tops	0.44 0.11 0.20 0.20 0.12	96 10 62 010
Germany, 1996 (Reka)	EC includes fenpropidin	0.098		300	2	0 15 23 27 39	whole plant whole plant leaves+tops leaves+tops leaves+tops	0.36 0.16 0.074 0.084 0.063	96 10 61 009
Germany, 1996 (Ribella)	EC includes fenpropidin	0.10		400	2	0 14 21 28	whole plant whole plant leaves+tops leaves+tops	0.39 0.44 0.50 0.087	gr 50596
Germany, 1996 (Ribella)	EC includes fenpropidin	0.1		400	2	0 14 21 28 35	whole plant whole plant leaves+tops leaves+tops leaves+tops	0.66 0.24 0.31 0.25 0.25	gr 49496
UK, 2004 (Veronica)	EC includes fenpropidin	0.1		300	2	27	leaves	0.09	04-6047

a - Inadequate supporting field data and analytical method

Table 62. Difenoconazole residues in oilseed rape fodder resulting from supervised trials in Germany

RAPE SEED FODDER	Application [a]					PHI	Commodity	Residues, mg/kg	Ref
country, year (variety)	Form	kg ai/ha	water (L/ha)	no.	BBCH stage, final ap	days		difenoconazole	
Germany, 1997 (Capitol)	SC includes carbendazim	0.13	400	2	BBCH 69-71	0 35 35	whole plant pods + seed stubble	1.4 0.18 0.14	gr 52297
Germany, 1997 (Express)	SC includes carbendazim	0.13	400	2	BBCH 71-75	0 35 35	whole plant pods + seed stubble	1.5 0.02 0.02	gr 51197
Germany, 1997 (Express)	SC includes carbendazim	0.13	400	2	BBCH 71	0 34 34	whole plant pods + seed stubble	1.7 0.12 0.04	gr 53497
Germany, 1996 (Evita)	SC includes carbendazim	0.13	400	1 2	BBCH 65	28 0 35 35	whole plant whole plant green pods stubble	< 0.02 1.8 < 0.02 0.06	gr 54696

RAPE SEED FODDER	Application [a]					PHI	Commodity	Residues, mg/kg	Ref
country, year (variety)	Form	kg ai/ha	water (L/ha)	no.	BBCH stage, final ap	days		difenoconazole	
Germany, 1996 (Lirajet)	SC includes carbendazim	0.13	400	1 2	BBCH 63-65	21 0 34 34	whole plant whole plant green pods stubble	< 0.02 2.3 < 0.02 0.04	gr 53496
Germany, 1996 (Synergy)	SC includes carbendazim	0.13	400	1 2	BBCH 63-65	28 0 35 35	whole plant whole plant green pods stubble	< 0.02 2.5 < 0.02 0.09	gr 51296
Germany, 1996 (Wotan)	SC includes carbendazim	0.13	400	1 2	BBCH 65	28 0 35 35	whole plant whole plant green pods stubble	< 0.02 1.6 < 0.02 0.05	gr 52396

a - BBCH growth stages for oilseed rape (Stauss, 1994)
63: 30% of flowers on main raceme open.
65: full flowering, 50% of flowers on main raceme open, older petals falling.
67: flowering declining, majority of petals fallen.
69: end of flowering.
71: 10% of pods have reached final size.
73: 30% of pods have reached final size.
75: 50% of pods have reached final size.

Table 63. Difenoconazole residues in sunflower plant and stubble resulting from supervised trials in France and Switzerland

SUNFLOWER PLANT & STUBBLE	Application [a]					PHI	Commodity	Residues, mg/kg	Ref
country, year (variety)	Form	kg ai/ha	water (L/ha)	no.	BBCH stage, final ap	days		difenoconazole	
France, 2004 (Alstars)	SC includes azoxystrobin	0.13	400	1 2	BBCH 51-55	15 0 11 21 32 42	whole plant whole plant whole plant whole plant whole plant whole plant	0.12 1.5 0.18 0.06 0.03 0.01	04-0416 FR-FR-04-0125
France, 2004 (DK 3792)	SC includes azoxystrobin	0.13	400	1 2	BBCH 51-55	21 0 10 20 31 45	whole plant whole plant whole plant whole plant whole plant whole plant	0.06 2.0 0.47 0.28 0.27 0.34	04-0415
France, 2004 (Galix)	SC includes azoxystrobin	0.13	300	1 2	BBCH 51-55	14 0 10 21 31 46	whole plant whole plant whole plant whole plant whole plant whole plant	2.4 6.2 1.8 1.0 0.87 0.28	04-0416 FR-FR-04-0123
France, 2004 (Kolda)	SC includes azoxystrobin	0.13	310	1 2	BBCH 59	15 0 10 21 29 45	whole plant whole plant whole plant whole plant whole plant whole plant	0.29 3.7 1.1 0.48 0.25 0.35	04-0416 FR-FR-04-0124
France, 2004 (LG5655)	SC includes azoxystrobin	0.13	210	1 2	BBCH 51-55	14 0 10 20 31 46	whole plant whole plant whole plant whole plant whole plant whole plant	0.29 5.3 0.61 0.39 0.36 0.23	04-0416 FR-FR-04-0126

SUNFLOWER PLANT & STUBBLE	Application [a]					PHI	Commodity	Residues, mg/kg	Ref
country, year (variety)	Form	kg ai/ha	water (L/ha)	no.	BBCH stage, final ap	days		difenoconazole	
Switzerland, 2004 (Prodisol)	SC includes azoxystrobin	0.13	400	1		15	whole plant	0.17	04-0311
				2	BBCH 51-55	0	whole plant	4.6	
						9	whole plant	0.90	
						20	whole plant	0.20	
						30	whole plant	0.16	
						44	whole plant	0.07	
						68	stubble	0.04	

a - BBCH growth stages for sunflower (Stauss, 1994)
51: inflorescence just visible between youngest leaves.
53: inflorescence separating from youngest leaves, bracts distinguishable from foliage leaves.
55: inflorescence separated from youngest foliage leaf.
57: inflorescence clearly separated from foliage leaves.
59: ray florets visible between the bracts, inflorescence still closed.

FATE OF RESIDUES IN STORAGE AND PROCESSING

In processing

The Meeting received information on the fate of difenoconazole residues during the processing of apples for juice, carrots for juice and canning, grapes for wine and raisins, olives for oil, rape seed for oil, sugar beet and tomatoes for juice and puree. Also information was provided on hydrolysis studies of difenoconazole to assist with identification of the nature of the residue during processing.

Processing factors have been calculated for difenoconazole residues in apples, carrots, grapes, olives and tomatoes. The data for rape seed and sugar beet could not be used because residue levels did not exceed the LOQ in the raw commodity.

Muir (2003, RJ3360B) measured the hydrolysis of [^{14}C]triazole-difenoconazole in aqueous buffers at pHs and temperatures experienced during food processing and cooking. Recoveries of total ^{14}C ranged from 102 – 111%. Very little of the difenoconazole was hydrolysed (Table 64). Low levels (< 1.1%) of a minor unknown were noted in the pH 5 pH 6 solutions when analysed by TLC. Difenoconazole was essentially stable during the hydrolysis conditions simulating food processing conditions.

Table 64. Hydrolysis of [^{14}C]triazole-difenoconazole under conditions representing food processes

Difenoconazole concentration	Hydrolysis conditions			Represent	% difenoconazole remaining
2 mg/L	pH 4	90 ˚C	20 mins	pasteurisation	95.6%
2 mg/L	pH 5	100 ˚C	60 mins	baking, brewing and boiling	98.0%
2 mg/L	pH 6	120 ˚C	20 mins	sterilisation	98.5%

Zietz (1998, IF-97/33628-00, IF-97/33752-00) processed samples of rape seed from supervised trials with difenoconazole in Germany (Smith, 1998, gr 52297, gr 51197). The pilot plant processing on 3 kg oilseed samples was designed to simulate the commercial processes of drying, expelling and filtering and produced dried seed, oil and oilseed cake. Residue data are summarised in Table 65.

In a trial in Spain, Richards (2006, 04-6067) treated olives with difenoconazole at 0.24 kg difenoconazole per hectare (2× label rate) and harvested samples of 20 kg for processing to olive oil. Whole olives were processed into virgin oil and refined oil (Figure 7). Difenoconazole residues from the olives partitioned into the oil fraction. The refining step had very little influence on the difenoconazole residues (Table 65).

Beinhauer (1997, 96 10 62 010) treated sugar beet twice with a formulation containing difenoconazole and fenpropidin and harvested the beets 28 days later for processing (Figure 8). Difenoconazole residues in the raw and processed commodities were below the LOQ (Table 65). The process and results were similar for a second sugar beet processing trial (Beinhauer, 1997, 96 10 61 009).

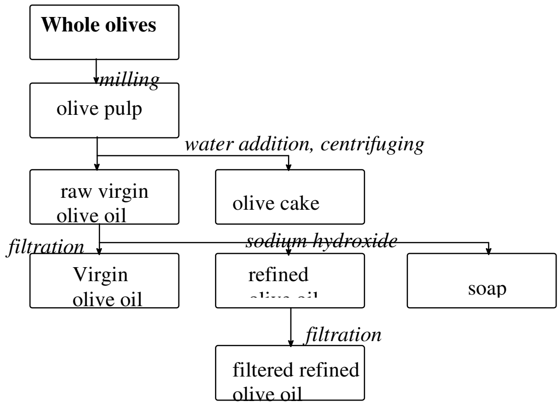


Figure 7. Olive processing

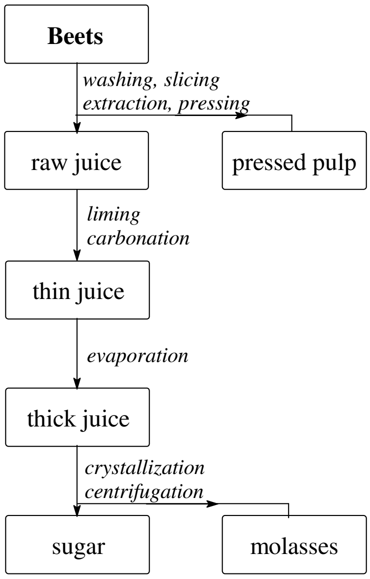


Figure 8. Sugar beet processing

Simon (2002, gap82901) treated apple trees with an exaggerated rate of difenoconazole and harvested fruit 18 days after the final of 4 applications for processing into juice and puree. Fruit (70 kg) were manually washed with cold water, then sliced, wrapped in press cloths and pressed in a juice press, which separated juice from pomace. Juice was pasteurised at 80 – 82 ˚C for 30 minutes. For puree, apples (12 kg) were manually washed in cold water and then boiled in water until the puree was passable through a sieve. Sugar, citric acid and ascorbic acid were added until the puree reached a pH of 3.0 – 4.5. A tin was filled with puree, and then sealed and heated at 95 ˚C for 20 minutes. Some difenoconazole residues were removed in the initial washing steps. Difenoconazole residues did not appear in the apple juice and were concentrated in the pomace. Much of the residue was lost in converting apples to puree (Table 65).

In a grape drying trial in Chile, Kühne-Thu (1999, 225/98) applied difenoconazole at 1× and 5× the label rate (0.05 and 0.25 kg ai/ha) and harvested grapes 63 days after the final of 3 applications. Grapes were washed for about one minute and then placed in wooden trays with mesh bottoms and subjected to sulphur dioxide fumigation for 12 h. The trays of grapes were then dried in ovens at 65 ˚C for about 36 – 40 h losing about two-thirds of their weight, 30 kg grapes producing 10 kg raisins. Residue data are summarised in Table 65.

Table 65. Difenoconazole residues in raw and processed commodities resulting from supervised trials on rape seed, olives, sugar beet and apples

CROP country, year (variety)	Application					PHI days	Commodity [a]	Residues, mg/kg difenoconazole	Ref
	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.				
RAPE SEED Germany, 1997 (Capitol)	SC includes carbendazim	0.13		400	2	56	seed dried seed oil presscake	< 0.02 < 0.02 < 0.02 < 0.02	gr 52297 IF-97/33628-00

CROP country, year (variety)	Application Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	PHI days	Commodity [a]	Residues, mg/kg difenoconazole	Ref
RAPE SEED Germany, 1997 (Express)	SC includes carbendazim	0.13		400	2	55	seed dried seed oil presscake	< 0.02 < 0.02 < 0.02 < 0.02	gr 51197 IF-97/33752-00
RAPE SEED Germany, 1997 (Express)	SC includes carbendazim	0.13		400	2	55	seed dried seed oil presscake	< 0.02 < 0.02 < 0.02 < 0.02	gr 53497 IF-97/33753-00
OLIVE Spain, 2004 (Alberquina)	EC	0.24		800	2	30	whole olives virgin oil refined oil	0.93 1.4 1.3	04-6067 AF/7872/SY/1
				replicate processing 2			whole olives virgin oil refined oil	0.86 1.4 1.3	
				replicate processing 3			whole olives virgin oil refined oil	0.75 1.1 0.89	
				replicate processing 4			whole olives virgin oil refined oil	0.80 1.2 1.2	
SUGAR BEET Germany, 1996 (Hilma)	EC includes fenpropidin	0.098		300	2	28	root raw juice pressed pulp thin juice thick juice raw sugar molasses	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02	96 10 62 010
SUGAR BEET Germany, 1996 (Reka)	EC includes fenpropidin	0.098		300	2	39	root raw juice pressed pulp thin juice thick juice raw sugar molasses	< 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02	96 10 61 009
APPLES Germany, 2001 (Mondial Gala)	EC	0.34		1000	4	18	fruit washed fruits pomace 22%dm pomace 95%dm juice before [b] juice after [b]	0.415 0.30 1.48 6.55 < 0.01 < 0.01	gap82901
APPLES Germany, 2001 (Mondial Gala)	EC	0.34		1000	4	18	fruit washed fruits puree	0.56 0.47 0.08	gap82901
APPLES Chile, 1994-95 (Red Spur)	EC	0.050	0.0031	1600	6	0 28 42 56 92 92 92	fruit fruit fruit fruit fruit apple juice pomace 17%dm	0.16 (< 0.02) 0.06 (0.02) 0.02 (< 0.02) 0.02 (< 0.02) < 0.02 (< 0.02) < 0.02 (< 0.02) 0.06 (0.02)	2207/94 [c]
APPLES Chile, 1994-95 (Golden Delicious)	EC	0.096	0.0065	1480	6	0 14 27 41 55 91 91 91	fruit fruit fruit fruit fruit fruit apple juice pomace 16%dm	0.24 (< 0.02) 0.17 (0.02) 0.16 (0.02) 0.06 (< 0.02) 0.05 (< 0.02) 0.02 (< 0.02) < 0.02 (< 0.02) 0.13 (0.02)	2205/94 [c]

CROP country, year (variety)	Application Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	PHI days	Commodity [a]	Residues, mg/kg difenoconazole	Ref
APPLES Chile, 1994-95 (Golden Delicious)	EC	0.050	0.0034	1470	6	0 14 27 41 55 91 91 91	fruit fruit fruit fruit fruit fruit apple juice pomace 22%dm	0.11 (< 0.02) 0.10 (< 0.02) 0.07 (< 0.02) 0.03 (< 0.02) 0.02 (< 0.02) 0.02 (< 0.02) < 0.02 (< 0.02) 0.075 (< 0.02)	2204/94 [c]

a - dm: dry matter. bw: before washing; aw: after washing.

b - Juice before and after pasteurization.

c - Studies 2204/94, 2205/94, 2207/94. Residues of metabolite CGA 205375 are in parentheses.

Table 66. Difenoconazole residues in raw and processed commodities resulting from supervised processing trials on grapes

CROP country, year (variety)	Application Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	PHI days	Commodity [a]	Residues, mg/kg difenoconazole	Ref
GRAPES Spain, 1992 (Malvasia)	EC	0.013 +0.018 +0.020 +0.026	0.005	250 +360 +400 +520	4	42	grapes wine	0.05 0.02 < 0.01 < 0.01 b	2051/92
GRAPES Spain, 1992 (Palamino)	EC	0.018 +0.025 +0.037 +0.043	0.005	360 +500 +730 +850	4	54	grapes wine	0.05 0.06 < 0.01 < 0.01 b	2053/92
GRAPES France, 1992 (Grenache)	EC	0.16		200	6	72	grapes wine	0.03 < 0.01	OF92010 G98
GRAPES France, 1992 (Pinot Noir)	EC	0.16		850 +110 +110 +110	4	82	grapes wine	0.02 < 0.01	OF92010 734
GRAPES France, 1992 (Semillon)	EC	0.16		200	4	63	grapes wine	0.02 < 0.01	OF92010 Q98
GRAPES France, 1992 (Sauvignon)	EC	0.030		150	4	68	grapes must wine, white pomace, dry	< 0.02 < 0.02 < 0.01 0.21	OF92016 U59
GRAPES France, 1992 (Gamay)	EC	0.030		250 +3×120	4	73	grapes must wine, red pomace, dry	0.05 0.05 < 0.01 0.77	OF92016 Y56
GRAPES France, 1992 (Pinot noir)	EC	0.030		850 +3×110	4	82	grapes must wine, red pomace, dry	< 0.02 < 0.02 < 0.01 0.11	OF92016 731
GRAPES France, 1992 (Chardonnay)	EC	0.030		150 +3×120	4	82	grapes must wine, white pomace, dry	0.03 < 0.02 < 0.01 0.28	OF92016 830
GRAPES France, 1992 (Cabernet Sauvignon)	EC	0.030		200	4	90	grapes must wine, red pomace, dry	0.03 0.02 < 0.01 0.31	OF92016 Q92

CROP	Application					PHI	Commodity	Residues, mg/kg	Ref
country, year (variety)	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	days	[a]	difenoconazole	
GRAPES France, 1992 (Tannat)	EC	0.030		200	4	81	grapes must wine, red pomace, dry	0.02 < 0.02 < 0.01 0.28	OF92016 Q93
GRAPES France, 1992 (Ugni Blanc)	EC	0.030		200	4	60	grapes must wine, white pomace, dry	0.05 < 0.02 < 0.01 6.6 [c]	OF92016 G94
GRAPES France, 1992 (Carignan)	EC	0.030		100	4	71	grapes must wine, red pomace, dry	< 0.02 < 0.02 < 0.01 < 0.04	OF92016 J97
GRAPES France, 1995 (Pinot Noir)	EC	0.030	0.023	130	4	68	grapes juice wine wine	0.02 (< 0.02) < 0.01 (< 0.01) < 0.01 (0.01) (u 0.01) [d]	OF95144 DE97
GRAPES Chile, 1996-97 (Thomson seedless)	EC	0.050		1040 +740 +1600	3	0 7 21 35 62 62 62	grapes grapes grapes grapes grapes juice pomace 26% dm	0.16 0.09 0.05 0.03 < 0.02 < 0.02 0.07	2217/96
GRAPES Chile, 1996-97 (Thomson seedless)	EC	0.050		810 +470 +1220	3	59	grapes juice pomace 27% dm	< 0.02 < 0.02 0.07	2219/96
GRAPES Chile, 1996-97 (Red Globe)	EC	0.055		1170 +1280 +1420	3	72	grapes juice pomace 28% dm	< 0.02 < 0.02 0.09	2220/96
GRAPES Chile, 1996-97 (Cabernet Sauvignon)	EC	0.046 +0.061 +0.049		730 +980 +1530	3	102	grapes juice pomace 45% dm	< 0.02 < 0.02 0.05	2221/96
GRAPES Chile, 1998-99 (Thompson Seedless)	EC	0.050	0.005	1000	3	63	grapes grapes bw grapes aw raisins	0.06 0.04 0.04 0.04 0.07 0.07	2258/98
GRAPES Chile, 1998-99 (Thompson Seedless)	EC	0.25	0.025	1000	3	63	grapes grapes bw grapes aw raisins	0.60 0.50 0.25 0.30 0.63 0.48	2258/98

a - dm: dry matter; bw: before washing; aw: after washing; u: sample from control (untreated) plot.

b - Duplicate plots and wine from duplicate plots.

c - Study OF92016, trial G94. The analytical result (6.6 mg/kg) was confirmed by repeat analysis. The study director investigated this apparently anomalous high result, but was not able to find a reason.

d - Study OF95144. Residues of metabolite CGA 205375 are in parentheses.

In a tomato processing trial in France, Ryan (2006, 04-6049) sprayed tomato vines with difenoconazole at 0.37 kg ai/ha and harvested tomatoes (200 kg) 7 days after the final of 3 applications. Tomatoes were processed in a set of full balance studies and then in 3 follow-up studies. Data are summarised in Table 67.

The effect on residue levels of sorting and washing tomatoes with water was first examined.

In processing to juice, unwashed tomatoes were crushed and juice and pomace were separated on a sieve. Dry pomace was produced by placing the wet pomace in an oven at 60 ˚C for 2 – 3 days. Citric acid (to pH 3.5) and salt (7 g/kg) were added to raw juice and the finished juice was produced by pasteurization for 1 minute at 82 – 85 ˚C.

In the production of puree, unwashed tomatoes were crushed and concentrated in a saucepan and then sieved. Salt and citric acid were added and the puree, in glass jars, was sterilised for 10 minutes at 115 ˚C.

In the simulation of canning, unwashed tomatoes were blanched and then immediately plunged into cold water to split and loosen the peel which was removed with a knife. The peeled tomatoes, in glass jars, were covered with tomato juice and sterilised for 10 minutes at 115 – 120 ˚C.

Table 67. Difenoconazole residues in raw and processed commodities resulting from supervised processing trials on tomatoes

TOMATOES	Application				PHI	Commodity	Difenoconazole residues, mg/kg			
country, year (variety)	Form	kg ai/ha	water (L/ha)	no.	days		Full balance study	Follow up study 1	Follow up study 2	Follow up study 3
France, 2004 (Netico)	EC	0.37	610 +720 +720	3	7	fruit received	0.18	0.26	0.19	0.28
sorting and washing						fruit sorted fruit washed	0.19 0.15	0.20 0.11	0.22 0.18	0.20 0.16
juicing						crushed pomace, wet juice, raw pomace, dry juice, finished	0.24 0.39 0.07 5.1 0.05	 0.54 5.2 0.04	 0.58 5.7 0.06	 0.47 4.8 0.04
puree						crushed sieved tomato pomace, wet puree, raw puree, finished	0.22 0.18 1.3 0.16 0.18	 0.15	 0.14	 0.15
canning						fruit, peeled peel canned canned & sterilized	< 0.01 1.9 0.02 0.01	 0.02	 < 0.01	 0.02

In a carrot processing trial in France, Anderson (2006, 05-6022-REG) sprayed a carrot crop with difenoconazole at 0.50 kg ai/ha and harvested carrots (160 kg) 7 days after the final of 3 applications. Carrots were processed in a set of full balance studies and then in 3 follow-up studies. Data are summarised in Table 68.

In the simulation of canning, carrots were sorted and peeled and both ends were removed. The peeled carrots were washed thoroughly and blanched in boiling water for 1 minute and placed in jars with brine and citric acid to produce pH 3.5 and then sealed and sterilized for 10 minutes at 115 – 120 ˚C. For cooked carrots, the washed carrots were cooked in boiling water for 15 minutes and packaged in plastic bags under vacuum.

For juicing, carrots were washed thoroughly after sorting, peeling and end removal. The washed carrots were processed in a juice extractor which separated juice from pulp in a centrifugal filter. After the pH of the juice was adjusted to 3.5 with citric acid, the juice was pasteurized at approximately 85 ˚C and packaged in glass jars.

Table 68. Difenoconazole residues in raw and processed commodities resulting from supervised processing trials on carrots

CARROTS	Application				PHI	Commodity	Difenoconazole residues, mg/kg			
country, year (variety)	Form	kg ai/ha	water (L/ha)	no.	days		Full balance study	Follow up study 1	Follow up study 2	Follow up study 3
France, 2005 (Maestro)	EC	0.50	400	3	7	carrots pre-processing	0.31	0.25	0.44	0.44
canning						carrots, peeled	0.04			
						carrots, washed	0.02	0.08	0.02	0.02
						carrots, blanched	0.03			
						carrots, canned	0.01	0.03	0.02	0.01
cooking						carrots, peeled	0.03			
						carrots, washed	0.04	0.03	0.02	0.02
						carrots, cooked	0.02	0.01	0.02	0.02
juicing						carrots, peeled	0.05			
						carrots, washed	0.05	0.04	0.02	0.03
						juice, raw	0.02			
						carrot pulp	0.03			
						juice, pasteurized	0.02	0.03	0.01	0.02

Table 69. Summary of processing factors for difenoconazole residues. The factors are calculated from the data recorded in tables in this section

Raw agricultural commodity (RAC)	Processed commodity	Calculated processing factors.	Median or best estimate
Apple	juice	< 0.02, < 1.0. < 1.0	< 0.02
Apple	dry pomace	15.4	15
Apple	puree	0.14	0.14
Carrot	canned	0.02, 0.03, 0.05, 0.12	0.04
Carrot	juice	0.02, 0.05, 0.06, 0.12	0.055
Grapes	juice	< 0.5	< 0.5
Grapes	dry pomace	9.3, 10.3, 14.0, 15.4	12
Grapes	raisins	1.01, 1.4	1.2
Grapes	wine	< 0.18, < 0.20, < 0.20, < 0.29, < 0.33, < 0.33, < 0.33, < 0.50, < 0.50, < 0.50, < 0.50	< 0.18
Olives	refined oil	1.19, 1.40, 1.50, 1.51	1.4
Olives	virgin oil	1.47, 1.50, 1.50, 1.63	1.5
Tomatoes	canned tomato	< 0.05, 0.06, 0.07, 0.08	0.065
Tomatoes	juice	0.14, 0.15, 0.28, 0.32	0.22
Tomatoes	puree	0.54, 0.58, 0.74, 1.00	0.66

RESIDUES IN ANIMAL COMMODITIES

Farm animal feeding studies

The meeting received a lactating dairy cow feeding study and a laying hen feeding study, which provided information on likely residues resulting in animal commodities, milk and eggs from difenoconazole residues in the animal diet.

Lactating dairy cows

Groups of 3 lactating Holstein dairy cows (animals weighing 498 – 608 kg and 522 – 636 kg on days 1 and 29 respectively) were dosed once daily via gelatin capsule with difenoconazole at 1 ppm (1×), 3 ppm (3×) and 10 ppm (10×) in the dry-weight diet, equivalent to doses of 0.035, 0.11 and 0.35 mg difenoconazole per kg body weight, for 29-30 consecutive days (Tribolet, 2000, 202/99). Milk was collected on 9 occasions for analysis (days 0, 2, 5, 8, 12, 15, 19, 22 and 28). On days 29 and 30, the animals were slaughtered for tissue collection. Tissues collected for analysis were liver, kidney, perirenal fat, omental fat, round muscle, diaphragm muscle and loin muscle. Animals consumed approximately 20 kg dry feed each per day and produced approximately 19 – 26 kg milk per animal per day (means for each animal through the test period).

Parent difenoconazole residues did not occur above LOQ in muscle, kidney or fat tissues or milk for any of the test doses (Table 70). Parent difenoconazole residues were present in liver at the 10 ppm feeding level. Metabolite CGA 205375 was present in each of the tissues at 3 and 10 ppm feeding levels and in the liver and fat at the 1 ppm feeding level. The concentration of metabolite CGA 205375 in fat was approximately 3.3 times its concentration in muscle.

The average concentration of metabolite CGA 205375 in the tissues at the 10 ppm feeding level were: muscle 0.020 mg/kg; liver 0.30 mg/kg; kidney 0.044 mg/kg; fat 0.072 mg/kg. For liver, the transfer factors for the 3 feeding levels were reasonably consistent for metabolite CGA 205375. For fat, the transfer factors for metabolite CGA 205375 apparently decreased as the feeding level increased.

Metabolite CGA 205375 was consistently present in the milk from day 2 onwards at 0.005 – 0.009 mg/kg for the 10 ppm feeding level (Table 70).

Table 70. Residues in milk and tissues of lactating Holstein dairy cows (3 per group) dosed once daily via gelatin capsule with difenoconazole at the equivalent of 1 ppm (1×), 3 ppm (3×) and 10 ppm (10×) in the dry-weight diet, for 29 – 30 consecutive days

Substrate	Residues, mg/kg – individual animals					
	Dosing, 1 ppm		Dosing, 3 ppm		Dosing, 10 ppm	
	difenoconazole	CGA 205375	difenoconazole	CGA 205375	difenoconazole	CGA 205375
Loin muscle	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)	0.01 0.012 < 0.01	< 0.01 (3)	0.021 0.024 0.02
Round muscle	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)	0.01 0.01 < 0.01	< 0.01 (3)	0.016 0.019 0.014
Diaphragm muscle	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)	0.022 0.01 < 0.01	< 0.01 (3)	0.023 0.028 0.013
Liver	< 0.01 (3)	0.035 0.038 0.044	< 0.01 (3)	0.13 0.13 0.094	0.012 0.02 0.01	0.27 0.35 0.29
Kidney	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)+	0.018 0.018 0.015	< 0.01 (3)	0.041 0.052 0.038
Omental fat	< 0.01 (3)	0.01 0.013 0.01	< 0.01 (3)	0.033 0.027 0.02	< 0.01 (3)	0.063 0.095 0.072
Perirenal fat	< 0.01 (3)	0.01 0.013 0.01	< 0.01 (3)	0.031 0.032 0.022	< 0.01 (3)	0.057 0.079 0.065
Milk, day 0 [a]	< 0.005 (3)	< 0.005 (3)	< 0.005 (3)	< 0.005 (3)	< 0.005 (3)	< 0.005 (3)
Milk, day 2	< 0.005 (3)	< 0.005 (3)	< 0.005 (3)	< 0.005 (3)	< 0.005 (3)	0.009 0.007 0.006
Milk, day 5	< 0.005 (3)	< 0.005 (3)	< 0.005 (3)	< 0.005 (3)	< 0.005 (3)	0.008 0.009 0.005
Milk, day 8	< 0.005 (3)	< 0.005 (3)	< 0.005 (3)	< 0.005 (3)	< 0.005 (3)	0.009 0.008 0.005
Milk, day 12	< 0.005 (3)	< 0.005 (3)	< 0.005 (3)	< 0.005 (3)	< 0.005 (3)	0.007 0.009 0.005
Milk, day 15	< 0.005 (3)	< 0.005 (3)	< 0.005 (3)	< 0.005 (3)	< 0.005 (3)	0.008 0.008 0.005
Milk, day 19	< 0.005 (3)	< 0.005 (3)	< 0.005 (3)	< 0.005 (3)	< 0.005 (3)	0.007 0.008 0.005
Milk, day 22	< 0.005 (3)	< 0.005 (3)	< 0.005 (3)	< 0.005 (3)	< 0.005 (3)	0.007 0.008 0.005
Milk, day 28	< 0.005 (3)	< 0.005 (3)	< 0.005 (3)	< 0.005 (3)	< 0.005 (3)	0.007 0.008 0.006

a - Milk concentrations were provided as µg/L. They have been converted to mg/kg on the assumption that milk density is 1 kg/L.

Groups of 3 lactating Holstein dairy cows (animals weighing 508 – 708 kg and 508 – 710 kg at study initiation and completion respectively) were dosed once daily via gelatin capsule with difenoconazole at 1 ppm (1×), 5 ppm (5×) and 15 ppm (15×) in the dry-weight diet, equivalent to doses of 20, 100 and 300 mg per animal, for 29 – 30 consecutive days (Ryan, 2006, T009107-04-REG). Milk was collected on 10 days for analysis, equal volumes of evening and morning milk, (days 0 before treatment, 2, 5, 8, 12, 15, 19, 22, 26 and 28). On days 29 and 30, the animals were slaughtered between 24 – 30 hours after the final dose for tissue collection. Tissues collected for analysis were liver, kidney, perirenal fat, mesenterial fat, subcutaneous fat, round muscle, diaphragm muscle and loin muscle. Animals consumed approximately 22 – 25 kg dry feed each per day and produced approximately 13 – 35 kg milk per animal per day (means for each animal through the test period).

Parent difenoconazole residues did not occur above LOQ in muscle, kidney or fat tissues or milk for any of the test doses (Table 71). Parent difenoconazole residues were present in liver at the 5 and 15 ppm feeding levels. Metabolite CGA 205375, the major part of the residue, was present in each of the tissues at 5 and 15 ppm feeding levels and in the liver, kidney and fat at the 1 ppm feeding level. At the 15 ppm feeding level, the concentration of metabolite CGA 205375 in fat was approximately 3.1 times its concentration in muscle. Metabolite 1,2,4-triazole did not occur above LOQ in tissues and milk at the 1 ppm dosing level or in fat at the 15 ppm dosing level; it was present in milk, muscle, liver and kidney at the 5 and 15 ppm feeding levels.

The average concentration of metabolite CGA 205375 in the tissues at the 15 ppm feeding level were: muscle 0.04 mg/kg; liver 0.57 mg/kg; kidney 0.11 mg/kg; fat 0.12 mg/kg. For liver, the transfer factors for the 5 ppm and 15ppm feeding levels were close for metabolite CGA 205375. For fat, the transfer factors for metabolite CGA 205375 were also consistent for the 5 ppm and 15ppm feeding levels.

Metabolite CGA 205375 reached a plateau level in milk of approximately 0.012 mg/kg within 2 days at the 15 ppm feeding level. Metabolite 1,2,4-triazole was consistently present in the milk at the 5 and 15 ppm feeding levels. Plateau concentrations in milk of approximately 0.017 mg/kg and 0.04 mg/kg were quickly reached for the 5 ppm and 15 ppm feeding levels respectively (Table 71).

Table 71. Residues in milk and tissues of lactating Holstein dairy cows (3 per group) dosed once daily via gelatin capsule with difenoconazole at the equivalent of 1 ppm (1×), 5 ppm (5×) and 15 ppm (15×) in the dry-weight diet, for 29 – 30 consecutive days

Substrate	Residues, mg/kg – individual animals								
	Dosing, 1 ppm			Dosing, 5 ppm			Dosing, 15 ppm		
	difeno-conazole	CGA 205375	1,2,4-triazole	difeno-conazole	CGA 205375	1,2,4-triazole	difeno-conazole	CGA 205375	1,2,4-triazole
Loin muscle	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)	< 0.01 0.01 0.01	0.01 0.02 0.01	< 0.01 (3)	0.04 0.03 0.04	0.03 0.04 0.03
Round muscle	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)	< 0.01 (2) 0.01	0.01 (3)	< 0.01 (3)	0.04 0.04 0.03	0.03 0.04 0.03
Diaphragm muscle	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)	0.01 (3)	0.01 (3)	< 0.01 (3)	0.05 0.05 0.04	0.03 0.04 0.03
Liver	< 0.01 (3)	0.06 0.07 0.05	< 0.01 (3)	0.01 0.01 0.02	0.14 0.23 0.22	0.01 0.01 < 0.01	0.03 (3)	0.66 0.52 0.53	0.03 0.02 0.02
Kidney	< 0.01 (3)	< 0.01 0.01 < 0.01	< 0.01 (3)	< 0.01 (3)	0.03 0.04 0.04	0.02 (3)	< 0.01 (3)	0.12 0.12 0.09	0.03 0.05 0.03
Perirenal fat	< 0.01 (3)	< 0.01 0.01 < 0.01	< 0.01 (3)	< 0.01 (3)	0.03 0.05 0.05	< 0.01 (3)	< 0.01 (3)	0.13 0.13 0.10	< 0.01 (3)

Substrate	Residues, mg/kg – individual animals								
	Dosing, 1 ppm			Dosing, 5 ppm			Dosing, 15 ppm		
	difeno-conazole	CGA 205375	1,2,4-triazole	difeno-conazole	CGA 205375	1,2,4-triazole	difeno-conazole	CGA 205375	1,2,4-triazole
Mesenterial fat	< 0.01 (3)	< 0.01 0.01 < 0.01	< 0.01 (3)	< 0.01 (3)	0.03 0.04 0.04	< 0.01 (3)	< 0.01 (3)	0.14 0.12 0.12	< 0.01 (3)
Subcutaneous fat	< 0.01 (3)	< 0.01 0.02 < 0.01	< 0.01 (3)	< 0.01 (3)	0.03 0.04 0.04	< 0.01 (3)	< 0.01 (3)	0.13 0.13 0.11	< 0.01 (3)
Milk, day 0	< 0.005 (3)	< 0.005 (3)	< 0.01 (3)	< 0.005 (3)	< 0.005 (3)	< 0.01 (3)	< 0.005 (3)	< 0.005 (3)	< 0.01 (3)
Milk, day 2	< 0.005 (3)	< 0.005 (3)	< 0.01 (3)	< 0.005 (3)	< 0.005 (2) 0.007	0.01 0.01 0.02	< 0.005 (3)	0.012 0.013 0.010	0.02 0.02 0.03
Milk, day 5	< 0.005 (3)	< 0.005 (3)	< 0.01 (3)	< 0.005 (3)	< 0.005 (2) 0.006	0.01 0.02 0.02	< 0.005 (3)	0.016 0.012 0.013	0.03 0.04 0.04
Milk, day 8	< 0.005 (3)	< 0.005 (3)	< 0.01 (3)	< 0.005 (3)	< 0.005 (3)	0.01 0.01 0.02	< 0.005 (3)	0.015 0.013 0.009	0.04 0.04 0.05
Milk, day 12	< 0.005 (3)	< 0.005 (3)	< 0.01 (3)	< 0.005 (3)	< 0.005 (3)	0.01 0.03 0.02	< 0.005 (3)	0.012 0.011 0.008	0.04 0.04 0.05
Milk, day 15	< 0.005 (3)	< 0.005 (3)	< 0.01 (3)	< 0.005 (3)	< 0.005 (3)	0.02 (3)	< 0.005 (3)	0.013 0.014 0.009	0.03 0.04 0.05
Milk, day 19	< 0.005 (3)	< 0.005 (3)	< 0.01 (3)	< 0.005 (3)	< 0.005 (3)	0.01 0.02 0.02	< 0.005 (3)	0.011 0.013 0.008	0.03 0.03 0.05
Milk, day 22	< 0.005 (3)	< 0.005 (3)	< 0.01 (3)	< 0.005 (3)	< 0.005 (3)	0.01 0.02 0.02	< 0.005 (3)	0.020 0.011 0.012	0.04 0.04 0.05
Milk, day 26	< 0.005 (3)	< 0.005 (3)	< 0.01 (3)	< 0.005 (3)	< 0.005 (3)	0.01 0.02 0.02	< 0.005 (3)	0.011 0.010 0.009	0.04 0.04 0.05
Milk, day 28	< 0.005 (3)	< 0.005 (3)	< 0.01 (3)	< 0.005 (3)	< 0.005 (3)	0.01 0.02 0.02	< 0.005 (3)	0.012 0.009 0.011	0.04 0.03 0.04

Laying hens

Four groups of 15 laying white leghorn hens (3 subgroups of 5 birds per group weighing approximately 1.6 kg/bird at study initiation and completion respectively) were fed rations treated with difenoconazole at 0.3 ppm, 1 ppm, 3 ppm and 10 ppm, for 28 consecutive days (Ryan, 2006, T000141-05-REG). Eggs were collected on 10 occasions for analysis (days 0 before treatment, 1, 3, 6, 9, 13, 16, 20, 23 and 28).The birds were slaughtered between 20 – 24 h after removal of the treated food. Tissues collected for analysis were skin plus attached fat, peritoneal fat, liver and breast plus thigh muscle. Birds consumed approximately 130 – 140 g feed each per day. Residues data for difenoconazole and metabolites CGA 205375 and 1,2,4-triazole are summarised in Table 72. Tissue samples from the 0.3 ppm feeding group were not analysed because residues were at or below LOQ in the 1 ppm feeding group.

Parent difenoconazole residues did not occur above LOQ in muscle, fat, liver or eggs for any of the test doses (Table 72). Metabolite CGA 205375 was not present in the tissues. Average levels of 1,2,4-triazole in the tissues at the 10 ppm feeding level were: skin + attached fat 0.012 mg/kg; peritoneal fat < 0.005 mg/kg; liver 0.02 mg/kg; muscle 0.022 mg/kg.

Metabolite CGA 205375 occurred in eggs at the 1, 3 and 10 ppm feeding levels. It reached a plateau after approximately 9 days with plateau levels of 0.037 mg/kg and 0.13 mg/kg at the 3 and 10 ppm feeding levels respectively. At the 1 ppm feeding level, CGA 205375 was present in eggs at close to the LOQ (0.01 mg/kg).

Metabolite 1,2,4-triazole occurred in eggs at the 1, 3 and 10 ppm feeding levels. It reached a plateau after approximately 6 days with plateau levels of 0.007, 0.020 and 0.060 mg/kg at the 1, 3 and 10 ppm feeding levels respectively.

Table 72. Residues in eggs and tissues of laying white leghorn hens fed rations treated with difenoconazole at 1 ppm, 3 ppm and 10 ppm, for 28 consecutive days

Substrate	Residues, mg/kg – data on 3 subgroups of 5 birds per group [a]								
	Dosing, 1 ppm			Dosing, 3 ppm			Dosing, 10 ppm		
	difeno-conazole	CGA 205375	1,2,4-triazole	difeno-conazole	CGA 205375	1,2,4-triazole	difeno-conazole	CGA 205375	1,2,4-triazole
Skin + attached fat	na [b]	na	< 0.005 (3)	< 0.01 (3)	< 0.01 (3)	0.005 < 0.005 (2)	< 0.01 (3)	< 0.01 (3)	0.013 0.014 0.009
Peritoneal fat	na	na	< 0.005 (3)	< 0.01 (3)	< 0.01 (3)	< 0.005 (3)	< 0.01 (3)	< 0.01 (3)	< 0.005 (3)
Liver	na	na	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)	0.01 < 0.01 (2)	< 0.01 (3)	< 0.01 (3)	0.02 (3)
Breast, thigh muscle	na	na	< 0.005 (3)	< 0.01 (3)	< 0.01 (3)	0.008 < 0.005 (2)	< 0.01 (3)	< 0.01 (3)	0.022 0.023 0.020
Eggs, day 0	< 0.01 (3)	< 0.01 (3)	< 0.005 (3)	< 0.01 (3)	< 0.01 (3)	< 0.005 (3)	< 0.01 (3)	< 0.01 (3)	< 0.005 (3)
Eggs, day 1	< 0.01 (3)	< 0.01 (3)	< 0.005 (3)	< 0.01 (3)	< 0.01 (3)	< 0.005 (3)	< 0.01 (3)	< 0.01 (3)	< 0.005 (3)
Eggs, day 3	< 0.01 (3)	< 0.01 (3)	0.005 0.005 0.006	< 0.01 (3)	< 0.01 (3)	0.016 0.017 0.013	< 0.01 (3)	0.03 (3)	0.052 0.041 0.046
Eggs, day 6	< 0.01 (3)	< 0.01 (3)	0.006 0.007 0.007	< 0.01 (3)	0.03 0.02 0.03	0.016 0.022 0.023	< 0.01 (3)	0.10 (3)	0.067 0.060 0.060
Eggs, day 9	< 0.01 (3)	0.01 < 0.01 (2)	0.008 0.007 0.006	< 0.01 (3)	0.04 0.03 0.04	0.015 0.022 0.022	< 0.01 (3)	0.13 0.12 0.13	0.067 0.055 0.056
Eggs, day 13	< 0.01 (3)	< 0.01 (3)	0.007 0.006 0.005	< 0.01 (3)	0.04 0.03 0.04	0.017 0.021 0.023	< 0.01 (3)	0.17 0.14 0.12	0.066 0.056 0.056
Eggs, day 16	< 0.01 (3)	0.01 < 0.01 (2)	0.007 0.007 0.006	< 0.01 (3)	0.04 0.03 0.04	0.016 0.022 0.024	< 0.01 (3)	0.13 0.12 0.14	0.065 0.056 0.059
Eggs, day 20	< 0.01 (3)	< 0.01 0.01 < 0.01	0.010 0.008 0.007	< 0.01 (3)	0.04 0.03 0.04	0.018 0.023 0.023	< 0.01 (3)	0.15 0.09 0.13	0.063 0.058 0.060
Eggs, day 23	< 0.01 (3)	0.01 (3)	0.008 0.007 0.007	< 0.01 (3)	0.04 (3)	0.016 0.021 0.022	< 0.01 (3)	0.13 (3)	0.068 0.056 0.055
Eggs, day 28	< 0.01 (3)	0.01 (3)	0.008 0.007 0.007	< 0.01 (3)	0.03 0.04 0.03	0.016 0.019 0.024	< 0.01 (3)	0.13 0.14 0.13	0.069 0.056 0.056

a - Residues of difenoconazole, CGA 205375 and 1,2,4-triazole did not exceed LOQ values in any egg sample from the 0.3 ppm feeding group.

b - na: not analysed because no residues found at the higher feeding levels.

RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

Difenoconazole was included in the list of analytes examined in foods in the 20th Australian Total Diet Survey (FSANZ, 2003). The dietary exposure for difenoconazole was estimated to be zero, because its concentration in the surveyed foods was less than the limit of detection (0.01 mg/kg).

Difenoconazole was included in the Australian National Residue Survey program of monitoring apples and pears in 2004-05 (NRS, 2005) and 2005-06 (NRS, 2006). No residues (limit of reporting 0.05 mg/kg) were reported in apples (471 samples) and pears (139 samples) for the two years.

NATIONAL MAXIMUM RESIDUE LIMITS

Information was provided on national residue definitions for difenoconazole.

Australia:- (FSANZ, 2007):- Plant and animal commodities: difenoconazole.

Brazil:- Difenoconazole.

Costa Rica:- Difenoconazole.

Europe:- Plants: Difenoconazole (for monitoring and risk assessment).

Livestock: Difenoconazole and CGA205375 (for monitoring). Difenoconazole (for risk assessment)

Indonesia: - Difenoconazole.

Japan:- Difenoconazole.

Netherlands (Muller, 2007):- Plant products: difenoconazole

Products of animal origin: sum of difenoconazole and 1-[2-[2-chlor-4(4-chlorphenoxy)-phenyl]-2-hydroxy-1-1ethyl]-H-1,2,4-triazole.

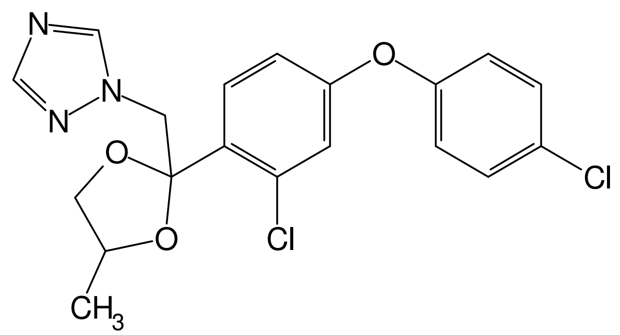
Switzerland:- Difenoconazole.

USA:- Difenoconazole.

APPRAISAL – RESIDUE AND ANALYTICAL ASPECTS

Difenoconazole was considered for the first time by the present meeting. It is a broad-spectrum fungicide used for disease control in many fruits, vegetables, cereals and other field crops. It has preventive and curative action. Difenoconazole acts by inhibition of demethylation during ergosterol synthesis.

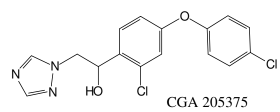
1-[2-[2-chloro-4-(4-chloro-phenoxy)-phenyl]-4-methyl[1,3]dioxolan-2-ylmethyl]-1H-1,2,4-triazole



Animal metabolism

The Meeting received animal metabolism studies with difenoconazole in rats, lactating goats and laying hens. Difenoconazole [^{14}C] labelled in the central phenyl ring or the triazole ring was used in most of the metabolism studies. Difenoconazole [^{14}C] labelled in the chlorophenoxy ring was used in some of the studies.

Difenoconazole is rapidly metabolized, initially to 1-[2-chloro-4-(4-chloro-phenoxy)-phenyl]-2-(1,2,4-triazol)-1-yl-ethanol (CGA 205375) and then with cleavage of the triazole moiety from the chlorophenoxyphenyl moiety. Conjugates are formed from hydroxylated metabolites. TRR levels are higher in the liver than in other tissues. Most of the TRR is rapidly excreted.



CGA 205375

Parent difenoconazole has a tendency to fat-solubility, but it is always a minor component of the residue. The major component of the residue in most animal commodities is CGA 205375, which appears to be fat-soluble because residue concentrations in fat are approximately 3 times as high as those in muscle. However, it is not strong fat-solubility because residue concentrations in fat are less than those in kidney and much less than those in liver (typically residues in liver are 6 – 8 times as high as in the fat).

When rats were orally dosed with labelled difenoconazole it was readily absorbed followed by extensive metabolism and excretion. The following metabolites were identified in excreta: CGA 205375, 1,2,4-triazole, 2-chloro-4-(4-chlorophenoxy)-benzoic acid, 2-chloro-4-(4-chlorophenoxy)-phenyl-hydroxyacetic acid, hydroxylated difenoconazole and hydroxylated CGA 205375. Sulphate conjugates of the hydroxylated metabolites were identified in urine. (See the toxicology report for more details of laboratory animal metabolism)

When two lactating goats were orally dosed with labelled ([^{14}C]triazole and [^{14}C]phenyl) difenoconazole for 10 consecutive days at 7.5 mg/animal/day, equivalent to 4.7 and 5.6 ppm in the feed, most of the administered [^{14}C] was excreted in the faeces (75% and 67%) and urine (31% and 21%). Residues in milk reached a plateau by day 2 (phenyl) and days 4 – 7 (triazole). Of the [^{14}C] in milk, 19% and 32% were distributed into the fat portion for the triazole and phenyl labels respectively (metabolite 1,2,4-triazole is water soluble). Residues of [^{14}C] were higher in liver (0.28 and 0.26 mg/kg) than in other tissues. Metabolite CGA 205375 constituted 57 – 58% of the TRR in liver, with parent difenoconazole at 1% or less. Triazole was the major component identified in milk, constituting 47% TRR.

When four lactating goats were orally dosed with labelled ([^{14}C]triazole and [^{14}C]phenyl) difenoconazole for 4 consecutive days at 150 mg/animal/day, equivalent to 100 ppm in the feed, [^{14}C] recovery was marginal at 40 – 64%. The TRR in liver (7.5 and 6.0 mg/kg) was much higher than other tissues. CGA 205375 was the major residue in each tissue, accounting for approximately 30–70% of the TRR. Difenoconazole residues in liver (0.62 and 0.40 mg/kg) were higher than in other tissues. Difenoconazole accounted for 1.5 – 8.3% of the TRR in each of the tissues. In milk, CGA 205375 accounted for 21% and 34% of the TRR (0.38 and 0.14 mg/kg), while difenoconazole (6 – 9% TRR) and triazole (6% TRR) were minor parts of the residue.

Two lactating goats were dosed orally once daily for 4 consecutive days by gelatin capsule with 150 mg/animal/day of [^{14}C-phenyl]difenoconazole, equivalent to 100 ppm in the feed and were slaughtered approximately 6 h after the final dose for tissue collection. CGA 205375 was the major component of the residue in all tissues and milk. Parent difenoconazole was present in all tissues and milk, but never exceeding 10% of the TRR. A number of metabolites resulted from hydroxylation and conjugation with glucuronic acid, sulphate and glycine. The concentration of the main component, CGA 205375, in fat was 2.3 times its concentration in muscle, but much below its concentration in liver and similar to that in kidney, suggesting borderline fat solubility.

When 4 laying hens were orally dosed with labelled ([^{14}C]triazole and [^{14}C]phenyl) difenoconazole for 14 consecutive days at 0.55 mg/bird/day, equivalent to 5 ppm in the feed, most of the administered [^{14}C] was excreted in the faeces (> 89%). Highest TRR appeared in the kidney (0.43 and 0.49 mg/kg) and liver (0.13 and 0.13 mg/kg). Apparent plateaus for TRR in egg whites and yolks were reached after approximately 4 and 7 days of dosing respectively. The plateau TRR values in egg whites were quite different for the two labels: 0.14 mg/kg for [^{14}C]triazole label and 0.011 mg/kg for [^{14}C]phenyl label, whereas the plateau levels in the yolks were essentially the same (0.28 and 0.29 mg/kg).

When 20 laying hens were orally dosed with labelled ([^{14}C]triazole and [^{14}C]phenyl) difenoconazole for 3 consecutive days at 7.5 mg/bird/day, equivalent to 68 ppm in the feed, most of the administered [^{14}C] was excreted in the faeces (76%). Highest TRR occurred in the liver (4.3 and 4.7 mg/kg) and kidney (1.9 and 2.2 mg/kg). CGA 205375 was the major identified component in each tissue: liver (30% and 34% TRR), kidney (20% and 22%), muscle (8.8% and 35%) and fat (46% and 64%). Parent difenoconazole accounted for less than 5% TRR in each tissue. For eggs from the phenyl label treatment, CGA 205375 was the main component of the residue (73 – 83% TRR). For the triazole label, triazole accounted for 67% of TRR in egg white and 33% TRR in egg yolk, while CGA 205375 accounted for 7.8% TRR in egg white and 36% TRR in egg yolk. Approximately 4 – 5% of the TRR in egg yolks was identified as parent difenoconazole.

Five laying hens were dosed orally once daily for 4 consecutive days by gelatin capsule with 12.5 mg/bird/day of [^{14}C-triazole]difenoconazole, equivalent to 121 ppm in the feed and were slaughtered approximately 6 h after the final dose for tissue collection. Significant [^{14}C] levels appeared in all tissues (liver 13 mg/kg, muscle 4.9 mg/kg, fat 10.4 mg/kg) and eggs (whites 4.0 mg/kg, yolks 4.5 mg/kg). CGA 205375 was a major component of the residue in tissues (liver 56% TRR, muscle 24% TRR, fat 61% TRR) and egg yolk (53% TRR). Triazole was also a significant component of the residue in tissues (liver 18% TRR, muscle 55% TRR, fat 4.6% TRR) and eggs (whites 75% TRR, yolks 31% TRR). Parent difenoconazole was a minor component of the residue in liver, muscle and egg yolk (< 5%TRR) but accounted for 18% of the TRR in fat.

The metabolism of difenoconazole in rats, goats and hens is qualitatively similar.

Plant metabolism

The Meeting received plant metabolism studies with difenoconazole in tomatoes, wheat, potatoes, grapes and oilseed rape. Difenoconazole [^{14}C] labelled in the central phenyl ring, in the triazole ring or in the chlorophenoxy ring was used in the metabolism studies.

Difenoconazole is generally slowly absorbed and metabolized. In most cases, particularly for parts of the plant directly exposed to the treatment, the parent difenoconazole is the dominant part of the residue. Parts of the plant not directly exposed are more likely to contain a residue dominated by a mobile water-soluble metabolite such as triazolylalanine.

The following plant metabolites apparently do not occur as animal metabolites of difenoconazole: triazolylalanine (2-amino-3-(1,2,4]triazol)-1-yl-propionic acid), triazolyl acetic acid (1,2,4-triazol-1-yl-acetic acid) and triazolyl-lactic acid (1,2,4-triazol-1-yl-lactic acid). At least some of these metabolites are common to other fungicides containing the 1,2,4-triazole moiety.

In a tomato metabolism study in USA, tomato plants in pots in a greenhouse were foliar sprayed 6 times at 7 day intervals with [^{14}C]phenyl and [^{14}C]triazole labelled difenoconazole at the equivalent of 0.12 kg ai/ha. Parent difenoconazole was the major part of the residue on foliage. Residue levels on tomato fruits sampled 7 days after the final treatment were insufficient for identification. A field-grown tomato metabolism study produced similar results.

In another tomato metabolism study in USA, tomato plants in pots in a greenhouse were foliar sprayed 6 times at 7 day intervals with [^{14}C]triazole labelled difenoconazole at the equivalent of 0.12 kg ai/ha. In tomato fruits sampled 33 days after the final treatment, parent difenoconazole (12 – 51% TRR) and metabolite triazolylalanine (19 – 42% TRR) were major components of the residue (TRR 0.13 – 0.20 mg/kg). In a parallel study with phenyl labelled difenoconazole, tomato fruits, sampled 33 days after the final treatment, contained parent difenoconazole (66% TRR) as the major part of the residue (TRR 0.17 mg/kg). In both of these studies low concentrations (< 2% TRR) of metabolite CGA 205375 and its ketone (1-(2-chloro-4-(4-chloro-phenoxy)-phenyl)-2-(1,2,4-triazol)-1-yl-ethanone) occurred in the fruits.

In a wheat metabolism study, triazole and triazolylacetic acid were identified in the mature stalks and grain produced from [^{14}C]triazole labelled difenoconazole treated seed. Metabolite CGA 205375 was identified in wheat tops from a parallel wheat metabolism study with [^{14}C]phenyl labelled difenoconazole.

In a greenhouse wheat metabolism study in USA, spring wheat seeds were treated with [^{14}C]phenyl and [^{14}C]triazole labelled difenoconazole at 0.25 and 0.30 g ai/kg seed and grown to maturity. Parent difenoconazole and metabolite CGA 205375 were identified at low levels in wheat tops at 25% maturity (40 days post sowing).

In a greenhouse wheat metabolism study in USA, spring wheat was foliar sprayed 4 times with [^{14}C]phenyl and [^{14}C]triazole labelled difenoconazole at a rate equivalent to 0.25 kg ai/ha. Mature samples of grain were harvested 29 days after the final application. In grain from the [^{14}C-triazole]difenoconazole treated crop, triazolylacetic acid and triazole accounted for 20% and 10% of the TRR (1.4 mg/kg) respectively. In grain from the [^{14}C-phenyl]difenoconazole treated crop, the TRR (0.064 mg/kg) was much lower, demonstrating that metabolic cleavage of the compound occurred before translocation to the grain. In the mature stalks, difenoconazole accounted for 50% of the TRR (54 and 47 mg/kg) for both labels.

Parent difenoconazole was not identified in mature grain from the wheat metabolism studies.

In a greenhouse potato metabolism study in USA, potato plants were foliar sprayed 6 times with [^{14}C]chlorophenoxy labelled difenoconazole at the equivalent of 0.12 kg ai/ha per application. Very little of the [^{14}C] translocated to the tubers (TRR 0.012 mg/kg) with parent difenoconazole and two primary metabolites identified as low-level components of the residue (< 10% TRR). Parent difenoconazole was the major component (76% TRR) of the foliage residue.

In a parallel study on potatoes with [^{14}C]triazole labelled difenoconazole, triazolylalanine (79% TRR) was the major part of the residue in tubers (TRR 0.087 mg/kg). Parent difenoconazole was again the major component (71% TRR) of the foliage residue.

In a field plot grape metabolism study in USA, grape vines were foliar sprayed 5 times with [^{14}C]phenyl and [^{14}C]triazole labelled difenoconazole. Parent difenoconazole was the major component (51% and 45% TRR) of the residue (TRR 0.13 and 0.12 mg/kg) in grapes harvested 20 days after 3 and 5 sprays. None of the identified metabolites exceeded 10% of the TRR in grapes. Parent difenoconazole was also the major identified component (17% TRR) of the residue (TRR 0.047 mg/kg) in grapes harvested 77 days after the second treatment.

In a field plot oilseed rape metabolism study in Switzerland, spring rape received two foliar sprays with [^{14}C]chlorophenoxy labelled difenoconazole at the equivalent of 0.13 kg ai/ha. Parent difenoconazole was the major identified component of the residue in stalks (17% TRR), seeds (15% TRR) and pods (17% TRR) taken at mature harvest 39 days after the second application and in oil (26% TRR) produced from the seed. Metabolite CGA 205375 exceeded 10% of TRR in the stalks (14%) and pods (11%).

In a parallel oilseed rape study with [^{14}C]triazole labelled difenoconazole, parent difenoconazole was a major identified component of the residue in stalks (17% TRR), pods (14% TRR) from samples taken at mature harvest, 39 days after the second application, and in oil (84% TRR) produced from the seed. Metabolite CGA 205375 exceeded 10% of TRR in the stalks (17%) and pods (13%). Triazolylalanine, the major residue component in the seed (56% TRR) also exceeded 10% in pods (12%). Triazolylalanine was also the major residue component in the meal (56% TRR). Other identified components of the residue in the meal were triazolylacetic acid, CGA 205375 and difenoconazole.

Parent difenoconazole is the main component of the residues in those parts of the crop directly exposed to treatment. For other parts of the crop, e.g., the grain of cereals and the tubers of potatoes, the main components of the residue are translocatable metabolites, e.g., triazolylalanine, which are common to other fungicides containing the 1,2,4-triazole moiety.

Environmental fate in soil

The Meeting received information on soil aerobic metabolism and soil photolysis properties of difenoconazole as well as studies on the behaviour of difenoconazole residues in crop rotations. Difenoconazole residues are reasonably persistent in soils and are expected to be present in the soil at harvest time for treated root and tuber crops. Difenoconazole residues are also expected to persist in the soil until the sowing of rotational crops. The confined rotational crops studies demonstrate that difenoconazole itself does not appear as a residue in the rotational crop. The water-soluble and mobile metabolites triazolylalanine, triazolylacetic acid and triazolyl-lactic acid have been identified in the rotational crops.

Aerobic soil degradation rates were influenced by the nature of the soil, temperature, moisture status of the soil and dose when [^{14}C]difenoconazole was subjected to laboratory soil incubation. Estimated aerobic soil metabolism half-lives for difenoconazole at 20 °C ranged from 63 to 700 days (n=12) with a median of 181 days. After 220 – 300 days, mineralization and unextractable residues (20 – 54% of dose) were major sinks for the [^{14}C] label. The degree of mineralization was different for the phenyl and triazole label positions, e.g., 0.8 – 4.6 % of the dose for the triazole label and 3.4 – 33% for the phenyl label.

CGA 205375 and 1,2,4-triazole were identified as soil metabolites. Metabolite CGA 205375 consistently reached a maximum (expressed as parent) of 5 – 10% of the dose and had begun to decline by the end of the observation period. Metabolite 1,2,4-triazole typically reached a maximum (expressed as parent) around 20% of the dose during the observation period. The aerobic soil metabolism of the metabolites, CGA 205375 and 1,2,4-triazole, was studied separately. The major metabolite of CGA 205375 was 1,2,4-Triazole.

Difenoconazole on a soil surface was stable to photolysis during the test period of 30 days.

In rotational crops with the [^{14}C] label in the phenyl moiety, the level of carry-over residues in rotational crops was too low for characterization or identification. With the [^{14}C] label in the triazole moiety and application to bare ground at 0.13 kg ai/ha, metabolites triazolylalanine, triazolylacetic acid and triazolyl-lactic acid were identified in rotational crops: maize grain TRR 0.21 mg/kg (66% triazolylalanine 66%); wheat grain 0.34 mg/kg (44% triazolylalanine, 26% triazolylacetic acid); lettuce heads 0.017 mg/kg (31% triazolylalanine, 43% triazolyl-lactic acid; and sugar beet tops 0.029 mg/kg (25% triazolylalanine, 54% triazolyl-lactic acid).

In outdoor non-confined rotational crop studies in Germany, bare ground was treated directly with difenoconazole at a rate equivalent to 0.75 kg ai/ha and the upper 10 cm soil layer was turned over to mix in the applied material. Carrots or spinach were sown 30 – 31 days after the difenoconazole application and harvested for analysis 97–136 days (carrots) and 62 – 77 days (spinach) after the application. Residues of difenoconazole (LOQ 0.02 mg/kg) and triazolylalanine (LOQ 0.05 mg/kg) in the carrots and spinach did not exceed the LOQs. Difenoconazole residue levels in the soil were in the range 0.15 – 0.23 mg/kg during rotational crop samplings.

Methods of residue analysis

The Meeting received descriptions and validation data for analytical methods for residues of parent difenoconazole in raw agricultural commodities, processed commodities, feed commodities, animal tissues, milk and eggs. Methods were provided also for metabolite CGA 205375 in animal tissues, milk and eggs.

In the methods for plant commodities, macerated samples are typically extracted with methanol or acetonitrile and the extract is cleaned up by solvent partitions and solid phase column chromatography. The final residue may be determined by GLC with ECD or NPD or alternatively by LC-MS-MS. LOQs are typically in the 0.01 – 0.05 mg/kg range. The analytical methods for animal commodities are similar, but with extraction methods tailored for milk, eggs or animal tissues. The LOQ for milk is 0.005 mg/kg and eggs and tissues 0.01 – 0.05 mg/kg.

Analytical recovery data were satisfactory for difenoconazole and CGA 205375 (in animal commodities) for numerous commodities.

Residue methods were tested by independent laboratories unfamiliar with the analysis and were found to have satisfactory recoveries and no background interferences.

DFG Method S19 (revision) was demonstrated to be suitable for analysis of difenoconazole residues in a number of crop commodities.

The acetonitrile-water extraction of poultry tissues and eggs, as in the analytical method, was applied to liver, fat, muscle and egg yolk samples from a [^{14}C-triazole]difenoconazole metabolism study and was shown to provide comparable extraction for difenoconazole, CGA 205375 and 1,2,4-triazole with the exhaustive extraction of the metabolism study.

Stability of residues in stored analytical samples

Information was received on the freezer storage stability of parent difenoconazole residues in plant and animal commodities, and of residues of CGA 205375 in animal commodities.

Difenoconazole residues were stable in the following crop commodities for the intervals tested, some for 1 year, but most for 2 years: banana, cotton seed, cotton seed meal, cotton seed oil, lettuce, potatoes, soya beans, tomatoes, wheat forage, wheat grain and wheat straw.

Difenoconazole and metabolite CGA 205375 spiked into animal tissues (0.2 mg/kg) and milk (0.05 mg/kg) were stable when stored at or below -18 °C for approximately 10 months.

Definition of the residue

Parent difenoconazole is the dominant component of the residue in crop commodities and is a suitable analyte for enforcement purposes.

Parent difenoconazole is generally no more than a minor component in animal commodities. The major component of the residue in most animal commodities is metabolite CGA 205375 (1-[2-chloro-4-(4-chloro-phenoxy)-phenyl]-2-(1,2,4-triazol)-1-yl-ethanol).

In the goat metabolism studies, the concentration of CGA 205375 in the fat was approximately 3 times as high as in the muscle, but much lower than in the liver. In the dairy cow feeding studies, the concentration of CGA 205375 in the fat was approximately 3 times as high as in the muscle, but much lower than in the liver. In the laying hen metabolism studies, the concentration of CGA 205375 in the fat was approximately 5 – 8 times as high as in the muscle, but also much lower than in the liver. The octanol-water partition coefficient of CGA 205375 (log P_{OW}=3.8) suggests fat-solubility.

The Meeting decided the residue would be defined as fat-soluble.

The Meeting recommended a residue definition for difenoconazole.

Definition of the residue (for compliance with the MRL and for estimation of dietary intake) for plant commodities: difenoconazole.

Definition of the residue (for compliance with the MRL and for estimation of dietary intake) for animal commodities: sum of difenoconazole and 1-[2-chloro-4-(4-chloro-phenoxy)-phenyl]-2-(1,2,4-triazol)-1-yl-ethanol), expressed as difenoconazole.

The residue is fat soluble.

Results of supervised residue trial on crops

The Meeting received supervised trials data for difenoconazole uses on oranges, pome fruits (apple, pear), stone fruits (cherries, peach, plum), grapes, olives, tropical fruits (banana, mango, papaya), bulb vegetables (garlic, leek), Brassica vegetables (broccoli, Brussels sprouts, cabbages, cauliflower), watermelon, fruiting vegetables (chilli peppers, tomatoes), lettuce, soya beans, root and tuber vegetables (carrot, potato, sugar beet), stalk and stem vegetables (asparagus, celeriac, celery), cereal grains (rice, wheat) and oilseeds (rape seed, sunflower seed). Residue data were also provided on wheat straw and fodder, rice straw and fodder, sugar beet leaves and tops, oilseed rape fodder and sunflower plant and stubble.

In trials where duplicate field samples from an unreplicated plot were taken at each sampling time and analysed separately, the mean of the two results was taken as the best estimate of the residue from the plot.

Labels (or translations of labels) were available from Australia, Belgium, Brazil, Central America (Belize, Costa Rica, Dominican Republic, El Salvador, Guatemala, Honduras, Nicaragua, and Panama), France, Germany, Indonesia, Italy, Poland, Spain, Switzerland and UK describing the registered uses of difenoconazole.

Citrus fruits

In Brazil, difenoconazole may be applied to citrus trees twice at a spray concentration of 0.005 kg ai/hL with a 30 days PHI. In two trials in Brazil matching GAP and two others with a spray concentration of 0.01 kg ai/ha, difenoconazole residue levels were < 0.05 mg/kg.

The number of trials was insufficient for an orange MRL recommendation.

Pome fruit

Spanish GAP allows five applications of difenoconazole to apple or pear trees at 0.075 kg ai/ha with a PHI of 14 days. In three trials from Spain, matching GAP, difenoconazole residues in apples were 0.10, 0.14 and 0.15 mg/kg.

In two apple trials from France with application parameters matching Spanish GAP, difenoconazole residues were 0.11 and 0.28 mg/kg.

In two trials from Greece, also with application parameters matching Spanish GAP, difenoconazole residues were 0.05 and 0.13 mg/kg.

In two trials from Italy also with application conditions matching Spanish GAP, difenoconazole residues were 0.06 and 0.08 mg/kg.

In one pear trial from France and one from Greece, matching Spanish GAP, difenoconazole residues in pears were 0.07 and 0.16 mg/kg, respectively.

The Meeting decided to combine the apple and pear data to support a pome fruit MRL. Residues in the 11 trials in ranked order (median underlined) were: 0.05, 0.06, 0.07, 0.08, 0.10, 0.11, 0.13, 0.14, 0.15, 0.16 and 0.28 mg/kg.

The Meeting estimated a maximum residue level, an STMR value and an HR value for difenoconazole in pome fruit of 0.5, 0.11 and 0.28 mg/kg respectively.

Stone fruits

Polish GAP allows 3 applications of difenoconazole to cherry trees at 0.05 kg ai/ha with a PHI of 14 days.

In a cherry trial from France and two from Germany, with application conditions matching Polish GAP, difenoconazole residues in cherries were 0.08, 0.06 and 0.10 mg/kg, respectively.

Italian GAP allows 3 applications of difenoconazole to peach trees with a spray concentration of 0.0075 kg ai/hL with a PHI of 7 days. In five Italian trials matching Italian GAP, difenoconazole residues on peaches were 0.07, 0.11, 0.14, 0.14 and 0.19 mg/kg.

In a peach trial from France and two from Greece with application conditions matching Italian GAP, difenoconazole residues in peaches were 0.18, 0.16 and 0.26 mg/kg, respectively.

In summary, the difenoconazole residues on peaches from eight trials (in ranked order, median underlined) were: 0.07, 0.11, 0.14, 0.14, 0.16, 0.18, 0.19 and 0.26 mg/kg.

French GAP allows 3 applications of difenoconazole to plum trees with a spray concentration of 0.005 kg ai/hL with a PHI of 14 days. In four French trials matching GAP (accepted variation on spray concentration 0.0035 – 0.0065 kg ai/hL) difenoconazole residues on plums were 0.02, 0.03, 0.07 and 0.10 mg/kg.

In four German trials on plums with application conditions matching French GAP (accepted variation on spray concentration 0.0035 – 0.0065 kg ai/hL), difenoconazole residues were < 0.01, 0.01, 0.02 and 0.04 mg/kg.

In two Spanish trials on plums with application parameters matched French GAP, difenoconazole residues were 0.03 and 0.08 mg/kg.

In summary, the difenoconazole residues on plums from 10 trials were: < 0.01, 0.01, 0.02, 0.02, 0.03, 0.03, 0.04, 0.07, 0.08 and 0.10 mg/kg.

The data from the peaches and plums were apparently of different populations and could not be combined.

The Meeting estimated a maximum residue level, an STMR value and an HR value for difenoconazole in peaches of 0.5, 0.15 and 0.26 mg/kg respectively. These values may also be used for nectarines.

The data from plums and cherries were combined for mutual support, residues in 13 trials in ranked order (median underlined) were: < 0.01, 0.01, 0.02, 0.02, 0.03, 0.03, 0.04, 0.06, 0.07, 0.08, 0.08, 0.10 and 0.10 mg/kg.

The Meeting estimated a maximum residue level, an STMR value and an HR value for difenoconazole in plums and cherries of 0.2, 0.04 and 0.10 mg/kg respectively.

Grapes

Italian GAP allows 4 applications of difenoconazole to grape vines with a spray concentration of 0.005 kg ai/hL with a PHI of 21 days. In six Italian trials from 2003 – 2004 matching GAP, difenoconazole residues on grapes were 0.01, 0.02, 0.02, 0.03, 0.03 and 0.04 mg/kg. In two French trials matching Italian GAP, residues in grapes were 0.04 and 0.07 mg/kg.

In summary, the difenoconazole residues on grapes from eight trials in ranked order (median underlined) were: 0.01, 0.02, 0.02, 0.03, 0.03, 0.04, 0.04 and 0.07 mg/kg.

The Meeting estimated a maximum residue level, an STMR value and an HR value for difenoconazole in grapes of 0.1, 0.03 and 0.07 mg/kg respectively.

Olives

In Spain, difenoconazole may be applied to olive trees three times at a spray concentration of 0.015 kg ai/hL with a 30 days PHI. In seven trials in Spain in 2003 – 2005 matching GAP, difenoconazole residue levels were 0.22, 0.29, 0.40, 0.42, 0.51, 0.90 and 1.2 mg/kg.

In an olive trial in France with application conditions matching Spanish GAP, difenoconazole residues on olives were 0.76 mg/kg.

In summary, difenoconazole residues in olives from eight trials in ranked order (median underlined) were: 0.22, 0.29, 0.40, 0.42, 0.51, 0.76, 0.90 and 1.2 mg/kg.

The Meeting estimated a maximum residue level, an STMR value and an HR value for difenoconazole in olives of 2, 0.465 and 1.2 mg/kg respectively.

Bananas

In Costa Rica, Guatemala and Honduras difenoconazole may be applied 8 times to bananas at 0.1 kg ai/ha with harvest permitted on the day of application. The use pattern includes aerial application.

In the banana trials in 1997 in Ecuador, Colombia and Honduras, unbagged fruit were chosen for study although these cropping conditions, approved as GAP, rarely occur in commercial banana production. The trials of 1993 in Costa Rica and Guatemala included both bagged and unbagged fruits. For the purposes of estimating an MRL, only data from unbagged fruit are considered in this case.

In three banana trials in Colombia with conditions matching the GAP of Costa Rica, residues of difenoconazole in whole fruit were < 0.02, 0.02 and 0.04 mg/kg, with residues in pulp all at < 0.02 mg/kg.

In two banana trials in Costa Rica with conditions matching GAP, difenoconazole in whole fruit were 0.03 and 0.04 mg/kg, with residues in pulp both at < 0.02 mg/kg.

In three banana trials in Ecuador with conditions matching the GAP of Costa Rica, difenoconazole in whole fruit were all < 0.02 mg/kg, with residues in pulp also all at < 0.02 mg/kg.

In one banana trial from Guatemala with conditions matching the GAP of Costa Rica, difenoconazole in whole fruit were 0.07 mg/kg, with residues in pulp at < 0.02 mg/kg.

In three banana trials in Honduras with conditions matching the GAP of Costa Rica, difenoconazole in whole fruit were < 0.02, < 0.02 and 0.03 mg/kg, with residues in pulp also all at < 0.02 mg/kg.

In summary, difenoconazole residues in whole bananas from the 12 unbagged trials were: < 0.02 (5), 0.02, 0.02, 0.03, 0.03, 0.04, 0.04 and 0.07 mg/kg. Residues in banana pulp were all < 0.02 mg/kg.

The Meeting estimated a maximum residue level, an STMR value and an HR value for difenoconazole in bananas of 0.1, 0.02 and 0.02 mg/kg respectively.

Mango

In Brazil, difenoconazole may be applied to mango trees three times at a spray concentration of 0.0125 kg ai/hL with a 7 days PHI. In four trials in Brazil in 2003 matching GAP, difenoconazole residues in mango whole fruits were 0.025, 0.025, 0.035 and 0.04 mg/kg. No data were available for residues in edible portion.

The Meeting estimated a maximum residue level, an STMR value and an HR value for difenoconazole in mangos of 0.07, 0.03 and 0.04 mg/kg respectively.

Papaya

In Brazil, difenoconazole may be applied to papayas four times at a spray concentration of 0.0075 kg ai/hL with a 14 days PHI. In four trials in Brazil in 2002 matching GAP, difenoconazole residues in papaya whole fruits were 0.02, 0.03, 0.07 and 0.10 mg/kg and residues in edible portion were all < 0.01 mg/kg. In four trials where the spray concentration was 0.015 kg ai/hL (2× label) residues in whole papaya fruit were 0.09, 0.09 0.12 and 0.20 mg/kg and residues in edible portion were < 0.01 (3) and 0.02 mg/kg, suggesting residues could occur in the edible portion, i.e., not a nil residue.

The double rate trials provided additional support, particularly in cases such as this for difenoconazole where the residue is generally external and essentially non-systemic.

The Meeting estimated a maximum residue level, an STMR value and an HR value for difenoconazole in papaya of 0.2, 0.01 and 0.02 mg/kg respectively.

Garlic

In Brazil, difenoconazole may be applied to garlic crops six times at a rate of 0.13 kg ai/ha with a 14 days PHI. In four trials in Brazil in 1995 with 6 applications of 0.19 or 0.38 kg ai/ha (1.5× and 3× label rates), difenoconazole residues in bulbs of garlic were all < 0.02 mg/kg at PHIs of approximately 0, 7, 14 and 21 days.

Data from the exaggerated rates and various sampling intervals suggest that difenoconazole residues do not reach garlic bulbs.

The Meeting estimated a maximum residue level, an STMR value and an HR value for difenoconazole in garlic of 0.02*, 0 and 0 mg/kg respectively.

Leeks

In Germany, difenoconazole may be applied to leek crops 3 times at a rate of 0.1 kg ai/ha with a 21 days PHI. In four trials in Germany with application in line with GAP, difenoconazole residues in whole plants with roots removed were 0.02, 0.07, 0.09 and 0.12 mg/kg.

In four leek trials in France with conditions matching German GAP, difenoconazole residues in whole plants were 0.03, 0.05, 0.13 and 0.21 mg/kg.

In two leek trials from Italy with conditions matching German GAP, difenoconazole residues in whole plants were 0.14 and 0.17 mg/kg.

In two leek trials from Switzerland with conditions matching German GAP, difenoconazole residues in edible portions were 0.02 and 0.04 mg/kg.

The Meeting accepted that the three descriptions of the commodity analysed, i.e., (1) whole plants with roots removed, (2) whole plants and (3) edible parts, were all intended to agree with the Codex description of the commodity for analysis: *Whole vegetable after removal of roots and adhering soil*.

In summary, difenoconazole residue in leeks from the 12 trials, in rank order (median underlined), were: 0.02, 0.02, 0.03, 0.04, 0.05, <u>0.07</u>, <u>0.09</u>, 0.12, 0.13, 0.14, 0.17 and 0.21 mg/kg.

The Meeting estimated a maximum residue level, an STMR value and an HR value for difenoconazole in leeks of 0.3, 0.08 and 0.21 mg/kg respectively.

Broccoli

In Belgium, difenoconazole may be applied twice to broccoli at a rate of 0.13 kg ai/ha with a 14 days PHI. In trials in France, Netherlands and Spain, difenoconazole was applied 3 times rather than twice. Difenoconazole is a reasonably persistent residue as found in the decline trials with residue remaining on the whole plant just prior to the final application. However, carryover on the flower heads is not expected as they were unlikely to be formed at the time of the first application.

In four broccoli trials in France with conditions matching Belgian GAP, except for 3 applications instead of 2, difenoconazole residues in flower heads on days 13 – 15 after the final application were 0.02, 0.05, 0.08 and 0.10 mg/kg.

In two broccoli trials from The Netherlands, with conditions matching Belgian GAP except for 3 applications instead of 2, difenoconazole residues in flower heads on day 14 after the final application were < 0.02 and 0.03 mg/kg.

In two broccoli trials in Spain, with conditions matching Belgian GAP except for 3 applications instead of 2, difenoconazole residues in flower heads on day 14 and day 21 (higher residues than on day 14) after the final application were 0.41 and 0.15 mg/kg.

In summary, difenoconazole residue in broccoli flower heads from the eight trials, in ranked order (median underlined), were: 0.02, 0.02, 0.03, <u>0.05</u>, <u>0.08</u>, 0.10, 0.15 and 0.41 mg/kg.

The Meeting estimated a maximum residue level, an STMR value and an HR value for difenoconazole in broccoli of 0.5, 0.065 and 0.41 mg/kg respectively.

Brussels sprouts

In France, difenoconazole may be applied to Brussels sprouts 3 times at a rate of 0.13 kg ai/ha with a 21 days PHI.

In four Brussels sprouts trials from Belgium in 1999, with conditions in line with French GAP, difenoconazole residues in buttons on days 20 – 21 and 28 (higher residues than on day 21) after the final application were 0.02, 0.05, 0.07 and 0.09 mg/kg.

In eight Brussels sprouts trials in the UK, with conditions matching French GAP, difenoconazole residues in buttons on days 21 – 22 after the final application were 0.04, 0.05, 0.05, 0.06, 0.07, 0.08, 0.10 and 0.14 mg/kg.

In summary, difenoconazole residues in Brussels sprouts buttons from the 12 trials, in ranked order (median underlined), were: 0.02, 0.04, 0.05, 0.05, 0.05, <u>0.06</u>, <u>0.07</u>, 0.07, 0.08, 0.09, 0.10 and 0.14 mg/kg.

The Meeting estimated a maximum residue level, an STMR value and an HR value for difenoconazole in Brussels sprouts of 0.2, 0.065 and 0.14 mg/kg respectively.

Cabbage

In France, difenoconazole may be applied to cabbage 3 times at a rate of 0.13 kg ai/ha with a 21 days PHI. In six trials from France, with application parameters in line with GAP, difenoconazole residues in cabbage heads were < 0.01 (2), 0.01, < 0.02 and < 0.05 (2) mg/kg.

In Germany, difenoconazole may be applied to cabbage 3 times at a rate of 0.1 kg ai/ha with a 21 days PHI. In two trials in Germany, with trial parameters in line with GAP, difenoconazole residues in cabbage heads were < 0.02 (2) mg/kg.

In five cabbage trials in Belgium in 1999, with conditions in line with French GAP, difenoconazole residues in cabbage heads on day 21 after the final application were < 0.02 (5) mg/kg.

In two cabbage trials in Germany in 2003, with conditions in line with French GAP, difenoconazole residues in cabbage heads on day 21 after the final application were < 0.02 and 0.19 mg/kg.

In two cabbage trials in The Netherlands in 2002, with conditions in line with French GAP, difenoconazole residues in cabbage heads on day 21 after the final application were < 0.02 (2) mg/kg.

In three cabbage trials in UK in 1990 with conditions in line with French GAP, difenoconazole residues in cabbage hearts on day 21 after the final application were 0.06, 0.10 and 0.13 mg/kg. The Meeting accepted that cabbage "hearts" meant the same as cabbage "heads".

In summary, difenoconazole residues in cabbages from the 20 trials, in rank order (median underlined), were: < 0.01 (3), 0.01, < 0.02 (10), < 0.05 (2), 0.06, 0.10, 0.13 and 0.19 mg/kg.

The Meeting estimated a maximum residue level, an STMR value and an HR value for difenoconazole in head cabbage of 0.2, 0.035 and 0.19 mg/kg respectively.

Cauliflowers

In France, difenoconazole may be applied to cauliflowers 3 times at a rate of 0.13 kg ai/ha with a 14 days PHI. In 12 trials from France matching GAP, difenoconazole residues in the flower heads were 0.01, < 0.02 (9), 0.03 and 0.10 mg/kg.

In a cauliflower trial in Switzerland in 2005, with conditions in line with French GAP, difenoconazole residues in flower heads on day 14 after the final application were < 0.01 mg/kg.

In two cauliflower trials in the UK in 1999 and 2005, with conditions matching French GAP, difenoconazole residues in flower heads on day 14 after the final application were < 0.02 and 0.02 mg/kg.

In summary, difenoconazole residues in cauliflowers from the 15 trials, in ranked order (median underlined), were: < 0.01, 0.01, < 0.02 (10), 0.02, 0.03 and 0.10 mg/kg.

The Meeting estimated a maximum residue level, an STMR value and an HR value for difenoconazole in cauliflowers of 0.2, 0.02 and 0.10 mg/kg respectively.

Watermelons

Residue data were available only on the edible portion of the watermelons in the four trials provided, so estimation of an MRL was not possible.

Chilli peppers

In Indonesia, difenoconazole may be applied at 7 day intervals to chilli pepper crops at a spray concentration of 0.0063 – 0.013 kg ai/hL with no required PHI.

One trial from Indonesia matched GAP for maximum spray concentration with harvest on day 6 after treatment. A second Indonesian trial used a spray concentration of 0.025 kg ai/hL (2× label rate). One trial from Malaysia matched Indonesian GAP for maximum spray concentration and harvest on the day of treatment. A second Malaysian trial used a spray concentration of 0.025 kg ai/hL (2× label rate).

The Meeting agreed that, for a minor use, a minimum of three trials matching GAP conditions is needed. The Meeting was not able to recommend a maximum residue level for difenoconazole residues in chilli peppers.

Tomatoes

In Italy, difenoconazole may be applied to tomato crops 4 times at a rate of 0.13 kg ai/ha with a 7 days PHI.

In two tomato trials (glasshouse and polytunnel) in France in 2005, with conditions in line with Italian GAP, difenoconazole residues in tomatoes on day 7 after the final application were 0.04 and 0.05 mg/kg.

In five tomato trials (field) in Greece in 2001 – 2003, with conditions in line with Italian GAP, difenoconazole residues in tomatoes on day 7 and 10 (higher residues than on day 10) after the final application were 0.10, 0.13, 0.18, 0.28 and 0.36 mg/kg.

In a tomato trial (glasshouse) in UK in 2005, with conditions in line with Italian GAP, difenoconazole residues in tomatoes on day 7 after the final application were 0.10 mg/kg.

In two tomato trials (field) in Spain in 2003, with conditions in line with Italian GAP, difenoconazole residues in tomatoes on day 7 after the final application were 0.03 and 0.09 mg/kg.

In a tomato trial (polytunnel) in Spain in 2005, with conditions in line with Italian GAP, difenoconazole residues in tomatoes on day 7 after the final application were 0.12 mg/kg.

In summary, difenoconazole residues in tomatoes from the field trials were: 0.03, 0.09, 0.10, 0.13, 0.18, 0.28 and 0.36 mg/kg; and from protected trials were: 0.04, 0.05, 0.10, and 0.12 mg/kg. The data appear to be from similar populations and can be combined.

In summary, difenoconazole residues in tomatoes from the 11 trials, in ranked order (median underlined), were: 0.03, 0.04, 0.05, 0.09, 0.10, 0.10, 0.12, 0.13, 0.18, 0.28 and 0.36 mg/kg.

The Meeting estimated a maximum residue level, an STMR value and an HR value for difenoconazole in tomatoes of 0.5, 0.10 and 0.36 mg/kg respectively.

Lettuce

In Spain, the registration document states that difenoconazole is registered for use on lettuce at a rate of 0.13–0.20 kg ai/ha with a 14 days PHI. The maximum application rate on the available label was 0.13 kg ai/ha. The Meeting agreed to use the GAP from the registration document.

In eight lettuce trials from Spain in 1991 and 2003 with application rates of 0.1 – 0.18 kg ai/ha (within 30% of GAP rate) the residues 13 – 14 days after the final application, in ranked order (median underlined), were: 0.07, 0.08, 0.29, 0.31, 0.51, 0.56, 0.65 and 1.0 mg/kg.

The Meeting estimated a maximum residue level, an STMR value and an HR value for difenoconazole in head lettuce and leaf lettuce of 2, 0.41 and 1.0 mg/kg respectively.

Soya beans

In Brazil, difenoconazole may be applied to soya bean crops once at a rate of 0.075 kg ai/ha with a 30 days PHI. In six soya bean trials in 2000 and 2003 in Brazil with conditions in line with GAP, except that there were 2 applications in place of 1, difenoconazole residues in the dry beans on day 30 and 31 after the final application were < 0.01 (3) and < 0.02 (3) mg/kg.

The Meeting estimated a maximum residue level and an STMR value for difenoconazole in soya beans of 0.02* and 0.02 mg/kg respectively.

Carrots

In France, difenoconazole may be applied to carrot crops 3 times at a rate of 0.13 kg ai/ha with a 14 days PHI. In nine carrot trials in 1991 – 1993, 1996 and 2000 in France, with conditions in line with GAP, difenoconazole residues in the carrots on days 14 or 15 after the final application were 0.02, 0.02, 0.03, 0.03, 0.04, 0.05, 0.07, 0.11 and 0.13 mg/kg.

In two carrot trials in 1987 in Switzerland, with conditions in line with French GAP, difenoconazole residues in carrots on day 14 after the final application were 0.07 and 0.12 mg/kg.

In summary, difenoconazole residues in carrots from the 11 trials, in rank order (median underlined), were: 0.02, 0.02, 0.03, 0.03, 0.04, 0.05, 0.07, 0.07, 0.11, 0.12 and 0.13 mg/kg.

The Meeting estimated a maximum residue level, an STMR value and an HR value for difenoconazole in carrots of 0.2, 0.05 and 0.13 mg/kg respectively.

Potatoes

In Spain, difenoconazole may be applied to potato crops 4 times at a rate of 0.2 kg ai/ha with a 30 days PHI. In seven potato trials in 2003 and 2005 in Spain with conditions in line with GAP except that only 2 applications were made, difenoconazole residues in the potato tubers on days 27 – 31 after the second and final application were < 0.01 (6) and 0.01 mg/kg.

In a trial in 2005 in Italy with the application rate in line with Spanish GAP, difenoconazole residues in potato tubers on day 29 after the second application were < 0.01 mg/kg.

The potato metabolism studies suggest that parent difenoconazole residues in tubers should be below LOQ. However, residues might be occasionally expected in tubers with surface exposure to spray application.

In summary, difenoconazole residues in potatoes from the eight trials, in rank order (median underlined), were: < 0.01 (7), 0.01 mg/kg.

The Meeting estimated a maximum residue level, an STMR value and an HR value for difenoconazole in potatoes of 0.02, 0.01 and 0.01 mg/kg respectively.

Sugar beet

In Germany, difenoconazole may be applied to sugar beet crops twice at a rate of 0.1 kg ai/ha with a 28 days PHI. In 14 sugar beet trials in 1987 – 88 and 1995 – 96 in Germany with conditions in line with GAP except that in some trials 3 applications were made, difenoconazole residues in the sugar beet roots on days 27 – 30, or later if higher residues, after the second application were < 0.02 (4), 0.02 (4), 0.03, 0.03, 0.06, 0.08, 0.08 and 0.10 mg/kg.

In three sugar beet trials in 1985 and 1991 in France, with conditions in line with German GAP, difenoconazole residues in sugar beet tubers on days 25, 29 and 33 after the second application were all < 0.02 mg/kg.

In a sugar beet trial in Denmark with conditions matching German GAP, difenoconazole residue in sugar beet root 37 days after the second application was 0.08 mg/kg.

In a sugar beet trial in the UK with conditions matching German GAP, difenoconazole residue in sugar beet root 35 days after the second application was 0.08 mg/kg.

In summary, difenoconazole residues in sugar beet from the 19 trials, in ranked order (median underlined), were: 0.01, < 0.02 (7), 0.02 (4), 0.03, 0.033, 0.06, 0.08, 0.08, 0.08 and 0.10 mg/kg.

The Meeting estimated a maximum residue level and an STMR value for difenoconazole in sugar beet of 0.2 and 0.02 mg/kg respectively.

Asparagus

In France, difenoconazole may be applied to asparagus crops 3 times at 0.13 kg ai/ha. In asparagus crops protected by 6 to 8 applications of fungicide per year, the difenoconazole product should be used for the first three treatments and other products that act in a different way should be used to complete the season.

In four asparagus trials in France, two in Italy and two in Switzerland where difenoconazole was applied 4 – 8 times at 0.13 kg ai/ha and asparagus shoots were harvested for analysis 179 –

290 days later (approximating French GAP), the resulting difenoconazole residues were < 0.02 (7) and 0.02 mg/kg.

The Meeting estimated a maximum residue level, an STMR value and an HR value for difenoconazole in asparagus of 0.03, 0.02 and 0.02 mg/kg respectively.

Celeriac

In Belgium, difenoconazole may be applied to celeriac 4 times at a rate of 0.13 kg ai/ha with a 14 days PHI. In three Belgian trials matching GAP, difenoconazole residues in celeriac roots 15 days after the final treatment were 0.08, 0.12 and 0.22 mg/kg.

The Meeting acknowledged that celeriac is a minor crop and decided to estimate an MRL based on the three trials. The Meeting estimated a maximum residue level, an STMR value and an HR value for difenoconazole in celeriac of 0.5, 0.12 and 0.22 mg/kg respectively.

Celery

In France, difenoconazole may be applied to celery crops 4 times at a rate of 0.13 kg ai/ha with a 14 days PHI.

The Codex description of the sample to be analysed is: "*Whole commodity as marketed after removal of obviously decomposed or withered leaves.*" For celery, the commodity marketed is usually trimmed celery, i.e., most foliage removed. In a number of the celery trials, leaf and stems had been detached and analysed separately. The Meeting agreed to use the stem data where stems and leaf were analysed separately.

In four celery trials in 2003 – 04 in France, with conditions in line with GAP, difenoconazole residues in celery stems on day 14 after the final application were 0.03, 0.04, 0.14 and 0.26 mg/kg.

In two celery trials in 1990 in Italy, with conditions in line with French GAP, difenoconazole residues in celery edible parts and celery stems on day 14 after the final application were 1.2 and 2.0 mg/kg respectively.

In two celery trials in 2004 in Spain and one in Switzerland in 1988, with conditions in line with French GAP, difenoconazole residues in celery stems on day 14 after the final application were 0.04, 0.05 and 0.17 mg/kg. Data from a second trial in Switzerland were not used because difenoconazole residues (0.02 mg/kg) in a sample from the control plot were significant with respect to the residue (0.058 mg/kg) in the treated plot.

In summary, difenoconazole residues in celery from the nine trials, in ranked order (median underlined), were: 0.03, 0.04, 0.04, 0.05, <u>0.14</u>, 0.17, 0.26, 1.2 and 2.0 mg/kg.

The Meeting estimated a maximum residue level, an STMR value and an HR value for difenoconazole in celery of 3, 0.14 and 2.0 mg/kg respectively.

Rice

In Indonesia, difenoconazole may be applied to rice at 0.050 to 0.10 kg ai/ha, with one application at the mid booting stage (45 days after sowing) and one at the 75% flowering stage (approximately 60 days after sowing). These growth stages are interpreted as equivalent to BBCH 43 – 45 and BBCH 63 – 67 growth stages.

In two rice trials in Indonesia with application rates of 0.063 kg ai/ha (37% below maximum GAP) and with timing to match GAP, residues in rice grain were 1.3 and 0.75 mg/kg.

In three rice trials in Malaysia with application rates of 0.064 – 0.075 kg ai/ha and timing to match Indonesian GAP, difenoconazole residues in rice grain harvested 28–30 days after the second application were 0.15, 0.16 and 0.37 mg/kg. In another trial in Malaysia at 0.12 kg ai/ha and with similar timing, residues of difenoconazole in rice grain were 0.76 mg/kg.

In summary, difenoconazole residues in rice grain from the six trials were: 0.15, 0.16, 0.37, 0.75, 0.76 and 1.3 mg/kg.

The Meeting decided that six trials (some at application rates not close enough to maximum GAP) were insufficient for a major commodity such as rice and did not estimate a maximum residue level.

Wheat

In Switzerland, difenoconazole may be applied once to wheat crops at a rate of 0.13 kg ai/ha up to growth stage BBCH 61.

In three wheat trials in Denmark, three in France and one in Switzerland where the difenoconazole was applied at 0.13 kg ai/ha up to growth stage BBCH 61, residues of difenoconazole in wheat grain were all < 0.02 mg/kg.

In nine wheat trials in France and seven in the UK, where the difenoconazole was applied at 0.12 – 0.15 kg ai/ha from growth stages BBCH 61 to 87, residues of difenoconazole in wheat grain were also all < 0.02 mg/kg.

In summary, difenoconazole residues in wheat grain from the 23 trials were all < 0.02 mg/kg.

The metabolism studies suggest that parent difenoconazole residues should not occur in the grain. The Meeting agreed that the evidence supported an STMR of nil residues in wheat.

The Meeting estimated a maximum residue level and an STMR value for difenoconazole in wheat of 0.02* and 0 mg/kg respectively.

Rapeseed

In the UK, difenoconazole may be applied twice to oilseed rape crops at a rate of 0.13 kg ai/ha up to the end of flowering (growth stage BBCH 69).

In four oilseed rape trials in 1996 in Germany, with conditions in line with GAP of the UK, difenoconazole residues in rape seed on days 56 – 80 after the second application were all < 0.02 mg/kg.

In three oilseed rape trials in 1997 in Germany with the second of two applications of difenoconazole of 0.13 kg ai/ha at growth stages BBCH 69 – 75, i.e., later than approved in UK GAP, difenoconazole residues in rape seed on days 55 – 56 after the second application were all < 0.02 mg/kg.

In two oilseed rape trials in 1988 in France with two applications of difenoconazole of 0.13 kg ai/ha and harvest 83 days after the second application (probably before end of flowering), i.e., within the conditions of UK GAP, difenoconazole residues in rape seed were both 0.04 mg/kg.

In summary, difenoconazole residues in rape seed from the nine trials, in ranked order (median underlined), were: < 0.02 (7), 0.04 and 0.04 mg/kg.

The Meeting estimated a maximum residue level and an STMR value for difenoconazole in rape seed of 0.05 and 0.02 mg/kg respectively.

Sunflower seed

In Switzerland, difenoconazole may be applied once to sunflower crops at a rate of 0.13 kg ai/ha up to growth stage BBCH 51. In three trials on sunflower in 2004 – 2005 in Switzerland according to the conditions of GAP, except that 2 applications were made instead of 1, difenoconazole residues in sunflower seed, harvested 68 – 73 days after the second application were all < 0.01 mg/kg.

In six sunflower trials in 2004 – 05 in France, with conditions matching Swiss GAP, except for 2 applications instead of 1, difenoconazole residues in sunflower seed harvested 59 – 101 days after the second application were < 0.01 (5) and 0.01 mg/kg.

In two sunflower trials in 2005 in Spain with conditions matching Swiss GAP, except for 2 applications instead of 1, difenoconazole residues in sunflower seed harvested 74 and 87 days after the second application were both < 0.01 mg/kg.

In summary, difenoconazole residues in sunflower seed from the 11 trials, in ranked order (median underlined), were: < 0.01 (10), 0.01 mg/kg.

The Meeting estimated a maximum residue level and an STMR value for difenoconazole in sunflower seed of 0.02 and 0.01 mg/kg respectively.

Wheat straw and fodder

In Switzerland, difenoconazole may be applied once to wheat crops at a rate of 0.13 kg ai/ha up to growth stage BBCH 61. In a Swiss trial on wheat in 1989 with conditions matching GAP, difenoconazole residues in wheat straw harvested 45 days after the single application were 1.2 mg/kg.

In three wheat trials in 1989 – 1990 in Denmark with conditions in line with Swiss GAP, difenoconazole residues in wheat straw on days 57, 58 and 75 after the single application were 0.26, 0.64 and 0.31 mg/kg.

In two wheat trials in 1989 in France, with conditions in line with Swiss GAP, difenoconazole residues in wheat straw on days 57 and 63 after the single application were 0.73 and 0.82 mg/kg.

In summary, difenoconazole residues in wheat straw from the six trials, in ranked order (median underlined), were: 0.26, 0.31, 0.64, 0.73, 0.82 and 1.2 mg/kg.

The Meeting estimated a maximum residue level, an STMR value and a highest residue value for difenoconazole in wheat straw and fodder of 3, 0.685 and 1.2 mg/kg respectively.

Sugar beet leaves or tops

In Germany, difenoconazole may be applied to sugar beet crops twice at a rate of 0.1 kg ai/ha with a 28 days PHI. In 14 sugar beet trials in 1987 – 1988 and 1995 – 1996 in Germany with conditions in line with GAP except that in some trials 3 applications were made, difenoconazole residues in the sugar beet leaves or tops on days 27 – 30 after the second application were 0.084, 0.087, 0.09, 0.11, 0.20, 0.25, 0.25, 0.26, 0.43, 0.43, 0.47, 0.53, 0.62 and 0.95 mg/kg.

In a sugar beet trial in 1985 in France, with conditions in line with German GAP, difenoconazole residues in sugar beet leaves 24 days after the second application were 0.17 mg/kg.

In a sugar beet trial in Denmark with conditions matching German GAP, difenoconazole residues in sugar beet leaves 37 days after the second application were 0.45 mg/kg.

In a sugar beet trial in the UK, with conditions matching German GAP, difenoconazole residues in sugar beet leaves 27 days after the second application were 0.09 mg/kg.

In summary, difenoconazole residues in sugar beet leaves or tops from the 17 trials in ranked order (median underlined), were: 0.084, 0.087, 0.09, 0.09, 0.11, 0.17, 0.20, 0.25, 0.25, 0.26, 0.43, 0.43, 0.45, 0.47, 0.53, 0.62 and 0.95 mg/kg.

The Meeting estimated an STMR value and a highest residue value for difenoconazole in sugar beet leaves or tops of 0.25 and 0.95 mg/kg (fresh weight), respectively.

Fate of residues during processing

The Meeting received information on the fate of difenoconazole residues during the processing of apples for juice, carrots for juice and canning, grapes for wine and dried grapes, olives for oil, rape seed for oil, sugar beet for sugar and molasses, and tomatoes for juice and puree. Also information was provided on hydrolysis studies of difenoconazole to assist with identification of the nature of the residue during processing.

Processing factors have been calculated for difenoconazole residues in apples, carrots, grapes, olives and tomatoes. The data for rape seed and sugar beet could not be used as residue levels in the raw commodity did not exceed the LOQ.

Difenoconazole was stable under the hydrolysis conditions (pH, temperature, time) representing the food processes pasteurisation, baking, brewing and boiling and sterilisation.

Apples from difenoconazole field trials at exaggerated application rates were washed, sliced and pressed to separate pomace from juice. The juice was pasteurised at 80 – 82 °C for 30 minutes. Puree was produced by boiling washed apples until the puree passed through a sieve. Sugar, citric acid and ascorbic acid were added until the puree reached a pH of 3.0 – 4.5 and then was heated at 95 °C for 20 minutes.

In a grape drying trial in Chile, grapes were harvested 63 days after the third of 3 applications of difenoconazole at 1× and 5× the label rate. The grapes were washed for about one minute and then placed in wooden trays with mesh bottoms and subjected to sulphur dioxide fumigation for 12 h. The trays of grapes were then dried in ovens at 65 °C for about 36 – 40 h losing approximately two-thirds of their weight, 30 kg grapes producing 10 kg dried grapes.

Wine was produced from grapes in a series of supervised field trials in France and Spain. Difenoconazole residues appeared in the pomace, but not in the wine. In grape trials in Chile, difenoconazole residues appeared in the pomace, but not in the juice.

Olives from a difenoconazole field trial at an exaggerated rate (2×) were processed into virgin oil and refined oil. The virgin oil was separated by centrifuging the mixture of olive pulp (from milling) and added water. The oil was refined by a sodium hydroxide process to produce soap from free acids. Residue levels in virgin and refined oil were essentially the same.

In a tomato processing trial in France, tomatoes were harvested 7 days after the final of 3 applications of difenoconazole at 0.37 kg ai/ha. In processing to juice, unwashed tomatoes were crushed and sieved to produce juice and pomace. Finished juice was produced by pasteurization for 1 minute at 82 – 85 °C after citric acid and salt were added to raw juice. In the production of puree, unwashed tomatoes were crushed and concentrated in a saucepan and then sieved. Salt and citric acid were added and the puree, in glass jars, was sterilised for 10 minutes at 115 °C. In the simulation of canning, unwashed tomatoes were blanched and then immediately plunged into cold water to split and loosen the peel which was removed with a knife. The peeled tomatoes, in glass jars, were covered with tomato juice and sterilised for 10 minutes at 115 – 120 °C.

In a carrot processing trial in France, carrots were harvested 7 days after the final of 3 difenoconazole applications at 0.50 kg ai/ha. In the simulation of canning, carrots were sorted and peeled with both ends removed. The peeled carrots were washed thoroughly and blanched in boiling water for 1 minute and placed in jars with brine and added citric acid to produce a pH of 3.5 and then sealed and sterilized for 10 minutes at 115 – 120 °C. For cooked carrots, the washed carrots were cooked in boiling water for 15 minutes and packaged in plastic bags under vacuum. For juicing, carrots were washed thoroughly after sorting, peeling and end removal and were then processed in a juice extractor which separated juice from pulp in a centrifugal filter. After the pH of the juice was adjusted to 3.5 with citric acid, the juice was pasteurized at approximately 85 °C and packaged in glass jars.

Calculated processing factors and the median or best estimate are summarized in the following table.

Raw agricultural commodity (RAC)	Processed commodity	Calculated processing factors.	Median or best estimate
Apple	juice	< 0.02, < 1.0. < 1.0	< 0.02
Apple	dry pomace	15.4	15
Apple	puree	0.14	0.14
Carrot	canned	0.02, 0.03, 0.05, 0.12	0.04
Carrot	juice	0.02, 0.05, 0.06, 0.12	0.055
Grapes	juice	< 0.5	< 0.5
Grapes	dry pomace	9.3, 10.3, 14.0, 15.4	12
Grapes	dried grapes	1.01, 1.4	1.2

Raw agricultural commodity (RAC)	Processed commodity	Calculated processing factors.	Median or best estimate
Grapes	wine	< 0.18, < 0.20, < 0.20, < 0.29, < 0.33, < 0.33, < 0.33, < 0.50, < 0.50, < 0.50, < 0.50	< 0.18
Olives	refined oil	1.19, 1.40, 1.50, 1.51	1.4
Olives	virgin oil	1.47, 1.50, 1.50, 1.63	1.5
Tomatoes	canned tomato	< 0.05, 0.06, 0.07, 0.08	0.065
Tomatoes	juice	0.14, 0.15, 0.28, 0.32	0.22
Tomatoes	puree	0.54, 0.58, 0.74, 1.00	0.66

The processing factors for dry apple pomace (15), apple juice (< 0.02) and apple puree (0.14) were applied to the estimated STMR for pome fruits (0.11 mg/kg) to produce STMR-P values for dry apple pomace (1.65 mg/kg), apple juice (0.0022 mg/kg) and apple puree (0.015 mg/kg).

The processing factors for dry grape pomace (12), grape juice (< 0.5) and wine (< 0.18) were applied to the estimated STMR for grapes (0.03 mg/kg) to produce STMR-P values for dry grape pomace (0.36 mg/kg), grape juice (0.015 mg/kg) and wine (0.0054 mg/kg).

The processing factor for dried grapes (1.2) was applied to the estimated STMR and HR for grapes (0.03 and 0.07 mg/kg) to produce STMR-P and HR-P values for dried grapes (raisins) of 0.036 and 0.084 mg/kg respectively.

The Meeting estimated a maximum residue level for difenoconazole in dried grapes (= currants, raisins, sultanas) of 0.1 mg/kg. The estimated maximum residue level is the same as for grapes, so no separate MRL recommendation is necessary.

The processing factors for canned carrots (0.04) and carrot juice (0.055) were applied to the estimated STMR for carrots (0.05 mg/kg) to produce STMR-P values for canned carrots (0.002 mg/kg) and carrot juice (0.0028 mg/kg).

The processing factors for tomato puree (0.66), tomato juice (0.22) and canned tomato (0.065) were applied to the estimated STMR for tomatoes (0.10 mg/kg) to produce STMR-P values for tomato puree (0.066 mg/kg), tomato juice (0.022 mg/kg) and canned tomato (0.0065 mg/kg).

The processing factors for virgin olive oil (1.5) and refined olive oil (1.4) were applied to the estimated STMR for olives (0.465 mg/kg) to produce STMR-P values for virgin olive oil (0.70 mg/kg) and refined olive oil (0.65 mg/kg)

Residues in animal commodities

Livestock feeding

The meeting received lactating dairy cow feeding studies and a laying hen feeding study, which provided information on likely residues resulting in animal commodities, milk and eggs from difenoconazole residues in the animal diet.

Lactating dairy cows

Groups of 3 lactating Holstein dairy cows were dosed once daily via gelatin capsule with difenoconazole at 1 ppm (1×), 3 ppm (3×) and 10 ppm (10×) in the dry-weight diet for 29 – 30 consecutive days. Parent difenoconazole residues did not occur above LOQ in muscle, kidney or fat tissues or milk for any of the test doses, but were present in liver from the 10 ppm feeding-level group. Metabolite CGA 205375 was present in each of the tissues from the 3 and 10 ppm feeding-level groups and in the liver and fat from the 1 ppm feeding-level animals. The concentration of metabolite CGA 205375 in fat was approximately 3.3 times its concentration in muscle. The average concentrations of metabolite CGA 205375 in the tissues from the 10 ppm feeding-level animals were: muscle 0.020 mg/kg; liver 0.30 mg/kg; kidney 0.044 mg/kg; fat 0.072 mg/kg. For metabolite CGA 205375 in liver, the transfer factors for the 3 feeding levels were reasonably consistent. For fat, the transfer factors for metabolite CGA 205375 apparently decreased as the feeding level increased.

For the 10 ppm feeding-level animals, metabolite CGA 205375 was consistently present in the milk from day 2 onwards at 0.005 – 0.009 mg/kg.

In a second study, groups of 3 lactating Holstein dairy cows were dosed once daily via gelatin capsule with difenoconazole at 1 ppm (1×), 5 ppm (5×) and 15 ppm (15×) in the dry-weight diet for 29 – 30 consecutive days. Parent difenoconazole residues did not occur above LOQ in muscle, kidney or fat tissues or milk for any of the test doses. Parent difenoconazole residues were present in liver from the 5 and 15 ppm feeding-level groups. Metabolite CGA 205375, the major part of the residue, was present in each of the tissues from the 5 and 15 ppm feeding-level animals and in the liver, kidney and fat from the 1 ppm feeding-level group. In the 15 ppm feeding-level group, the concentration of metabolite CGA 205375 in fat was approximately 3.1 times its concentration in muscle. The average concentrations of metabolite CGA 205375 in the tissues from the 15 ppm feeding-level animals were: muscle 0.04 mg/kg; liver 0.57 mg/kg; kidney 0.11 mg/kg; fat 0.12 mg/kg. For metabolite CGA 205375 in liver, the transfer factors for the 5 ppm and 15ppm feeding levels were close. For fat, the transfer factors for metabolite CGA 205375 were also consistent for the 5 ppm and 15 ppm feeding levels. Metabolite CGA 205375 reached a plateau level in milk of approximately 0.012 mg/kg within 2 days from the 15 ppm feeding-level animals. Metabolite 1,2,4-triazole (not included in the difenoconazole residue definition) was consistently present in the milk from the 5 and 15 ppm feeding levels groups where plateau concentrations in milk of approximately 0.017 mg/kg and 0.04 mg/kg respectively were quickly reached.

The two feeding studies were generally in good agreement of transfer factors. The Meeting decided to use the study with the 1 and 3 ppm feeding levels as most closely bracketing the dietary burdens.

Laying hens

Laying white leghorn hens were fed rations treated with difenoconazole at 0.3 ppm, 1 ppm, 3 ppm and 10 ppm for 28 consecutive days. Parent difenoconazole residues did not occur above LOQ (0.01 mg/kg) in muscle, fat, liver or eggs for any of the test doses. Metabolite CGA 205375 also was not present in the tissues above LOQ (0.01 mg/kg). Average levels of 1,2,4-triazole in the tissues from the 10 ppm feeding-level birds were: skin plus attached fat 0.012 mg/kg; peritoneal fat < 0.005 mg/kg; liver 0.02 mg/kg; muscle 0.022 mg/kg. Metabolite CGA 205375 occurred in eggs from the 1, 3 and 10 ppm feeding-level groups reaching a plateau after approximately 9 days with levels of 0.037 mg/kg and 0.13 mg/kg in eggs from the 3 and 10 ppm feeding-level groups respectively. At the 1 ppm feeding level, CGA 205375 was present in eggs at close to the LOQ (0.01 mg/kg). Metabolite 1,2,4-triazole occurred in eggs from the 1, 3 and 10 ppm feeding-level birds. It reached a plateau after approximately 6 days with plateau levels of 0.007, 0.020 and 0.060 mg/kg in eggs from the 1, 3 and 10 ppm feeding-level birds respectively.

Livestock dietary burden

The Meeting estimated the dietary burden of difenoconazole in livestock on the basis of the diets listed in Annex 6 of the 2006 JMPR Report (OECD Feedstuffs Derived from Field Crops). Calculation from highest residue, STMR (some bulk commodities) and STMR-P values provides the levels in feed suitable for estimating MRLs, while calculation from STMR and STMR-P values for feed is suitable for estimating STMR values for animal commodities. The percentage dry matter is taken as 100% when the highest residue levels and STMRs are already expressed as dry weight.

Estimated maximum and mean dietary burdens of livestock

Dietary burden calculations for beef cattle, dairy cattle, broilers and laying poultry are provided in Annex 6. The calculations were made according to the livestock diets from US-Canada, EU and Australia in the OECD Table (Annex 6 of the 2006 Report of the JMPR).

	Livestock dietary burden, difenoconazole, ppm of dry matter diet					
	US-Canada		EU		Australia	
	max	mean	max	mean	max	mean
Beef cattle	0.62	0.48	1.85	0.81	1.42	0.9 [b]
Dairy cattle	0.44	0.30	2.10 [a]	0.76 [c]	0.59	0.44
Poultry - broiler	0.01	0.01	0.12	0.05	0.01	0.01
Poultry - layer	0.01	0.01	0.54 [d]	0.20 [e]	0.01	0.01

a - Highest maximum beef or dairy cattle dietary burden suitable for MRL estimates for mammalian meat and milk

b - Highest mean beef or dairy cattle dietary burden suitable for STMR estimates for mammalian meat.

c - Highest mean dairy cattle dietary burden suitable for STMR estimates for milk.

d - Highest maximum poultry dietary burden suitable for MRL estimates for poultry meat and eggs.

e - Highest mean poultry dietary burden suitable for STMR estimates for poultry meat and eggs.

Animal commodities, MRL estimation

For MRL estimation, the residues in the animal commodities are the sum of difenoconazole and CGA 205375 (1-[2-chloro-4-(4-chloro-phenoxy)-phenyl]-2-(1,2,4-triazol)-1-yl-ethanol)) expressed as difenoconazole.

Cattle

For MRL estimation, the high residues in the tissues were calculated by interpolating the maximum dietary burden (2.10 ppm) between the relevant feeding levels (1 and 3 ppm) from the dairy cow feeding study and using the highest tissue concentrations from individual animals within those feeding groups.

The STMR values for the tissues were calculated by taking the STMR dietary burden (0.95 ppm) as a proportion of the lowest feeding level (1 ppm) multiplied by the feeding-level residue (mean of the 3 animals).

Residues in the milk were below LOQ (0.005 mg/kg) for all samples from the 1 ppm and 3 ppm feeding groups, so the dietary burdens (2.10 and 0.95 ppm) were taken as a proportion of the 3 ppm to calculate the residues resulting from the dietary burdens.

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.

Dietary burden (ppm) Feeding level [ppm]	Milk	Muscle	Liver	Kidney	Fat
MRL					
	mean	highest	highest	highest	highest
MRL dairy cattle (2.10) [1, 3]	< 0.004 [< 0.005, < 0.005]	0.019 [< 0.01, 0.026]	0.11 [0.051, 0.15]	0.016 [< 0.01, 0.021]	0.028 [0.015, 0.038]
STMR					
	mean	mean	mean	mean	mean
STMR beef cattle (0.95) [0, 1]		< 0.01 [0, < 0.01]	0.043 [0, 0.045]	< 0.01 [0, < 0.01]	0.012 [0, 0.013]
STMR dairy cattle (0.76) [0, 1, 3]	< 0.001 [0, < 0.005, < 0.005)]				

The data from the cattle feeding studies were used to support mammalian meat and milk MRLs.

The Meeting estimated a maximum residue level and an STMR value for difenoconazole in milks of 0.005* and 0.001 mg/kg, respectively. No information was available on the distribution of residue between the fat and non-fat milk fractions.

For muscle, the residue arising from a dietary burden of 2.10 ppm was 0.019 mg/kg, while the residue resulting from a dietary burden of 0.95 ppm was < 0.01 mg/kg. For fat, the residue arising from a dietary burden of 2.10 ppm was 0.028 mg/kg, while the residue resulting from a dietary burden of 0.95 ppm was 0.012 mg/kg.

The Meeting estimated a maximum residue level for difenoconazole in mammalian meat (fat) of 0.05 mg/kg. The Meeting estimated STMR and HR values for meat (fat) of 0.012 and 0.028 mg/kg respectively. The Meeting estimated STMR and HR values for meat (muscle) of 0.01 and 0.019 mg/kg respectively.

For liver, the residue arising from a dietary burden of 2.10 ppm was 0.11 mg/kg, while the residue resulting from a dietary burden of 0.95 ppm was 0.043 mg/kg. The Meeting estimated a maximum residue level, an STMR value and an HR value for difenoconazole in liver of 0.2, 0.043 and 0.11 mg/kg, respectively.

For kidney, the residue arising from a dietary burden of 2.10 ppm was 0.016 mg/kg, while the residue resulting from a dietary burden of 0.95 ppm was < 0.01 mg/kg. Although the residue levels in kidney were somewhat below those in liver, the Meeting decided that it was preferable to have an offal MRL which would be supported by the liver data.

The Meeting estimated a maximum residue level, an STMR value and an HR value for difenoconazole in mammalian edible offal of 0.2, 0.043 and 0.11 mg/kg, respectively.

Poultry

In the table, 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.

Dietary burden (ppm) Feeding level [ppm]	Eggs	Muscle	Liver	Fat	Skin + attached fat
MRL					
	highest	highest	highest	highest	highest
MRL laying hens (0.54) [0, 1]	0.0054 [0, 0.01]				
MRL laying hens (0.54) [0, 3, 10]		< 0.00054 [0, < 0.01, < 0.01]	< 0.00054 [0, < 0.01, < 0.01]	< 0.00054 [0, < 0.01, < 0.01]	< 0.00054 [0, < 0.01, < 0.01]
STMR					
	mean	mean	mean	mean	mean
STMR laying hens (0.20) [0, 1]	< 0.0020 [0, < 0.01]				
STMR laying hens (0.20) [0, 3, 10]		< 0.0002 [0, < 0.01, < 0.01]	< 0.0002 [0, < 0.01, < 0.01]	< 0.0002 [0, < 0.01, < 0.01]	< 0.0002 [0, < 0.01, < 0.01]

The data from the laying hen feeding studies were used to support poultry meat and egg MRLs.

The residue levels of difenoconazole + CGA 205375, expressed as difenoconazole, in poultry tissues and eggs arising from the dietary burdens (0.54 and 0.20 ppm difenoconazole in feed, dry weight) were all less than the analytical method LOQ (0.01 mg/kg).

For poultry tissues, residues were below LOQ (0.01 mg/kg) even at the 10 ppm feeding level, so an estimate of the STMRs was made by dividing the dietary burden (0.20 ppm) by 10 ppm and multiplying by the LOQ (0.01 mg/kg) to produce a value of 0.00020 mg/kg. An estimate of the HRs was made by dividing the dietary burden (0.54 ppm) by 10 ppm and multiplying by the LOQ (0.01 mg/kg) to produce a value of 0.00054 mg/kg.

For eggs, residues were below LOQ (0.01 mg/kg) at the 1 ppm feeding level, so an estimate of the STMR was made by dividing the dietary burden (0.20 ppm) by 1 ppm and multiplying by the LOQ (0.01 mg/kg) to produce a value of 0.0020 mg/kg. Similarly, a calculation for the HR for eggs produced a value of 0.0054 mg/kg.

The Meeting estimated maximum residue levels of 0.01* mg/kg for poultry eggs, poultry meat (fat) and poultry edible offal.

The Meeting estimated STMRs of 0.0020 mg/kg for eggs and 0.00020 mg/kg for poultry meat and poultry edible offal.

The Meeting estimated HRs of 0.0054 mg/kg for eggs and 0.00054 mg/kg for poultry meat and poultry edible offal.

RECOMMENDATIONS

On the basis of the data from supervised trials, the Meeting concluded that the residue concentrations listed below are suitable for establishing MRLs and for assessing IEDIs and IESTIs.

Definition of the residue (for compliance with the MRL and for estimation of dietary intake) for plant commodities: *difenoconazole.*

Definition of the residue (for compliance with the MRL and for estimation of dietary intake) for animal commodities*: sum of difenoconazole and 1-[2-chloro-4-(4-chloro-phenoxy)-phenyl]-2-(1,2,4-triazol)-1-yl-ethanol), expressed as difenoconazole.*

The residue is fat soluble.

CCN	Commodity	MRL, mg/kg	STMR or STMR-P, mg/kg	HR or HR-P, mg/kg
VS 0621	Asparagus	0.03	0.02	0.02
FI 0327	Banana	0.1	0.02	0.02
VB 0400	Broccoli	0.5	0.065	0.41
VB 0402	Brussels sprouts	0.2	0.065	0.14
VB 0041	Cabbages, head	0.2	0.035	0.19
VR 0577	Carrots	0.2	0.05	0.13
VB 0404	Cauliflowers	0.2	0.02	0.10
VR 0578	Celeriac	0.5	0.12	0.22
VS 0624	Celery	3	0.14	2.0
FS 0013	Cherries	0.2	0.04	0.10
DF 0269	Dried grapes (= currants, Raisins and Sultanas) [a]		0.036	0.084
MO 0105	Edible offal (Mammalian)	0.2	0.043	0.11
PE 0112	Eggs	0.01*	0.0020	0.0054
VA 0381	Garlic	0.02*	0	0
FB 0269	Grapes	0.1	0.03	0.07
VA 0384	Leek	0.3	0.08	0.21
VL 0482	Lettuce, Head	2	0.41	1.0
VL 0483	Lettuce, Leaf	2	0.41	1.0
FI 0345	Mango	0.07	0.03	0.04

CCN	Commodity	MRL, mg/kg	STMR or STMR-P, mg/kg	HR or HR-P, mg/kg
MM 0095	Meat (from mammals other than marine mammals)	0.05 (fat)	0.01 muscle 0.012 fat	0.019 muscle 0.028 fat
ML 0106	Milks	0.005*	0.001	
FS 0245	Nectarine	0.5	0.15	0.26
FT 0305	Olives	2	0.465	1.2
FI 0350	Papaya	0.2	0.01	0.02
FS 0247	Peach	0.5	0.15	0.26
FS 0014	Plums (including prunes)	0.2	0.04	0.10
FP 0009	Pome fruits	0.5	0.11	0.28
VR 0589	Potato	0.02	0.01	0.01
PM 0110	Poultry meat	0.01* (fat)	0.0002 muscle 0.0002 fat	0.00054 muscle 0.00054 fat
PO 0111	Poultry, Edible offal of	0.01*	0.0002	0.00054
SO 0495	Rape seed	0.05	0.02	
VD 0541	Soya bean (dry)	0.02*	0.02	
VR 0596	Sugar beet	0.2	0.02	
SO 0702	Sunflower seed	0.02	0.01	
VO 0448	Tomato	0.5	0.10	0.36
GC 0654	Wheat	0.02*	0	
AS 0654	Wheat straw and fodder, Dry	3	0.685	1.2
JF 0226	Apple juice	-	0.0022	
AB 0226	Apple pomace, dry	-	1.65	
-	Apple puree	-	0.015	
-	Carrot, canned	-	0.002	
-	Carrot, juice	-	0.0028	
JF 0269	Grape juice	-	0.015	
AB 0269	Grapes pomace, dry	-	0.36	
OR 0305	Olive oil, refined	-	0.65	
OC 0305	Olive oil, virgin	-	0.70	
JF 0048	Tomato juice	-	0.022	
-	Tomato puree	-	0.066	
-	Tomato, canned	-	0.0065	
-	Wine	--	0.0054	

* at or about the LOQ.

a - Dried grapes (= currants, Raisins and Sultanas). The estimated maximum residue level is the same as for grapes, so no separate MRL recommendation is necessary.

DIETARY RISK ASSESSMENT

Also see the General Report on triazoles.

Long-term intake

The evaluation of difenoconazole resulted in recommendations for MRLs and STMR values for raw and processed commodities. Where data on consumption were available for the listed food commodities, dietary intakes were calculated for the 13 GEMS/Food Consumption Cluster Diets. The results are shown in Annex 3 of the 2007 Report of the JMPR.

The IEDIs in the thirteen Cluster Diets, based on estimated STMRs were 1 – 10% of the maximum ADI (0.01 mg/kg bw). The Meeting concluded that the long-term intake of residues of difenoconazole from uses that have been considered by the JMPR is unlikely to present a public health concern.

Short-term intake

The IESTI of difenoconazole calculated on the basis of the recommendations made by the JMPR represented 0 – 10% of the ARfD (0.3 mg/kg bw) for children and 0 – 7% for the general population.

The Meeting concluded that the short-term intake of residues of difenoconazole resulting from uses that have been considered by the JMPR is unlikely to present a public health concern.

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03-0523	Benazeraf L	2004	Residue study with difenoconazole (CGA 169374) in or on olives in Spain. ADME - Bioanalyses, Vergèze, France. Report no. 03-0523. Unpublished.
03-0524	Benazeraf L	2004	Residue study with difenoconazole (CGA 169374) in or on olives in Spain. ADME - Bioanalyses, Vergèze, France. Report no. 03-0524. Unpublished.
03-0613	Solé C	2004	Residue study with difenoconazole (CGA 169374) in or on outdoor tomatoes in Greece. ADME - Bioanalyses, Vergèze, France. Report no. 03-0613. Unpublished.
03-0614	Solé C	2004	Residue study with difenoconazole (CGA 169374) in or on outdoor tomatoes in Greece. ADME - Bioanalyses, Vergèze, France. Report no. 03-0614. Unpublished.
04-0306	Benazeraf L	2005	Difenoconazole (GA169374): residue study in or on celery in Spain. ADME - Bioanalyses, Vergèze, France. Report no. 04-0306. Unpublished.
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04-0309	Benazeraf L	2005	Residues of difenoconazole after application of A7402T in cherries in France. North. ADME – Bioanalyses, Vergèze, France. Syngenta report no 04-0309, Syngenta. Unpublished.

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04-0404	Benazeraf L	2005	Difenoconazole (CGA 169374): residue study in or on leek in France (south) and Italy. ADME - Bioanalyses, Vergèze, France. Report no 04-0404. Includes AF/7894/SY/1, AF/7894/SY/2, AF/7894/SY/3. Unpublished.
04-0405	Benazeraf L	2005	Difenoconazole (CGA 169374): residue study in or on broccoli in France (north). ADME - Bioanalyses, Vergèze, France. Syngenta report no 04-0405. Unpublished.
04-0412	Benazeraf L	2005	Residue study with difenoconazole (CGA 169374) in or on peach in Greece. ADME – Bioanalyses, Vergèze, France. Syngenta report no 04-0412. Includes GR/FR/04-0093, GR/FR/04-0094. Unpublished.
04-0415	Benazeraf L	2005	Azoxystrobin (ICI5504) and difenoconazole (CGA 169374): residue study in or on sunflowers in northern France. ADME - Bioanalyses, Vergèze, France. Report no. 04-0415. Unpublished.
04-0416	Benazeraf L	2005	Azoxystrobin (ICI5504) and difenoconazole (CGA 169374): residue study in or on sunflowers in southern France. ADME - Bioanalyses, Vergèze, France. Report no. 04-0416. Includes FR-FR-04-0123, FR-FR-04-0124, FR-FR-04-0125, FR-FR-04-0126, Unpublished.
04-0426	Benazeraf L	2005	Difenoconazole (CGA 169374): residue study in or on broccoli in Spain. ADME - Bioanalyses, Vergèze, France. Report no. 04-0426. Includes AF/7867/SY/2, AF/7866/SY/3. Unpublished.
04-0427	Benazeraf L	2005	Difenoconazole (CGA 169374): residue study in or on celery in France (north). ADME - Bioanalyses, Vergèze, France. Report no. 04-0427. Includes AF/7868/SY/1, AF/7868/SY/2. Unpublished.
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04-6047	Richards S	2005	Fenpropidin (CGA114900) and difenoconazole (CGA 169374): residue study in or on sugar beet in France (north). Syngenta, Jealott's Hill, UK, report no. 04-6047. Unpublished.
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05-0411	Bour D	2006	Azoxystrobin (ICI5504) and difenoconazole (CGA 169374): residue study on sunflowers in France (south) and Spain. ADME - Bioanalyses, Vergèze, France. Report no. 05-0411. Includes AF/8542/SY/1, AF/8542/SY/2, AF/8542/SY/3, AF/8542/SY/4. Unpublished.

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05-0530	Bour D	2006	Difenoconazole (CGA 169374): residue study on cauliflower in Switzerland. ADME - Bioanalyses, Vergèze, France, Syngenta report no. 05-0530. Unpublished.
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202/99	Tribolet R	2000	Residues of difenoconazole (CGA 169374) and its metabolite CGA 205375 in milk, blood, and tissues (muscle, fat, liver, kidney) of dairy cattle resulting from feeding of difenoconazole at three dose levels. Novartis Crop Protection AG, Basel, Switzerland. Report 202/99. Unpublished.
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