CLOFENTEZINE (156)

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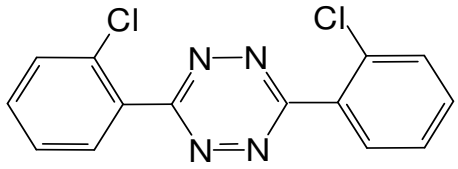
EXPLANATION

Clofentezine, an acaricide, was first evaluated by the JMPR in 1986 and re-evaluated for residues several times up to 1992. At the 37th session of the CCPR (ALINORM 05/28/24, Appendix XIV), clofentezine was scheduled for the CCPR periodic residue review by the 2007 JMPR.

For clofentezine TC and for clofentezine SC formulation specification were established through the Joint FAO/WHO Meeting on Pesticide Specification (JMPS) and published as FAO Specifications and Evaluations for Agricultural Pesticides-Clofentezine (2007) (http://www.fao.org/ag/agp/agpp/Pesticid/Default.htm).

The manufacturer supplied information on identity; metabolism and environmental fate; residue analysis; use pattern; residues resulting from supervised trials on citrus, pome fruits, stone fruits, grapes, strawberries, currants, melons, tree nuts, tomatoes, and cucumbers; and the fate of residues on apple, peach, almond and animal tissues during storage and orange, apple, grape and strawberry in processing. GAP information and enforcement methods were supplied by the manufacturer and the governments of Netherlands and Australia.

IDENTITY

ISO Common name	Clofentezine (BSI, ANSI, draft-ISO)
Chemical name	
IUPAC name	3,6-bis (2-chlorophenyl)-1,2,4,5-tetrazine
CAS name	3,6-bis (2-chlorophenyl)- 1,2,4,5-tetrazine
CAS Registry Number	74115-24-5
CIPAC Number	418
Synonyms and trade names	Apollo
Manufacturer's codes	NC21314 = SN 84866 = AE B084866
Structural formula	
Molecular formula	Empirical formula: C14H8Cl2N4
Molecular weight	303.1 g/mol
Minimum content of ai	980 g/kg

PHYSICAL AND CHEMICAL PROPERTIES

Pure active ingredient

Property	Description or result	Reference
Appearance	Magenta crystalline solid	Bright, A.A.S., 1987, R-12515
Odour	Odourless	Bright, A.A.S., 1987, R-12515
Vapour pressure (purity 99.7%):	6.0×10^{-7} Pa at 20 °C 1.4×10^{-6} Pa at 25 °C 6.1×10^{-5} Pa at 50 °C	Smeykal, H., 2000c, R-13285
Henry's law constant	0.168 Pa·m³/mol	Heimann, S., 2001, DHD-03/01

Property	Description or result	Reference
Boiling point (purity 99.7%)	The melting stage is directly followed by exothermal effect. The test substance decomposes before reaching the boiling point. No boiling point does exist.	Smeykal, H., 2000, R-13283
Melting point (purity 99.7%)	183.04 °C	Smeykal, H., 2000, R-13283
Octanol-water partition coefficient:	Log Pow = 4.1 at 40 °C at pH 2.0, 7.0 , 9.0, and	Mühlberger, B., 2001, R-13314
	Log Pow = 4.1 at 25 °C in unbuffered water.	Bright, A.A.S., Stalker, A.M., 1990, R-12519
Solubility in water at 22 °C (purity 98.2%)[a/]:	pH 5.0: 2.52 µg/L pH 7.0: < 2.0 µg/L pH 9.2: < 2.0 µg/L	Smith, S., Kelly, I.D., 1985, R-12523
Solubility in organic solvents at 20 – 25 °C (purity > 99.0%): 20 °C	Ethylacetate: 5.67 g/L n-Heptane: 111.4 mg/L	Mühlberger, B., 2001, R-13315
25 °C	Acetone: 9.3 g/L Dichloromethane: 37.4 g/L	Bright, A.A.S., 1987, R-12514A
	Ethanol: 0.49 g/L Xylene: 5.0 g/L	Bright, A.A.S., 1987, R-12514B
25 °C	DMSO: 11.8 g/L	Bright, A.A.S., 1988, R-12513
Relative density (purity 99.7%)	1.52 g/cm³ at 21.2 °C	Smeykal, H., 2000, R-13284
Absolute density (>95%)	1.51 g/cm³ at 20 °C	Johnson, M.K., 1987, R-12516
Dissociation constant in water	Due to the low solubility of clofentezine in water, as well as the hydrolytical instability at the pH range where a dissociation of clofentezine might be expected (estimated pKa > 9.0), the dissociation constant for clofentezine cannot be determined experimentally.	Heintze, A., 2003, R-16523
Hydrolysis rate in absence of light	Pre-test at 49 °C: pH 4: No significant hydrolysis in 5 days pH 7: Hydrolysis of ca. 20% within 0.1 days pH 9: DT_{50} < 0.1 days Main test at pH 7: DT_{50} = 1.1 d, DT_{90} = 3.7 d DT_{50} = 0.6 d, DT_{90} = 1.4 d at 35 °C. Clofentezine was first hydrolysed to AEC 593600 which was then rapidly hydrolysed to at least two additional radioactive fractions characterized as AEF 023666 and AEF 092117.	van der Gaauw, A., 2001, R-13318

Property	Description or result	Reference
Photolysis in water	Photochemical half-life at pH 5.05 (DT_{50}): Natural sunlight: < 7 days Dark control: > 31 days	Kelly, I.D., 1985, R-12521

a - The method for the determination of the solubility which is prescribed by the EEC directive cannot determine more accurate values for the solubility of clofentezine due to its fast hydrolysis and low solubility (Heintze, A., 2002, R-15521).

nd: not detected

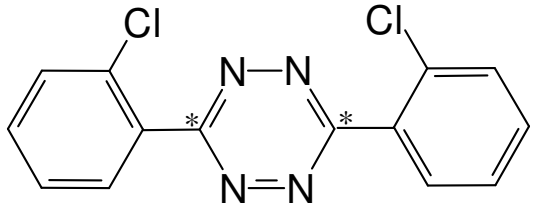
FORMULATIONS

Clofentezine is available in the following formulations:

Trade Names	Formulation		Country
	Type	Conc. (g ai/L)	
Acaristop	SC	500	Austria, Ecuador, Peru
Acaristop SC	SC	500	Colombia
Acaristop 50 SC	SC	500	Argentina, Belize, Chile, Costa Rica, Guatemala, Honduras, Mexico, Uruguay
Acaristop 500 SC	SC	500	Brazil, Moldova, Poland
Acaristop 20 SC	SC	200	Italy
Achilles	SC	500	New Zealand
Acarus	SC	500	Italy
Agristop	SC	500	Italy
Apollo	SC	500	Australia, Belgium, Denmark, Germany, Luxemburg, New Zealand, Portugal, The Netherlands, Turkey
Apollo 20 SC	SC	200	France, Italy
Apollo 50 SC	SC	500	Algeria, Bulgaria, Croatia, France, Greece, Israel, Kenya, Macedonia, Mexico,Morocco, New Zealand, Norway, Romania, Russia, Slovakia, Slovenia, Spain, Tunisia, United Kingdom, Yugoslavia
Apollo Plus	OF	60	Romania
Apollo Plus 060 OF	OF	60	Poland
Apolo Plus 60EK	OF	60	Bulgaria
Apollo SC	SC	500	Australia, Canada, Italy, South Africa, Switzerland, U.S.A
Apolo 20 SC	SC	200	Spain
Appolo	SC	500	Kazakhstan, Ukraine
Cara Flowable	WP	400	Japan
Colvert	SC	500	France
Hsine-Po-Lu	SC	500	Taiwan, China
Saran	SC	500	South Korea
Ulisses	SC	500	Portugal

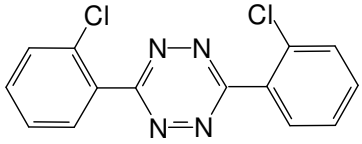
METABOLISM

The Meeting received information on animal and plant metabolism and environmental fate studies using clofentezine labelled at the tetrazine ring.

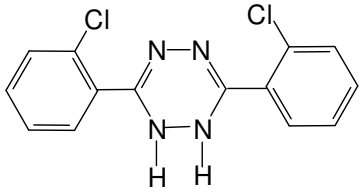


Structures, names and codes for clofentezine and its metabolites in animal, plant and environmental fate studies are summarized below.

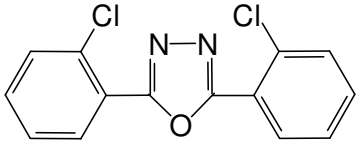
Clofentezine (NC 21 314)
3,6-bis (2-chlorophenyl)-1,2,4,5-tetrazine



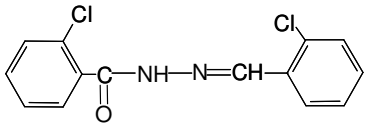
NC 22 505 (AEC 522505)
3,6-bis (2-chlorophenyl)-1,2-dihydro-1,2,4,5-tetrazine



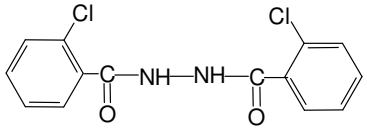
NC 12 940
3,6-bis (2-chlorophenyl)-1,3,4- oxadiazole



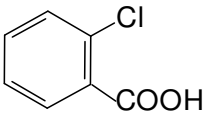
(AEC 593600) (FBC 93600)
2-chlorobenzoic (2-chlorobenzylidene) hydrazide



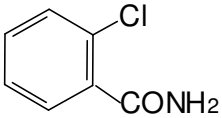
NC 12 898 (AEC 512898)
N',N-bis (2-chlorobenzoyl) hydrazine



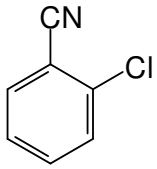
NC 233 (AEC 500233)
2-chlorobenzoic acid



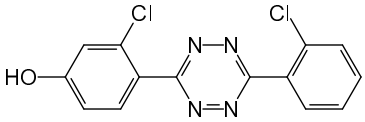
(AEF 092117)
2-chlorobenzamide



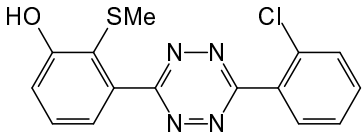
(AEF 023666)
2-chlorobenzonitrile



4-OH clofentezine



2-SMe-3-OH clofentezine
3-(2'-methylthio-3'-hydroxyphenyl)-6-(2'-chlorophenyl)-1,2,4,5-tetrazine



Animal metabolism

The Meeting received animal metabolism studies with clofentezine in mice, rats, rabbits, dogs, baboons, lactating cows, lactating goats and laying hens. Mouse, rat, rabbit, dog and baboon metabolism studies were evaluated by the WHO Core Assessment Group of the 2005 JMPR. Several comparative metabolism studies between laboratory animals and livestock are summarised at the end of this section.

Dairy cattle

A lactating Guernsey cow weighing 434 kg was orally dosed with [^{14}C]-clofentezine daily for five days at the rate of 0.27 mg/kg bw/day (Campbell, J.K., Needham, D., 1983, R-12545). According to authors of the report the dose corresponded to 22 mg/kg in apple pomace used as feed. Residues in milk were monitored for five days and the cow was killed for tissue analysis 18 h after the last dose. Residues of ^{14}C in milk reached a plateau level of approximately 0.007 mg/L on the second day with only minor variations in concentration on subsequent days (Table 1). Residues were highest in bile (1.09 mg/kg), intestinal tract (0.02 – 0.23 mg/kg) and liver (0.09 mg/kg) (Table 2).

Table 1. Residues of [^{14}C]-clofentezine equivalent in whole milk, cream and skimmed milk from a cow dosed with [^{14}C]-clofentezine for 5 days at 0.27 mg/kg bw/day

Day	TRR in whole milk (mg/L)		TRR in the cream (mg/L)		TRR in the skimmed milk (mg/L)	
	Morning	Afternoon	Morning	Afternoon	Morning	Afternoon
0[a]	NS	< 0.001	NS	< 0.001	NS	< 0.001
1	< 0.001	0.001	< 0.001	0.001	0.001	< 0.001
2	0.005	0.005	0.006	0.005	0.005	0.005
3	0.007	0.006	0.007	0.005	0.006	0.005
4	0.007	0.006	0.007	0.005	0.007	0.006
5	0.006	0.006	0.007	0.005	0.006	0.006
6 [b]	0.006		0.006		0.006	

a - Sample taken on the afternoon of the day prior to the first dose.

b - Sample taken 18 hours after the last dose.

NS: no sample

Table 2. Total radioactive residues in cow tissues dosed for 5 days with 0.27 mg/kg bw/day [^{14}C]-clofentezine

Tissue	TRR (mg/kg)	Tissue	TRR (mg/kg) (mean ± SD)
Liver	0.09 ± 0.01	Abomasum	0.02 ± 0.00
Kidney	0.04 ± 0.01	Duodenum	0.04 ± 0.00
Heart	0.01 ± 0.00	Jejunum	0.02 ± 0.00
Thyroid	0.04 ± 0.01	Ileum	0.19 ± 0.00
Ovaries	0.01 ± 0.00	Caecum	0.12 ± 0.00
Brain	0.01 ± 0.00	Colon	0.23 ± 0.00
Tongue	0.01 ± 0.00	Pancreas	0.01 ± 0.00
Rumen	0.02 ± 0.00	Bile	1.09 ± 0.13
Reticulum	0.02 ± 0.00	Other [a]	< 0.01
Omasum	0.05 ± 0.00		

a - Lung, spleen, skin, muscle, bone, fat, adrenals, mammary gland, uterus, eyes

A lactating Friesian cow of bodyweight of 480 kg was administered with [^{14}C]-clofentezine twice daily at milking by oral capsule for 3 days (2.2 mg/kg bw/day) (Phillips, M.W.A., Swalwell, L., 1988, R-12559). The cow was sacrificed 16 h after the final dose and the residues were characterized in order to elucidate the metabolism.

Residues of TRR in milk reached a plateau level of 0.18 mg equiv/L on the third day post initiation of dosing (Table 3). Methanolic soxhlet extraction liberated 93% of the activity from milk samples. A further extraction of cows milk including a snail juice (in high concentration containing the enzymes β-glucuronidase and sulphatase) hydrolysis step confirmed that 75% of the activity

present in milk co-chromatographed with 4-OH clofentezine. Small amounts of the radioactivity remained unresolved, however there was no evidence of significant quantities of metabolites other than the 4-OH clofentezine.

Table 3. Levels of total radioactivity detected in milk during and after twice daily oral administration of [^{14}C]-clofentezine at 2.2 mg/kg bw/day for 3 days

Day	TRR in the morning (mg/L)	TRR in the afternoon (mg/L)
1	< 0.001 [a]	0.013
2	0.11	0.17
3	0.18	0.16
4	0.15	-

a - pre-dose

The major tissue residue was detected in the liver (0.76 mg equiv/kg), the residues in other tissues are shown in Table 4. Of the total radioactive residue in liver 60% was extracted by soxhlet extraction with diethyl ether and methanol. The methanolic extract contained 53.5% TRR which appeared to correspond to 4-OH clofentezine when subjected to TLC using chloroform:methanol:ammonia eluent. The methanolic extract was further extracted with snail juice to liberate 44.1% of the TRR which was further extracted with 12.5% ethyl acetate in hexane. This extract was then washed with acetonitrile, which removed 14.8% of the residue. This co-chromatographed with 4-OH clofentezine. The ethyl acetate:hexane fraction required further clean up with an LC Diol cartridge, acetone precipitation then repeated hydrolysis with snail juice. Of the total residue from a repeated analysis, 68% was found to be solvent extractable. Hydrolysis of this extractable residue with partitioning into ethyl acetate/hexane produced a single component co-chromatographing with 4-OH clofentezine. The majority of the residue was 4-OH clofentezine.

Table 4. Levels of total radioactivity detected in the tissues collected at sacrifice 16 hours after oral administration of [^{14}C]-clofentezine at 2.2 mg/kg bw/day for 3 days

Tissue	TRR (mg/kg)
Liver	0.76
Kidney	0.36
Muscle	0.016
Renal fat	0.26
Subcutaneous fat	0.02

Samples of liver, kidney and renal fat from previous cow study (Phillips, M.W.A., Swalwell, L., 1988, R-12559) were extracted again and the radiolabelled residues characterised (Phillips, M.W.A., Swalwell, L., 1989, R-12561).

From the liver, 67% of the residue was extracted with solvent. The extract contained a single radioactive component identified as 4-OH clofentezine by chromatographic comparison with a standard. The unextracted residue (33%) was subjected to more severe digestion and hydrolysis procedures. Analysis of the released material showed several peaks including 4-OH clofentezine and it was concluded that the unextracted component in the liver had broken down in the digestion procedure and that the liver residue, both extractable and bound, was 4-OH clofentezine.

From kidney samples approximately 83% of the residue was solvent extractable and was identified as 4-OH clofentezine by TLC comparison with standard. Following hydrolysis and clean up of the remaining unextracted residue, the products of hydrolytic breakdown of 4-OH clofentezine were also observed. It was concluded that the major residue in kidney was 4-OH clofentezine.

The majority of the radioactivity (90%) present in renal fat was solvent extracted and identified as 4-OH clofentezine.

Goats

A lactating goat (Type Saanen) weighing 35 kg was given a single oral dose of [^{14}C]-clofentezine corresponding to a single day's intake of apple pomace (one third of feed per day) containing 22 mg/kg clofentezine. Residues in milk and plasma were monitored for 72 h, after which time the goat was killed and the tissues examined for residues (Table 5 and 6) (Campbell, J.K., Needham, D., 1983, R-12541).

TRR in milk reached a maximum 0.049 mg/L 24 h after dosing. Residues in milk were below 0.001 mg/L after 72 hours. Maximum total radioactive residues in tissues at 72 h after dosing were found in the eye and liver (0.03 mg/kg). All other tissues contained less than 0.01 mg/kg. Excretion of clofentezine by goats was virtually complete after 72 h.

Table 5. Total Radioactive residues in milk of a goat after a single oral dose of [^{14}C]-clofentezine at 7.3 ppm in feed

Time (hours)	TRR in milk (mg/L)
0	< 0.001
7	0.025
24	0.049
31	0.027
48	0.007
55	0.003
72	< 0.001

Table 6. Total radioactive residues in goat tissues at 72 h after a single oral dose of [^{14}C]-clofentezine at 7.3 ppm in feed

Tissue	TRR (mg/kg) (mean ± SD)
Liver	0.03 ± 0.005
Kidney	0.01 ± 0.001
Adrenals	0.01 ± 0.003
Eyes	0.03 ± 0.003
Other [a]	< 0.01

a - Heart, lung, spleen, skin, muscle, bone fat, thyroid, gonads, mammary gland, uterus, brain, tongue, rumen, reticulum, omasum, abomasum, intestine, pancreas, rumen contents, bile.

A lactating Anglo-Nubian goat weighing 48 kg was dosed with [^{14}C]-clofentezine at the exaggerated rate of 2.2 mg/kg bodyweight per day for 7 consecutive days to ensure quantifiable residues in milk (Campbell, J.K., 1987, R-12555). Milk samples were collected twice per day and several urine samples were also obtained and analysed.

Residues in milk reached a plateau level of approximately 0.2 mg/L on the third day of dosing (Table 7). A total of 93 ± 3% of the residue in milk was extractable with methanol. Of the extractable residue, 83.5% consisted of hydroxy-clofentezine isomers with 4-OH clofentezine being the largest single component. A fraction of 16.5% did not respond to enzyme hydrolysis and is probably not a conjugate of hydroxy-clofentezine. The residue in milk was mostly hydroxy-clofentezine complexed with endogenous material. There was no evidence for the presence of significant residues of 2-SMe-3-OH clofentezine. The major urinary metabolite was identified as 4-OH clofentezine, both free and conjugated.

Table 7. Analysis of milk for residues of [^{14}C]-clofentezine by liquid scintillation counting

Day	TRR in whole milk (mg/L)	
	Morning	Afternoon
1	0	0.044
2	0.18	0.20
3	0.23	0.21
4	0.24	0.22
5	0.16	0.14

Day	TRR in whole milk (mg/L)	
	Morning	Afternoon
6	0.17	0.16
7	0.18	0.12

Hens

Eight laying hens (ISA/Warren brown hybrid strain) with bodyweight in the range of 1.84-2.21 kg at study initiation were administered with [14C]-clofentezine orally for 3 consecutive days at a dose level of 17 mg clofentezine/kg bw/day (Creedy, C.L., Challis, I.R., 1988, R-12557). The animals were killed 12 h after receiving the last dose and necropsied.

In general, analysis of the residues present in the edible tissues and eggs showed that unchanged clofentezine was the major component (Table 8). The 3- and 4-OH-derivatives of clofentezine were also present and the remaining unidentified polar metabolites probably included conjugates of the hydroxy-metabolites. Analysis of the liver showed that 33% of the total residue was present as clofentezine and 19% as 3- and 4-OH clofentezine. A further 31% of the total residue was convertible to ortho-chlorobenzoic acid (OCBA) by treatment with hydrobromic acid (HBr) and was thus shown to be clofentezine related. Unchanged clofentezine was a major constituent of the total residues in fat (70%), skin (7.0%), unlaid developing eggs (32%) and muscle (34%). The metabolites, 3- and 4-OH-derivatives of clofentezine, were also shown to be present in fat (5%), skin (6%) and muscle (18%). In a separate extraction of eggs with hexane/acetonitrile 47% TRR was identified as clofentezine.

Table 8. Level and nature of residues in tissues and eggs of hens after 3 days oral dosing with [^{14}C]-clofentezine at 17 mg/kg bw/day

Tissue	TRR (mg/kg) (mear ± SD)	% TRR extracted (%)	% TRR as clofentezine (%)	% TRR as 3- / 4-OH-Clofentezine
Liver	0.70 ± 0.09	63.5	33.1	19.4
Abdominal fat	3.04 ± 0.65	79.0	70.3	4.66
Skin	0.87 ± 0.33	88.6	68.2	5.94
Muscle	0.14 ± 0.04	100.7	33.5	17.7
Unlaid eggs	0.60 ± 0.10	64.7	32.1	Not determined
Egg yolk (day 2)	0.17 ± 0.09	Not determined		
Egg white (day 2)	0.02 ± 0.005	Not determined		

The radiolabelled dose was rapidly eliminated with at least 70% of each daily dose being excreted during the subsequent 24 h period. The major radiolabelled constituent in the excreta was unchanged clofentezine (56 – 70%). The pattern of the metabolites detected was similar to that found previously in the urine of rats orally dosed with clofentezine and included 3- and 4-OH clofentezine and the glucuronide conjugate of the 4-OH isomer.

Comparative metabolism

Cow and goat

Milk samples from the previous cow study (Phillips, M.W.A., Swalwell, L., 1988, R-12559) and goat study (Campbell, J.K., 1987, R-12555) as well as from one further goat (type British Saanen) treated with clofentezine were extracted and analysed for the presence of radioactive metabolites (Phillips, M.W.A., Swalwell, L., 1989, R-12560).

Most of the ^{14}C residue in milk from cows or goats dosed orally with [^{14}C]clofentezine was extractable (> 94%). A single major metabolite, 4-OH clofentezine, was identified in milk. No 3-, 5- or 6-OH clofentezine or 3-OH-2-S-Me-clofentezine was observed.

Rat, goat and calf

Six male rats (Sprague Dawley) with bodyweight in the range of 218 – 238 g at study initiation were administered [^{14}C]-clofentezine orally at a dose level of 20 mg clofentezine/kg bodyweight/day at 4:45 pm. Three were killed after 16 h and the remaining three after 48 h. A female British Saanen goat weighing 75 kg were dosed orally with clofentezine in two capsules per day for 2 days at the time of milking, each capsule containing a dose equivalent to 2.5 mg clofentezine/kg bodyweight. The goat was killed approximately 19 h following the administration of the final dose. A British Friesian bull calf weighing 85 kg was administered orally at a dose rate of 5.1 mg/kg bodyweight at 10:00 pm. The calf was killed 12 h later (Needham, D., Challis, I.R., 1985, R-12549). Following oral administration of radiolabelled clofentezine to the rat, goat and calf, the livers were extracted with methanol. Unextracted residues were converted by hydrobromic acid to ortho-chlorobenzoic acid (OCBA) and quantified.

The methanolic extract contained 32 – 69% of the residue from the livers of rats killed 16 h after dosing; 15 – 28% from the livers of rats killed 48 h after dosing; 50% from the liver of a goat killed 19 h after dosing and 80% from the liver of a calf killed 12 h after dosing (Table 9). In each case the extracted residues had a chromatographic profile which was qualitatively similar to that found in rat urine, with conjugates of 3-, 4- and 5-hydroxylated clofentezine and a 3-(methylthio-hydroxyphenyl)-6-(2'-chlorophenyl)-1,2,4,5-tetrazine isomer. The unextracted residues could be converted almost quantitatively to orthochlorobenzoic acid by hydrobromic acid reflux, suggesting that the overall clofentezine moiety was still present in the residue. There is no qualitative difference in the metabolism of rat, goat and calf.

Table 9. Nature of residues in the liver of the rat, goat and calf (Needham, D., Challis, I.R., 1985, R-12549)

Animal	Time after dosing (h)	% of original residue		OCBA as % of ether extract	Recovery [a] (%)
		Methanol extract	Ether extract after HBr reflux		
Rat 1	16	46.5	17.7	83.8	.0
Rat 2	16	68.7	10.9	88.3	.9
Rat 3	16	32.2	29.1	88.3	.6
Rat 4	48	14.8	31.8	93.9	.5
Rat 5	48	22.5	32.6	64.8	.7
Rat 6	48	27.7	28.2	87.3	.9
Goat	19	49.9	23.2	76.9	.6
Calf	12	80.1	12.1	96.1	3.4

a - The recovered OCBA contains only 50% of the radioactivity and is therefore counted twice.

Rat, mouse, rabbit, calf, dog and baboon.

Samples from several studies (M36, M30, M31, M15, M18, M12, M37) were analysed and their residue composition compared (Challis, I.R., 1985, R-12550).

The metabolism of clofentezine in rat, mouse, rabbit, calf, dog and baboon was qualitatively similar, with hydroxylation and replacement of chlorine with a methylthio group being major pathways. Many minor metabolites were formed in all species (Figure 1). Quantitative interspecies differences were apparent, most notably the fact that in the calf and the baboon, hydroxylation and subsequent conjugation were the most prominent pathways, with methylthiolation being a very minor route of metabolism. The latter pathway was apparently more prominent in rodents – especially rat - and the rabbit and also present in dog. There are two major metabolic routes in mammals, namely: replacement of chlorine by a methylthio- group and hydroxylation of the phenyl ring, usually followed by conjugation.

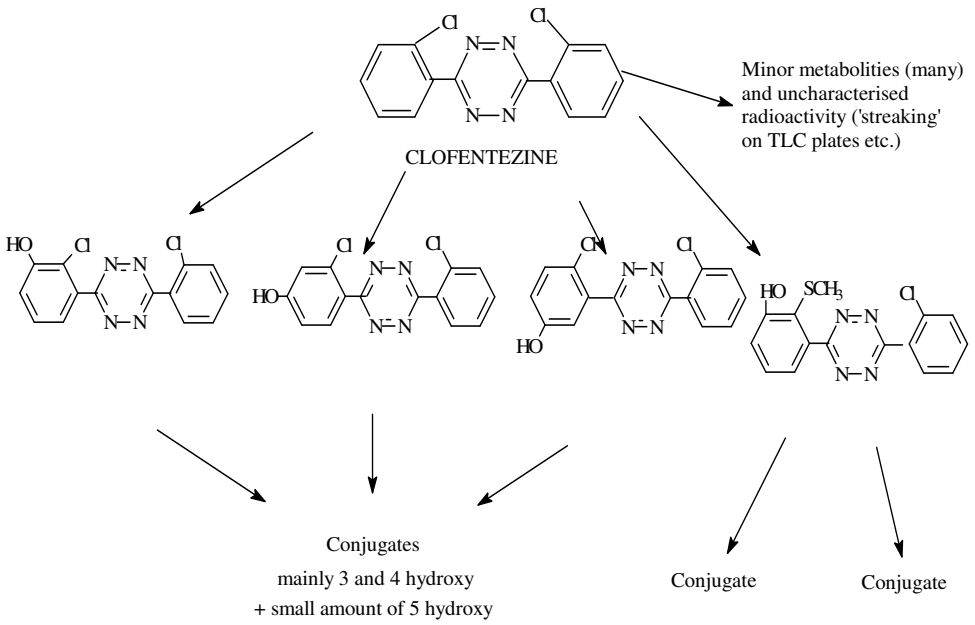


Figure 1. Proposed metabolism for Clofentezine in animals (Challis, I.R., 1985, R-12550).

Plant metabolism

The Meeting received plant metabolism studies with clofentezine on lemon, apple, peach and grapes.

In each crop tested, unchanged compound was the major extractable residue present (66 – 90%). Fibre bound residues accounted for up to a further 23% of the total radioactivity. However, it has been shown that this residue is not bio-available. All other compounds identified accounted for less than 10% of the recovered radioactivity. The most significant of these was 2-chlorobenzonitrile.

Lemon

Two container-grown lemon trees (variety: Eureka, grafted onto Poncirus rootstock) were maintained under glasshouse conditions with a photoperiod of 16 hours in the UK. The metabolism of [^{14}C]-clofentezine formulated as a 50 WP on lemon foliage was studied over a period of 103 days (Smith, S., Kelly, I.D., 1986, R-12727). Clofentezine was applied as a wettable powder at a field application rate of 0.03% (equivalent to 0.3 kg/ai/ha) on trees. A total of 36 semi-mature leaves were treated each with 350 µL of formulation to simulate treatment in the field where a "runoff" method is used. Samples were taken 0, 10, 25, 54 and 103 DAT.

There was a steady decline in the radioactivity isolated from the leaves, falling to 26.8% of the applied dose at day 103 (Table 10). The majority of the recovered activity remained associated with the leaf surface. After 103 days only 12.7% of the recovered and extracted activity had penetrated the leaf tissue.

Chromatographic analysis showed that the majority of radioactivity still associated with the leaves from a dichloromethane wash of the foliage (surface wash) remained as unchanged clofentezine (77% at day 103). At least 20 breakdown products were observed, the largest of which was 2-chlorobenzonitrile (6.8% of TRR at day 103=1.8% of AR), the principle photodegradation product of clofentezine. Dissipation of this volatile metabolite from the leaf surface probably accounted for the loss of radioactivity with time. No other single metabolite accounted for more than 2.1% of the total recovered activity. Only 2.4% of the total radioactive residue at final harvest was associated with fibre bound residues.

Table 10. Characterization of clofentezine residues in/on lemon foliage (Smith, S., Kelly, I.D., 1986, R-12727)

Substance	% of recovered radioactivity				
	Day 0	Day 10	Day 25	Day 54	Day 103
Surface wash	99.8	98.2	97.4	95.7	87.3
• Clofentezine [a]	97.6	91.1	88.2	84.3	77.2
• 2-chlorobenzonitrile [a]	1.2	5.5	8.1	8.5	6.8
Non-polar extract	0.16	1.25	1.15	1.74	6.15
Polar extract	0.04	0.34	0.73	1.60	4.18
Fibre-bound	0.04	0.25	0.75	1.01	2.39
Total	100	100	100	100	100
Total in % of AR	93.1	91.3	74.4	50.2	26.8

a - In surface wash only

Apple

Three young apple trees (approximately 1.5 m tall, variety: Cox) were grown in individual 0.7 m diameter drums containing medium loam soil. The trees were placed in a sheltered position outdoors. The metabolism of [^{14}C]-clofentezine formulated as 35 WP was investigated in apple foliage (Warner, P.A., 1981, R-12723). Clofentezine was painted on leaves, petioles, twigs and flowers on two apple trees at an application rate corresponding to 0.5 kg ai/ha. Samples of leaves and stems were excised 10, 25, 50 and 100 days after treatment (DAT) and analysed.

The majority of applied radioactivity was easily washed off the surface of the foliage with dichloromethane. Even after 100 days, 67.1% of recovered radioactivity was found in the wash fraction (Table 11). Analysis of recovered radioactivity by thin layer chromatography (TLC) showed that clofentezine remained largely unchanged as parent active substance but there was a steady increase of fibre bound material with time (17.1% TRR after 100 days).

Table 11. Distribution of total recovered radioactive residues in solvent extracts of [^{14}C]-clofentezine treated apple foliage (mean of duplicate determinations) expressed as % of dose

Fraction	DAT			
	10	25	50	100
Dichloromethane washing	77.0%	81.3%	65.2%	67.1%
Dichloromethane extraction	15.5%	8.9%	14.7%	8.1%
Methanol extraction	3.8%	4.0%	8.6%	7.8%
Fibre bound	3.7%	5.8%	11.5%	17.0%

A metabolite or degradation product, identified as NC 22505, was found in the 10 day (11%) and 100 day (6%) extracted samples (Table 12). It was not found at 25 and 50 days. No other single metabolite accounted for more than 1% of the recovered radioactivity was observed.

Table 12. Characterization of clofentezine residues in/on [^{14}C]-clofentezine treated apple foliage (results expressed as % total radioactivity recovered)

Substance	Day 10	Day 25	Day 50	Day 100
Clofentezine	81.9	86.8	78.4	65.9
NC 22505	11.0	0.0	0.0	6.0
Uncharacterised [a]	1.7	7.4	7.9	7.0
Polar materials	1.4	0.0	2.2	4.1
Fibre-bound	4.0	5.8	11.5	17.0

a - Poorly defined radioactivity on TLC plates with no single metabolite > 1% of TRR.

The metabolism of ^{14}C-clofentezine formulated as a 50 WP was investigated in apples (variety: Cox Orange Pippin) grown outside in the UK and treated at a field application rate of 0.03 kg ai/L and at the exaggerated rate of 0.76 kg ai/hL to aid the identification of any metabolites that might become significant if higher treatment rates were employed (Kelly, I.D., 1985, R-12726). The formulation was applied in 2µL drops to the surface of the fruit. Fruit was treated 10 weeks

before they were expected to be fully ripened. All apples were harvested 72 DAT with the exception of a few fruits which were removed one hour after the initial treatment to check the accuracy of the application. The apples harvested at 72 DAT were allowed to fully ripen for an extra 3 days under glass. Peel and flesh were analysed separately.

The radioactivity associated with the apples had decreased to 14.2% of the applied radioactivity 75 DAT (mature harvest) yielding a residue of only 0.031 mg/kg clofentezine equivalents (Table 13). A similar decline was seen in the apples treated at the exaggerated rate (Table 14). The decrease in radioactivity associated with apples was similar to that following the 0.76% treatment, reaching 11.5% (0.995 mg/kg clofentezine equivalents) of the applied value after 75 days.

Table 13. Distribution of the average radioactivity in apples harvested at maturity following treatment with 0.03 kg ai/L.

Extract	% of applied radioactivity	% of recovered radioactivity	Residue level (mg/kg) [a]
Peel – Dichloromethane extraction	5.28%	37.1%	0.012
Peel – Dichloromethane/ acetone extraction	1.42%	10.0%	0.004
Peel – Acetone/ water extraction	0.38%	2.6%	< 0.001
Peel fibre	5.44%	38.4%	0.012
Flesh – Dichloromethane/ acetone extraction	1.12%	7.8%	0.003
Flesh – Acetone/ water extraction	0.19%	1.4%	< 0.001
Flesh fibre	0.37%	2.7%	< 0.001
Total	14.2%	100%	0.031

a - Residue level expressed as clofentezine equivalent

Table 14. Distribution of the average radioactivity in apples harvested at maturity following treatment with 0.7 kg ai/L

Extract	% of applied radioactivity	% of recovered radioactivity	Residue level (mg/kg) [a/]
Peel – Dichloromethane extraction	8.37%	72.4%	0.721
Peel – Dichloromethane/ acetone extraction	1.30%	11.4%	0.113
Peel – Acetone/ water extraction	0.12%	1.0%	0.010
Peel fibre	1.01%	8.8%	0.087
Flesh – Dichloromethane/ acetone extraction	0.64%	5.5%	0.055
Flesh – Acetone/ water extraction	0.04%	0.4%	0.004
Flesh fibre	0.06%	0.5%	0.005
Total	11.5%	100%	1.00

a - Residue level expressed as clofentezine equivalent

Unchanged clofentezine was the major extractable residue present, accounting for 33% (0.011 mg/kg) of the recovered radioactivity (Table 15). Fibre-bound residues accounted for a further 38% (0.012 mg/kg). No other single metabolites accounted for greater than 7% (0.002 mg/kg) of the recovered radioactivity. The quantity of radioactive residue obtained from the 0.76% treatment is much greater in the dichloromethane extract and the extractable radioactivity consisted mainly of the unchanged clofentezine.

Table 15. Characterisation of metabolites in apples harvested at maturity following treatment with 0.03% formulation

Identity	% of recovered radioactivity	Residue level (mg/kg) [a]
Clofentezine	33.2%	0.011
Aqueous soluble metabolites	7.1%	0.002
Volatile residues [b]	4.4%	0.001
Peel fibre bound residues	38.3%	0.012
Flesh fibre bound residues	2.6%	0.001
Unknown metabolites in peel dichloromethane extract	6.4%	0.002

Identity	% of recovered radioactivity	Residue level (mg/kg) [a]
Unknown metabolites in other extracts	3.8%	0.001
Unanalysed material	4.2%	0.001
Total	100.0%	0.031

a - Residue level expressed as mg/kg clofentezine equivalents of whole apple.

b - 2-chlorobenzonitrile (a volatile photodegradation product of clofentezine in aqueous solution found on the apple surface).

Unchanged clofentezine was the major residue present accounting for 81.8% (0.81 mg/kg) of the recovered radioactivity (Table 16). Even at 25 times the normal treatment rate, no other metabolite accounted for more than 0.02 mg/kg (2%) of the recovered radioactivity. The fibre-bound residues were proportionally much less in 0.76% rate treated apples, accounting for only 8.8% (0.09 mg/kg clofentezine equivalents). Partial fragments, including 2-chlorobenzoic acid, derived from clofentezine or its metabolites could be extracted from the fibre-bound residue under extremely harsh hydrolytic conditions. This indicates that the fibre-bound residue results from the bonding of clofentezine or its metabolites to insoluble plant material and not from the incorporation $^{14}CO_2$ into natural products

Table 16. Characterisation of metabolites in apples harvested at maturity following treatment with 0.76% formulation

Identity	% of recovered radioactivity	Residue level (mg/kg) [a/]
Clofentezine	81.8%	0.814
Aqueous soluble metabolites	1.2%	0.013
Volatile residues [b]	0.1%	0.001
Peel fibre bound residues	8.8%	0.087
Flesh fibre bound residues	0.5%	0.005
Unknown metabolites in peel dichloromethane extract	3.9%	0.039
Unknown metabolites in other extracts	2.3%	0.023
Unanalysed material	1.4%	0.013
Total	100.0%	0.995

a - Residue level expressed as mg/kg clofentezine equivalents of whole apple.

b - 2-chlorobenzonitrile (a volatile photodegradation of product of clofentezine in aqueous solution found on the apple surface).

The nature of the fibre-bound residues of clofentezine in apples was investigated following application of ^{14}C-clofentezine formulated as a 50 SC to three separate apple varieties (Golden Delicious, Top Red and Granny Smith) at field rates of 0.06 kg ai/L and 0.48 kg ai/L in South Africa (Edwards, R., 1987, R-12728). These high application rates were used to assist in the identification of both extractable metabolites and bound residues. The apple trees were grown and treated outside in an orchard and the fruits harvested 25 and 64 days after application. The apples were separated into peel and flesh samples for the residue determination.

The total radioactive residues in apples at the lower application rate (Table 17) ranged from 0.08 – 0.224 mg/kg clofentezine equivalents (20.9 – 42.4% of applied radioactivity). In all cases the majority of the recovered residue was found in the peel (90 – 96%). Bound residues from samples treated at the lower application rate were largely restricted to the peel fraction and accounted for 4.5 - 11.3% of the recovered radioactivity (0.009 – 0.010 mg/kg). This proportion was dependent upon apple variety and increased with time. Bound radioactivity was effectively released by 16 h base hydrolysis and partially solubilised by enzymatic treatment with pectinase and cellulase. Analysis showed that the insoluble residue consisted of unchanged clofentezine (approximately 50% of the fibre-bound residue) and breakdown products (2-chlorobenzoic acid and 2-chlorobenzyl alcohol) which had become incorporated into peel components.

Table 17. Characterization of clofentezine residues in apples harvested at maturity

Substance	% of recovered radioactivity			
	Variety 'Top Red' 25 days after 0.06% treatment	Variety 'Golden Delicious' 25 days after 0.06% treatment	Variety 'Granny Smith' 64 days after 0.06% treatment	Variety 'Granny Smith' 64 days after 0.48% treatment
Clofentezine	72.0	85.4	65.0	84.0
2-chlorobenzonitrile	nd	nd	nd	4.3
Non-polar extracts	17.5	9.9	21.3	7.3
Polar extracts	2.1	0.9	2.5	1.2
Fibre-bound	8.2	4.5	11.3	3.3
Total	100.0	100.0	100.0	100.0
Total in % of AR	20.9	42.4	22.7	28.3

nd: not determined.

Peach

The metabolism of [^{14}C]-clofentezine formulated as a 50 SC was studied in immature peach (variety: Rochester) fruit (at fruit set) grown under glasshouse conditions in the UK. Treatments were made at 0.01% and 0.1% 62 days prior to harvest (Edwards, R., 1988, R-12729). A separate application was made to leaves of peach trees adjoining untreated fruits at 0.01%.

Immediately following the treatments of fruits with 0.01% and 0.1% spray concentrations, 87.7% and 96.1% of TRR were identified as parent compound, respectively (Table 18). Of the recovered total radioactive residue at 62 days (0.047 mg/kg clofentezine equivalents) at normal field treatment rate, the surface wash contained 74.9% (0.036 mg/kg) of the total recovered radioactivity as clofentezine and 8.4% (0.004 mg/kg) as 2-chlorobenzonitrile. The fibre-bound residue was negligible (< 0.005 mg/kg). No other metabolites were observed. At the exaggerated field treatment rate, a total residue of 0.70 mg/kg clofentezine equivalents was recovered. Analysis showed that 90.9% (0.633 mg/kg) of the recovered radioactivity was present as clofentezine, 5.4% (0.038 mg/kg) as 2-chlorobenzonitrile and only 0.6% (0.004 mg/kg) as fibre-bound residue.

Negligible translocation of residue from foliar applications was observed to peach leaves and to developing untreated fruit (0.0005 mg/kg). The majority of the radioactivity on the outside of these treated leaves remained as unchanged clofentezine. A separate extraction using the residue method for parent clofentezine accounted for 91.4% of the total residue.

Table 18. Characterization of clofentezine residues in peach fruit

Substance	0.01% treatment				0.1% treatment			
	Day 0		Day 62		Day 0		Day 62	
	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
Surface wash	91.2	1.77	83.5	0.040	69.8	13.2	85.2	0.60
Clofentezine [a]	87.7	1.70	74.9	0.036	67.6	12.8	79.0	0.55
2-chlorobenzonitrile [b]	nd		8.4	0.004	nd		5.2	0.037
Non-polar extracts	7.5	0.14	10.8	0.005	29.2	5.40	12.4	0.087
Clofentezine	nd		nd		28.5	5.26	11.9	0.083
Polar extracts	1.3	0.025	3.5	0.001	0.9	0.17	1.8	0.014
Fibre-bound	0.1	0.002	2.2	0.001	0.1	0.019	0.6	0.004
Total	100.0	1.93	100.0	0.047	100.0	18.8	100.0	0.70

a - In surface wash only

b - nd: not detected.

Grapes

The metabolism of [^{14}C]-clofentezine was investigated in grapes (variety: Muller Thurgeau) grown under glasshouse conditions in the UK (Campbell, J.K., 1989, R-12730). The formulation was applied in droplets directly to the surface of grape berries at rates corresponding to 0.01% and 0.1% at different growth stages of development to obtain two separate post harvest intervals (day 24/25 and 45/46) at harvest.

Results in Table 19 indicate that the major part of the extractable radioactivity was in the dichloromethane wash. The quantities of fibre bound residues extracted with the Bligh-Dyer method increased with time after treatment. After treatment with the 0.01% and 0.1% formulation at DAT=0, 1.05 and 8.30 mg/kg (equivalent to 99.7% and 99.9% of the extracted radioactivity, respectively) were removed with three kinds of solvent system wash. At 24 days after treatment this residue had decreased to 0.38 and 2.49 mg/kg (97.3% and 98.3% of the radioactivity), respectively. At day 45 after application the total fibre bound radioactivity increased to 23% and 11.5% (equivalent to 0.03 and 0.05 mg/kg).

Table 19. Distribution of extracted radioactivity in grapes treated with 0.01% and 0.10% [^{14}C]-clofentezine formulations

Extract	% total activity in extract		Total residue concentration (mg/kg)	
	0.01% ai	0.10% ai	0.01% ai	0.10% ai
Harvest at DAT = 0				
DCM Wash	93.5	97.3	0.99	8.08
Chloroform Phase	5.3	1.6	0.06	0.13
Methanol/Water Phase	0.9	1.0	0.01	0.09
Fibre	0.3	0.1	< 0.01	0.01
TOTAL	100	100	1.06	8.31
Harvest at DAT = 24/25				
DCM Wash	78.1	86.9	0.30	2.19
Chloroform Phase	12.0	8.0	0.05	0.21
Methanol/Water Phase	7.2	3.4	0.03	0.08
Fibre	2.7	1.7	0.01	0.04
TOTAL	100	100	0.39	2.52
Harvest at DAT = 45/46				
DCM Wash	48.0	71.6	0.05	0.32
Chloroform Phase	18.6	11.0	0.02	0.05
Methanol/ Water Phase	10.4	5.9	0.01	0.03
Fibre	23.0	11.5	0.03	0.05
TOTAL	100	100	0.11	0.45

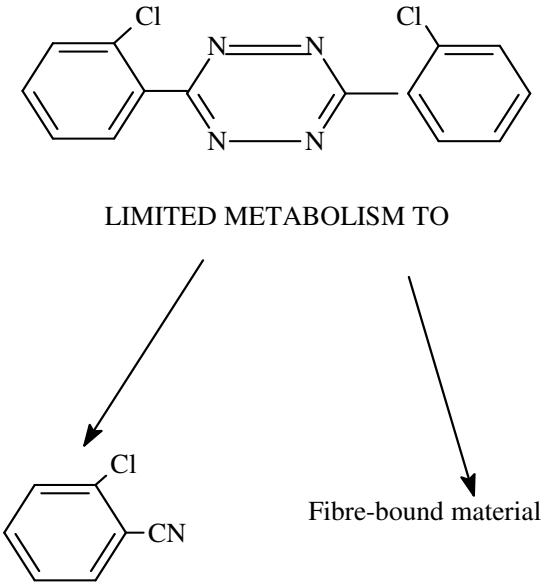
Characterisation of the radioactivity found at these stages confirmed that by far the majority of the residue was present as parent clofentezine with 76.9% and 85.5% of the extracted radioactivity 24 days after treatment with 0.01% and 0.1% formulation, respectively. This is equivalent to a total residue of 0.3 and 2.15 mg/kg. At the same treatment rate and analysis time, the remaining residue was made up predominantly of 2-chlorobenzonitrile (0.04 mg/kg, 9.61% of extracted radioactivity and 0.18 mg/kg, 7.13% of extracted radioactivity) and polar compounds comprising 2-chlorobenzoic acid, 2-chlorobenzamide and 2-chlorobenzyl alcohol (1.39% of extracted radioactivity, 0.005 mg/kg and for the low dosage and 0.76% of extracted radioactivity, 0.02 mg/kg in the case of high dosage). At 45 DAT with the same treatment rate, the quantities of clofentezine present were significantly lower, due to a lower overall residue and also the incorporation of radioactivity into fibre bound residue. At this point the extractable radioactivity composed of for low and high dosage rates respectively: clofentezine (55.4% and 69.2% or 0.06 and 0.31 mg/kg). 2-chlorobenzonitrile (5.11% and 7.34% or 0.006 and 0.033 mg/kg) and polar compounds (3.79% and 2.63%, 0.004 and 0.012 mg/kg). The remaining radioactivity was present as fibre bound residue. Analysis of the fibre bound material by solubilisation under harsh hydrolytic conditions indicated that, as in the case of apples, it was partially degradable to 2-chlorobenzoic acid. Application of a residue method detecting the parent compound to samples of grapes demonstrated that the method accounted for the majority of the clofentezine in the samples (Table 20).

Table 20. Characterisation of residues in grapes after treatment with 0.01% and 0.10% [^{14}C]-clofentezine formulations, % values in % of TRR

Treatment	Extract	Polar Compound [a]	2-chlorobenzonitrile	Clofentezine	% of extract characterised
DAT = 0 days					
0.01% ai	Total extracts	1.08% (0.01 mg/kg)	1.40% (0.015 mg/kg)	94.2% (1.00 mg/kg)	96.7
0.10% ai	Total extracts	0.39% (0.03 mg/kg)	1.24% (0.10 mg/kg)	96.4% (8.01 mg/kg)	98.0
DAT = 24-25 days					
0.01% ai	Total extracts	1.39% (0.005 mg/kg)	9.61% (0.04 mg/kg)	76.9% (0.29 mg/kg)	87.9
0.10% ai	Total extracts	0.76% (0.02 mg/kg)	7.13% (0.18 mg/kg)	85.5% (2.15 mg/kg)	93.4
DAT = 45-46 days					
0.01% ai	Total extracts	3.79% (0.004 mg/kg)	5.11% (0.006 mg/kg)	55.4% (0.06 mg/kg)	64.3
0.10% ai	Total extracts	2.63% (0.012 mg/kg)	7.34% (0.033 mg/kg)	69.2% (0.31 mg/kg)	79.2

a - Polar compounds comprised of 2-chlorobenzoic acid, 2-chlorobenzamide and 2-chlorobenzyl alcohol.

The majority of the remaining residue on plants above at harvest was clofentezine. Apart from clofentezine the largest single component was always 2-chlorobenzonitrile which is the main photolysis product of clofentezine. The bound residue was shown to arise from the binding of clofentezine or its metabolites to insoluble plant components and largely restricted to the peel fraction.



2-Chlorobenzonitrile

Figure 2. Proposed metabolic pathway for clofentezine in plants.

Environmental fate in soil

The Meeting received information on the environmental fate of clofentezine in soil, including studies on aerobic soil metabolism, field dissipation and crop rotational studies. Because clofentezine is used on sugar beet and asparagus (edible portion in soil) the studies on aerobic soil metabolism and field dissipation were evaluated by the Meeting.

Soil metabolism

The degradation of [^{14}C]-clofentezine has been investigated in two soils: a clay loam (Bottisham, pH 6.2, 14.7% OC, 50% max water holding capacity) and loamy sand (Cottenham, pH 6.5, 1.9% OC, 50% max water holding capacity) incubated under aerobic conditions at 15 ºC for up to 67 days (Leake, C.R., Arnold, D.J., 1983, R-12693). The soils were treated with ^{14}C-clofentezine at a concentration of 1.89 mg/kg dry weight of soil (equivalent to a field application rate of 1.4 kg ai/ha assuming a homogenous distribution in the top 5 cm soil layer and a soil density of 1.5 g/cm^3) and incubated at 50% of their maximum water holding capacity in the current of CO_2-free moist air. Volatile radioactivity evolved from the soil was collected. At 0, 7, 14, 30 and 67 DAT soil samples were extracted with solvents and the radioactivity quantified by liquid scintillation counting and analysed by thin layer chromatography (Table 21). The amounts of radioactivity remaining 'bound' to the soils were determined by combustion.

Cleavage of the tetrazine ring led to the formation of small amounts (up to 6.8%) of 2-chlorobenzoic acid. A small percentage of the compound was mineralised to $^{14}CO_2$ (3 – 6% in both soils after 67 days) and generated soil 'bound' residues (maximum of 20% after 67 days). No other major radiolabelled products were present in any of the soil extracts. All DT_{50} and DT_{90} calculated with single first order kinetics are summarized in Table 21.

Table 21. Distribution of radioactivity in clay loam and loamy sand

Fraction	% of applied radioactivity				
	0 days	7 days	14 days	30 days	67 days
Clay loam					
Extractable	102.2	97.4	94.8	86.7	55.3
• Clofentezine	91.1	94.0	88.4	67.8	50.0
• 2-chlorobenzoic acid	0.9	1.7	0.8	1.6	0.6
• Polar material	1.8	1.3	2.6	4.4	1.5
• Remainder	8.4	0.6	3.0	12.8	3.2
Bound residue	1.5	5.6	8.6	11.7	20.3
$^{14}CO_2$	--	0.2	0.5	1.4	5.7
Total recovered	103.7	103.2	103.9	99.8	81.3
DT_{50}	66 days		DT_{90}	229 days	
Loamy sand					
Extractable	101.9	102.7	101.3	98.3	71.8
• Clofentezine	97.2	95.5	96.6	83.2	62.6
• 2-chlorobenzoic acid	0.8	0.4	0.8	6.8	1.2
• Polar material	0.3	0.0	1.1	1.9	3.6
• Remainder	3.6	6.6	2.9	6.5	4.5
Bound residue	0.3	1.4	1.8	5.2	14.4
$^{14}CO_2$	--	0.2	0.2	0.8	2.5
Total recovered	102.2	104.3	103.3	104.3	88.7
DT_{50}	102 days		DT_{90}	335 days	

The degradation of clofentezine was investigated in three soils; a clay (Shelford, pH 6.6, 4.5% OC, 50% maximum water holding capacity), a loamy sand (Cottenham, pH 6.5, 1.9% OC, 50% maximum water holding capacity) and a clay loam (Bottisham, pH 6.2, 14.7% OC, 50% maximum water holding capacity) each incubated in the laboratory for up to one year under aerobic conditions (Leake, C.R., Arnold, D.J., 1983, R-12698). Samples of the soils were treated with [^{14}C]-clofentezine at a concentration of 1.61 mg/kg dry weight of soil in the clay, and 2.1 mg/kg in the loamy sand and clay loam (equivalent to a field application rate of 1.2 and 1.6 kg ai/ha assuming 5 cm depth and soil density of 1.5 g/cm³). The soils were incubated in flasks at 50% maximum water holding capacity in the dark and at a temperature of 25 ºC. During incubation, the flasks were continuously purged with carbon dioxide-free moist air. Radiolabelled volatile products evolved from the soils were collected in solvent traps for quantification and analysis. At various time intervals, soil samples were extracted with solvents and the radioactivity assayed by liquid scintillation counting and thin layer chromatography using two different solvents (Tables 22 and 23). The amounts of radioactivity remaining 'bound' to the soils were determined by combustion and a balance of radioactivity was determined for each sample.

Under aerobic conditions $^{14}CO_2$ was steadily evolved from all three soils and after 1 year 56% of the applied compound had been mineralised to $^{14}CO_2$ in the loamy sand compared with approximately 38% and 25% in the clay and clay loam, respectively. The quantity of "bound" radioactivity increased with time in all three soil types: up to 30% in the loamy sand, 38% in the clay and 40% in the clay loam. The decrease of extractability appeared to correspond with the organic matter. The degradation of the parent compound proceeded via hydrolytic cleavage of the tetrazine ring, leading to the formation of 2-chlorobenzoic (2-chlorobenzylidene)hydrazide (max. 13%). Further degradation led to the formation of several other minor metabolites, N, N'-bis-(2-chlorobenzoyl)-hydrazine (maximum 1.6%), 2-chlorobenzamide (maximum 0.8%) and 2-chlorobenzoic acid (maximum 6.2%), with mineralization to $^{14}CO_2$ and the formation of soil bound residue. An unidentified metabolite was detected at a maximum of 10.8% after 21 days in the clay loam soil and at not significant levels in the other two soils. Clofentezine was degraded in all three soils under aerobic conditions with a "half life" of approximately 4, 6 and 8 weeks in the clay, loamy sand and clay loam respectively.

Table 22. Distribution of radioactivity [% of applied radioactivity]

Fraction	Clay														
Days incubation	0	1	2	7	14	21	30	61	90	120		180	217	272	360
Extractable	104.9	95.2	97.3	86.0	75.7	54.9	48.7	42.0	30.4	33.0		18.5	21.0	18.1	18.4
Bound residue	3.0	5.9	5.8	11.8	16.3	23.3	30.1	28.3	28.2	31.9		28.4	32.2	36.2	37.9
$^{14}CO_2$	--	0.2	0.2	1.8	5.7	15.8	17.1	27.5	37.5	31.3		43.7	35.7	39.1	38.3
Total recovered	107.9	101.3	103.3	97.8	97.7	94.0	95.9	97.8	96.1	96.2		90.6	88.9	93.4	94.6
	Loamy sand														
Days incubation	0	1	2	7	14	21	30	62	87	120	150	181	220	269	360
Extractable	99.8	97.5	102.3	102.5	89.9	91.6	82.5	77.9	56.4	45.3	35.4	35.0	35.3	30.8	23.8
Bound residue	0.4	1.0	1.3	2.6	3.8	6.7	8.2	14.9	35.5	21.0	21.6	23.5	27.4	28.7	29.7
$^{14}CO_2$	--	0.1	0.2	0.7	1.6	2.5	6.1	13.8	26.7	36.9	43.6	41.3	46.0	48.5	55.6
Total recovered	100.2	98.6	103.8	105.8	95.3	100.8	96.8	106.6	118.7	103.2	100.6	99.8	108.6	108.0	109.1
	Clay loam														
Days incubation	0	1	2	7	14	21	30	59	90	120	150	239	269	300	360
Extractable	98.9	98.7	98.2	96.4	91.4	85.7	81.0	66.5	58.8	52.1	49.6	41.5	41.6	37.2	33.3
Bound residue	1.2	3.1	3.8	6.7	9.3	12.3	15.4	34.5	29.0	33.1	35.5	45.3	53.2	48.7	46.4
$^{14}CO_2$	--	0.1	0.5	1.6	2.7	4.3	6.0	11.4	15.7	20.1	17.2	20.7	13.4	23.9	24.9
Total recovered	100.1	101.9	102.5	104.7	103.4	102.3	102.4	112.4	103.5	105.3	102.3	107.5	108.2	109.8	104.6

Table 23. Distribution and characterisation of radioactivity in soil extracts

	^{14}C as % of dose											
Days incubated	0/1	7	14	21	30	61	90	120	150	181	269	360
Clay												
Clofentezine	--	68.6	60.8	--	--	17.7	18.6	24.9	--	--	--	--
unidentified	--	0.9	1.5	--	--	2.3	0.6	< 0.1	--	--	--	--
AE C593600	--	3.3	1.1	--	--	2.7	0.5	1.4	--	--	--	--
AE C512898	--	1.0	0.8	--	--	1.4	0.5	0.6	--	--	--	--
2-chlorobenzoic acid	--	4.4	2.8	--	--	1.9	0.5	1.1	--	--	--	--
2-chlorobenzamide	--	< 0.1	< 0.1	--	--	< 0.1	< 0.1	< 0.1	--	--	--	--
Origin	--	1.9	2.0	--	--	2.7	0.4	1.2	--	--	--	--
Remainder	--	5.3	5.2	--	--	6.5	2.1	3.5	--	--	--	--
Acetonitrile/water uncharacterised	--	0.8	nd	--	--	1.2	1.2	1.3	--	--	--	--
Non extracted "bound" residues	--	9.1	nd	--	--	24.4	29.4	32.3	--	--	--	--
$^{14}CO_2$	--	1.3	3.3	--	--	26.0	35.5	26.9	--	--	--	--
Total recovered	--	96.6	nd	--	--	86.8	89.4	93.2	--	--	--	--
Loamy sand												
Clofentezine	68.4	62.1	--	--	54.7	--	--	32.9	20.2	19.9	14.7	--
unidentified	5.5	7.1	--	--	1.0	--	--	2.4	2.4	1.9	1.8	--
AE C593600	5.7	5.2	--	--	13.0	--	--	2.9	6.0	1.2	0.7	--
AE C512898	0.6	2.2	--	--	0.8	--	--	0.7	0.7	0.5	0.4	--
2-chlorobenzoic acid	0.9	6.2	--	--	3.6	--	--	0.7	0.8	1.0	0.7	--
2-chlorobenzamide	0.5	0.1	--	--	0.6	--	--	< 0.1	< 0.1	0.8	0.2	--
Origin	1.4	9.9	--	--	2.9	--	--	1.4	1.5	1.8	0.9	--

	^{14}C as % of dose											
Days incubated	0/1	7	14	21	30	61	90	120	150	181	269	360
Remainder	5.2	18.1	--	--	7.4	--	--	3.2	3.0	4.0	3.3	--
Acetonitrile/water uncharacterised	0.1	0.4	--	--	0.2	--	--	1.0	0.9	1.1	1.0	--
Non extracted "bound" residues	1.0	2.6	--	--	6.6	--	--	20.8	24.3	23.6	22.3	--
$^{14}CO_2$	< 0.1	0.6	--	--	4.8	--	--	40.7	47.4	48.4	51.8	--
Total recovered	89.3	114.5	--	--	102.8	--	--	106.7	107.2	104.2	97.8	--
Clay loam												
Clofentezine	63.7	60.9	70.3	56.6	56.7	--	42.0	34.2	--	--	--	22.4
unidentified	9.2	9.7	7.0	10.8	7.6	--	1.7	6.0	--	--	--	3.2
AE C593600	3.7	1.2	0.9	2.0	1.3	--	3.1	1.6	--	--	--	1.6
AE C512898	1.6	1.1	0.7	1.5	1.2	--	1.2	1.0	--	--	--	0.6
2-chlorobenzoic acid	2.9	3.5	2.9	2.2	2.8	--	1.0	2.0	--	--	--	1.6
2-chlorobenzamide	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	--	< 0.1	< 0.1	--	--	--	< 0.1
Origin	4.3	3.7	2.4	2.6	4.7	--	1.8	3.8	--	--	--	2.6
Remainder	13.7	18.3	8.8	10.1	10.5	--	10.0	6.6	--	--	--	3.2
Acetonitrile/water uncharacterised	0.2	1.0	0.6	0.7	1.0	--	--	--	--	--	--	--
Non extracted "bound" residues	1.5	6.1	8.5	10.6	13.3	--	25.7	29.4	--	--	--	42.6
$^{14}CO_2$	-	2.0	2.9	4.3	6.3	--	15.7	18.6	--	--	--	26.6
Total recovered	100.8	107.4	105.0	100.7	105.4	--	102.2	103.2	--	--	--	104.4

The experimental data above were evaluated by fitting the first-order-kinetic model to the concentrations observed in the aerobic soil degradation. The calculated DT_{50} and DT_{90} values for clofentezine are presented in the Table 24. The amounts of the metabolite AEC 593600 detected in the soil laboratory study were generally very low. The quantity of AEC 593600 peaked at 0.053 mg/kg in clay (Shelford) at 7 days after dosing, 0.27 mg/kg in loamy sand (Cottenham) at 30 days after dosing and 0.078 mg/kg in clay loam (Bottisham) respectively.

Table 24. First-order-kinetic evaluation of the aerobic soil degradation study

Location	Soil type	Clofentezine	
		DT_{50}	DT_{90}
Shelford	Clay	64	167
Cottenham	Loamy sand	115	304
Bottisham	Clay loam	227	585

A laboratory study of clofentezine degradation in two standard West German soil types was conducted using two sandy loams, BBA soil types 2.2 (Speyer 2.2, pH 6.2, 4.5% OC, 5.3% clay, 40% max water holding capacity) and 2.3 (Speyer 2.3, pH 7.8, 1.9% OC, 10.8% clay, 40% max water holding capacity) (Snowdon, P.J., 1982, R-12691). The soils were fortified at a rate of 2 mg/kg dry soil equivalent to 2 kg ai/ha, incubated at 40% maximum water holding capacity in the dark at a temperature of 22 °C for 147 days.

A two phase, first order rate of dissipation was observed with a rapid initial degradation, which was followed by a slower second phase of decline (Table 25). In type 2.2, clofentezine half-lives of 32.2 and 174.0 days were computed over the periods from 0 to 14 and from 28 to 147 days, respectively. In type 2.3, clofentezine half-lives of 15.6 and 87.1 days were computed over the periods from 0 to 28 and from 56 to 147 days, respectively. The experimental data above were evaluated by fitting the first-order-kinetic model to the concentrations observed in the aerobic soil degradation. The calculated DT_{50} and DT_{90} values for clofentezine are presented in the Table 25.

Table 25. Degradation of clofentezine in two soils (Snowdon, P.J., 1982, R-12691).

Time (days)	Residue level (mg/kg)	
	Speyer 2.2: Sandy loam	Speyer 2.3: Sandy loam
0	1.96	2.16
7	1.65	1.63
14	1.45	1.15
28	1.22	0.63
56	0.97	0.27
84	0.82	0.21

Time (days)	Residue level (mg/kg)	
	Speyer 2.2: Sandy loam	Speyer 2.3: Sandy loam
147	0.74	0.13
DT_{50}: 1st phase	32.2 (0 – 14 days)	15.6 (0 – 28 days)
2nd phase	174.0 (28 - 147 days)	87.1 (56 – 147 days)
DT_{50} (first-order-kinetics)	78 days	14.5 days
DT_{90} (first-order-kinetics)	330 days	98 days

The available data indicates that the degradation of clofentezine in soil may be represented by the following flow diagram.

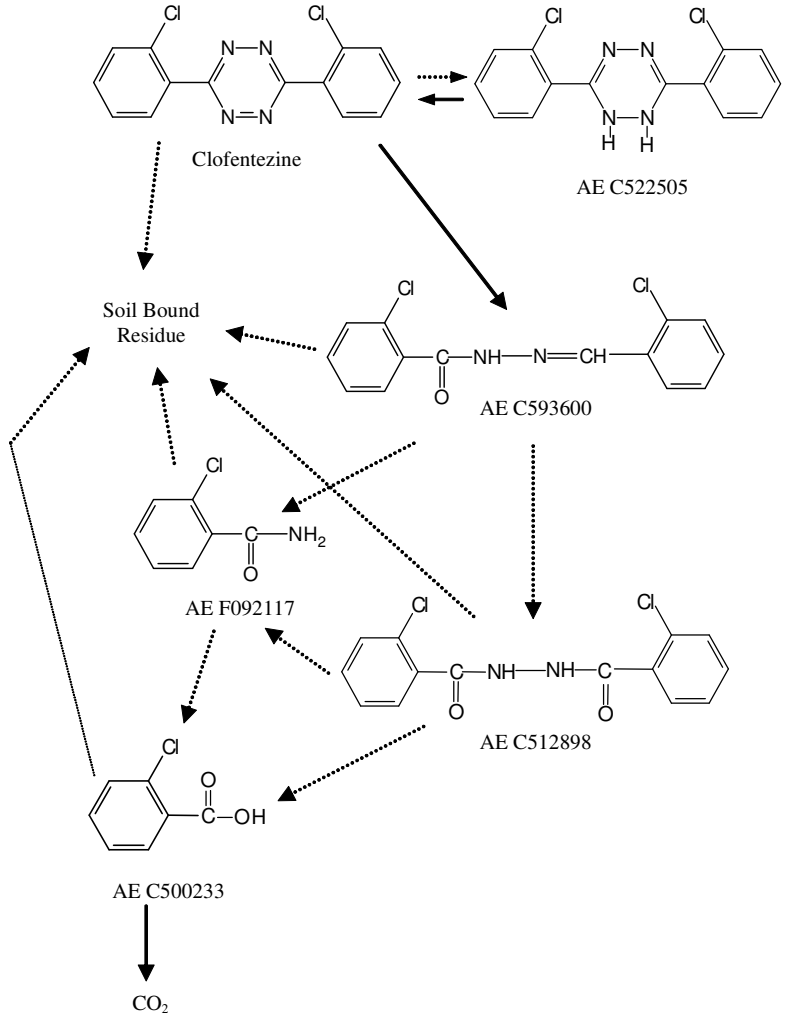


Figure 3. Proposed metabolic pathway for clofentezine in soil.

Field dissipation

Four trials were conducted at four sites in Germany during 1993 and 1994 involving application of clofentezine as the SE formulation to bare soil plots (Patmore, I.R., Godfrey, T.L., Peatman, M.H., 1996, R-12802; Nohl-Weiler, C., 1995, R-12804). The plots received a single application of clofentezine at a nominal rate of 225 g ai/ha, and the actual rates ranged from 0.20 to 0.32 kg ai/ha. Soil samples were taken down to 20 cm 10 times during a period of one year. Almost all of clofentezine were found in the top layer (0 – 10 cm). At all four trials the top soil residues declined over the sampling intervals with residues at or below the LOQ of 0.02 mg/kg being observed at or

after three months in three of four trials. In the fourth site (Rheinheim) residues of 0.02 mg/kg remained until one year (Figure 4).

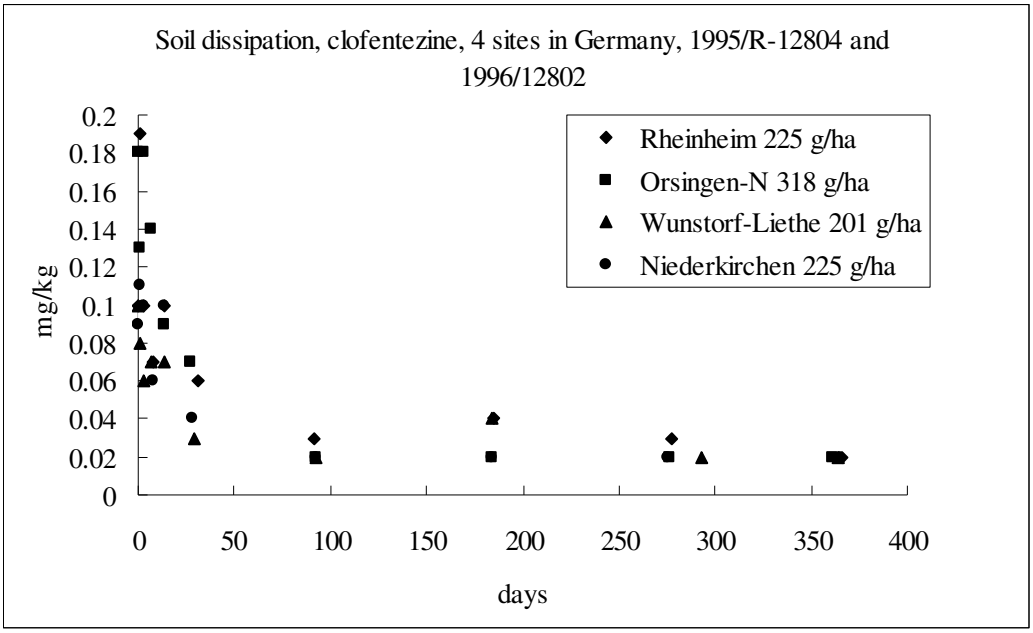


Figure 4. Field dissipation of clofentezine on bare soils in 4 sites in Germany in 1993.

Six trials were conducted in Germany in 1991 applying clofentezine as the 50 SC formulation to bare soil plots (Peatman, M.H., 1992, R-12799). The plots received a single application of clofentezine at a nominal rate of 0.4 kg ai/ha. Soil samples were taken down to 30 cm 11 times during 1 year. Following single application, initial residues found in the air dried top soil (0 – 10 cm) ranged from 0.04 to 0.43 mg/kg (Figure 5). All six trials demonstrated a decline in residues over one year, with levels dropping towards LOQ, apart from the trial at Schwichteler, where the residues remained at round 0.1 mg/kg over the 6 – 12 month period.

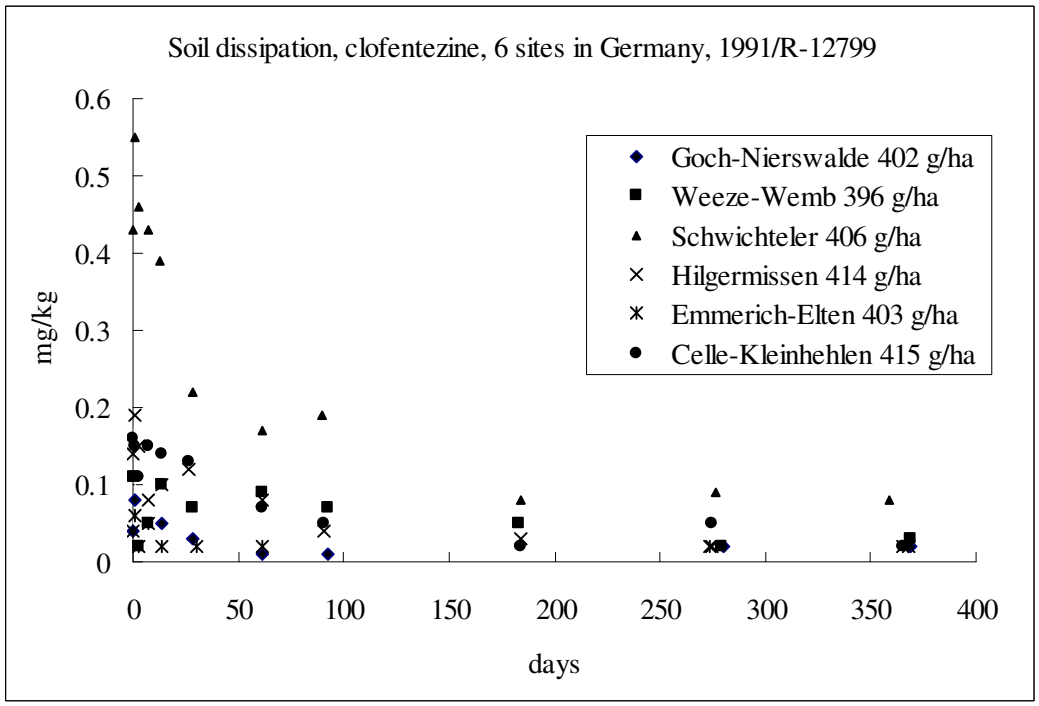


Figure 5. Field dissipation of clofentezine on bare soils in 6 sites in Germany in 1991

Four trials were conducted in Germany in 1989 applying clofentezine as the 50 SC formulation to bare soil plots (Snowdon, P.J., 1991, R-13076). The plots received a single application of clofentezine at a nominal rate of 0.4 kg ai/ha. Soil samples were taken at 11 times up to 1 year. Following single application, initial residues found in the top soil (0 – 10 cm) ranged from < 0.01 to 0.68 mg/kg (Figure 6). All four trials demonstrated a decline of residues over one year or 180 days, with levels dropping towards LOQ, apart from the trial at Goch-Nierswalde. There is no evidence of clofentezine residues in the deeper soil layers. At Niederkirchen, consistently low residues showed very poor correlation to the application rate. This trial should be regarded as anomalous.

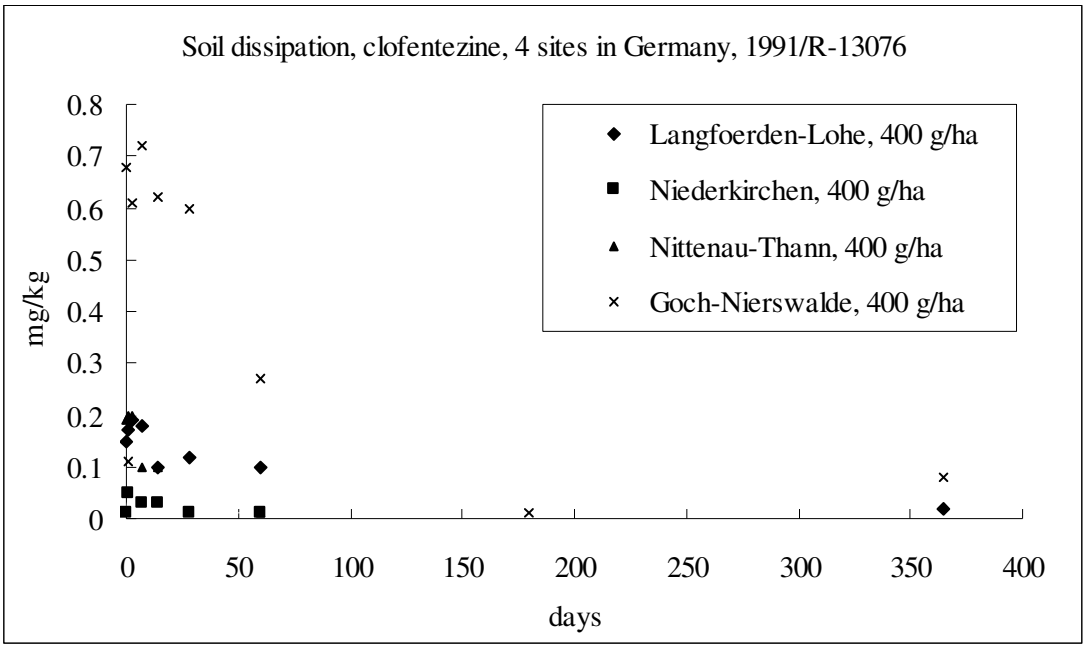


Figure 6. Field dissipation of clofentezine on bare soils in 4 sites in Germany in 1989

Clofentezine was applied to bare soil in Fresno, CA as a 42% suspension concentrate (Castro, L., 1991, R-12800) in 1990. One broadcast application was made at a rate of 0.29 kg ai/ha. Triplicate soil samples each consisting of five cores taken to a depth of 90 cm were collected at selected intervals during the trial. These intervals were pre-application, days 0 (immediately after application), 3, 7, 14, and 28; and months 2, 3, 4, 6, 7.5, 8, 8.5, and 9. The samples were subdivided into seven sections (0 – 7.5, 7.5 – 15, 15 – 30, 30 – 45, 45 – 60, 60 – 75, and 75 – 90 cm) for analysis. Analysis was done by high performance liquid chromatography (HPLC).

Except for one replicate, clofentezine was detected only in the topmost soil horizon. No apparent movement of clofentezine into lower horizons was noted. No detectable residues of AEC 593600, the major soil degradate of clofentezine, were found throughout the study. The average concentration of clofentezine in the top soil horizon at Day 0 was 0.23 mg/kg. The average residues in the top horizon had declined to 0.02 mg/kg by month 8 (243 days after application). The magnitude of the residue of clofentezine was determined by adding the residues detected in the top two horizons (0 – 7.5 and 7.5 – 15 cm) for each subplot and then averaging these sums (Figure 7).

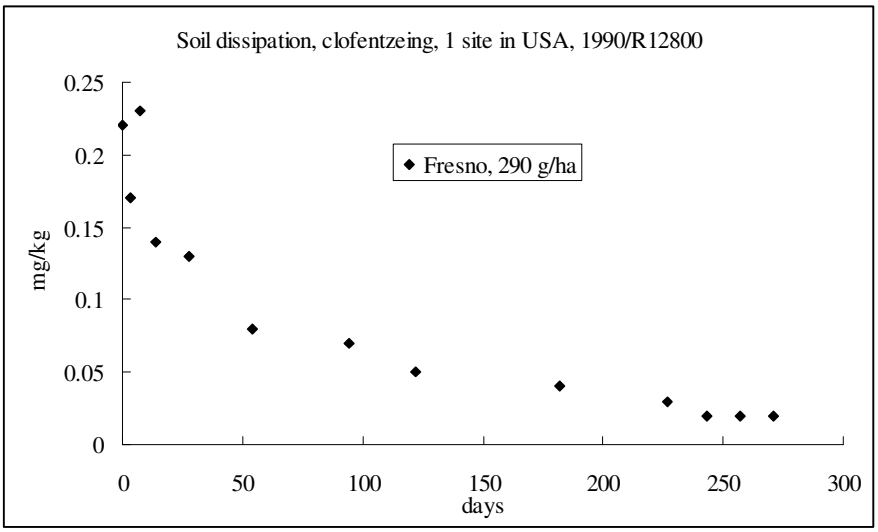


Figure 7. Field dissipation studies on bare soil in 1990 in USA (Castro, L., 1991, R-12800).

A site near Trenton, Ontario, was treated with two applications of 600 mL 50% (w/v) suspension concentrate (0.30 kg ai) per hectare (Bardalaye, P., 1988, R-12709). The first application to bare ground was made on May 27, 1987, and the second one on August 1, 1987. Duplicate plots 15 m apart were sprayed; the untreated control plot was 50 m from the treated plots. At each sampling time three sets of five cores were sampled from both Plot 1 and Plot 2. The cores were subdivided into 0 – 7.6 cm (1st layer), 7.6 – 15 cm (2nd layer), 15 – 23 cm (3rd layer) and 23 – 30 cm (4th layer) horizons. Soil samples were collected before the first treatment, immediately after 1st treatment (1 hour), and 1, 3, 7,14, 28,and 60 days, and then immediately after the second treatment, and 1, 3, 7,14, 28, 28, and 60 days.

The highest level of clofentezine residues found in soil was 0.26 mg/kg. There was no evidence of the chemical leaching to depths below 0 – 7.6 cm. Clofentezine residues were located almost exclusively in the 0 – 7.6 layer, the mean residues in the 7.6 – 15 cm layer was 0.02 mg/kg or less in all but one sample(at day 3 after second treatment in plot 2,). Analysis of the 15 – 23 cm and 23 – 30 cm cores from this sample together with representative cores from 0, 7 and 28 days at the same depths showed no clofentezine residues above the limit of determination. The rate of decline following the second application was similar to that from the first treatment. Clofentezine dissipates quickly in soil (Table 26). The highest level of clofentezine residues in soil treatment with the chemical at maximum use rates was 0.26 mg/kg.

Table 26. Field dissipation studies on bare soil in 1987 in Canada

Trial	Applic rate, kg/ha	Initial conc, mg/kg	clofentezine as % of original concentration in 0 – 7.6 cm soil.					
			1 days	3 days	7 days	14 days	28 days	56 days
Replicate I	Plot area 392 sq m. Soil nature: pH 5.0, 2.6% organic matter, 91% Sand.							
1980 treat in May	0.30	0.09	211%	111%	144%	67%	56%	< 22%
			1 days	3 days	7 days	14 days	28 days	56 days
Replicate IV	Plot area 392 sq m. Soil nature: pH 5.5, 2.7% organic matter, 93% Sand.							
1980 treat in May	0.30	0.11	118%	100%	118%	64%	27%	18%
			1 days	3 days	7 days	14 days	28 days	60 days
Replicate II	Plot area 392 sq m. Soil nature: pH 5.6, 3.0% organic matter, 91% Sand.							
1980 treat in May	0.30	0.13	100%	92%	46%	31%	27%	15%

Trial	Applic rate, kg/ha	Initial conc, mg/kg	clofentezine as % of original concentration in 0 – 7.6 cm soil.					
			1 days	3 days	7 days	14 days	28 days	56 days
Replicate III 1980 treat in May			1 days	3 days	7 days	14 days	28 days	60 days
	Plot area 392 sq m. Soil nature: pH 5.6, 2.4% organic matter, 91% Sand.							
	0.30	0.16	113%	81%	31%	25%	19%	19%

Three soil residue trials were conducted in Italy during 1990 applying clofentezine as a 50% suspension concentrate formulation, CR 16244 to apple or pear trees (Godfrey, T.L., Peatman, M.H., 1993, R-12711). The soil was sampled from the plots along a diagonal line underneath the tree spray area. Soil was sampled on three occasions after application (between June and October) to a depth of 30 cm. Following single applications of clofentezine to fruiting apple and pear trees residues in the 0 – 10 cm layer of soil on the day of application were < 0.01, 0.01 and 0.06 mg/kg at the three sites. At the second sampling time (60 – 63 days) and at the final time (121 – 126 days) mean residues were < 0.01 mg/kg (LOD). No residues were detected in the lower soil layers.

Six soil residue trials were conducted in the UK during 1982 applying clofentezine as a 50% wettable powder formulation, CR 15456 to apple trees (Manley, J.D., Snowdon, P.J., 1983, R-12697). Soil was sampled on one occasion close to normal harvest in August to a depth of 7.5 cm. Following single applications of clofentezine to apple trees residues in the top layer of soil were 0.02 mg/kg or less (96 – 120 days after application). After two spray applications residue levels were 0.06 mg/kg or less (49 – 59 days after the second application, and 105 to 109 days after the first application)

An orchard soil residue decline trial was conducted at Prosser, Washington, U.S.A., 1985, applying clofentezine 50% suspension formulation (Manley, J.D. and P.J. Snowdon, 1985, R-12708). Duplicate plots of apple trees were sprayed at petal fall at a rate of 0.69 kg ai/ha – the dosage for apples - and then again at the same rate when the developing fruit were between 2.5 cm and 5.1 cm in diameter. The soil below the trees was sampled at intervals beginning on the day after the first spray (May, 1985) until 90 days after the second spray (September, 1985). 2 cores were taken from each of the two replicate plots of eight trees and divided into 0 to 7.5 cm and 7.5 to 15 cm depth segments.

A similar decline of clofentezine residues in the orchard soil occurred after each application to apples (Table 27).

Table 27. Summary of clofentezine residues in orchard soil in USA.

Application rate (g ai/ha)	Interval (days since last application)	Clofentezine residue level (mg/kg)	
		0 - 7.5 cm	7.5 – 15.0 cm
692	Pre-treatment [a]	0.008, 0.006	N.D., 0.004
	1	0.20, 0.18	0.11
	3	0.35	0.11
	7	0.05	0.09
	14	0.05	0.04
	28	0.05	0.04
	60	< 0.01	< 0.01
692+692	1	0.40	0.13
	3	0.39, 0.28	0.10
	7	0.10	0.13
	14	0.09	0.04
	28	0.06	0.05
	69	< 0.01	< 0.01
	90	< 0.01	< 0.01

a - Apparent residues (N.D. denotes non-detectable residues).

An orchard soil residue decline trial was conducted at Sodus, New York, U.S.A., 1984, applying clofentezine 50 SC formulation, CR 16244 (Snowdon, P.J., 1986, R-12705). Triplicate plots of apple trees were sprayed pre-bloom at a rate of 0.02% ai and then the same rate when developing fruit were between 2.5 and 4.0 cm in diameter. The soil below the trees was sampled at intervals

beginning on the day after the first spray (May 1984) until 156 days after the second spray (December, 1984). Two cores were taken around each tree to a depth of 15 cm and divided into 0 to 7.5 cm and 7.5 to 15.0 cm depth segments. Mean clofentezine residue levels found in the top 7.5 cm of the orchard soil remained close to the determination limit for the duration of the study, ranging from < 0.05 to 0.07 mg/kg (Table 28). The highest residue found in an individual replicate was 0.10 mg/kg on the day after the second application. Residues in soil at the lower sampling depth of 7.5 to 15.0 cm were determined only at 14 days after the first and second sprays, respectively. All other values were below the determination limit.

Table 28. Summary of clofentezine residues in orchard soil in USA (Snowdon, P.J., 1986, R-12705).

Application rate (% ai)	Interval (days since last application)	Mean clofentezine residue level (mg/kg)	
		0 - 7.5 cm	7.5 – 15.0 cm
0.02	Pre-treatment	< 0.05	< 0.05
	1	< 0.05	
	3	0.05	
	7	< 0.05	
	14	0.06	
	28	< 0.05	
	63	< 0.05	
0.02+0.002	1	0.07	< 0.05
	3	0.07	
	7	< 0.05	
	14	0.07	
	35	< 0.05	
	67	< 0.05	
	123	< 0.05	
	156	0.05	

Mature apple trees from one site in Canada (Osoyoos, British Columbia) were sprayed with clofentezine 50% suspension concentrate (Castro, L., 1987, R-12805). Two application was made during the early season and at 48 days before normal harvest. The nominal rate of both treatments was 0.28 ai/ha. Litter and soil samples were collected before and immediately after the first treatment, and at 1, 3, 6, 13, 27 and 61 days. Similar sampling plan was applied after the second treatment. Residues of clofentezine were only found in the orchard litter. No leaching into soil was found at any time point. Clofentezine residues were comparable in the litter between trees (0.67 mg/kg) and the drip-line 0.88 mg/kg). Residues of clofentezine in the litter decline rapidly with apparent half-life of two weeks or less. Residues had declined to less than 0.1 mg/kg within four weeks of the second treatment. Clofentezine binds strongly onto litter and does not leach into orchard soil.

Crop rotation studies

Information on the fate of clofentezine in follow-on crop studies was made available to the meeting.

A three-step evaluation procedure to assess the potential for residues of clofentezine in following crops was undertaken (Allen, R., 1997, R-12853). Two possible scenarios were assessed:

1. Clofentezine used in cut flowers with a follow-on crop of leafy vegetables (lettuce or spinach) in the same year.
2. Clofentezine used in strawberries for 3 successive years followed by leafy vegetables (lettuce or spinach) in the late summer of the same year.

The low water solubility and relatively high octanol/water partition coefficient of clofentezine mitigate against the uptake of clofentezine residues form soil into follow-on crops. Residues of clofentezine and any bound residues are expected to be low (Table 29).

Table 29. Calculated availability of clofentezine residues to follow-on crops (Allen, R., 1997, R-12853)

	PECs at sowing (mg/kg soil)[a]	Maximal solubility in water (µg/L)	Available residue in 0.2 L soil water (µg/kg soil)	% of total residue in soil water available for uptake
Scenario 1	0.14	2.5	0.5	0.4
Scenario 2	0.04	2.5	0.5	1.3

a - PEC: pesticide equivalent concentration

The uptake of residues into the fruit and foliage of miniature orange trees (*Citrus mitis* Blanco) and the foliage of apple seedlings following soil application of [^{14}C]-clofentezine formulated as 50% (w/w) WP has been investigated under glasshouse conditions (Kelly, I.D., 1985, R-12789). Two applications, 61 days apart, were made at rates equivalent to 0.6 kg ai/ha and 1.2 kg ai/ha. Plant samples were analysed at intervals of up to 360 days from the first treatment.

After 360 days, 27.6% of the radioactivity applied in the orange trial and 21.0% of that applied in the apple trial was still associated with the soil. The majority of this radioactivity (> 80%) was in the form of bound residues. Extractable residues in orange fruit were less than 0.005 mg/kg at all sampling times (Table 30). Fibre bound residues were less than 0.01 mg/kg. Residues in fruit did not show a steady increase with time, but reached a maximum after 120 days. Extractable residues in orange foliage (maximum value 0.011 mg/kg) were reasonably constant at each time point studied, whereas fibre bound residues increased, reaching a value of 0.028 mg/kg after 360 days. Similarly, in apple foliage extractable residues (maximum value 0.016 mg/kg) remained fairly constant between 61 and 360 days, while the fibre bound residue increased to a level of 0.057 mg/kg after 360 days (Table 31).

Table 30. Residue distribution of clofentezine in uptake experiments with orange trees

Time (days)	Residue levels (mg/kg)					
	Orange fruit		Orange leaves		Soil	
	Extractable	Fibre-bound	Extractable	Fibre-bound	Extractable	Bound
61	0.002	< 0.001	0.011	0.002	--	--
120	0.002	0.008	0.008	0.013	--	--
240	0.004	0.002	0.007	0.014	--	--
360	0.003	0.003	0.010	0.028	0.062	1.01

Table 31. Residue distribution of clofentezine in uptake experiments with apple seedlings

Time (days)	Residue levels (mg/kg)			
	Apple leaves		Soil	
	Extractable	Fibre-bound	Extractable	Bound
61	0.004	0.003	--	--
120	0.016	0.018	--	--
240	0.010	0.014	--	--
360	0.012	0.057	0.083	0.507

RESIDUE ANALYSIS

Analytical methods

The Meeting received descriptions and validation data for analytical methods for residues of clofentezine in various raw agricultural commodities of plant and animal origin and processed products.

Methods rely on HPLC-UV, HPLC-PDA, GC and GC-MSD for analysis of clofentezine in the various matrices. Clofentezine and its metabolites are determined and reported.

Data collection methods

Analytical method for residues of Clofentezine and metabolites in animal tissues and milk. (Manley, J.D., Peatman, M.H., Snowdon, P.J., 1985, R-21211A)

Analyte:	2-chlorobenzoic acid	GC-ECD
LOQ:	0.05 mg/kg in both animal tissues and milk	
Description	Clofentezine residues in minced tissues or whole milk are hydrolysed to 2-chlorobenzoic acid by addition of hydrobromic acid (figure 11), cleaned up by partition into diethyl ether and back-partition between alkali and ether. The extracts are then concentrated, methylated with diazomethane and the residues finally determined by gas chromatography with electron capture detection (GC-ECD).	

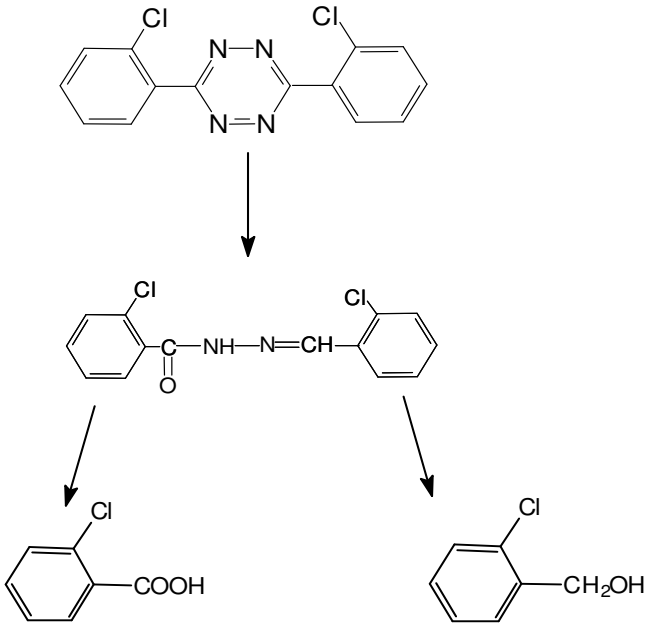


Figure 9. Possible hydrolysis scheme for clofentezine with hydrobromic acid.

Addendum to Report RESID/85/32: Analytical method for residues of Clofentezine and metabolites in milk. (Ferreira, E.M., 1997, R-21211B)

Analyte:	2-chlorobenzoic acid	GC-MSD
LOQ:	0.05 mg/kg in egg	
Description	Clofentezine residues in egg are hydrolysed to 2-chlorobenzoic acid by addition of hydrobromic acid, cleaned up by partition into diethyl ether and back-partition between alkali and ether. The extracts are then concentrated, methylated with diazomethane and the residues finally determined by gas chromatography with mass selective detection (GC-MSD).	

Residues of Clofentezine and metabolites in the tissues and eggs of laying hens following a 28 day feeding study in the UK 1986. (Peatman, M.H., Snowdon, P.J., 1987, R-12735)

Analyte:	2-chlorobenzoic acid	GC-ECD
LOQ:	0.05 mg/kg in both animal tissues and egg	
Description	Clofentezine residues in minced tissues or whole milk are hydrolysed to 2-chlorobenzoic acid by addition of hydrobromic acid, cleaned up by partition into diethyl ether and back-partition between alkali and ether. The extracts are then concentrated, methylated with diazomethane and the residues finally determined by gas chromatography with electron capture detection (GC-ECD).	

Analytical method for the determination of residues of Clofentezine and metabolites in animal tissues and milk. 2nd edition. (Peatman, M.H., Snowdon, P.J., 1988, R-21212)

Analyte:	2-chlorobenzoic acid	GC-ECD
LOQ:	0.01 mg/kg for milk and 0.05 mg/kg for animal tissues	
Description	Clofentezine residues in minced tissues or whole milk are hydrolysed to 2-chlorobenzoic acid by addition of hydrobromic acid, cleaned up by partition into diethyl ether and back-partition between alkali and ether. The extracts are then concentrated, methylated with diazomethane and the residues finally determined by gas chromatography with electron capture detection (GC-ECD).	

Analytical method for the determination of residues of Clofentezine and 4-hydroxy-Clofentezine in animal tissues by gas chromatography. (Peatman, M.H., Snowdon, P.J., 1989, R-21213)

Analyte:	2-chlorobenzoic acid	GC-MSD
LOQ:	0.05 mg/kg in egg	
Description	Clofentezine and 4-hydroclofentezine residues in animal tissues are hydrolysed to 2-chlorobenzoic acid by addition of hydrobromic acid, cleaned up by partition into diethyl ether and back-partition between alkali and ether. The extracts are then concentrated, methylated with diazomethane and the residues finally determined by gas chromatography with mass selective detection (GC-MSD).	

Analytical method for the determination of residues of 4-hydroxy Clofentezine in milk and animal fat by high performance liquid chromatography. 2nd edition. (Peatman, M.H., Snowdon, P.J., 1989, R-12716)

Analyte:	4-OH clofentezine	HPLC-UV
LOQ:	0.01 mg/kg for milk and 0.05 mg/kg for fat	
Description	The method involves extraction of residues by homogenisation/ultrasonic agitation with acetone (milk) or hexane/ acetone reflux (fat). Complexed/conjugated forms in milk are released by enzymic hydrolysis with snail digestive juice before partitioning into ethyl acetate/ hexane. Fat extracts are cleaned up by elution through a diol solid phase cartridge. Residues are determined by normal phase HPLC-UV. Confirmation was carried out with reverse phase HPLC. No interferences were observed.	

Analytical method for the determination of free Clofentezine residues in milk and animal fat by high performance liquid chromatography. (Peatman, M.H., Snowdon, P.J., 1989, R-13121)

Analyte:	Clofentezine	HPLC-UV
LOQ:	0.01 mg/kg for milk, 0.02 mg/kg for fat	
Description	Clofentezine was determined in milk and in fatty tissue from dairy cows by solvent extraction, clean-up by acetonitrile/hexane partitioning followed by elution through a Sep-pak silica cartridge, and determination by HPLC-UV. Confirmation was carried out with reverse phase HPLC No interferences were observed.	

Analytical method for residues of NC21314 (Clofentezine) in apples. (Snowdon, P.J., Crofts, M, 1981, R-13160A)

Analyte:	Clofentezine	HPLC-UV
LOQ:	0.01 mg/kg for apple	
Description	Clofentezine residues are extracted with methanol: dichloromethane (1:9), cleaned up by silica Sep-Pak cartridge and determined with HPLC-UV using methanol:water (3:1) as mobile phase.	

Analytical method for residues of NC21314 (Clofentezine) in apples and pears. (Improved method.) (Snowdon, P.J., 1983, R-18736)

Analyte:	Clofentezine	HPLC-UV
LOQ:	0.01 mg/kg for apple and pear	
Description	Clofentezine residues are extracted with acetone, cleaned up by silica Sep-Pak cartridge and determined with HPLC-UV.	

Analytical method for residues of NC21314 (Clofentezine) in apples. (2nd edition.). (Manley, J.D., Snowdon, P.J., 1985, R-13160)

Analyte:	Clofentezine	HPLC-UV
LOQ:	0.01 mg/kg for apple	
Description	Clofentezine residues are extracted with acetone, cleaned up by silica Sep-Pak cartridge and determined with HPLC-UV.	

Analytical method for residues of Clofentezine in miscellaneous fruit crops. (Manley, J.D., Snowdon, P.J., 1986, R-12714)

Analyte:	Clofentezine	HPLC-UV
LOQ:	0.01 mg/kg for apple, pear, grape, peach and strawberry	
Description	Clofentezine residues are extracted with acetone, cleaned up by silica Sep-Pak cartridge and determined with HPLC-UV.	

Residue analysis of Clofentezine in strawberries and apples. (Wende, S., 2001, R-14208)

Analyte:	Clofentezine	HPLC-PDA
LOQ:	0.02 mg/kg for apple and strawberry	
Description	A residue method validation for Clofentezine determination in strawberries and apples was successfully performed according to a modified DFG multiresidue method S19. Residues were extracted with acetone/water (2:1), a clean-up step performed by partitioning into cyclohexane/ethyl acetate (1:1) followed by gel-permeation chromatography, and analysed by HPLC/PDA. No interferences were observed.	

Independent laboratory validation for the determination of clofentezine residues in strawberries and apple based on the DFG-S19 multiresidue enforcement methods. (Chambers, J.G., 2006, R-18909)

Analyte:	Clofentezine	HPLC-PDA
LOQ:	0.02 mg/kg for apple and strawberry	
Description	The method reported by Wende 2001 (20011171/01-RVP) was independently validated in strawberries and apples The method was based on DFG multiresidue method S19. Residues were extracted with acetone/water (2:1), a clean-up step performed by partitioning into cyclohexane/ethyl acetate (1:1) followed by gel-permeation chromatography, and analysed by HPLC/UV. No interferences were observed.	

Analytical method for residues of Clofentezine in citrus fruit. (Manley, J.D., Snowdon, P.J., 1985, R-13148)

Analyte:	Clofentezine	HPLC-PDA
LOQ:	0.01 mg/kg for citrus fruit and 0.05 mg/kg in orange jam	
Description	Clofentezine was extracted from citrus fruit with acetone, followed by separation into hexane and clean-up with Sep-pac cartridge, and determination by HPLC-UV.	

Residue of Clofentezine in almond hulls and nutmeats following application of Apollo 50 SC at early and mid-season. (Castro, L., Powley, C., 1986, R-13184)

Analyte:	Clofentezine	HPLC-UV
LOQ:	0.01 mg/kg for almond hulls and nutmeats	
Description	Almond hulls were blended with acetone. In a partition clean-up process the filtered acetone is diluted with water and extracted with hexane. After taking the sample to dryness the residues are reconstituted in hexane. In a second clean-up step the sample is loaded onto a silica gel cartridge, eluted with a Dichloromethane/hexane mixture, taken to dryness, and reconstituted in marker solution before determination with HPLC-UV. Almond nutmeats are blended with acetonitrile, which is partitioned with hexane, diluted with water and then extracted with hexane, which is taken to dryness. Silica clean-up and determination are done as for nutmegs. With almond hulls, interferences of 0.01 – 0.05 mg/kg are regularly observed, but rarely for almond nuts (0.004 mg/kg).	

Stability of Clofentezine in almond hulls and nutmeats under freezer storage for a period of two years. (Castro, L., 1988, R-13240)

Analyte:	Clofentezine	HPLC-UV
LOQ:	0.01 mg/kg for almond hulls and nutmeats	
Description	Almond hulls were blended with acetone. In a partition clean-up process the filtered acetone is diluted with water and extracted with hexane. After taking the sample to dryness the residues are reconstituted in hexane. In a second clean-up step the sample is loaded onto a silica gel cartridge, eluted with a Dichloromethane/hexane mixture, taken to dryness, and reconstituted in marker solution before determination with HPLC-UV. Almond nutmeats are blended with acetonitrile, which is partitioned with hexane, diluted with water and then extracted with hexane, which is taken to dryness. Silica clean-up and determination are done as for nutmegs. With almond hulls, interferences of 0.01 – 0.05 mg/kg are regularly observed, but rarely for almond nuts (0.004 mg/kg).	

Enforcement methods

Validation of an analytical method for the determination of Clofentezine and metabolites in animal tissue. Independent Laboratory Validation. (Witte, A., 2004, R-17532)

Analyte:	2-chlorobenzoic acid	GC-MSD
LOQ:	0.01 mg/kg for eggs and milk, 0.02 mg/kg for fat and meat and 0.05 mg/kg for liver	
Description	This report validates the method which was developed based on the methods of Resid/85/32 and Resid/87/30. Clofentezine residues in minced tissues or whole milk are hydrolysed to 2-chlorobenzoic acid by addition of hydrobromic acid, cleaned up by partition into diethyl ether / hexane and back-partition between alkali and ether. The extracts are then concentrated and cleaned up by anion-exchange chromatography. 2-chlorobenzoic acid is derivatised using n-methyl-n-trimethylsilyltrifluroacetamide (MSTFSA) and the residues finally determined within 24 hours by gas chromatography with mass selective detection (GC-MSD).	

Independent Validation of an analytical method for the determination of Clofentezine and its metabolites in animal tissues. (Chambers, J. G., 2006, R-20408)

Analyte:	2-chlorobenzoic acid	GC-MSD
LOQ:	0.01 mg/kg for eggs and milk, 0.02 mg/kg for muscle and fat and 0.05 mg/kg for liver and kidney	
Description	This report includes the validation and confirmation of the method which was developed and validated by Witte (2004, R-17532) based on modifications to the methods of Resid/85/32 and Resid/87/30.	

Independent laboratory validation of an analytical method for determination of residues of Clofentezine in miscellaneous fruit crops. (Pires, A., 2000, R-12959)

Analyte:	Clofentezine	HPLC-DAD
LOQ:	0.01 mg/kg for apple, pear, grape, peach and strawberry	
Description	This report validates and confirms the method of Manley and Snowdon, 1986a. In the validation experiments, a slightly changed mobile phase allowed better separation of interfering peaks near to the Clofentezine retention time. Clofentezine residues are extracted with acetone, cleaned up by silica Sep-Pak cartridge and determined with HPLC-DAD.	

Residue analytical method for the determination of Clofentezine technical (dried) in cores of sunflower (rich fat) and wheat grains (low fat). (Geffke, T., 2003, R-16387)

Analyte: Clofentezine HPLC-DAD
LOQ: 0.01 mg/kg for sunflower seeds and wheat grain
Description Sunflower seeds were blended with acetone. In a partition clean-up process the filtered acetone is diluted with water and extracted with hexane. After taking the sample to dryness the residues are reconstituted in hexane. In a second clean-up step the sample is loaded onto a silica gel cartridge, eluted with a Dichloromethane/hexane mixture, taken to dryness, and reconstituted in marker solution before determination with HPLC-DAD. Wheat grains are blended with acetonitrile, which is partitioned with hexane, diluted with water and then extracted with hexane, which is taken to dryness. Silica clean-up and determination are done as for sunflower seeds.

Residue analytical method for the determination of Clofentezine technical (dried) in cores of sunflower (rich fat) and wheat grains (low fat). (Bardalaye, P., Kelly,I. D., 1988, R-12715)

Analyte: Clofentezine GC-ECD
LOQ:
Description Clofentezine has been screened through EPA multi-residue protocols I and III, with apples as the substrate. Residues of clofentezine were extracted with acetonitrile or acetone followed by liquid-liquid partitioning, Florisil column clean-up and gas chromatography determination with electron capture detection. At high column temperatures, decomposition of clofentezine during gas chromatographic analysis was observed and this is believed to contribute significantly to the low recoveries observed (15 to 50% at fortification levels of 0.05 to 0.5 mg/kg). It is thus concluded that the EPA multi-residue protocols I and III may be used for the qualitative detection of clofentezine but quantitative analysis may be irreproducible.

Recovery data of fortified in various matrices from the internal and independent laboratory validation testing are summarized in Table 32.

Table 32. Validation data for analytical methods for the determination of clofentezine residues in food

Sample Matrix	Fortific. Level [mg/kg]	Average recovery [%]	RSD [%]	No. of analyses	Ref
Data collection methods					
Liver	0.20 - 6.0	98.4	6.4	10	R-21211A
Kidney	0.20 - 2.0	95.9	8.0	7	
Muscle	0.06 - 0.60	92.5	4.4	9	
Heart	0.04 - 0.20	98.8	15.8	4	
Fat	0.05 - 0.40	93.6	7.6	8	
Milk	0.10 - 1.0	88.0	17.2	30	
Egg	0.40 - 1.0	86.0	9.5	3	R-21211B
Liver	0.05 - 0.20	104.0	28.6	2	R-12735
Kidney	0.40 - 1.0	82.0	17.2	2	
Muscle	0.10 - 0.40	88.8	11.2	6	
Fat	0.10 - 0.40	114.5	13.8	4	
Eggs	0.40 - 1.0	86.0	9.5	3	
Liver	0.05 - 0.20	78.1	14.8	19	R-21212
Muscle	0.05 - 0.20	92.7	10.1	7	
Fat	0.05 - 0.20	81.0	12.9	7	
Milk	0.01 - 0.20	83.6	11.4	14	
Liver	0.10 - 0.25	101.2	13.6	16	R-21213
Kidney	0.10 - 0.25	99.8	14.5	5	
Muscle	0.10 - 0.25	88.4	11.9	9	
Liver (4-hydroxy-derivate)	0.05 - 0.25	86.6	10.1	11	

Sample Matrix	Fortific. Level [mg/kg]	Average recovery [%]	RSD [%]	No. of analyses	Ref
Kidney (4-hydroxy-derivate)	0.025 - 0.25	94.8	15.2	8	
Muscle (4-hydroxy-derivate)	0.05 - 0.25	82.9	14.2	14	
Fat (4-hydroxy-derivate)	0.03 - 0.14	84.0	15.0	23	R-12716
Milk (4-hydroxy-derivate)	0.02 - 0.20	93.6	15.0	11	
Animal fat	0.01 – 0.2	88.1	6.6	12	R-13121
Milk	0.01 – 0.2	79.8	9.4	10	
Animal fat [a]	0.04 – 0.08	74.5		4	
Milk [a]	0.08	71.0		3	
Apple	0.05 - 0.50	84.5	11.0	42	R-13160A
Apple and pear	0.01 - 1.00	90.9	9.7	31	R-18736
Apple	0.01 - 1.00	91.5	5.6	51	R-13160
Apple, pear, grape, peach and strawberry	0.01 - 0.20	92.7	7.1	28	R-12714
Apple	0.02-0.2	81	9	10	R-14208
Strawberry	0.02-0.2	80	8	10	
Apple	0.02-0.2	89	4.3	10	R-18909
Strawberry	0.02-0.2	87	4.4	10	
Citrus fruit	.01 – 1.0	86.0	8.7	7	R-13148
Orange fruit	0.01-0.10	76.7	5.0	10	R-15921
Orange jam	0.05-0.50	83.1	5.6	10	
Almond nuts	.05 – 1.0	79	6.1		R-13184
Almond hulls	.05 – 100	89	14	6	
Almond nuts	.02 – 0.20	85	19	0	R-13240
Almond hulls	.02 – 2.0	75	8.9		
Enforcement Methods					
Egg	0.01-0.1	96	3	10	R-17532
Fat	0.02-0.2	91	14	10	
Liver	0.05-0.5	100	2	10	
Meat	0.02-0.2	74	12	10	
Milk	0.01-0.1	105	8	10	
Muscle	0.02-0.2	96	3.8	10	R-20408
Liver	0.05-0.5	74	8.2	14	
Kidney	0.05-0.5	79	8.1	10	
Milk	0.01-0.1	91	16.9	10	
Fat	0.02-0.2	90	12.2	14	
Eggs	0.01-0.1	94	6.9	10	
Apple	0.01-0.10	84.6	14.2	10	R-12959
Peach	0.01-0.10	99.1	6.6	10	
Grape	0.01-0.10	98.0	11.7	9	
Strawberry	0.01-0.10	88.3	12.4	9	
Sunflower seeds	0.01-0.1	87	11	10	R-16387
Wheat grain	0.01-0.1	95	4	10	

a - Confirmatory method

Stability of residues in stored analytical samples

The Meeting received information on the stability of residues of clofentezine in apple, peach, almond (hulls and nutmeats), muscle, liver, fat, and milk.

Individual whole apple fruits were each fortified with 10 pg of [^{14}C]-clofentezine and stored in deep freezer at -18 °C (Snowdon, P.J., 1984, R-13127). Fruit were removed at intervals of up to 2 years (three apples per interval) for immediate residue analysis. Residues were extracted initially by a surface rinse in dichloromethane followed by maceration of the whole fruit with a dichloromethane/methanol mixture and subsequent clean-up through a Sep-pak silica cartridge. Radioactivity in final extracts and remaining solids was determined by high performance liquid chromatography and/or liquid scintillation counting (after oxidative combustion of solids).

Clofentezine residue levels in laboratory fortified whole apples remained reasonably stable and extractable after 379 days (1 year) storage with 86% of the nominal residue still present (Table 33). Regression analysis of the storage data indicated a first order decline of extractable residues over the 769 day (2 year) test period with a computed half-life of 1480 days (approx. 4.0 years). At all intervals of the study, fibre bound activity accounted for the remainder of the fortified residue.

Table 33. Stability of clofentezine residues in frozen apples (Snowdon, P.J., 1984, R-13127)

Time (days)	Extractable residue (%)	Fibre-bound residue (%)
0	100.3	< 0.1
34	99.5	< 0.1
98	98.5	2.8
187	94.8	5.2
379	86.4	8.6
558	79.7	20.0
769	69.8	25.5

Untreated peaches were chopped in half. Fifty gram samples were fortified at 0.02 mg/kg or 1.0 mg/kg levels with clofentezine by pipetting an acetone solution onto peach skin. These samples were stored in a freezer at -15 °C and analysed at designated time points (nominally 3, 6, 12, 18 and 24 months) (Castro, L., 1988, R-13242). Peach fruit was extracted by blending with acetone which was then filtered and diluted with water. The acetone-water mixture was extracted with hexane, which was then evaporated to dryness. The residues were reconstituted in hexane and purified through silica gel. The extracts evaporated again to dryness, reconstituted in a known volume of marker solution (40 µg/mL N-2-(2-propyl) phenylbenzamide in the HPLC mobile phase) and quantified by HPLC using UV detection. Clofentezine is relatively stable in peaches under freezer conditions. The data is scattered (Table 34), however, and the low correlation coefficients of the best-fit line indicates that the recoveries experienced throughout the study were a result of experimental variation rather than a trend. Based on the experimental results, however, clofentezine appears to be very stable in peaches during freezer storage.

Table 34. Stability of Clofentezine residues in frozen peaches

Time (days)	% residue remaining at 0.02 ppm (%)	% residue remaining at 1.0 ppm (%)
0	74	88
98	73	80
246	92	89
365	87	100
637	81	94
736	80	92

Samples of almond hulls and almond nutmeats were coarsely ground. Samples of hull were fortified at 0.02 mg/kg or 2.0 mg/kg levels with clofentezine. Samples of nutmeat were fortified at 0.02 or 0.2 mg/kg levels. These samples were stored in a freezer at -15 °C and analysed at designated time points (3, 6, 12, and 24 months) (Castro, L.E., 1988, R-13240).

Untreated hull samples freshly fortified from 0.02 to 2.0 mg/kg gave an average recovery of 75% with a standard deviation of 8.9%. Freshly fortified (0.02 – 0.2 mg/kg) almond nutmeat gave a recovery of 85% with a standard deviation of 19%. Clofentezine is stable in almond hulls and nutmeats under freezer conditions for at least one year (Table 35).

Table 35. Stability of Clofentezine residues in frozen almond and nutmeat

Time (months)	% residue remaining in almond nutmeat (%)		% residue remaining in almond hulls (%)	
	0.02 ppm	0.20 ppm	0.02 ppm	2.0 ppm
0	109	94	81	81 [a]
3	114	72	69	62
6	65	75	78	83
12	71	90	62	76
24	62	69	62	76

a - Accidentally fortified with 0.2 mg/kg.

Samples of muscle, liver, peritoneal fat and whole milk taken from dairy cattle were each fortified with 13 – 26 µg [^{14}C]-clofentezine (equivalent to 1.04 mg/kg in each tissue type and 0.26 mg/kg in milk) and stored in deep freeze at -20 °C (Peatman, M.H., Snowdon, P.J., 1985, R-13164). At 0 and 6 months storage, unchanged clofentezine was determined with a cold solvent extraction followed by acetonitrile/hexane partitioning and concentration with Sep-Pak cartridge. From 6 months storage onward, total clofentezine-derived residues were determined by derivation to 2-chlorobenzoic acid (2-CBA). The acid hydrolysis effectively splits the tetrazine ring of clofentezine an derivatives non-symmetrically, with only one of the two tetrazine carbon atoms becoming part of a 2-CBA molecule, the hydrolysis proceeding on a 1:1 mole ratio basis. Only half of the ^{14}C-carbons from the hydrolysed residues in this study will be incorporated into 2-CBA molecules. Therefore, total residues were derived from the method by multiplication of the activity recovered as 2-CBA by a factor of two.

After 6 months storage, the mean percentage of parent had fallen to 38.3% (muscle), 71.7% (liver), 50% peritoneal fat), and 50.1% (milk) (Table 36). Comparison with the data on radioactivity showed that more than 90% of the original residue in muscle, liver and fat, and approximately 84% in milk was accounted for after 15 months storage. Clofentezine is relatively unstable in products of animal origin, but the total clofentezine-derived residue is stable in animal products for at least 15 months.

Table 36. Stability of clofentezine residues in frozen animal tissue

Time (months)	% extractable residues											
	Muscle			Liver			Peritoneal fat			Milk		
	Parent [a]	2-CBA [b]	% of residue accounted for [c]	Parent	2-CBA	% of residue accounted for	Parent	2-CBA	% of residue accounted for	Parent	2-CBA	% of residue accounted for
0	83.3		83.3	82.7		82.7	85.7		85.7	87.7		87.7
1	55.0		55.0	65.5 [d] 61.7		65.5 61.7	61.2 76.1		61.2 76.1	63.0		63.0
3	52.9		52.9	80.1		80.1	62.3		62.3	80.4		80.4
6	38.3		38.3	71.7		71.7	50.0		50.5	50.1		50.1
6		41.5	83.0		33.5	67.0					35.5	71.0
15		46.0	92.0		50.7 50.0	101.3		55.5 47.0	111.0 94.0		42.0	84.0

a - mean % of radioactivity quantified as unchanged clofentezine.

b - mean % of radioactivity quantified as 2-chlorobenzoic acid (2-CBA).

c - in final extract as determined by analytical method.

d - two sets of samples analysed at these intervals.

USE PATTERN

Clofentezine is a specific contact acaricide for use on a range of fruits, vegetables and other crops. It acts primarily as an ovicide with some effect on young motile stages and with long residual activity. It is particularly effective against winter eggs of the European red mite (*Panonychus ulmi*). Labels and English translations were available for all the uses. Information on registered uses included in this monograph is generally limited to countries where supervised trials had been conducted, and is summarized in Table 37.

Table 37. Registered field uses of clofentezine

Crop	Country or Region	Formulation End-use product	F/G[a]	Application					PHI [days]
				Method	No. per crop season min. max.	kg ai/hL max.	Water L/ha per appl. Min. max.	kg ai/ha per applic. min. max.	
Apricot Plum									
		20 SC							
Almond	California, USA								14
Almond	USA								
Apple	Argentina	50 SC	F	Spray	1	0.01-0.015	2500-3000		28
Apple	Belgium	50 SC	F	Spray	1		1500	0.1325	none
Apple	Canada	50 SC	F	Spray	1	0.032-0.064	> 475	0.15-0.30	45
Apple	Canada					> 0.004	950		
Apple	France	50 SC	F	Spray	1	0.02			42
Apple	Greece	50 SC	F	Spray	1	0.015	2000-2500		45
Apple	Italy	50 SC	F	Spray	1	0.02	1500-2000	> 0.2 (winter egg)	30
Apple	South Africa	50 SC		Spray		0.02			30
Apple	UK	50 SC	F	Spray	1	0.02-0.045	440-1000	0.2	28
Apple	USA	50 SC	F	Spray	1		470-3740	0.118-0.235	45
Apricot	USA	50 SC	F	Spray	1		470-3740	0.059-0.235	21
Apricot, Peach, Plum Cherry	Greece	50 SC	F	Spray	1	0.015	2000-2500		45
Cherry	UK								
Citrus	Greece	50 SC	F	Spray	1	0.015			30
Citrus	Italy	50 SC	F	Spray	1	0.015-0.02	500-2000		30
Citrus	Spain	50 SC or 20 SC	F	Spray		0.005-0.01	2000-4000		21
Cucumber	Greece	50 SC	F/G	Spray	1	0.015			4
Cucumber	Switzerland	50 SC	G	Spray	1	0.02			14
Cucurbits	France	50 SC		Spray		0.02		0.2	3
Currant	France	50 SC		Spray		0.02		0.2	45
Grapevine	France	50 SC		Spray	1	0.02	1000	0.2	42
Grape	France	20 SC							
Grapevine	Germany	50 SC	F	Spray	1			0.06-0.24	35
Grapevine	Italy	50 SC	F	Spray	1	0.01-0.015	1500-2000	> 0.2 (winter egg)	30
Grape	Italy	20 SC					Min 500	> 0.25 (during vegetation)	

Crop	Country or Region	Formulation End-use product	F/G [a]	Application					PHI [days]
				Method	No. per crop season min. max.	kg ai/hL max.	Water L/ha per appl. Min. max.	kg ai/ha per applic. min. max.	
Grapevine	Spain	50 SC				0.01-0.03	1000-1500		30
Melon	France	50 SC		Spray				0.2	3
Melon	Italy	20 SC, 50 SC	F/G	Spray	1	0.015-0.02	1500-2000 Min 500	> 0.2(winter egg)	15
Melon	Italy							> 0.25 (during vegetation)	
Melon	Spain	20 SC, 50 SC		Spray		0.01-0.02	500-1000		3
Nectarine	USA)								
Peach	USA,(California								21(10[b])
Pear	Argentina	50 SC	F	Spray	1	0.01-0.015	2500-3000		20
Pear	Argentina					0.01-0.015			
Pear	Argentina					0.015-0.02			
Pear	Belgium						(mini 1000)		
Pear	Canada	50 SC	F	Spray	1	0.032-0.064	> 475	0.15-0.30	21
Pear	Canada					> 0.004	950		
Pear	Greece								
Pear	Italy	20 SC					mini 500	> 0.25 (during vegetation)	
Pear	South Africa								
Pear	UK								
Pear	USA								21
Plum	Germany	50 SC	F	Spray	1	0.02	500-1500	0.075	
Plum	Italy	50 SC	F	Spray	1	0.02	1500-2000	> 0.2(winter egg)	30
Plum	Italy	20 SC					mini 500	> 0.25 (during vegetation)	
Plum	UK	50 SC	F	Spray	1	0.02-0.045	440-1000	0.2	56
Pome fruit	Australia	50 SC	F	Spray	1	0.015	1500-5000		21
Pome fruit	Germany	50 SC	F	Spray	1 per year	0.02	500-1500	0.075 0.1	35
Stone fruit	Australia	50 SC	F	Spray	1	0.015	run-off 1500-3000 (up to 5000)		21
Strawberry	France	50 SC		Spray				0.2	3
Strawberry	Germany	50 SC	F	Spray	1			0.3	NR [c]
Strawberry	Spain	50 SC, 20 SC		Spray		0.01-0.02	1000		3
Strawberry	The Netherlands	50 SC	F	Spray	1-2 interval 14-21 days	0.015	500-1000	0.075-0.15	NR [c]
Tomato	Italy	20 SC, 50 SC	F/G	Spray	1	0.02-0.03	1500-2000 Min 500	> 0.2(winter egg)	15
Tomato	Italy							> 0.25 (during vegetation)	

Crop	Country or Region	Formulation End-use product	F/G [a]	Application					PHI [days]
				Method	No. per crop season min. max.	kg ai/hL max.	Water L/ha per appl. Min. max.	kg ai/ha per applic. min. max.	
Tomato	Netherlands	50 SC		Spray	1-2	0.015	500-1500	0.075-0.225	3
Tomato	Spain	20 SC, 50 SC		Spray		0.01-0.02	1000-1500		3
Walnut	USA	50 SC		Spray	1		470-3740	0.059-0.235	30

a - F: field use. G: greenhouse use

b - PHI of 10 day is for California only.

c - NR = Not relevant: Application limited by growth stage of the crop.

RESIDUES RESULTING FROM SUPERVISED TRIALS

The Meeting received information on supervised field trials for clofentezine uses on the following crops.

Commodity	Country	Table No
Almond	USA	Table 49
Citrus	Greece, Italy and Spain	Table 38
Cucurbits	The Netherlands, France, Greece, Switzerland	Table 45
Currants	France	Table 44
Grapes	France, Italy, Spain, Germany	Table 42
Melons	Spain, France, Italy, Portugal, Greece	Table 46
Plum	USA, Germany	Table 41
Pome fruit	Argentina, Australia, Canada, UK, France, Greece, Germany, Belgium, Italy, South Africa, USA	Table 39
Stonefruit except plums	Australia, Italy, Greece, USA, UK	Table 40
Strawberry	France, Germany, Spain, the Netherlands	Table 43
Tomatoes	France, Germany, Greece, Italy, the Netherlands, Spain	Table 47
Walnut	USA	Table 48

Where residues were not detected, they are reported as below the LOQ. Residue data, application rates and spray concentrations have generally been rounded to two significant figures or, for residues near the LOQ, to one significant figure. Although trials included control plots, no control data are given in the tables except where residues in control samples exceeded the LOQ. Residue data are recorded unadjusted for percent recovery. Multiple results are recorded in the data tables where the trial design included replicate plots and where separate samples have been identified as being from these replicate plots. Results used for estimating maximum residues, HR and STMRs are double underlined.

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.

Conditions of the supervised residue trials were generally well reported in detailed field reports. Most field reports provided data on the sprayers used, plot size, residue sample size and sampling date. In multiple applications, the application rate, spray concentration and water volume may not have been exactly the same for all applications; the recorded values in the supervised trials summary tables are for the final application.

All samples were frozen soon after harvest and kept frozen in storage until shipment to the residue analysis lab. Upon receipt, all samples were transferred to deep freeze storage (≤ 15 °C). The samples of apples and peaches from supervised were stored unchopped. There were some samples of

almond from supervised trials stored unsplit and some samples were stored split. Intervals between sampling and analysis were reported for most trials and were within the acceptable proven storage stability duration. Stability studies performed on peach and apple indicate that there is no decline in clofentezine extractability and accountability for at least one and half years, and on almond no decline for at least one year.

Table 38. Results of residue trials with clofentezine conducted in citrus, foliar treatment

CITRUS Country, Year *Location* (variety)	Application					PHI days	Commodity	Residues, mg/kg clofentezine	Ref.
	Formulation	kg ai/ha	kg ai/hL	Water L/ha	No.				
ORANGE									
Greece, 1990 *Skala Lakonias*	SC	0.75	0.015	5000	1		control peel	0.09	R-13064
							control fruit	0.01	
						20	flesh	0.03	
						32		0.02	
						20	peel	0.86	
						32		0.67	
						20	whole	0.32	
						32		0.10	
Greece, 1990 *Nafplio*	SC	0.75	0.015	5000	1	30	flesh	0.03	
							peel	0.58	
							whole	0.18	
		1.5	0.03	5000	1	30	flesh	< 0.01	
							peel	0.33	
							whole	0.12	
Italy, 1984 *Catania*	SC		0.01	spray to run off	1		control peel	0.02	R-13146 (A/10-3)
						21	flesh	0.02	
							peel	0.29	
							whole	0.09	
						38	flesh	0.03	
(Tarocco)							peel	0.22	
							whole	0.09	
			0.02	spray to run off	1	21	flesh	0.05	
							peel	0.65	
							whole	0.26	
						38	flesh	0.02	
							peel	0.26	
							whole	0.08	
Italy, 1984 *Catania* (Moro)	SC		0.01	spray to run off	1		control peel	0.02	(A/10-4)
						21	flesh	0.02	
							peel	0.40	
							whole	0.14	
						38	flesh	0.02	
							peel	0.21	
							whole	0.08	
			0.02	spray to run off	1	21	flesh	0.07	
							peel	0.66	
							whole	0.27	
						38	flesh	0.05	
							peel	0.90	
							whole	0.31	

CITRUS Country, Year *Location* (variety)	Application					PHI days	Commodity	Residues, mg/kg clofentezine	Ref.
	Formulation	kg ai/ha	kg ai/hL	Water L/ha	No.				
Spain, 2001 *Valencia* (Navelina)	SC	0.481	0.01	4542	1	0	whole	0.18	R-15921 (R9)
						10	whole	0.08	
						20	whole	0.09	
						40	peel	0.24	
							flesh	< 0.01	
							juice	0.01	
							jam	< 0.05	
							whole	0.07	
						60	peel	0.16	
							flesh	< 0.01	
							whole	0.05	
Spain, 2001 *Valencia* (Navel)	SC	0.257	0.01	2674	1	0	whole	0.16	(R10)
						10	whole	0.12	
						20	whole	0.06	
						40	peel	0.32	
							flesh	0.02	
							juice	< 0.01	
							jam	< 0.05	
							whole	0.09	
						60	peel	0.08	
							flesh	< 0.01	
							whole	0.02	
Spain, 2001 *Valencia* (Satustiana)	SC	0.481	0.01	4542	1	0	whole	0.13	(R11)
						40	peel	0.16	
							flesh	0.02	
							juice	< 0.01	
							jam	< 0.05	
							whole	0.06	
Spain, 2001 *Valencia* (Navelina)	SC	0.340	0.01	3220	1	0	whole	0.10	(R12)
						40	peel	0.09	
							flesh	< 0.01	
							juice	< 0.01	
							jam	< 0.05	
							whole	0.03	
Spain, 2001 *Valencia* (Navel Lane Late)	SC	0.263	0.01	2479	1	0	whole	0.16	(R13)
						10	whole	0.19	
						20	whole	0.10	
						40	peel	0.22	
							flesh	< 0.01	
							juice	< 0.01	
							jam	< 0.05	
							whole	0.05	
						60	peel	0.11	
							flesh	< 0.01	
							whole	0.03	

CITRUS Country, Year *Location* (variety)	Application					PHI days	Commodity	Residues, mg/kg clofentezine	Ref.
	Formulation	kg ai/ha	kg ai/hL	Water L/ha	No.				
Spain, 2001 *Valencia* (Valencia Late)	SC	0.345	0.01	3261	1	0	whole	0.23	(R14)
						10	whole	0.16	
						20	whole	0.11	
						40	peel	0.42	
							flesh	< 0.01	
							juice	< 0.01	
							jam	< 0.05	
							whole	0.12	
						60	peel	0.26	
							flesh	< 0.01	
							whole	0.07	
Spain, 2001 *Valencia* (Navel late)	SC	0.499	0.01	4714	1	0	whole	0.16	(R15)
						20	peel	0.25	
							flesh	< 0.01	
							juice	< 0.01	
							jam	< 0.05	
							whole	0.07	
						40	peel	0.14	
							flesh	< 0.01	
							juice	< 0.01	
							jam	< 0.05	
							whole	0.04	
Spain, 2001 *Valencia* (Navel Lane Late)	SC	0.259	0.01	2447	1	0	whole	0.22	(R16)
						20	peel	0.23	
							flesh	0.01	
							juice	< 0.01	
							jam	< 0.05	
							whole	0.06	
						40	peel	0.22	
							flesh	< 0.01	
							juice	< 0.01	
							jam	< 0.05	
							whole	0.06	
LEMON									
Greece, 1990 *Nafplio*	SC	0.75	0.015	5000	1	30	flesh	0.03	R-13064
							peel	0.31	
							whole	0.15	
Italy, 1984 *Catania* (Primo Fiore)	SC		0.01	spray to run off	1	21	flesh	0.02	R-13146 (A/10-1)
							peel	0.18	
							whole	0.09	
						38	flesh	< 0.01	
							peel	0.17	
							whole	0.07	
			0.02	spray to run off	1	21	flesh	0.03	
							peel	0.32	
							whole	0.15	
						38	flesh	0.02	
							peel	0.56	
							whole	0.26	

CITRUS Country, Year *Location* (variety)	Application					PHI days	Commodity	Residues, mg/kg clofentezine	Ref.
	Formulation	kg ai/ha	kg ai/hL	Water L/ha	No.				
TANGERINE									
Italy, 1984 *Catania*	SC		0.01	spray to run off	1		control peel	0.02	R-13146 (A/10-2)
						21	flesh	0.03	
							peel	0.92	
							whole	0.24	
						38	flesh	0.01	
							peel	0.16	
							whole	0.05	
			0.02	spray to run off	1	21	flesh	0.03	
							peel	1.85	
							whole	0.49	
						38	flesh	0.06	
							peel	0.38	
							whole	0.14	
MANDARIN									
Spain, 2001 *Valencia* (Hermandina)	SC	0.310	0.01	2927	1	0	whole	0.11	R-15921 (R1)
						10	whole	0.11	
						20	whole	0.08	
						40	peel	0.16	
							flesh	< 0.01	
							whole	0.04	
						60	peel	0.16	
							flesh	< 0.01	
							whole	0.04	
Spain, 2001 *Valencia* (Clemenvilla)	SC	0.256	0.01	2419	1	0	whole	0.18	(R2)
						10	whole	0.13	
						20	whole	0.08	
						40	peel	0.11	
							flesh	< 0.01	
							whole	0.03	
						60	peel	0.07	
							flesh	< 0.01	
							whole	0.02	
Spain, 2001 *Valencia* (Clemenules)	SC	0.423	0.01	3990	1	0	whole	0.13	(R3)
						40	peel	0.11	
							flesh	< 0.01	
							whole	0.03	
Spain, 2001 *Valencia* (Hermandina)	SC	0.168	0.01	1589	1	0	whole	0.13	(R4)
						40	peel	0.20	
							flesh	< 0.01	
							whole	0.05	
Spain, 2001 *Valencia* (Ortanique)	SC	0.167	0.01	1573	1	0	whole	0.33	(R5)
						10	whole	0.20	
						20	whole	0.17	
						40	peel	0.24	
							flesh	< 0.01	
							whole	0.07	
						60	peel	0.17	
							flesh	< 0.01	
							whole	0.05	

CITRUS Country, Year *Location* (variety)	Application					PHI days	Commodity	Residues, mg/kg clofentezine	Ref.
	Formulation	kg ai/ha	kg ai/hL	Water L/ha	No.				
Spain, 2001 *Valencia* (Ortanique)	SC	0.394	0.01	3741	1	0	whole	0.24	(R6)
						10	whole	0.17	
						20	whole	0.15	
						40	peel flesh whole	0.60 < 0.01 0.13	
						60	peel flesh whole	0.35 < 0.01 0.10	
Spain, 2001 *Valencia* (Fortuna)	SC	0.443	0.01	4179	1	0	whole	0.16	(R7)
						20	peel flesh whole	0.23 0.02 0.08	
						40	peel flesh whole	0.25 0.01 0.06	
Spain, 2001 *Valencia* (Ortanique)	SC	0.422	0.01	3975	1	0	whole	0.25	(R8)
						20	peel flesh whole	0.76 0.17 0.18	
						40	peel flesh whole	0.49 0.01 0.12	

Table 39. Results of residue trials with clofentezine conducted in apple and pear, foliar treatment

POME FRUITS Country, Year *Location* (variety)	Application					PHI days	Commodity	Residues, mg/kg clofentezine	Ref.
	Formulation	kg ai/ha	kg ai/hL	Water L/ha	No.				
APPLE									
Argentina, 1987 *Rio Negro* (Red delicious)	SC		0.01	Not recorded	1	49 70 98 27	1-4 cm fruit	0.10 0.05 0.02 0.01	R-13007
						25 46 74 03	1-4 cm fruit	0.20 0.08 0.04 0.01	
						0 21 49 78	4 cm fruit	0.69 0.17 0.10 0.03	
						29 58	5 cm fruit	0.33 0.17 0.04	
			0.015	Not recorded	1	49 70 98 27	1-4 cm fruit	0.11 0.07 0.02 0.04	
						25 46 74 03	1-4 cm fruit	0.39 0.06 0.05 0.03	

POME FRUITS Country, Year *Location* (variety)	Application					PHI days	Commodity	Residues, mg/kg clofentezine	Ref.
	Formulation	kg ai/ha	kg ai/hL	Water L/ha	No.				
						0	4 cm fruit	0.93	
						21		0.38	
						49		0.18	
						78		0.09	
							5 cm fruit	0.72	
						29		0.23	
						58		0.06	
Australia, 1982	80 WP						control fruit	0.09	R-13102
Victoria			0.01		2	20	mature fruit	0.15	
(Red delicious)			0.02		2	20	mature fruit	0.36	
			0.03		2	20	mature fruit	0.44	
Canada, 1986	50 SC	0.24	0.01	3300	1	107	mature fruit	< 0.01	R-13201
Ontario					2	21	mature fruit	0.18	(R15.06-86-JES-01)
(McIntosh)									
Canada, 1986		0.28			1	113	mature fruit	< 0.01	(R15.06-86-JES-02)
Ontario					2	23	mature fruit	0.09	
(Red delicious)									
Canada, 1986		0.28			1	112	mature fruit	0.02	(R15.06-86-TWN-01)
British Columbia					2	50	mature fruit	0.04 [a]	
(Red delicious)									
Canada, 1987	50 SC	0.28	0.01	3370	2	21	mature fruit	0.09	R-13238 (R152.12-87-GGH-02)
Ontario									
(McIntosh)									
Canada, 1987		0.30	0.01	3333	2	20	mature fruit	0.35	(R152.12-87-GGH-03)
Nova Scotia									
(Delicious)									
Canada, 1987		0.30	0.06	470	2	22	mature fruit	0.09	(R152.12-87-GGH-04)
Quebec									
(McIntosh)									
UK, 1983	50 SC		0.03	1000	1	160	mature fruit	< 0.01	R-13126
Surrey			0.06					< 0.01	
(Jonathan)									
UK, 1983			0.02	800	1	113	mature fruit	< 0.01	
Hereford			0.04					0.03	
(Discovery)									
UK, 1983			0.04	400	1	137	mature fruit	< 0.01	
Kent			0.075					< 0.01	
(Cox's Orange Pippin)									
UK, 1983			0.03	500	1	87	mature fruit	< 0.01	
Hereford			0.06					< 0.01	
(Discovery)									
UK, 1983			0.05	393	1	126	mature fruit	< 0.01	
Essex			0.076					< 0.01	
(Cox's Orange Pippin)									
UK, 1983			0.06		2	64	mature fruit	0.02	
Essex									
(Cox's Orange Pippin)									
UK, 1983			0.02		1	91	mature fruit	< 0.01	
Cambs			0.14					0.09	
(Cox's Orange Pippin)			0.27					0.27	
UK, 1983			0.02		1	91	mature fruit	0.02	
Cambs			0.14					0.11	
(Ida Red)			0.27					0.40	

POME FRUITS Country, Year *Location* (variety)	Application					PHI days	Commodity	Residues, mg/kg clofentezine	Ref.
	Formulation	kg ai/ha	kg ai/hL	Water L/ha	No.				
UK, 1983 *Essex* (Bramley)	50 SC		0.015	to run off	1	14	mature fruit	0.18	
						21		0.20	
						28		0.17	
						35		0.10	
			0.02			14		0.19	
						21		0.28	
						28		0.23	
						35		0.16	
			0.03			14		0.30	
						21		0.26	
						28		0.36	
						35		0.34	
UK, 1983 *Essex* (Cox's Orange Pippin)			0.015	to run off	1	14	mature fruit	0.09	
						21		0.15	
						28		0.22	
						35		0.24	
			0.02			14		0.37	
						21		0.22	
						28		0.18	
						35		0.17	
			0.03			14		0.45	
						21		0.29	
						28		0.26	
						35		0.26	
France, 1984 *La Force* (Golden)	50 SC		0.015		1	14	mature fruit	0.13	R-13133
						28		0.04	
						42		0.07	
			0.02			14	mature fruit	0.12	
						28		0.14	
						42		0.06	
			0.03			14	mature fruit	0.29	
						28		0.13	
						42		0.09	
France, 1984 *Loriol* (Golden)			0.015		1	14	mature fruit	0.01	
						28		0.02	
						42		0.01	
			0.02			14	mature fruit	0.04	
						28		0.01	
						42		< 0.01	
			0.03			14	mature fruit	0.05	
						28		0.05	
						42		0.03	
France, 1984 *Vertou* (Richared)			0.015		1	14	mature fruit	< 0.01	
						28		< 0.01	
						42		< 0.01	
			0.020			14	mature fruit	0.01	
						28		< 0.01	
						42		< 0.01	
			0.030			14	mature fruit	0.01	
						28		< 0.01	
						42		< 0.01	

POME FRUITS Country, Year *Location* (variety)	Application					PHI days	Commodity	Residues, mg/kg clofentezine	Ref.
	Formulation	kg ai/ha	kg ai/hL	Water L/ha	No.				
France, 1983 *Le Thor* (Granny Smith)	50 SC		0.015		1	27	mature fruit	0.20	R-13113
						44		0.03	
						58		0.02	
(Golden Delicious)						27	mature fruit	0.02	
						44		0.02	
						58		0.05	
(Starkingson)						27	mature fruit	0.10	
						44		0.01	
						58		0.08	
(Granny Smith)			0.02			27	mature fruit	0.06	
						44		0.10	
						58		0.02	
(Golden Delicious)						27	mature fruit	0.14	
						44		0.11	
						58		0.04	
(Starkingson)						27	mature fruit	0.19	
						44		0.22	
						58		0.02	
(Granny Smith)			0.025			27	mature fruit	0.04	
						44		0.02	
						58		0.05	
(Golden Delicious)						27	mature fruit	0.04	
						44		0.07	
						58		0.04	
(Starkingson)						27	mature fruit	0.07	
						44		0.07	
						58		0.05	
(Granny Smith)			0.03			27	mature fruit	0.09	
						44		0.12	
						58		0.17	
(Golden Delicious)						27	mature fruit	0.06	
						44		0.14	
						58		0.18	
(Starkingson)						27	mature fruit	0.08	
						44		0.20	
						58		0.10	
Greece, 1986 *Alexandria* (Imperial)	50 SC		0.01	1650	1	0	mature fruit	0.27	R-13187
						32		0.03	
						42		0.03	
			0.015			0	mature fruit	0.34	
						32		0.04	
						42		0.04	
			0.02			0	mature fruit	0.68	
						32		0.11	
						42		0.07	
Germany, 1992 *Tönisvorst-Vorst* (Jonagold)	SE (60 g/L)	0.1125	0.015	750	1	0	mature fruit	0.47	R-13072
						14		0.10	
						28		0.08	
						35		0.05	
						42		0.04	
Germany, 1992 *Orsingen-Nenzingen* (Idared)		0.225	0.03	750		0	mature fruit	0.34	
						28		0.13	
						35		0.11	
						42		0.10	

POME FRUITS Country, Year *Location* (variety)	Application					PHI days	Commodity	Residues, mg/kg clofentezine	Ref.
	Formulation	kg ai/ha	kg ai/hL	Water L/ha	No.				
Germany, 1992 *Ersdorf* (Golden Delicious)		0.21	0.042	500		0	mature fruit	0.27	
						14		0.14	
						28		0.09	
						35		0.05	
						42		0.07	
Germany, 1984 *Ahrweiler* (James Grieve)	50 SC	0.3+0.3	0.03+0.02	1000+1500	2	0	mature fruit	0.87	R-13131
						14		0.21	
						28		0.08	
						35		0.09 [a]	
						42		0.04	
Germany, 1984 *Kempen – St. T Huber* (James Grieve)		0.3+0.3	0.02+0.02	1500+1500	2	0	mature fruit	0.13	
						14		0.02	
						28		< 0.01	
						35		0.02 [a]	
						42		< 0.01	
Germany, 1984 *Kirchheim* (Cox's Orange Pippin)		0.3+0.3	0.06+0.06	500+500	2	0	mature fruit	0.19	
						14		0.16	
						28		0.17	
						35		0.16	
						42		0.04	
Germany, 1984 *Stockuch Wahlwies* (Jonagold)	50 SC	0.3+0.3	0.06+0.06	500+500	2	0	mature fruit	0.40	
						14		0.17	
						28		0.13	
						35		0.08	
						42		0.13	
Belgium, 1993 *St. Truiden* (Jonagold)	SE (60 g/L) 50 SC	0.216	0.0144	1500	1	7	mature fruit	0.31	R-13075
						14		0.28	
						21		0.22	
						30		0.13	
		0.225	0.015	1500		7	mature fruit	0.22	
						14		0.17	
						21		0.17	
						30		0.15	
		0.225	0.015	1500		7	mature fruit	0.11	
						14		0.24	
						21		0.08	
						30		0.17	
		0.432	0.0288	1500	1	7	mature fruit	0.71	
						14		0.54	
						21		0.30	
						30		0.39	
		0.450	0.030	1500		7	mature fruit	0.34	
						14		0.36	
						21		< 0.05	
						30		0.30	
		0.450	0.030	1500		7	mature fruit	0.43	
						14		0.49	
						21		0.24	
						30		0.23	
Germany, 1985 *Kippenhausen /Immenstaad* (James Grieve)	50 SC	0.3+0.3	0.02+0.02	1500+1500	2	0	mature fruit	0.36	R-13167
						14		0.13	
						28		0.05	
						35		0.03 [a]	
						43		0.03	

POME FRUITS Country, Year *Location* (variety)	Application					PHI days	Commodity	Residues, mg/kg clofentezine	Ref.
	Formulation	kg ai/ha	kg ai/hL	Water L/ha	No.				
Germany, 1985 *Ahrweiler* (James Grieve)	50 SC	0.3+0.3	0.03+0.02	1000+1500	2	0	mature fruit	0.73	R-13167
						14		0.13	
						28		0.05	
						35		0.04 [a]	
						42		0.02	
Germany, 1985 *Kempen 1* (James Grieve)	50 SC	0.3+0.3	0.075+ 0.075	400+400	2	0	mature fruit	0.35	
						14		0.10	
						28		0.05	
						35		0.05	
						42		0.02	
Germany, 1985 *Bönnigheim* (James Grieve)	50 SC	0.3+0.3	0.075+ 0.075	400+400	2	0	mature fruit	0.44	
						14		0.15	
						28		0.10	
						35		0.05	
						42		0.07	
South Africa, 1980/81 *Elgin* (Star Crimson)	50 WP		0.01		2	25	mature fruit	0.08	R-13090 (trial 13)
						39		0.04	
						47		0.03	
						61		0.03	
					1	71		0.02	
						85		0.01	
			0.02		2	25	mature fruit	0.09 [a]	
						39		0.04	
						47		0.05	
						61		0.04	
					1	71		0.03	
						85		0.02	
			0.04		2	25	mature fruit	0.22	
						39		0.16	
						47		0.12	
						61		0.05	
					1	71		0.04	
						85		0.03	
			0.04		1	32	mature fruit	0.22	(trial 17)
						39		0.15	
						46		0.19	
						54		0.14	
					1	32	mature fruit	0.33	
						39		0.30	
						46		0.34	
						54		0.27	
South Africa, *Grabouw* (Granny Smith)	50 SC	0.75	0.025	3000	1	0	mature fruit	0.68	R-13159
						15		0.38	
						20		0.20	
						30		0.09	
						40		0.09	
						50		0.07	
						60		0.03	
		0.9	0.03	3000	1	0	mature fruit	0.06	
						15		0.45	
						20		0.25	
						30		0.14	
						40		0.09	
						50		0.04	
						60		0.04	

POME FRUITS Country, Year *Location* (variety)	Application Formulation	kg ai/ha	kg ai/hL	Water L/ha	No.	PHI days	Commodity	Residues, mg/kg clofentezine	Ref.
		.8	0.06	3000	1	)	mature fruit	1.19	
						.5		0.50	
						20		0.62	
						30		0.57	
						40		0.18	
						50		0.16	
						60		0.07	
USA, 1985 *Oregon* (Red Delicious)	50 SC	).28).56	0.015 0.030	1870 1870	2	21	mature fruit	0.22 0.23	R-13162 (R15.03-85-T WM-02)
USA, 1985 *Washington* (Red Delicious)		).28).56	0.015 0.030	3740 3740	2	29	mature fruit	0.06 0.11	(R15.03-85-T WM-01)
USA, 1985 *New York* (McIntosh)		).28).56	0.030 0.060	935 935	2	35	mature fruit	0.11 0.30	(R15.03-85-JES-01)
USA, 1985 *New Jersey* (McIntosh)		).28	0.020	1403	2	36	mature fruit	0.02	(R15.02-85-JES-08)
USA, 1985 *Pennsylvania* (Golden Delicious)		).21).43	0.007 0.015	2805 2805	2	38	mature fruit	0.25 0.44	(R15.03-85-JES-02)
USA, 1985 *Oregon* (Red Delicious)		).28	0.015	1870	2	42	mature fruit	0.05 [a]	(R15.03-85-T WM-01)
USA, 1985 *Michigan* (Red Delicious)		).28	0.015 0.030	1870 935	2	45	mature fruit	0.04 [a] 0.04 [a]	(R15.02-85-G GH-01)
USA, 1985 *Michigan* (Red Delicious)	50 SC	).28	0.030 0.030	935 935	2	45	mature fruit	0.12 [a] 0.11 [a]	(R15.02-85-G GH-03)
USA, 1985 *Michigan* (Golden Delicious)		).28	0.030 0.030	935 935	2	45	mature fruit	0.05 [a] 0.04 [a]	(R15.02-85-G GH-06)
USA, 1985 *Washington* (Red Delicious)		).28	0.015	3740	2	45	mature fruit	0.12 [a]	(R15.02-85-T WM-03)
USA, 1985 *Pennsylvania* (Red Delicious)		).28	0.007	2338	2	45	mature fruit	0.02 [a]	(R15.02-85-JES-07)
USA, 1985 *New York* (McIntosh)		).28	0.030	935	2	45	mature fruit	0.07 [a]	(R15.02-85-JES-02)
USA, 1985 *Pennsylvania* (Red Delicious)		).28			2	46	mature fruit	0.01 [a]	(R15.02-85-JES-16)
USA, 1985 *California* (Rome)		).21	0.011	1870	2	47	mature fruit	0.07 [a]	(R15.02-85-JCA-01)
USA, 1985 *Washington*		).28	0.015	3740	2	50	mature fruit	0.05 [a]	(R15.02-85-T WM-02)

POME FRUITS Country, Year *Location* (variety)	Application					PHI days	Commodity	Residues, mg/kg clofentezine	Ref.
	Formulation	kg ai/ha	kg ai/hL	Water L/ha	No.				
(Red Delicious)									
USA, 1985 *New Jersey* (Stayman)		0.28			2	60	mature fruit	0.06	(R15.02-85-JES-01)
USA, 1985 *Michigan* (Red Delicious)	50 SC	0.28	0.022	1300	2	0	mature fruit	0.40	R-13163 (R15.03-85-G GH-01)
						7		0.13	
						14		0.09	
						21		0.18	
						28		0.10	
		0.56	0.043	1300	2	0	mature fruit	0.41	
						7		0.41	
						14		0.47	
						21		0.32	
						28		0.16	
USA, 1985 *Michigan* (Red Delicious)	50 SC	0.28	0.030	935	2	0	mature fruit	0.19	(R15.03-85-G GH-02)
						7		0.10	
						14		0.16	
						21		0.11	
						28		0. 08	
		0.28	0.030	935	2	0	mature fruit	0.20	
						7		0.19	
						14		0.16	
						21		0.11	
						28		0. 10	
		0.56	0.060	935	2	0	mature fruit	0.35	
						7		0.41	
						14		0.27	
						21		0.23	
						28		0. 23	
		0.56	0.060	935	2	0	mature fruit	0.39	
						7		0.31	
						14		0.30	
						21		0.17	
						28		0. 20	
USA, 1985 *New York* (McIntosh)	50 SC	0.28	0.030	935	2	21	mature fruit	0.22	(R15.03-85-JES-01)
						28		0.21	
						35		0.11	
		0.56	0.060	935	2	21	mature fruit	0.48	
						28		0.39	
						35		0.30	
USA, 1985 *Pennsylvania* (Golden Delicious)	50 SC	0.21	0.007	2805	1	114	mature fruit	-	(R15.03-85-JES-02)
						121		0.01	
						127		< 0.01	
						135		< 0.01	
		0.43	0.015	2805	1	114	mature fruit	0.02	
						121		0.02	
						127		0.03	
						134		0.02	
		0.21	0.007	2805	2	17	mature fruit	0.37	
						24		0.34	
						30		0.33	
						38		0.25	
		0.43	0.015	2805	2	17	mature fruit	0.67	
						24		0.79	
						30		0.70	
						38		0.44	

POME FRUITS Country, Year *Location* (variety)	Application					PHI days	Commodity	Residues, mg/kg clofentezine	Ref.
	Formulation	kg ai/ha	kg ai/hL	Water L/ha	No.				
USA, 1985	50 SC	0.28	0.015	3740	2	21	mature fruit	0.05	(R15.03-85-T
Washington						29		0.06	WM-01)
(Red Delicious)		0.56	0.030	3704	2	21	mature fruit	0.10	
						29		0.11	
USA, 1986	50 SC	0.28	0.015	3740	2	1	mature fruit	0.16	R-13228
Washington						3		0.09	
(Golden Delicious)						7		0.12	
						4		0.04	
						28		0.05	
						45		0.01 [a]	
USA, 1985	50 SC	0.28	0.007	3740	1	20	mature fruit	0.27	R-13169
California		0.56	0.015					0.62	(R15.03-85-JCA-01)
(Rome)									
USA, 1985	50 SC	0.28			2	45	whole apple	0.02 [a]	R-12732
Pennsylvania									(R15.01-85-JE
(Red Delicious)									S-03)
USA, 1985	50 SC	0.28			2	40	whole apple	0.02 [a]	(R15.01-85-JE
New Jersey									S-04)
(Staymen)									
PEAR									
Australia, 1982	80 WP		0.03		2	7	mature fruit	0.26	R-13102
Victoria									
(Beurre Bosc)									
Australia, 1982	80 WP		0.03		2	22	mature fruit	0.54	
Victoria									
(Beurre Bosc)									
Australia, 1982	50 SC	0.278	0.02	1387	2	21	mature fruit	0.02 [a]	R-13170
Victoria						28	mature fruit	0.01	
(Packham)						35	mature fruit	0.02	
		0.417	0.03	1388	2	21	mature fruit	0.04	
						28	mature fruit	0.05	
						35	mature fruit	0.03	
Canada, 1987	50 SC	0.30	0.01	3370	2	21	mature fruit	0.13 [a]	R-15402
Ontario									(R152.12-87-GGH-02)
(Bartlett)									
Canada, 1987		0.30	0.01	3333	2	25	mature fruit	0.12 [a]	(R152.12-87-
Nova Scotia									GGH-03)
(Bartlett)									
Canada, 1988	50 SC	0.28			1	21	mature fruit	0.14	R-15401
British Columbia									(GGH-01)
(Bartlett)									
Canada, 1988		0.28			1	21	mature fruit	0.19	(GGH-02)
Nova Scotia									
(Bartlett)									
Italy, 1984	50 SC		0.01	to run off	1	45	mature fruit	0.05	R-13144
Bolzano			0.02					0.12	(A/8-3)
(Buona Luigia)									
Italy, 1984	50 SC		0.01		1	45	mature fruit	0.01	(A/8-4)
Ferrara			0.02					0.04	
(Abate)									
Italy, 1984	50 SC		0.02		1	16	mature fruit	0.21	(A/9-1)
Ferrara						30		0.04	
(Abate)						45		0.01	
						62		< 0.01	

POME FRUITS Country, Year *Location* (variety)	Application Formulation	kg ai/ha	kg ai/hL	Water L/ha	No.	PHI days	Commodity	Residues, mg/kg clofentezine	Ref.
South Africa, 1984 *Hamlet* (Beurre Hardy)	50 SC	0.675	0.025	2700	1	20	mature fruit	0.42	R-13159
						30		0.20	
						40		0.10	
						50		0.14	
						60		0.09	
		0.81	0.03	2700	1	0	mature fruit	1.15	
						20		0.30	
						30		0.37	
						40		0.12	
						50		0.11	
						60		0.08	
		1.62	0.06	2700	1	0	mature fruit	1.30	
						20		0.09	
						30		0.24	
						40		0.36	
						50		0.35	
						60		0.03	
South Africa, 1984 *Hamlet* (Winter Nellis)	50 SC	0.675	0.025	2700	1	20	mature fruit	0.32	
						30		0.22	
						40		0.10	
						50		0.18	
						60		0.22	
		0.81	0.03	2700	1	0	mature fruit	0.08	
						20		0.41	
						30		0.37	
						40		0.14	
						50		0.14	
						60		0.19	
		1.62	0.06	2700	1	0	mature fruit	1.54	
						20		0.99	
						30		0.63	
						40		0.41	
						50		0.45	
						60		0.32	
USA, 1985 *California* (Rome)	50 C	0.28	0.007	3740	1	97	mature fruit	< 0.01	R-15405
			0.007+0.015	3740+1870	2	21		0.09 [a]	(R15.02-85-J
		0.56	0.015		1	97		0.04	CA-01)
			0.015+0.030	3740+1870	2	21		0.03	
		0.28	0.030	935	1	138	mature fruit	< 0.01	(R15.02-85-J
					2	21		0.15 [a]	CA-02)
		0.56	0.060		1	97		0.04	
					2	21		0.24	
USA, 1986 *Washington* (Bartlett)		0.28	0.007	3740	1	90	mature fruit	< 0.01	(R15.02-86-T
					2	21		0.18 [a]	WM-01)
		0.56	0.015		1	90		< 0.01	
					2	21		0.13	
USA, 1986 *Oregon* (Aujou)		0.28	0.007	3740	1	136	mature fruit	< 0.01	(R15.02-86-T
					2	21		0.09 [a]	WM-02)
		0.56	0.015		1	136		< 0.01	
					2	21		0.15	
USA, 1986 *New York* (Bartlett)	50 SC	0.28	0.010	2805	1	102	mature fruit	< 0.01	(R15.02-86-J
					2	21		0.20 [a]	ES-01)
		0.56	0.020		1	102		< 0.01	
					2	21		0.32	
USA, 1993	SE	0.29	0.014	2030	1	21	mature fruit	0.06	R-15403

POME FRUITS Country, Year *Location* (variety)	Application					PHI days	Commodity	Residues, mg/kg clofentezine	Ref.
	Formulation	kg ai/ha	kg ai/hL	Water L/ha	No.				
California (Bartlett)	50 SC							0.09	(DRC-01)
USA, 1993 *New York* (Bartlett)	SE	0.27	0.030	906	1	21	mature fruit	0.05	(DRS-01)
	50 SC	0.28		944				0.06	
USA, 1993 *Washington* (Bartlett)	SE	0.28	0.030	935	1	21	mature fruit	0.08	(TWM-01)
	50 SC	0.28	0.030	935				0.08	
USA, 1993 *Oregon* (Red Bartlett)	SE	0.28	0.030	935	1	21	mature fruit	0.06	(TWM-02)
	50 SC	0.28	0.030	935				0.04	

a - Interval between applications >> PHI (1st application in spring). In such case, the last application can be considered as unique and resulting residue data are used for MRL calculation.

Table 40. Results of residue trials with clofentezine conducted with foliar treatment in apricots, cherries, nectarines and peaches

STONE FRUITS Country, Year *Location* (variety)	Application					PHI days	Commodity	Residues, mg/kg clofentezine	Ref.
	Formulation	kg ai/ha	kg ai/hL	Water L/ha	No.				
PEACH									
Australia, 1986 *New South Wales* (Glenalton)	50 SC		0.02		1	22	mature (flesh)	0.06 (0.06)	R-13017 (I/HO 1-84)
						45		0.03 (0.03)	
						68		0.05 (0.06)	
			0.03		1	22	mature (flesh)	0.14 (0.15)	
						45		0.06 (0.06)	
						68		0.08 (0.08)	
Australia, 1986 *Victoria* (Yanco Queen)	50 SC	0.45	0.015	3000	2	7	mature fruit	0.32	R-13017 (A/V 2-88)
						14		0.22	
						22		0.13 [a]	
		0.45+0.90	0.015+0.030		2	7		0.68	
						14		0.38	
						22		0.29	
Italy, 1984 *Como* (Baby Golden)	50 SC		0.01		1	48	mature (flesh)	0.06 (0.07)	R-13137 (A/8-2)
			0.02		1	48		0.21 (0.23)	
Italy, 1984 *Padova* (Vesuvio)			0.02			64		< 0.01 (0.01)	(A/9-3)
						50		0.04 (0.04)	
						85		0.07 (0.08)	
						19		0.07 (0.08)	
Italy, 1991 *Revello* (Star-sungloo)	50 SC		0.015	2000	1	141	mature fruit	< 0.01	R-13077 (A1091DC)
			0.020			75		0.01	
Italy, 1991 *San Giovanni di Osteloo* (Andros)						81	mature fruit	0.02	(A1091RR)
USA, 1986 *California* (Andrus)	50 SC	0.28	0.007	3740	2	1	mature fruit	0.09	R-13200 (R15.03-86-JC A-02)
						3		0.17	
						7		0.08	
						14		0.07	
						21		0.05 [a]	
USA, 1986	50 SC	0.28	0.007	3740	2	1	mature fruit	0.67	(R15.03-86-G

STONE FRUITS Country, Year *Location* (variety)	Application					PHI days	Commodity	Residues, mg/kg clofentezine	Ref.
	Formulation	kg ai/ha	kg ai/hL	Water L/ha	No.				
Michigan (Red Haven)						3		0.69	GH-01)
						7		0.34	
						14		0.17	
						21		0.08 [a]	
USA, 1986	50 SC	0.28		run-off	1	77	mature fruit	< 0.01	R-13202
South Carolina					2	21		0.35 [a]	(R15.03-86-W
(Coronet)		0.56			1	77		0.05	KT-01)
					2	21		0.58	
USA, 1986	50 SC	0.28	0.007	3740	1	115	mature fruit	< 0.01	(R15.03-86-J
California			0.015		2	21		0.02 [a]	CA-01)
(Andrus)		0.56	0.015		1	115		< 0.01	
			0.030		2	21		0.16	
USA, 1986	50 SC	0.28	0.030	935	1	108	mature fruit	< 0.01	(R15.03-86-J
California			0.060		2	21		0.24 [a]	CA-04)
(Carson)		0.56	0.060		1	108		0.02	
			0.12		2	21		0.25	
USA, 1986	50 SC	0.28	0.030	935	1	64	mature fruit	0.03	(R15.03-86-G
Michigan			0.060		2	21		0.09	GH-02)
(Red Skin)		0.56	0.060		1	64		0.07	
			0.12		2	21		0.29	
USA, 1987	50 SC	0.28	0.012	2338	2	14	mature fruit	0.27	R-13239
California						20		0.26	(R152.06-87-J
(Loadel)									CA-01)
(Andrus)		0.28	0.012	2338	2	13	mature fruit	0.41	(R152.06-87-J
						21		0.40	CA-02)
(Faye Elberta)		0.28	0.012	2338	2	11	mature fruit	0.33	(R152.06-87-J
						20		0.22	CA-03)
(Angelus)		0.28	0.031	916	2	13	mature fruit	< 0.02	(R152.06-87-J
						20		0.10	FS-04)
USA, 1987	50 SC	0.28	0.030	935	2	21	mature fruit	0.19	(R152.06-87-G
Michigan (New Haven)									GH-01)
USA, 1987	50 SC	0.28	0.030	935	2	21	mature fruit	0.06	(R152.06-87-G
Michigan (Garnet Beauty)									GH-02)
USA, 1987 *South Carolina* (Summer Gold)	50 SC	0.28		run-off	2	21	mature fruit	0.11	(R152.06-87-WKT-01)
USA, 1988		0.28	0.056	496	1	21	mature fruit	0.09	R-13062
Pennsylvania									(R152.02-88-J
(Early Rio Oso Gem)									ES-01)
USA, 1988		0.28	0.054	520	1	14	mature fruit	0.22	(R152.02-88-J
New Jersey (Loring)						21		0.18	ES-02)
USA, 1988 *South Carolina* (Elberta)	50 SC	0.28	0.031	898	1	14	mature fruit	0.18	(R152.02-88-WKT-01)
						21		0.08	
USA, 1988	50 SC	0.28	0.75	37.4	1	21	mature fruit	0.11	(R152.02-88-J
California									CA-01)

STONE FRUITS Country, Year *Location* (variety)	Application					PHI days	Commodity	Residues, mg/kg clofentezine	Ref.
	Formulation	kg ai/ha	kg ai/hL	Water L/ha	No.				
(Walford)									
(Starn)		0.28	0.75	37.4		21	mature fruit	0.13	(R152.02-88-J CA-02)
USA, 1988 *California* (Suncrest)	50 SC	0.28	0.45	62.4	1	20	mature fruit	0.06	(R152.02-88-J FS-01)
(Elegant ladies)		0.28	0.50	56.4	1	21	mature fruit	0.03	(R152.02-88-J FS-02)
USA, 1993 *California* (Elegant lady)	50 SC	0.28	0.45	1421	1	21	mature fruit	0.12	R-12803 (J-93R-04-FSCA)
	SE (60 g/L)							0.14	
USA, 1993 *Georgia* (Red Skin)	50 SC	0.26	0.019	1365	1	21	mature fruit	0.18	(J-93R-04-WKT-02)
	SE (60 g/L)	0.28	0.021					0.04	
USA, 1993 *South Carolina* (June Prince)	50 SC	0.27	0.016	1711	1	21	mature fruit	0.14	(J-93R-04-WKT-01)
	SE (60 g/L)	0.29	0.017					0.09	
USA, 2002 *California* (Last Chance)	50 SC	0.55	0.066	836	1	10	mature fruit	0.08	R-21214 (22084-CA04)
(Fairtime)		0.56	0.062	904	1	10	mature fruit	0.11	(22084-CA05)
(Starn)		0.57	0.063	900	1	10	mature fruit	0.10	(22084-CA06)
(Sweet September)		0.57	0.090	635	1	10	mature fruit	0.04	(22084-CA07)
APRICOT									
Greece, 1989 *HE-Mauplion Argolidos* (Chassiotica)	50 SC	0.38	0.015	2500	1	38	mature fruit	0.16	R-13031
						65		0.04	
						73		0.04	
USA, 1987 *California* (Tilton)	50 SC	0.28	0.007	3857	2	21	mature fruit	0.15	R-13241 (R152.13.87-J CA-01)
(Blenheim)		0.28	0.007	3918	2	22	mature fruit	0.72	(R152.13.87-J CA-02)
USA, 1993 *California* (Patterson)	50 SC	0.27	0.030	904	1	21	mature fruit	0.13	R-13081 (J-93R-05-DRC-01)
	SE (60 g/L)	0.30	0.031	958				0.14	
CHERRY									
USA, 1987 *Washington* (Lambert) (sweet)	50 SC	0.28	0.007	3740	2	21	mature fruit	0.12	R-13241 (R152.13-87-T WM-03)
USA, 1987 *Oregon* (VAN) (sweet)	50 SC	0.28	0.007	3740	2	21	mature fruit	0.33	(R152.13-87-T WM-04)
USA, 1987 *Michigan* (Montmoreney) (sour)	50 SC	0.28	0.030	935	2	21	mature fruit	0.07	(R152.13-87-G GH-01)
USA, 1987		0.28	0.030	935	2	21	mature fruit	0.22	(R152.13-87-J

STONE FRUITS Country, Year *Location* (variety)	Application					PHI days	Commodity	Residues, mg/kg clofentezine	Ref.
	Formulation	kg ai/ha	kg ai/hL	Water L/ha	No.				
New York (Montmoreney) (sour)									ES-01)
UK, 1983 *Herefordshire* (Noire de Guben)	50 SC	0.24	0.03	800	1	51	mature fruit	0.02	R-13112 (GB 83-339)
		0.48	0.06	800				0.03	
(Merton Favorite)		0.24	0.03	800	1	41	mature fruit	0.02	
		0.48	0.06					0.04	
UK, 1983 *Kent* (Merton Favorite)		0.24	0.03	800	1	47	mature fruit	0.01	(GB 83-338)
		0.48	0.06					0.01	
NECTARINE									
USA, 1986	50 SC	0.28		run-off	1	91	mature fruit	< 0.01	R-13202
South Carolina					2	21		0.17 [a]	(R15.03-86-W
(Columbia)		0.56			1	91		0.01	KT-01)
					2	21		0.41	
USA, 1986	50 SC	0.28	0.007	3740	1	92	mature fruit	< 0.01	(R15.03-86-J
California			0.007+0.015	3740+1870	2	21		0.04 [a]	CA-04)
(Red Diamond)		0.56	0.015	3740	1	92		< 0.01	
			0.015+0.030	3740+1870	2	21		0.33	
USA, 1986	50 SC	0.28	0.050	561	1	115	mature fruit	< 0.01	(R15.03-86-J
New Jersey					2	21		0.11 [a]	ES-01)
(Red Gold)		0.56	0.10		1	115		< 0.01	
					2	21		0.34	
USA, 1987	50 SC	0.28	0.012	2338	2	14	mature fruit	0.13	R-13239
California						21		0.04	(R152.06-87-J
(Summer Grande)									CA-04)
USA, 1987		0.28	0.012	2338	2	20	mature fruit	0.20	(R152.06-87-J
California (Fire Bright)									FS-01)
USA, 1987		0.28	0.009	3216	2	16	mature fruit	0.16	(R152.06-87-J
California						21		0.09	FS-02)
(Fantasin)									
USA, 1987		0.28	0.009	3216	2	14	mature fruit	0.20	(R152.06-87-J
California						21		0.19	FS-03)
(Royal Giant)									
USA, 1987 *South Carolina* (VPI-62N o Lovell)	50 SC	0.28		run-off	2	20	mature fruit	0.07	(R152.06-87-WKT-02)

a - Interval between applications >> PHI (1st application in spring). In such case, the last application can be considered alone and resulting residue data are used for MRL calculation.

Table 41. Results of residue trials with clofentezine conducted with foliar treatment in plums

PLUMS Country, Year *Location* (variety)	Application					PHI days	Commodity	Residues, mg/kg clofentezine	Ref.
	Formulation	kg ai/ha	kg ai/hL	Water L/ha	No.				
USA, 1987 *California* (Wickson)	50 SC	0.28	0.007	3918	2	22	mature fruit	0.58	R-13241 (R152.13.87-J CA-03)
(Satsum)								0.02	
USA, 1987 *Michigan*		0.28	0.030	935	2	21	mature fruit	0.03	(R152.13.87-G GH-02)

PLUMS Country, Year *Location* (variety)	Application					PHI days	Commodity	Residues, mg/kg clofentezine	Ref.
	Formulation	kg ai/ha	kg ai/hL	Water L/ha	No.				
(Stanley)									
USA, 1987 *Oregon* (Late Hallans)		0.28	0.007	3740	2	21	mature fruit	< 0.02	(R152.13.87-TWM-01)
USA, 1987 *Oregon* (Presidents)		0.28	0.007	3740	2	21	mature fruit	< 0.02	(R152.13.87-TWM-02)
Germany, 1985 *Hösbach* (Ruth Gerstetter)	50 SC	0.30	0.02	1500	2	0	mature fruit	0.02	R-13235
						14		0.01	
						28		< 0.01	
						35		< 0.01	
						42		< 0.01	
Germany, 1985 *Ahrweiler* (Zimmers Frühzwetschge)		0.30	0.03+0.02	1000+1500	2	0	mature fruit	0.13	
						12		0.07	
						26		0.04	
						35		0.04	
						42		0.02	
Germany, 1985 *Bodensee* (Nacy)		0.30	0.06	500	2	0	mature fruit	0.09	
						15		0.07	
						28		0.06	
						35		0.04	
						42		0.03	
Germany, 1985 *Bonningheim* (Italienische)		0.30	0.03+0.02	1000+1500	2	0	mature fruit	0.07	
						14		0.05	
						28		0.04	
						35		0.02	
						42		0.03	
Germany, 1986 *Ahrweiler* (Zimmers Frühzwetschge)	50 SC	0.30	0.02	1500	2	0	mature fruit	0.07	R-13024 (SCH 00114)
						14		0.03	
						21		0.04	
						28		0.06	
						35		0.10	
Germany, 1986		0.30	0.02	1500	2	0	mature fruit	0.27	(SCH 00115)
Ahrweiler						14		0.02	
(Stanley)						21		0.02	
						28		< 0.01	
						35		< 0.01	
Germany, 1986		0.30	0.075	400	2	0	mature fruit	0.15	(SCH 00116)
Eisenbach						14		0.13	
(Stanley)						21		0.09	
						28		0.09	
						35		0.07	

Table 42. Results of residue trials with clofentezine conducted with foliar treatment in grapes

GRAPES Country, Year *Location* (variety)	Application					PHI days	Commodity	Residues, mg/kg clofentezine	Ref.
	Formulation	kg ai/ha	kg ai/hL	Water L/ha	No.				
France, 2001 *Sorede* (Muscat)	50 SC	0.317	0.030	1056	1	0	grape	0.32	R-13464 (F01N040R)
						8		0.26	
						16		0.15	
						24		0.15	
						29		0.11	
France, 2001 *Montesquieu des Alberes* (Macabeu)	50 SC	0.300	0.030	996	1	30	grape	0.09	(F01N041R)
Spain, 2001	50 SC	0.301	0.030	1002	1	0	grape	0.49	(S01N019R)

GRAPES Country, Year *Location* (variety)	Application					PHI days	Commodity	Residues, mg/kg clofentezine	Ref.
	Formulation	kg ai/ha	kg ai/hL	Water L/ha	No.				
Casas de Eufemia (Bobal)						9		0.44	
						15		0.34	
						23		0.23	
						30		0.25	
Spain, 2001 *Requena* (Royal)	50 SC	0.300	0.03	1001	1	30	grape	0.27	(S01N020R)
Italy, 2001 *Imola* (Trebbiano)	50 SC	0.304	0.030	1002	1	0	grape	0.19	(I01N021R)
						9		0.13	
						16		0.15	
						23		0.10	
						30		0.12	
Greece, 2001 *Kalokastro* (Cardinal)	50 SC	0.313	0.030	1043	1	0	grape	1.05	(GR01N001R)
						9		1.18	
						15		0.88	
						23		0.80	
						30		0.67	
France, 1991 *St Martin de Sescas* (Merlot)	50 SC	0.10	0.10	100	1	23	grape	0.11	R-18418
		0.10	0.10					0.30	(R9114)
		0.225	0.225					0.14	
France, 1991 *Chaintré* (Chardonnay)	50 SC	0.10	0.10	100	1	26	grape	0.04	
		0.10	0.10					0.12	
		0.225	0.225					0.04	
Italy, 1987 *Broni* (Riesling)	50 SC	0.2	0.02	1000	1	127	grape	< 0.01	R-12739
		0.2+0.225	0.02+0.015	1000+1500	2	61		0.02	(R/12-87 A)
		0.15	0.01	1500	1	61		0.02	
		0.3	0.02			61		0.03	
		0.3	0.02			42		0.24	
Germany, 1992 *Niederkirchen* (Riesling)	35 EC	0.30	0.045	670	1	0	grape	1.2	R-18419
						15		0.66	(F43)
						28		0.55	
						35		0.39	
						42		0.38	
Germany, 1992 *Ilsfeld-Schozach* (Kerner)		0.30	0.03	1000	1	0	grape	1.7	(U92 R06)
						14		0.94	
						28		0.82	
						35		0.79	
						42		0.53	
Germany, 1992 *Ilsfeld-Schozach* (Spatburgunder)		0.30	0.03	1000	1	0		2.1	(U92 R07)
						14		1.1	
						28		0.87	
						35		0.89	
						42		0.72	
Germany, 1992 *Muller Thurgau* (Hagnau)		0.30	0.015	2000	1	0		0.93	(I02 A)
						27		0.32	
						34		0.23	
						41		0.20	
Germany, 1986 *Angelbachtal* (Muller Thurgau)	co-formu-lation with cyhexatin	0.113+0.281	0.014	800+2000	2	0	grape	0.36	R-18417
						16		0.28	
						29		0.20	
						37		0.22 [a]	
						44		0.16	

GRAPES Country, Year *Location* (variety)	Application					PHI days	Commodity	Residues, mg/kg clofentezine	Ref.
	Formulation	kg ai/ha	kg ai/hL	Water L/ha	No.				
Germany, 1986 *Ungstein* (Blauer Portugieser)		0.113+0.281	0.014	800+2000	2	0	grape	0.22	
						14		0.29	
						28		0.17	
						35		0.14 [a]	
						42		0.14	
Germany, 1986 *Schozach* (Blauer Spätburgunder)		0.113+0.281	0.014	800+2000	2	0		0.30	
						14		0.18	
						28		0.17	
						35		0.12 [a]	
						42		0.12	
Germany, 1986 *Trennfurt* (Müller Thurgau)		0.113+0.281	0.014	800+2000	2	0	grape	0.48	
						14		0.18	
						28		0.13	
						35		0.20 [a]	
						42		0.08	
Germany, 1986 *Angelbachtal* (Muller Thurgau)	50 SC	0.16+0.40	0.02	800+2000	2	0	grape	1.12	R-21166
						16		0.53	
						29		0.49	
						37		0.34	
						44		0.28	
Germany, 1986 *Ungstein* (Blauer Portugieser)						0	grape	1.75	
						14		1.04	
						28		0.47	
						35		0.46	
						42		0.43	
Germany, 1986 *Schozach* (Blauer Spätburgunder)		0.16+0.40	0.06	267+670	2	0	grape	0.88	
						14		0.46	
						28		0.35	
						35		0.20	
						42		0.18	
Germany, 1986 *Trennfurt* (Müller Thurgau)						0	grape	0.86	
						14		0.51	
						28		0.29	
						35		0.33	
						42		0.20	
Germany, 1985 *Erlenbach* (Muller Thurgau)	co-formu-lation with cyhexatin EC	0.112+0.28	0.014	800+2000	2	0	grape	0.16	R-21165
						14		0.12	
						28		0.14	
						35		0.09 [a]	
						42		0.09	
Germany, 1985 *Ungstein* (Blauer Portugieser)		0.112+0.28	0.014	800+2000	2	0	grape	1.5	
						13		0.87	
						27		0.52	
						34		0.61 [a]	
						41		0.32	
Germany, 1985 *Meersburg* (Müller Thurgau)		0.107+0.268	0.040	267+670	2	0	grape	2.90	
						14		1.20	
						22		0.89	
						28		1.00	
						35		0.67	
						42		0.73 [a]	

GRAPES Country, Year *Location* (variety)	Application					PHI days	Commodity	Residues, mg/kg clofentezine	Ref.
	Formulation	kg ai/ha	kg ai/hL	Water L/ha	No.				
Germany, 1985 *Schozach* (Blauer Spätburgunder)		0.107+0.268	0.040	267+670	2	0	grape	1.70	
						14		0.94	
						28		0.66	
						35		0.69 [a]	
						42		0.29	
Italy, 1984 *Pavia* (Riesling)	50 SC		0.01		1	53	grape	0.13	R-18416
			0.02					0.24	(A/8-1)
Italy, 1984 *Padova* (Merlot)			0.02		1	15		0.65	(A/9-2)
						33		0.35	
						48		0.49	
						63		0.34	

a - Interval between applications >> PHI (1st application in spring). In such case, the last application can be considered alone and the resulting residue data are used for MRL calculation.

Table 43. Results of residue trials with clofentezine conducted outdoor with foliar treatment in strawberries

STRAWBERRIES Country, Year *Location* (variety)	Application					PHI days	Commodity	Residues, mg/kg clofentezine	Ref.
	Formulation	kg ai/ha	kg ai/hL	Water L/ha	No.				
France, 1989 *Aiguillon SEU* (Selva)	50 SC	0.21	0.070	300	1	0	fruit	0.29	R-13018
								0.32	(I8957)
						3		0.24	
						7		0.21	
						14		0.15	
						28		0.03	
France, 1989 *Soucelles NEU* (Hummi gento)		0.21	0.070	300	1	0		0.28	
								0.21	
						3		0.17	
						7		0.18	
						14		0.20	
						28		0.14	
France, 1990 *Castillon* (Selva)	50 SC	0.20	0.02	1000	1	0	friut	0.36	R-13057
								0.29	
						3		0.13	
						7		0.06	
						14		0.04	
France, 2001 *Entzheim NEU* (Elvira)	50 SC	0.216	0.025	863	1	0	fruit	0.29	R-13465
								0.23	(F01N037R)
						3		0.24	
						5		0.18	
						7		0.17	
France, 2001 *Wissembourg NEU* (Elsanta)		0.189	0.025	758	1	0		0.29	(F01N038R)
								0.21	
						3		0.19	
						5		0.10	
						7		0.10	
France, 2001 *Blaesheim NEU* (Darselect)		0.195	0.025	780	1	3		0.09	(F01N039R)
Germany, 2001 *Stutensee-Staffort* (Elsanta)		0.206	0.025	824	1	0		0.25	(G01N036R)
								0.17	
						3		0.16	
						5		0.08	

STRAWBERRIES Country, Year *Location* (variety)	Application					PHI days	Commodity	Residues, mg/kg clofentezine	Ref.
	Formulation	kg ai/ha	kg ai/hL	Water L/ha	No.				
						7		0.08	
Germany, 2001 *Eberdingen* (Elsanta)		0.190	0.025	760	1	0		0.18	(G01N037R)
						1		0.11	
						3		0.09	
						5		0.07	
						7		0.07	
		1.0	0.125	800	1	0		0.75	
Germany, 2001 *Kleinsachsen* (Elsanta)		0.194	0.025	777		3		0.23	(G01N038R)
		0.939	0.125	751		0		2.38	
France, 2000 *Clery-en-Vexin NEU* (Gariguette)	50 SC	0.20	0.07	300	1	0	fruit	0.10	R-14527
						3		0.08	(FRA0101)
France, 2000 *Les Alluets le Roi NEU* (Mami)		0.20	0.07	300	1	0		0.19	(FRA0102)
						3		0.08	
Germany, 2000 *Meckenheim* (Elsanta)		0.20	0.03	600	1	0		0.12	(DEU0201)
						3		0.09	
Germany, 2000 *Koerlitz* (Korona)		0.20	0.07	300	1	0		0.27	(DEU0601)
						3		0.18	
Netherlands, 1990 *Brielle* (Elsanta)	50 SC	0.15	0.015	1000	1	1	mature fruit	0.09	R-13055
						3		0.08	(1990-677)
						7		0.04	
Netherlands, 1990 *Bruchmen* (Elsanta)					1	1		0.19	(1990-678)
						3		0.13	
						7		0.10	
Spain, 1992 *Moguer-Heulva* (Oso Grande)	50 SC	0.2	0.02	1000	1	0	fruit	1.2	R-13068
						3		0.73	(32/92)
						7		0.44	
						14		0.22	
(Chadler)						0		1.1	(33/92)
						3		0.60	
						7		0.29	
						14		0.21	
(Chadler)						0		0.93	(34/92)
						3		0.81	
						7		0.53	
						14		0.25	
(Muy)	50 SC	0.2	0.02	1000		0		1.8	(35/92)
						3		1.1	
						7		0.60	
						14		0.35	
(Oso Grande)						0		1.1	(36/92)
						3		0.75	
						7		0.43	
						14		0.26	
(Chadler)						0		0.66	(37/92)
						3		0.56	
						7		0.24	
						14		0.12	
(Turla)						0		0.87	(38/92)
						3		0.70	
						7		0.31	
						14		0.17	
(Chadler)						0		0.97	(39/92)
						3		0.50	

STRAWBERRIES Country, Year *Location* (variety)	Application					PHI days	Commodity	Residues, mg/kg clofentezine	Ref.
	Formulation	kg ai/ha	kg ai/hL	Water L/ha	No.				
						7		0.35	
						4		0.13	
Spain, 1992 *Los Palacies* (Chadler)						0		1.2	(48/92)
						3		0.72	
						7		0.43	
						4		0.25	

Table 44. Results of residue trials with clofentezine conducted in black currants.

CURRANTS Country, Year *Location* (variety)	Application					PHI days	Commodity	Residues, mg/kg clofentezine	Ref.
	Formulation	kg ai/ha	kg ai/hL	Water L/ha	No.				
France, 2001 *St Leonard en Beauce* (Tenah)	50 SC	0.21	0.052	403.7	1	14	whole fruit without stalk	< 0.04	R-18971 (RE01051)
France, 2001 *Roches* (Black Down)		0.21	0.067	312.4	1	14		< 0.04	(RE01052)
France, 2001 *Bapaume* (Tenah)		0.21	0.051	407.8	1	16		< 0.04	(RE01053)
France, 2001 *Sigognee* (Tenah)	50 SC	0.199	0.028	700	1	11	whole fruit without stalk	0.09	R-20184 (RE04083)

Table 45. Results of residue trials with clofentezine conducted in cucurbits

CUCURBITS Country, Year *Location* (variety)	Application					PHI days	Commodity	Residues, mg/kg clofentezine	Ref.
	Formulation	kg ai/ha	kg ai/hL	Water L/ha	No.				
GHERKINS									
Netherlands, 1988	50 SC	0.45	0.015	3000	1	1	whole fruit	0.24	R-12999
Horst						3		0.14	(654-88)
(Osias)						7		0.03	
Netherlands, 1988						1		0.13	(655-88)
HegeIsunn						3		0.08	
(Osias)						7		0.04	
CUCUMBER									
France, 1987	50 SC	0.16	0.02	800	1	1	whole fruit	0.09	R-13225
St Lambert Levées						4		0.07	(I8753)
						10		0.04	
						14		0.01	
Greece, 1988	50 SC	0.3	0.015	2000	1	0	whole fruit	0.15	R-13011
Esovalta						1		0.17	
(Palwera)						3		0.12	
		0.6	0.030	2000	1	1		0.23	
						3		0.21	
Greece, 1991	50 SC	0.3	0.015	2000	1	1	whole fruit	0.19	R-13063
Aspro						3		0.14	(No. 2)
(Sandra)		0.6	0.03	2000	1	1		0.37	

CUCURBITS Country, Year *Location* (variety)	Application					PHI days	Commodity	Residues, mg/kg clofentezine	Ref.
	Formulation	kg ai/ha	kg ai/hL	Water L/ha	No.				
						3		0.35	
Greece, 1991 *Esovalta* (Sandra)		0.3	0.015	2000	1	1		0.10	(No. 1)
						3		0.16	
		0.6	0.03	2000	1	1		0.25	
						3		0.35	
Greece, 1991 *Esovalta* (Daleva)		0.3	0.015	2000	1	1		0.12	(No. 3)
						3		0.12	
						7		0.07	
						14		0.02	
		0.6	0.03	2000	1	1		0.18	
						3		0.20	
						7		0.12	
						14		0.05	
Switzerland, 1985 *Dielsdorf* (?)	50 SC	0.30	0.02	1500	1	0	whole fruit	0.21	R-13197 (?)
						3		0.13	
						7		0.08	
						14		0.03	
						21		< 0.01	

Table 46. Results of residue trials with clofentezine conducted in melons

MELONS Country, Year *Location* (variety)	Application					PHI days	Commodity	Residues, mg/kg clofentezine	Ref.
	Formulation	kg ai/ha	kg ai/hL	Water L/ha	No.				
Spain, 1999 *Brenes* (Piel de sapo)	50 SC	0.20	0.067	300	1	0	whole fruit	< 0.05	R-13287 (ESP0201)
						3		< 0.05	
						3	peel	< 0.05	
						3	pulp	n.d.	
France, 1999 *Meilhan sur Garrone* (Buffalo)		0.20	0.08	250	1	0	whole fruit	< 0.05	(FRA0301)
						3		< 0.05	
						3	peel	0.08	
						3	pulp	n.d.	
Italy, 1999 *Molfetta* (Galia)	50 SC	0.20	0.04	500	1	0	whole fruit	0.17	(ITA0201)
						3		0.06	
						3	peel	0.19	
						3	pulp	n.d.	
Portugal, 1999 *Alpiarca* (Branco do Ribatejo)		0.20	0.033	600	1	0	whole fruit	< 0.05	(PRT0101)
						3		< 0.05	
						3	peel	0.07	
						3	pulp	n.d.	
Portugal, 1999 *Villa Franca de Xira* (Branco do Ribatejo)		0.20	0.033	600	1	0	whole fruit	< 0.05	(PRT0102)
						3		< 0.05	
						3	peel	0.07	
						3	pulp	n.d.	
Spain, 2000 *Brenes* (Extra rica miel)	50 SC	0.20	0.04	500	1	0	whole fruit	0.01	R-14529 (ESP0201)
						3		< 0.01	
						3	peel	0.02	
						3	pulp	n.d.	
France, 1999 *Ouzilly* (Dalton)		0.20	0.08	250	1	0	whole fruit	0.08	(FRA0201)
						3		0.05	
						3	peel	0.18	

MELONS Country, Year *Location* (variety)	Application Formulation	kg ai/ha	kg ai/hL	Water L/ha	No.	PHI days	Commodity	Residues, mg/kg clofentezine	Ref.
						3	pulp	n.d.	
Greece, 2000 *Chalkidona* (Galli)		0.20	0.04	500	1	0	whole fruit	0.11	(GRC0101)
						3		0.03	
						3	peel	0.04	
						3	pulp	n.d.	
Italy, 2000 *Giovani in Persiceto* (Calipso)		0.20	0.033	600	1	0	whole fruit	0.1	(ITA0101)
						3		0.03	
						3	peel	0.09	
						3	pulp	n.d.	

n.d.=not detectable (< LOQ×0.3).

Table 47. Results of residue trials with clofentezine conducted in tomato

TOMATO Country, Year *Location* (variety)	Application Formulation	kg ai/ha	kg ai/hL	Water L/ha	No.	PHI days	Commodity	Residues, mg/kg clofentezine	Ref.
Spain, 1999 *Brenes* (Inca)	50 SC	0.2	0.04	500	2	0	fruit	0.16	R-13288
						3		0.09	(ESP0201)
France, 1999 *Boe* (Perfect Peel)		0.2	0.08	250	2	0		0.21	
						3		< 0.05	(FRA0301)
Greece, 1999 *Korifi-Imatia* (Rio Grande)		0.2	0.04	500	2	0		0.06	(GRC0101)
						3		< 0.05	
Italy, 1999 *Molfetta* (Tondo Barletta)		0.2	0.04	500	2	0		0.06	(ITA0201)
						3		< 0.05	
Italy, 2005 Castagnito (Tomito)	50 SC	0.195	0.033	584	1	0	fruit	0.69	R-21215
						3		0.23	(125.I.SAG05
						7		0.12	/r)
						14		0.05	
(Missouri)	50 SC	0.219	0.033	656	1	0		0.59	(126.I.SAG05
						3		0.25	/r)
						7		0.15	
						14		0.10	
(H 34 02)		0.188	0.025	750	1	0		0.35	(127.I.SAG05
						3		0.23	/r)
						7		0.14	
						14		0.07	
(Guadalete/Ruphis)		0.191	0.033	572	1	0		0.50	(128.I.SAG05
						3		0.23	/r)
						7		0.10	
						14		0.06	
France, 1992 *Lisac* (Ferline)	50 SC	0.2	0.02	1000	1	3	whole fruit	0.05	R-13067 (XI 0201-1)
France, 1992 *St Michel* (Ferline)					1	3		0.09	(XI 0201-2)
Italy, 1986	50 SC		0.025	400-600	1	17	whole fruit	0.01	R-12997

TOMATO Country, Year *Location* (variety)	Application Formulation	kg ai/ha	kg ai/hL	Water L/ha	No.	PHI days	Commodity	Residues, mg/kg clofentezine	Ref.
Suzzomo (Peto Sed 220)						31		0.02	(A/986 B)
						45		< 0.01	
						59		ND	
			0.04	400-600	1	17		0.03	
						31		0.01	
						45		< 0.01	
						59		ND	
Italy, 1986 *Strada la Rizza* (Fandango)		0.375	0.025	1500	1	13		0.05	(A/986 E)
		0.6	0.04	1500		29		0.10	
Netherlands, 1999 *NL-9751 PE Haren* in glasshouse (Aromata)	50 SC	0.2	0.013	1500	2	0	whole fruit	0.08	R-14625
						3		< 0.05	(NLD0101)
						0		0.09	(NLD0102)
						3		0.09	
						0		0.08	(NLD0103)
						3		0.10	
(Ferrari)						0		0.14	(NLD0104)
						3		0.11	
(Kapika)						0		0.12	(NLD0105)
						3		0.12	
(Ferrai)						0		0.14	(NLD0106)
						3		0.16	
(Kapika)						0		0.15	(NLD0107)
						3		0.18	
France, 1999 *Montboissier* (Valina)	50 SC	0.224	0.08	280	2	0	whole fruit	0.07	R-13279
		0.2	0.08	250		3		0.06	(FRA0101)
Germany. 1999 Lambsheim (Vanessa)		0.2	0.05	400	2	0		0.07	(DEU0201)
						3		0.06	
						0		0.11	(DEU0202)
						3		0.11	
Germany, 1999 *Alfter-Oedekoven* (Hillshel)		0.2	0.033	600	2	0		< 0.05	(DEU0501)
						3		< 0.05	

Table 48. Results of residue trials with clofentezine conducted in walnut

WALNUT Country, Year *Location* (variety)	Application Formulation	kg ai/ha	kg ai/hL	Water L/ha	No.	PHI days	Commodity	Residues, mg/kg clofentezine	Ref.
USA, 1987 *California* (Chandler)	50 SC	0.28	0.012	2338	2	30	nutmeat	< 0.02	R-13243 (R152.09-87-J CA-01)
(Tahamee)					2	25		< 0.02	(R152.09-87-J CA-02)
(Chandler)					2	30		< 0.02	(R152.09-87-J CA-03)
(Sunland)					2	30		< 0.02	(R152.09-87-J

WALNUT Country, Year *Location* (variety)	Application Formulation	kg ai/ha	kg ai/hL	Water L/ha	No.	PHI days	Commodity	Residues, mg/kg clofentezine	Ref.
									FS-01)
USA, 1988 *California* (Howard)	50 SC	0.28	0.005	2840	1	30	nutmeat	< 0.02	R-13002 (R152.04-88-J CA-01)
(Tahamee)					1	25		< 0.02	(R152.04-88-J CA-02)
(Chandler)					1	30		< 0.02	(R152.04-88-J CA-03)
(Chandler)					1	30		< 0.02	(R152.04-88-J CA-04)

Table 49. Residues in almond meat and hulls from supervised trials with clofentezine

ALMOND Country, Year *Location* (variety)	Application Formulation	kg ai/ha	kg ai/hL	Water L/ha	No.	PHI days	Commodity	Residues, mg/kg clofentezine	Ref.
USA, 1987 *California* (Tioga)	50 SC	0.28			2	29	meat	< 0.02 [a]	R-13244 (R152.05-87-J CA-01)
							hulls	1.5 [a]	
(Ne Plus)					2	36	meat	< 0.02	(R152.05-87-J CA-02)
							hulls	1.7	
(Carmel)					2	38	meat	< 0.02	(R152.05-87-J CA-03)
							hulls	0.91	
(Carmel)					2	40	meat	< 0.02	(R152.05-87-J FS-01)
							hulls	1.2	
(Butte)					2	40	meat	< 0.02	(R152.05-87-J FS-02)
							hulls	1.2	
USA, 1988 *California* (Non-pariel)	50 SC	0.28			1	29	meat/hull	< 0.05/0.5	R-13003 (R152.01-88-J FS-01)
						45	(pre-hull split)	< 0.05/0.5	
						30	meat/hull (10% hull split)	< 0.05/0.2	
						24	meat/hull	< 0.05/0.9	
						30	(25% hull split)	< 0.05/0.5	
						17	meat/hull	< 0.05/0.7	
						28	(50% hull split)	< 0.05/0.6	
(Carmel)						30	meat/hull (pre-hull split)	0.1/1.4	(R152.01-88-J FS-02)
(Carmel)						32	meat/hull	< 0.05/0.7	(R152.01-88-J CA-01)
						40	(pre-hull split)	< 0.05/0.6	
						32	meat/hull (10% hull split)	0.1/2.0	
USA, 1988 *California*	50 SC	0.28				32	meat/hull (25% hull split)	0.2/1.1	
						32	meat/hull (50% hull split)	< 0.05/0.1	
(Carmel)						30	meat/hull (pre-hull split)	0.1/1.2	(R152.01-88-J CA-02)
						30	meat/hull	0.3/1.8	

ALMOND Country, Year *Location* (variety)	Application Formulation	kg ai/ha	kg ai/hL	Water L/ha	No.	PHI days	Commodity	Residues, mg/kg clofentezine	Ref.
							(10% hull split)		
						24	meat/hull (20-30% hull split)	0.3/1.5	
						24	meat/hull (50% hull split)	0.3/2.5	
						30	meat/hull (20-30% hull split)	0.2/2.2	
						30	meat/hull (20-30% hull split)	0.3/2.7	
(Carmel)						30	meat/hull (pre-hull split)	< 0.05/0.7	(R152.01-88-JCA-03)
						48	meat/hull (pre-hull split)	< 0.05/0.4	
						30	meat/hull (10% hull split)	< 0.05/1.4	
						24	meat/hull (20-30% hull split)	< 0.05/0.4	
						30	meat/hull (20-30% hull split)	< 0.05/0.3	
						24	meat/hull (50% hull split)	< 0.05/0.4	
						30	meat/hull (50% hull split)	< 0.05/0.2	
USA, 1993	50 SC	0.28	0.017	1684	1	30	meat/hull	< 0.01/0.11	R-13082
California (Carmel)	SE (60 g/L)	0.28	0.017	1684	1	30	(post-hull split)	< 0.01/0.06	(FSCA)
(Monterey)	50 SC	0.27	0.039	692	1	47	meat/hull	< 0.01/0.03	(DRC-01)
	SE (60 g/L)	0.28	0.040	692	1	47	(pre-hull split)	< 0.01/0.04	
(Pariel)	50 SC	0.27	0.044	608	1	30	meat/hull	< 0.01/0.12	(JFS-01)
	SE (60 g/L)	0.28	0.046	608	1	30	(pre-hull split)	< 0.01/0.12	
USA, 1985	50 SC	1.12	0.030	3741	1	21	meat/hull	0.02/30	R-13184
California		0.56	0.015	3741	1	21	(pre-hull split)	0.02/19	(R15.06-84-JCA-01)
		1.12	0.030	3741	1	36	meat/hull	0.24/60	
		0.56	0.015	3741	1	36	(post-hull split)	0.04/20	
		1.12	0.030	3741	1	16	meat/hull	0.08/39	(R15.06-84-JCA-03)
		0.56	0.015	3741	1	16	(pre-hull split)	0.06/26	
		1.12	0.030	3741	1	31	meat/hull	0.04/20	
		0.56	0.015	3741	1	31	(post-hull split)	0.04/14	
		0.14	0.004	3741	2	46	meat/hull (pre-hull split)	< 0.01/2.0	(R15.12-85-JCA-01)
		0.28	0.007	3741	2	46	meat/hull (pre-hull split)	< 0.01/0.89	
USA, 1985	50 SC	0.56	0.015	3741	1	46	meat/hull	< 0.01/1.9	
California							(pre-hull split)		
		0.14	0.004	3741	2	29	meat/hull (pre-hull split)	< 0.01/2.0	(R15.12-85-JCA-02)
		0.28	0.007	3741	2	29	meat/hull (pre-hull split)	< 0.01/2.3	
		0.56	0.015	3741	1	29	meat/hull (pre-hull split)	< 0.01/10.5	

ALMOND Country, Year *Location* (variety)	Application Formulation	kg ai/ha	kg ai/hL	Water L/ha	No.	PHI days	Commodity	Residues, mg/kg clofentezine	Ref.
		0.14	0.004	3741	2	25	meat/hull (pre-hull split)	< 0.01/0.68	(R15.12-85-JCA-03)
		0.28	0.007	3741	2	25	meat/hull (pre-hull split)	< 0.01/1.1	
		0.56	0.015	3741	1	25	meat/hull (pre-hull split)	< 0.01/2.1	
(Nonpareil)		0.14	0.003	4209	1	35	meat/hull (post-hull split)	< 0.01/0.43	(R15.12-85-JFS-01)
		0.28	0.007	4209	1	35	meat/hull (post-hull split)	< 0.01/0.60	
		0.56	0.013	4209	1	35	meat/hull (post-hull split)	< 0.01/1.2	
(Nonpareil)		0.14	0.003	4209	1	35	meat/hull (post-hull split)	< 0.01/1.2	(R15.12-85-JFS-02)
		0.28	0.007	4209	1	35	meat/hull (post-hull split)	< 0.01/1.6	
		0.56	0.013	4209	1	35	meat/hull (post-hull split)	< 0.01/6.6	
(Nonpareil)		0.14	0.002	7483	1	26	meat/hull (post-hull split)	< 0.01/0.26	(R15.12-85-JFS-03)
		0.28	0.004	7483	1	26	meat/hull (post-hull split)	< 0.01/1.0	
		0.56	0.007	7483	1	26	meat/hull (post-hull split)	< 0.01/0.72	
USA, 2002 *California*	SC 50	0.235	0.028	843	1	14	nutmeat	< 0.01	R-21214
							hull	0.38	(22084-CA01)
(Carmel)		0.238	0.028	853	1	14	nutmeat	< 0.01	(22084-CA02)
(Mission)							hull	1.06	
(Non Pareil)		0.232	0.025	920	1	14	nutmeat	< 0.01	(22084-CA03)
							hull	1.51	

a - Interval between applications >> PHI (1st application in spring). In such case, the last application can be considered alone and the resulting residue data are used for MRL calculation.

FATE OF RESIDUES IN STORAGE AND PROCESSING

In processing

The Meeting received information on the fate of clofentezine residues during aqueous hydrolysis under conditions of pasteurisation and baking, brewing and boiling and sterilisation. Information was also provided on the fate of clofentezine residues during the food processing of citrus, apples, grapes, and strawberries.

The hydrolytic stability of [^{14}C]-clofentezine was investigated in aqueous buffer solutions at three pH values and temperatures aimed at simulating normal processing practice (van der Gaauw, A., 2001, R-13317). The study was performed at pH 4, 5 and 6, and at temperatures of 90 °C, 100 °C and 120 °C, respectively, for between 20 and 60 minutes. The range of hydrolytic conditions represents the processes of pasteurisation, baking/brewing/boiling and sterilization (Table 50). For this, duplicate samples (except time 0) were set-up for each pH value. Each sample, consisting of 100 mL buffer solution containing the radio-labelled test substance, was incubated in a tightly closed glass vessel. The vessels were placed in an oil bath, under reflux conditions or in an autoclave. The test substance was tested at a concentration of about 2 µg/L buffer solution. At time 0 and after 20 or 60 minutes incubation, the samples were taken and analysed by HPLC. Selected samples were additionally

submitted to TLC analysis. The temperatures were maintained constant throughout the incubation period and no significant variation of the pH values was observed in the buffered solutions.

Table 50. Hydrolysis products of clofentezine at simulated processing

Temperature (°C)	Time (min)	pH	Process Represented
90	20	4	Pasteurisation
100	60	5	Baking, Brewing, Boiling
120	20	6	Sterilisation

The mean recoveries of radioactivity were 100.0 ± 1.8% (pH 4), 100.7 ± 2.3% (pH 5) and 99.8 ± 3.0% (pH 6) of the applied radioactivity. [^{14}C]-clofentezine was shown to be hydrolytically stable at pH 4 with no degradation occurring after 20 minutes at 90 °C (Table 51). At pH 5, [^{14}C]-clofentezine degraded by about 10% to form one known hydrolysis product, characterised as 2-chlorobenzoic (2-chlorobenzylidene)hydrazide. This fraction accounted for 12.4% of the applied radioactivity at the end of the incubation period. At pH 6, [^{14}C]-clofentezine degraded completely to three known metabolites, characterised as 2-chlorobenzoic (2-chlorobenzylidene)hydrazide, 2-chlorobenzo- nitrile and 2-chlorobenzamide. These compounds represented 77.6%, 4.9% and 17.0% of the applied radioactivity after 20 minutes at 120 °C, respectively.

Table 51. Hydrolysis products of clofentezine at simulated processing

Substance	Composition of reaction mixture as % of starting material		
	pH 4 (90 °C)	pH 5 (100 °C)	pH 6 (120 °C)
clofentezine	99.3	89.4	n.d.
2-chlorobenzoic (2-chlorobenzylidene) hydrazide	n.d.	12.4	77.6
2-chlorobenzonitrile	n.d.	n.d.	4.9
2-chlorobenzamide	n.d.	n.d.	17.0

n.d. = not detected

One trial were conducted in USA in 1987 involving application of clofentezine as the 50 SC formulation to mature oranges using a hand-held sprayer at 350 g ai/ha (Kelly, I.D., 1987, R-12738). One plot was treated 29 days, another 2 days before harvest. The treated fruit were then processed and clofentezine residues determined. The mean recovery ranged between 83 – 115% from the matrices analysed. In the unprocessed fruit, a mean residue level of 0.04 mg/kg clofentezine was found at harvest, with nearly all of the residues on the peel. Residues in processed products did not concentrate except in orange oil, where concentration factors of 65 – 120 (calculated from whole fruit) were found (Table 52).

Table 52. Clofentezine residues in orange and processed fractions resulting from supervised trials in USA

CITRUS country, year, location (variety)	Application			PHI days	Commodity	Residues, mg/kg clofentezine	Ref
	Form	kg ai/ha	no.				
USA, 1987, *California* (Washington Navel)	50 SC	0.35	1	2	whole fruit [a] (unwashed orange)	0.04	R-12738 (R15.07-87-JFS-01)
					flesh (unwashed orange)	0.02	
					peel (unwashed orange)	0.10	
					whole fruit [a] (washed orange)	0.03	

CITRUS country, year, location (variety)	Application Form	kg ai/ha	no.	PHI days	Commodity	Residues, mg/kg clofentezine	Ref
					flesh (washed orange)	< 0.01	
					peel (washed orange)	0.11	
					juice	< 0.01	
					oil	2.6	
					wet peel	< 0.05	
					dried peel	0.06	
					dried fine from peel	< 0.05	
					molasses	< 0.05	
				29	whole fruit [a] (unwashed orange)	0.04	
					flesh (unwashed orange)	0.01	
					peel (unwashed orange)	0.12	
					whole fruit [a] (washed orange)	0.04	
					flesh (washed orange)	< 0.01	
					peel (washed orange)	0.13	
					juice	< 0.01	
					oil	4.8	
					wet peel	< 0.05	
					dried peel	0.05	
					dried fine from peel	0.06	
					molasses	< 0.05	

a - Residues in whole fruit were calculated values.

Eight field trials were conducted in Spain to determine the residue level of clofentezine in oranges and processed fractions during the 2001 growing season (Serano C., 2002, R-15921). Clofentezine was applied only one time with an application rate of 0.01 kg ai/hL. Fruit harvested at 40 days after application were processed to peel and pulp and further to juice and jam, and clofentezine residues were determined in each fraction. Residues in whole fruit have been calculated from residues in peel and pulp. The results are summarized in Table 47. In most cases, distinct processing factors for pulp, juice and jam could not be calculated. All three categories of processed goods had mostly residue values below the LOQ.

Apples received a late season application of clofentezine at a rate of 0.03% ai, 14 days before harvest in 1981 (Snowdon, P.J., 1983, R-12731). This late treatment was deliberately made to ensure significant residues would be present to follow the processing operation. At harvest, the fresh fruit were sent for processing. The resulting products were canned apple juice and sauce, fresh cider and wet and dry apple pomace. The recovery efficiency in the fortification experiments gave a mean recovery of 83.6 ± 6.5%. In the unprocessed apples, a mean residue level of 1.29 mg/kg clofentezine was found at harvest. Subsequent mean residue levels found in samples of fresh cider, apple juice, apple sauce and wet pomace were < 0.01, 0.02, 0.01 and 7.34 mg/kg respectively (Table 53).

Table 53. Clofentezine residues in processed fractions of field treated apples in USA

APPLES country, year, location (variety)	Application Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	PHI days	Commodity	Residues, mg/kg clofentezine	Ref
USA, 1981 *New York* (McIntosh)	80 WP		0.03		1	14	whole fruit	1.29	R-12731
							pressed juice [a]	0.02	
							wet pomace	7.34	
							dry pomace	19.5	
							fresh cider	< 0.01	
							Sauce	0.01	

a - Before division into cider and juice.

Apples grown at four sites in the USA received early (½" green) to late (21 day PHI) season applications of clofentezine as 50 SC formulation at rates of 0.28 and 0.56 kg ai/ha in 1985 (Peatman, M.H., Snowdon, P.J., 1985, R-12732). At harvest, the fresh fruit were sent for processing. The resulting products were mash, apple juice and wet and dry pomace (Table 54). Clofentezine residues were enriched in pomace, with transfer factors of approximately 5 – 6 for wet pomace and 8 – 12 for dry pomace. The residue levels in juice were very low.

Table 54. Clofentezine residues in processed fractions of field treated apples derived from supervised trials in USA

APPLES country, year, location (variety)	Application Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	PHI days	Commodity	Residues, mg/kg clofentezine	Ref
USA, 1985 *New York* (McIntosh)	50 SC	0.28	0.030	935	1	21	whole apple	0.10	R-12732
							juice	0.02	(R15.01-85-JES
							wet pomace	0.55	-01)
							dry pomace	0.86	
						107	whole apple	< 0.01	
							juice	< 0.01	
							wet pomace	0.02	
							dry pomace	0.04	
						149	whole apple	< 0.01	
							juice	< 0.01	
							wet pomace	< 0.01	
							dry pomace	0.01	
		0.56	0.060	935	1	21	whole apple	0.19	
							mash	0.27	
							juice	0.02	
							wet pomace	1.10	
							dry pomace	2.20	
						107	whole apple	0.02	
							mash	0.02	
							juice	< 0.01	
							wet pomace	0.06	
							dry pomace	0.11	
						149	whole apple	< 0.01	
							mash	< 0.01	
							juice	< 0.01	
							wet pomace	0.02	
							dry pomace	0.05	
USA, 1985 *Ohio* (Red Delicious)	50 SC	0.28			1	158	whole apple	< 0.01	(R15.01-85-JRS
							juice	< 0.01	-01)
							wet pomace	< 0.01	
							dry pomace	< 0.01	
USA, 1985 *Pennsylvania*	50 SC	0.28			2	45	whole apple	0.02	(R15.01-85-JES
							juice	< 0.01	-03)

APPLES country, year, location (variety)	Application Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	PHI days	Commodity	Residues, mg/kg clofentezine	Ref
(Red Delicious)							wet pomace	0.03	
							dry pomace	0.16	
USA, 1985 New Jersey (Staymen)	50 SC	0.28			2	40	whole apple	0.02	(R15.01-85-JES -04)
							mash	< 0.01	
							juice	< 0.01	
							wet pomace	< 0.01	
							dry pomace	0.07	

Apples grown at three sites in the USA received application at 144 to 170 days PHI at rates of 0.28, 0.84 and 1.40 kg ai/ha (Castro, L., 1992, R-12740). At harvest, the fresh fruit were sent for processing and the residues determined in whole fruit and dried pomace. Before processing, the apples derived from Michigan and Washington trials were washed, but the fruits from New York trial were not washed. Drying time was 14 h at 60 °C in a vacuum oven for the Michigan samples, 2 ¼ h in a forced air oven at 170 – 190 °C for the New York samples, and 9 ¾ h in a fluidized bed at 175 – 200 °C for the Washington samples. Water content of pomace after drying ranged from 5 – 8% (NY, WA) to 57 – 62% (MI). No sample of whole fruit gave detectable samples. No residues were found in the trials from Michigan, and in those from Washington, only those with an application rate of 1.4 kg ai/ha led to measurable residues. Clofentezine residues were concentrated in dry pomace. Measurable residues were only found when the pomace was dried sufficiently (0.02 – 0.06 mg/kg). When apples were washed before processing, resides in dry pomace decreased to levels just above the LOQ (0.01 mg/kg). The possible concentration factors for clofentezine in apples could not be determined since even at highly exaggerated application rates, no residues were detected in fresh fruit.

Apples grown at four sites in the USA received application with 205 to 15 days PHI at rates between 0.14 and 1.4 kg ai/ha in 1993 (Cole, M.G., 1996, R-13083). At harvest, the fresh fruit were sent for processing and the residues determined in whole fruit and wet pomace. Residues in dry pomace were not determined since this item was removed by EPA from the list of processed commodities after the initiation of the study. Residues were determined in unwashed apples and wet pomace (Table 55).

Table 55. Clofentezine residues in apples and processed fractions resulting from supervised trials in USA

APPLES country, year, location (variety)	Application Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	PHI days	Commodity	Residues, mg/kg clofentezine	Ref
USA, 1993 *California* (Granny Smith)	50 SC	0.28	0.030	935	1	15	whole fruit	0.03	R-13083
		0.70	0.075	935	1	200	whole fruit	0.01	(J-93R-01-DRC -01)
USA, 1993 *Michigan* (MacIntosh)		0.28	0.030	935	1	15	whole apple	0.02	(J-93R-01-JRS -01)
							wet pomace	0.04	
		0.70	0.075	935	1	111	wet pomace	0.03	
		1.4	0.150	935	1	111	whole apple	0.01	
							wet pomace	0.06	
						118	wet pomace	0.02	
						121	wet pomace	0.03	
USA, 1993 *New York* (Twenty Ounce)		0.14		935		15	whole apple	0.05	(J-93R-01-DRS -01)
							wet pomace	0.06	
		0.28		935	1	15	whole apple	0.09	
							wet pomace	0.19	
		1.4		935	1	112	whole apple	0.02	
							wet pomace	0.04	
						118	whole apple	0.02	
							wet pomace	0.03	
						124	wet pomace	0.01	

APPLES country, year, location (variety)	Application Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	PHI days	Commodity	Residues, mg/kg clofentezine	Ref
USA, 1993 *Washington* (Red Delicious)		0.14		935	1	15	whole apple	0.05	(J-93R-01-TWM -01)
							wet pomace	0.12	
		0.28		935	1	15	whole apple	0.09	
							wet pomace	0.31	
		0.70		935	1	136	wet pomace	0.01	
						142	wet pomace	0.01	
		1.4		935	1	136	whole apple	0.02	
							wet pomace	0.04	
						142	whole apple	0.02	
							wet pomace	0.04	
						148	wet pomace	0.01	

Apples purchased commercially were treated with clofentezine dissolved in acetone by application with a checked syringe at an actual rate of 0.40 mg/kg and then processed to apple sauce (Pollmann, B., 2003, R-15918). Treated apples, washed apples, washing water, apple peel, peeled apple, and the apple sauce were analysed for clofentezine and the potential degradation products AEC593600, 2-chlorobenzamide and 2-chlorobenzonitrile. Clofentezine was detected in all samples but its concentration was below the LOQ in wash water, peeled apples, and in 6 out of 7 apple sauce samples including three samples analysed in addition to the original study plan. Of the degradation products, only 2-chlorobenzonitrile was detected in any of the samples, and only in a single apple peel sample contained it in quantifiable amount (0.02 mg/kg). The analytical results are listed in Table 56. Transfer factors and concentration factors were calculated only for clofentezine. Below the quantification limit, the LOQ was used for calculation of the factors. Clofentezine residues were significantly decreased by processing apples to apple sauce, with concentration factors below 0.1.

Table 56. Clofentezine residues in apples and processed fractions resulting from the spiked samples in 2003

Sample No.	Matrix	Clofentezine (mg/kg)	AEC 593600 (mg/kg)	2-Chloro-benzamide (mg/kg)	2-Chloro-benzonitrile (mg/kg)
G03N014R-8	unwashed apples	0.40	n.a.	n.a.	n.a.
G03N014R-9	wash water	< LOQ	n.d.	n.d.	n.d.
G03N014R-10	washed apples	0.29	n.d.	n.d.	< LOQ
G03N014R-11	apple cores	0.59	n.d.	n.d.	< LOQ
G03N014R-12	apple peel	1.32	n.d.	n.d.	0.02
G03N014R-13	peeled apples	< LOQ	n.d.	n.d.	n.d.
G03N014R-14	apple sauce	0.03	n.d.	n.d.	n.d.
G03N014R-17	unwashed apples	0.41	n.a.	n.a.	n.a.
G03N014R-18	apple sauce	< LOQ	n.d.	n.d.	n.d.
G03N014R-19	apple sauce	< LOQ	n.d.	n.d.	n.d.
G03N014R-20	apple sauce	< LOQ	n.d.	n.d.	n.d.

LOQ: 0.02 mg/kg n.d.: not detectable n.a.: not analysed

In nine USA trials clofentezine was applied to the vines once at the first sign of mite infestation. Two rates were used (0.28 or 0.56 kg/ha) (Castro, L., 1987, R-12736). The mature fruit was then harvested as normal resulting in pre-harvest intervals (PHI) of 39 and 46 days. Grapes were then dried into raisins. Residue levels were determined in whole grapes and raisins (Table 57).

Table 57. Clofentezine residues in grapes and processed raisins resulting from supervised trials in USA, 1984 and 1985

GRAPE country, year, location (variety)	Application					PHI days	Commodity	Residues, mg/kg clofentezine	Ref
	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.				
USA, CA, 1985 (Thomson)		0.28	0.020	1403	1	46	whole fruit	0.10	(R15.04-85-JFS -01)
							raisin	0.17	
		0.56	0.040	1403	1	46	whole fruit	0.26	
							raisin	0.29	
(Thomson)		0.28	0.012	2338	1	41	whole fruit	0.11	(R15.04-85-JFS -02)
							raisin	0.12	
		0.56	0.024	2338	1	41	whole fruit	0.06	
							raisin	0.14	
(Thomson)		0.28	0.020	1403	1	39	whole fruit	0.22	(R15.04-85-JFS -03)
							raisin	0.14	
		0.56	0.040	1403	1	39	whole fruit	0.12	
							raisin	0.35	

In Germany, grape vines were treated twice with clofentezine at 0.11 and 0.28 kg ai/ha in June and September, 1986 (Peatman, M.H., Snowdon, P.J., 1989, R-18417). At the penultimate sampling time, the additional grapes were taken for processing into must and wine. Residues were determined in grapes, must and wine (Table 58).

Table 58. Clofentezine residues in grapes and processed products derived from supervised trials in Germany, 1986

GRAPES Country, Year *Location* (variety)	Application					PHI days	Commodity	Residues, mg/kg clofentezine	Ref. Reg.DocID. (Trial No.)
	Formulation	kg ai/ha	kg ai/hL	Water L/ha	No.				
Germany, 1986 *Angelbachtal* (Muller Thurgau)	co-formulation with cyhexatin	0.113+0.281	0.014	800+2000	2	0	Grape	0.36	R-18417
						16		0.28	
						29		0.20	
						37		0.22	
						44		0.16	
						37	Must	no sample	
							Wine	nd	
Germany, 1986 *Schozach* (Blauer Spätburgunder)		0.113+0.281	0.014	800+2000	2	0		0.30	
						14		0.18	
						28		0.17	
						35		0.12	
						42		0.12	
						33	Must	< 0.01	
							Wine	< 0.01	

nd: non-detectable residue.

In Germany, grape vines were treated twice with clofentezine at 0.16 and 0.40 kg ai/ha in May and September, 1986 (Peatman, M.H., Snowdon, P.J., 1989, R-21166). At the penultimate sampling time, the additional grapes were taken for processing into must and wine. Residues were determined in grapes, must and wine (Table 59).

Table 59. Clofentezine residues in grapes and processed products derived from supervised trials in Germany, 1986

GRAPES Country, Year *Location* (variety)	Application					PHI days	Commodity	Residues, mg/kg clofentezine	Ref. Reg.DocID. (Trial No.)
	Formulation	kg ai/ha	kg ai/hL	Water L/ha	No.				
Germany, 1986 *Angelbachtal* (Muller Thurgeau)	50 SC	0.16+0.40	0.02	800+2000	2	0	grape	1.12	R-21166
						16		0.53	
						29		0.49	
						37		0.34	
						44		0.28	
						37	must	no sample	
							wine	< 0.01	
Germany, 1986 *Schozach* (Blauer Spätburgunder)		0.16+0.40	0.06	267+670	2	0	grape	0.88	
						14		0.46	
						28		0.35	
						35		0.20	
						42		0.18	
						33	must	< 0.01	
							wine	< 0.01	

In German trials grapes received double applications of clofentezine with 0.11 or 0.28 kg ai/ha as the EC co-formulation with cyhexatin between in June and September, 1985 (Peatman, M.H., Snowdon, P.J., 1988, R-21165). At the penultimate sampling time, the additional grapes were taken for processing into must and wine. Residues were determined in grapes, must and wine (Table 60).

Table 60. Clofentezine residues in grapes and processed products resulting from supervised trials in Germany, 1986

GRAPES Country, Year *Location* (variety)	Application					PHI days	Commodity	Residues, mg/kg clofentezine	Ref. Reg.DocID. (Trial No.)
	Formulation	kg ai/ha	kg ai/hL	Water L/ha	No.				
Germany, 1985 *Ungstein* (Blauer Portugieser)	co-formu-lation with cyhexatin EC	0.112+0.28	0.014	800+2000	2	0	grape	1.5	R-21165
						13		0.87	
						27		0.52	
						34		0.61	
						41		0.32	
						34	must	0.34	
							wine	0.03	
Germany, 1985 *Meersburg* (Müller Thurgau)		0.107+0.268	0.040	267+670	2	0	grape	2.90	
						14		1.20	
						22		0.89	
						28		1.00	
						35		0.67	
						42		0.73	
						35	must	0.33	
							wine	0.10	
Germany, 1985 *Schozach* (Blauer Spätburgunder)		0.107+0.268	0.040	267+670	2	0	grape	1.70	
						14		0.94	
						28		0.66	
						35		0.69	
						42		0.29	
						35	must	0.02	
							wine	-	

Clofentezine was applied to vines at mid-season at 0.28 kg ai/ha in USA, 1986 (Castro, L., 1987, R-12737). Mature grapes from the treated vines were collected 30 days after spraying. The harvested grapes were separated into three groups: fresh grapes for analysis, grapes to be crushed for juice and pomace, and grapes to be dried into raisins. The second group of grapes was crushed to produce a must, half of which was pressed to yield juice and wet pomace. The other half of the must was fermented for four days and then pressed to yield the fermented pomace. Both wet and fermented pomace were further dried in the laboratory to about 10% moisture content and will be referred to as oven-dried pomace. The third group of grapes was sun-dried to raisins for ten days and stored at 13 °C at low humidity to await processing (separation into grades). The raisins were separated into grade and off-grade. Clofentezine was detected whole grapes and derived products (Table 61).

Table 61. Clofentezine residues in grapes and processed fractions resulting from supervised trials in USA, 1986

GRAPE country, year, location (variety)	Application					PHI days	Commodity	Residues mg/kg clofentezine	Ref
	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.				
USA, 1986 *California* (Thompson Seedless)	50 SC	0.28	0.015	1870	1	30	whole grape	0.16	R-12737 (R15.04-86-J CA-01)
							juice	0.26	
							fresh wet pomace	0.20	
							fermented pomace	0.15	
							fresh oven-dried pomace	< 0.50	
							fermented oven-dried pomace	< 0.50	
							grade raisin	0.16	
							off-grade raisin	0.37	
							raisin waste	0.93	

In Italy, grape received single or double applications of clofentezine at rates of 0.01 and 0.02 kg ai/hL to crop stages ranging from 10 cm high shoots to 45 days pre-harvest in 1987 (Snowdon, P.J., Godfrey, T.L., 1991, R-12739). Grapes were sampled, between 42 and 127 days after the last application. Part of each sample was processed into wine. Residues were determined in grapes, must and wine fermented from macerated or peeled grapes. Clofentezine was detected whole grapes and derived products (Table 62).

Table 62. Clofentezine residues in grapes and processed fractions resulting from supervised trials in Italy, 1987

GRAPE country, year, location (variety)	Application					PHI days	Commodity	Residues mg/kg clofentezine	Ref
	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.				
Italy, 1987	50 SC	0.2	0.02	1000	1	127	whole grape	< 0.01	R-12739
Broni		0.2+0.225	0.02+0.015	1000+1500	2	61	whole grape	0.02	(R/12-87 A)
(Riesling)		0.15	0.01	1500	1	61	whole grape	0.02	
		0.30	0.02	1500	1	61	whole grape	0.03	
		0.30	0.02	1500	1	42	whole grape	0.24	
(Riesling)		0.2+ 0.225	0.02+ 0.015	1000+ 1500	2	61	must	nd	(R/6-87 A)
							wine making with maceration	nd	
							blank wine making (without peel)	< 0.01	

GRAPE country, year, location (variety)	Application Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	PHI days	Commodity	Residues mg/kg clofentezine	Ref
(Riesling)		0.15	0.01	1500	1	61	must	nd	
							wine making with maceration	nd	
							blank wine making (without peel)	< 0.01	
		0.30	0.02	1500	1	61	must	nd	
							wine making with maceration	nd	
							blank wine making (without peel)	nd	
		0.30	0.02	1500	1	42	must	nd	
							wine making with maceration	nd	
							blank wine making (without peel)	< 0.01	
Italy, 1987 *Selvazzano Dentro* (Merlot)		0.2+	0.02+	1000+	2	63	must	< 0.01	(R/6-87 D)
		0.225	0.015	1500			wine making with maceration	< 0.01	
							blank wine making (without peel)	< 0.01	
		0.15	0.01	1500	1	63	must	< 0.01	
							wine making with maceration	0.03	
							blank wine making (without peel)	< 0.01	
		0.30	0.02	1500	1	63	must	0.02	
							wine making with maceration	0.11	
							blank wine making (without peel)	< 0.01	
		0.30	0.02	1500	1	47	must	< 0.01	
							wine making with maceration	0.02	
							blank wine making (without peel)	nd	

Evaluation of residues in processed commodities of grapes was carried out in one USA trial in 1993 (Scott Brady, S., 1995, R-12742). Grapes received single treatments of clofentezine at a rate of 290.7 or 580.5 g ai/ha 21 days before harvest. Whole grapes were then processed to either raisins and raisin waste, or to juice and pomace. Residue levels were established and concentration factors calculated. Clofentezine was detected in whole grapes and derived products (Table 63).

Table 63. Clofentezine residues in grapes and processed fractions resulting from supervised trials in USA, 1993

GRAPE country, year, location (variety)	Application Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	PHI days	Commodity	Residues mg/kg clofentezine	Ref
USA, 1993 *California* (Thompson Seedless)	50 SC	0.291	0.037	785	1	21	field grapes	0.18	R-12742 (J-93R-02-FSCA)
							processor grapes	0.09	
							juice	nd	
							wet pomace	0.17	
							dry pomace	0.11	
							raisins	0.02	
							raisin waste	0.25	
		0.581	0.074	785	1	21	field grapes	0.56	
							processor grapes	0.25	
							juice	nd	
							wet pomace	0.47	
							dry pomace	0.37	
							raisins	0.07	
							raisin waste	0.79	

nd: non-detectable residue.

Evaluation of residues in processed commodities of grapes was carried out in USA trials in 1991 (Scott Brady, S., 1992, R-12741). Vines received single treatments of clofentezine at a rate of 0.245 kg ai/ha 35 days before harvest. Whole grapes were then processed to either raisins and raisin waste, or to juice and pomace. Residue levels were established and concentration factors calculated. Clofentezine was detected whole grapes and derived products (Table 64).

Table 64. Clofentezine residues in grapes and processed fractions resulting from supervised trials in USA, 1991

GRAPE country, year, location (variety)	Application Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	PHI days	Commodity	Residues mg/kg clofentezine	Ref
USA, 1991 *California* (Thompson Seedless)	50 SC	0.245	0.019	1311	1	35	whole grapes	0.03	R-12741 (J-91R-02-JFS -01)
							juice	0.06	
							wet pomace	0.06	
							dry pomace	0.10	
							raisins	< 0.02	
							raisin waste	0.29	

Strawberries treated with clofentezine as the 50 SC formulation were processed to simulate canning in Germany, 2001 (Pollmann, B., 2002, R-14209). Fresh strawberries were sorted, washed, sugared, canned and then pasteurized. Residues were determined from fresh strawberries as well as after pasteurization. Table 65 summarizes the results.

Table 65. Clofentezine residues in strawberries and processed fractions resulting from supervised trials in Germany, 2001

STRAWBERRY country, year, location (variety)	Application Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	PHI days	Commodity	Residues mg/kg clofentezine	Ref
Germany, 2001 *Eberdingen* (Elsanta)	50 SC	1.0	0.125	800	1	0	whole fruit	0.75	R-14209 (F01N037R)
							canned strawberries	0.23	

STRAWBERRY country, year, location (variety)	Application					PHI days	Commodity	Residues mg/kg clofentezine	Ref
	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.				
Germany, 2001 *Kleinsachsenheim* (Elsanta)		0.939	0.125	751	1	0	whole fruit	2.38	(F01N038R)
							canned strawberries	0.38	

The processing factors calculated from the trial data reported under supervised trials or under processing are summarised in Table 66.

Table 66. Summary of processing factors for clofentezine residues

Raw agricultural commodity (RAC)	Processed commodity	Calculated processing factors. [a]	Median or best estimate
Orange	Flesh	0.06, < 0.08(3), 0.08(2), 0.09, < 0.10, 0.13, < 0.14(5), 0.14, 0.16, < 0.17, 0.17(3), 0.19, < 0.20(4), 0.20(4), 0.22(3), < 0.25(4), 0.25(4), 0.26, < 0.33(5), 0.33(2), 0.43, < 0.50(2), 0.50, 0.94	0.20
	Peel	2.00, 2.07, 2.13, 2.15, 2.43, 2.44(2), 2.50(2), 2.63, 2.67, 2.69, 2.71, 2.75, 2.86, 2.88, 2.90, 3.00(2), 3.20(2), 3.22(2), 3.25(2), 3.40, 3.43(2), 3.50(4), 3.56, 3.57, 3.67(5), 3.71, 3.78, 3.83(2), 4.00(4), 4.08, 4.17, 4.22, 4.40, 4.62, 6.70	3.43
	Juice	< 0.08, < 0.11, < 0.14, 0.14, < 0.17(3), < 0.20, < 0.25(2), < 0.33(2)	0.14
	Oil	86.7, 120	103
	Wet peel	<1.25, <1.70	<1.25
	Dried peel	1.25, 2.0	1.63
	Dried fine from peel	1.50, <1.67	1.50
	Molasses	<1.25, <1.67	<1.25
Apples	Washed apples	0.73	0.73
	Peeled apples	< 0.050	< 0.050
	Peel	3.30	3.30
	Wet pomace	< 0.50, 1.20, 1.50 (2), 2.00 (4), 2.11, 2.40, 3.00, 3.44, 5.50, 5.69, 5.79, 6.00	2.06
	Dried pomace	3.50, 5.50, 5.79, 6.00, 8.00, 8.60, 11.6, 15.1	7.00 8.30
	Juice	0.016, 0.11, 0.20, < 0.5 (3)	0.11
	Fresh cider	< 0.008	0.008
	Sauce	0.008, < 0.049(3), 0.075	0.042
Grapes	Raisins	0.22, 0.28, 0.64, < 0.67, 1.09, 1.12, 1.70, 2.33, 2.92	1.11
	Juice	nd(2)	0
	Wet pomace	1.88, 1.89	1.89
	Dry pomace	1.22, 1.48	1.35
	White wine making	< 0.042, < 0.50 (2)	< 0.042
Strawberries	Canned strawberries	0.16, 0.31	0.24

a - 'Less-than' (<) values are derived from cases where residues were not detected in the processed commodity. The 'less-than' processing factor is then calculated from the LOQ of the analyte in the processed commodity and the residue in the raw agricultural commodity.

RESIDUES IN ANIMAL COMMODITIES

Direct animal treatments

Clofentezine is not used for direct animal treatments.

Farm animal feeding studies

Dairy cows

The Meeting received a lactating dairy cow feeding study, which provided information on likely residues resulting in animal tissues and milk from residues in the animal diet.

At the end of the feeding studies, tissues from the sacrificed animals were frozen and despatched to residue analysis lab with further 500 mL sub-samples of all the milk samples previously provided. Upon receipt, these samples were all transferred to deep freeze storage (-20 °C). The storage stability of clofentezine residues showed that more than 90% and 80% of the initial (day 0) clofentezine-derived residue by conversion to 2-chlorobenzoic acid (2-CBA) in cattle tissues (liver, muscle, fat) and milk may be accounted for after 15 months' storage in deep freeze (Peatman, M.H., Snowdon, P.J., 1985, R-13164). Intervals between sampling and analysis were reported for animal feeding studies and were within the acceptable proven storage stability duration.

Four groups of three Friesian dairy cows were fed clofentezine in the diet (technical grade) at dose levels of 0, 200, 600 and 2000 mg/animal/day (equivalent to 0, 0.57, 1.7 and 5.7 mg/kg bw/day using an average beef cattle bodyweight of 350 kg or 0, 0.4, 1.2 and 4.0 mg/kg bw/day using an average dairy cattle bodyweight of 500 kg, and equivalent to 0, 10, 30 and 100 mg/kg feed using an average feed consumption of 20 kg), over a 28 day period (Snowdon, P.J., 1985, R-12733). Taking into account the quantity of beef cattle daily feed which could encompass dry apple pomace (50%) and the potential residues in dried apple pomace a dose of 200 mg/animal/day was selected as being the lowest dose level in a worst case scenario. Levels at 3 and 10 times this were also included in the study. Daily milk samples were taken and at the termination of the study liver, kidney, heart, muscle, peritoneal fat and subcutaneous fat were analysed (Table 67 and 68). The limit of determination was 0.05 mg/kg equivalent clofentezine.

Table 67. Clofentezine derived residues in milk from lactating dairy cows dosed with clofentezine at 10 (1×), 30 (3×) and 100 ppm (10×) in the dry-weight diet, for 28 consecutive days

Treatment group (mg/animal.day)	Animal number	Total clofentezine-derived residue level (mg/kg) [b]					
		Day 1	Day 7	Day 10	Day 14	Day 20	Day 28
Control	1	n.d. [a]	n.d.	n.d.	n.d.	0.008	n.d., n.d.
	2	n.d.	n.d.	0.009	n.d.	n.d.	0.022
	3		n.d.		n.d.		
200 (10 ppm)	4	< 0.05	n.d.	< 0.05	< 0.05	< 0.05	< 0.05
	5	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05
	6	n.d.	n.d.	< 0.05	n.d.	< 0.05	< 0.05
600 (30 ppm)	7	< 0.05	0.07	0.07	< 0.05	0.08	< 0.05
	8	< 0.05	< 0.05	0.07	0.06	< 0.05	< 0.05
	9	n.d.	< 0.05	0.06	0.14	0.08	< 0.05
2000 (100 ppm)	10	< 0.05	0.15	0.13	0.17	0.18	0.20, 0.22
	11	< 0.05	0.24	0.17	0.27, 0.21	0.17	0.20, 0.21
	12	< 0.05	0.11	0.12	0.12	0.14	0.11, 0.15

a - n.d.: no detectable residue.

b measured as 2-chlorobenzoic acid but expressed in terms of equivalent clofentezine by multiplication with molecular weight factor of 1.936.

Table 68. Clofentezine derived residues in animal tissue from lactating dairy cows dosed with clofentezine at 10 (1×), 30 (3×) and 100 ppm (10×) in the dry-weight diet, for 28 consecutive days

Treatment group (mg/animal/day)	Animal number	Total clofentezine-derived residue level (mg/kg) [b]					
		Liver	Kidney	Heart	Muscle	P. fat	S. fat
Control	1	n.d. [a]	0.037	n.d.	n.d.	n.d.	n.d.
	2	0.013	0.059	0.006	n.d.	n.d.	
	3	n.d.	0.018	0.005	n.d.		n.d.
200	4	0.33	< 0.05	< 0.05	< 0.05	< 0.05	n.d.
	5	0.24	< 0.05	< 0.05	< 0.05	n.d.	n.d.
	6	0.22	< 0.05	< 0.05	< 0.05	n.d.	n.d.
600	7	1.4	0.22	< 0.05	< 0.05	< 0.05	< 0.05
	8	1.1	0.25	< 0.05	< 0.05	< 0.05	< 0.05
	9	0.95	0.06	< 0.05	< 0.05	< 0.05	< 0.05
2000	10	3.1	0.54	< 0.05	< 0.05	< 0.05	< 0.05
	11	1.8	0.55	0.05	< 0.05	< 0.05	< 0.05
	12	1.7	0.12	< 0.05	< 0.05	< 0.05	< 0.05

a - n.d.: no detectable residue.

b - measured as 2-chlorobenzoic acid but expressed in terms of equivalent clofentezine by multiplication with molecular weight factor of 1.936.

Calves

In order to determine any residue levels occurring in the tissues of animals maintained on a diet containing the maximum anticipated residue (equivalent to 0.02 mg/kg bw/day using an average beef cattle bodyweight of 100 kg and equivalent to 0.5 mg/kg feed using an average feed consumption of 3.5 kg) resulting from early season application of clofentezine, a group of dairy calves were fed for a period of 28 days during which clofentezine was administered daily by capsule in UK, 1986 (Peatman, M.H., Snowdon, P.J., 1986, R-12734). Four animals were maintained in a treatment group dosed with clofentezine at 0.02 mg/kg bw/day, with a further two animals forming a control group. At the termination of the live phase of the study, selected tissues from the animals (liver and kidney only) were frozen and retained for residue analysis. Tissues were analysed for total clofentezine-derived residues by acid hydrolysis of all components to 2-chlorobenzoic acid (Table 69). The limit of determination was 0.05 mg/kg equivalent clofentezine.

Table 69. Clofentezine derived residues in animal tissue from dairy calves dosed with clofentezine at 0.02 mg/kg bw/day, for 28 consecutive days

Treatment group (mg/kg bw/day)	Mean total clofentezine derived residue level (mg/kg) [a]							
	Liver				Kidney			
	Rep I	II	III	IV	I	II	III	IV
Control [b]	n.d.	n.d.			n.d.	0.025		
0.02	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05

a - measured as 2-chlorobenzoic acid but expressed in terms of equivalent clofentezine by multiplication with molecular weight factor of 1.936.

b - two replicates only.

Poultry

In a 28-day feeding study laying hens (*Gallus gallus domesticus*) were dosed with clofentezine incorporated into a daily diet in UK, 1986 (Peatman, M.H., Snowdon, P.J., 1987, R-12735). Ten birds were maintained in each of five treatment groups fed on diet containing clofentezine at levels of 0, 0.05, 0.15, 0.50 and 6.0 mg/kg. Egg samples were taken daily during the study and kept frozen. At the end of the live phase of the study, selected tissues from the birds (liver, kidney, muscle, abdominal fat and skin plus subcutaneous fat) were pooled from the 10 birds in each treatment group. Muscle samples, however, were pooled from only five of the birds, the remainder being retained as separate samples. Tissues and eggs were analysed for total clofentezine-derived residues by acid hydrolysis of all components to 2-chlorobenzoic acid (Table 70 and 71). The limit of determination was 0.05 mg/kg equivalent clofentezine.

Table 70. Clofentezine derived residues in eggs expressed as clofentezine

Time (days)	Feeding level	Residues in eggs (mg/kg) [b]		
		control	0.5 ppm	6.0 ppm
1		0.014	--	n.d.
7		--	n.d.	< 0.05
14		n.d. [a]	n.d.	< 0.05
21		--	n.d.	< 0.05
28		--	--	< 0.05
29		0.03	< 0.05	0.06

a - n.d. = not detected

b - measured as 2-chlorobenzoic acid but expressed in terms of equivalent clofentezine by multiplication with molecular weight factor of 1.936.

Table 71. Clofentezine derived residues in tissues expressed as clofentezine

Tissue	Feeding level	Residues (mg/kg) [b]			
		0.05 ppm	0.15 ppm	0.5 ppm	6.0 ppm
Liver		< 0.05	< 0.05	< 0.05	0.08
Kidney		n.d. [a]	n.d.	n.d.	0.06
Muscle		n.d.	n.d.	n.d.	< 0.05
Abdominal fat		n.d.	n.d.	n.d.	0.13
Subcutaneous fat + skin		n.d.	< 0.05	< 0.05	0.09

a - n.d. = not detected

b - measured as 2-chlorobenzoic acid but expressed in terms of equivalent clofentezine by multiplication with molecular weight factor of 1.936.

RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

An internet search was conducted on 27.09.2006 for residue monitoring of clofentezine. Several monitoring reports in EU and EU member states as well as the USA were found. However, none of them shows assessments of clofentezine.

In the EU monitoring project, for example, clofentezine is not listed in 1997 – 2003 (see http://ec.europa.eu/food/fvo/specialreports/pesticides_index_en.htm). The same is the case for the German Monitoring program of the last years (see http://www.bvl.bund.de/cln_027/nn_491394/DE/01__Lebensmittel/01__Sicherheit__Kontrollen/03__Monitoring/Monitoring__node.html__nnn=true) and for the USA monitoring program in 2000 – 2002 (see http://www.ams.usda.gov/Science/pdp/Download.htm).

However, the information found shows that clofentezine was rarely found in products of plant origin. Altogether, out of a total of 22652 samples only 52 fruit or in fruiting vegetables samples (0.3%) contained residues at or above the 0.01 mg/kg reporting limit. (Table 72).

Table 72. Summary of available monitoring data for clofentezine in plant products

Country	Year	Commodity	Number of samples	Samples above reporting limit	Maximum values detected (mg/kg)	Detection limit / reporting level (mg/kg)	Reference
Estonia	2000	Fruit, vegetables	124	0		0.01	[1]
Estonia	2002	Fruit, vegetables	59	0		0.01	[2]
Estonia	2004	Fruit, vegetables	75	0		0.01	[3]
Netherlands	1999	Plant products	1459	0		0.01	[4]
Netherlands	2000	Fruit, vegetables	2491	1		0.01	[5]
		Strawberry	120	1	0.13	0.01	
Netherlands	2001	Fruit, vegetables	2627	0		0.01	[6]
Netherlands	2002	Fruit, vegetables	3203	0		0.1	[7]
		Cereals	50	0		0.1	

Country	Year	Commodity	Number of samples	Samples above reporting limit	Maximum values detected (mg/kg)	Detection limit / reporting level (mg/kg)	Reference
Netherlands	2003	Fruit, vegetables	1051	3		0.01	[8]
			3196	2		0.1	
		Cereals	56	0		0.1	
		Grape	265	2	0.04	0.01	
		Strawberry	153	1	0.16	0.01	
		Currant	8	1	0.13	0.01	
		Passion fruit	12	1	0.02	0.01	
Netherlands	2004	Fruit, vegetables	2765	8		0.01	[9]
		Cereals	51	0		0.01	
		Strawberry	153	7	0.17	0.01	
		Tomato	120	1	0.01	0.01	
Netherlands	2005	Lemon	16	1	0.03	0.01	[10]
		Grape	135	3	0.15	0.01	
		Strawberry	127	13	0.51	0.01	
		Pepper	107	1	0.01	0.01	
		Herbs	78	1	0.02	0.01	
Sweden	2000	Fruit, vegetables	84	0		0.1	[11]
Sweden	2001	Fruit, vegetables	30	0		0.1	[12]
Sweden	2002	Fruit, vegetables	15	0		0.1	[13]
Sweden	2003	Fruit, vegetables	1734	1 [a]	0.011	0.01	[14]
Sweden	2004	Fruit, vegetables	1736	5	n.d.	0.01	[15]
USA	2003	Pear juice	66	0		0.006	[16]
		Pear	54	0		0.007	
USA	2004	Apples	216	0		0.007	[17]
		Pears	216	2	0.011	0.007	

a - in lemon

APPRAISAL – RESIDUE AND ANALYTICAL ASPECTS

Clofentezine, an acaricide first evaluated by the JMPR in 1986 and re-evaluated for residues several times up to 1992. A toxicological review was conducted in 2005, when an ADI of 0-0.02 mg/kg bw was established. The 2005 JMPR concluded that an ARfD was not necessary. At the 37th session of the CCPR, clofentezine was scheduled for Periodic Re-evaluation of residues by the 2007 JMPR.

The manufacturer supplied information on identity; metabolism and environmental fate; residue analysis; use patterns; residues resulting from supervised trials on citrus, pome fruits, stone fruits, grapes, strawberries, currants, melons, tree nuts, tomatoes and cucumbers; and the fate of residues on apple, peach, almond and animal tissues during storage and orange, apple, grape and strawberries in processing. GAP information and enforcement methods were supplied by the manufacturer and the governments of the Netherlands and Australia.

Animal metabolism

The Meeting received animal metabolism studies with clofentezine in lactating cows, goats and laying hens. Clofentezine [^{14}C] labelled in the tetrazine ring was used in the animal metabolism studies.

The metabolism of clofentezine in rat, mouse, rabbit, calf and cow, dog, baboon and hen was qualitatively similar (details on laboratory animal metabolism are given in the toxicology report), with hydroxylation of the phenyl ring and/or replacement of chlorine with a methylthio group being the 2 major pathways.

A lactating cow was orally dosed with [^{14}C] labelled clofentezine for 5 consecutive days at about 0.27 mg/kg bw per day. Residues in milk reached a plateau level of approximately 0.007 mg/L on the second day. Residues were highest in bile (1.1 mg/kg) and liver (0.09 mg/kg). Heart, muscle and fat contained residues less than 0.01 mg/kg.

A lactating cow was orally administered with [^{14}C] labelled clofentezine for 3 consecutive days at an exaggerated rate (2.2 mg/kg bw per day) in order to produce quantifiable residues. Levels of radioactivity in milk were shown to plateau at a level of 0.17 mg/kg clofentezine equivalents on day 3 after treatment. The major component of the residue in milk was 4-OH clofentezine (75% TRR, total radioactive residues). The [^{14}C] residue was higher in the liver (0.76 mg/kg) than in other tissues, of which at least 67% was identified as 4-OH clofentezine. In renal fat, 90% of the TRR (0.24 mg equivalents/kg) was confirmed to be 4-OH clofentezine. Residues in kidney were also found to be composed predominantly of 4-OH clofentezine (83% TRR, 0.30 mg equivalents/kg). The remaining components appeared to be hydrolysis products of 4-OH clofentezine.

A lactating goat was given a single oral dose (0.63 mg/kg bw per day) of [^{14}C]clofentezine. The results showed [^{14}C] in all tissues at levels below 0.05 mg/kg, with all the [^{14}C] being excreted within 72 hours of dosing. Highest TRR was in goat milk at a level of 0.049 mg/L clofentezine equivalents at a time of 24 h after dosing.

Another study at an exaggerated rate (2.2 mg/kg bw per day) was undertaken for 7 consecutive days, in which a plateau for TRR in milk was reached at days 3 or 4 of the test, with a maximum residue of 0.2 mg/L being obtained. Over 95% of the TRR was confirmed as 4-OH clofentezine.

In summary, most of the [^{14}C]clofentezine fed to ruminants was excreted within 72 h. Liver was the target tissue and the major part of the residue was 4-OH clofentezine. There are no qualitative differences in the comparative metabolism studies of rodents (rats) and ruminants (goats and cattle).

Laying hens were administered [^{14}C]clofentezine orally for 3 consecutive days at a dose level of 17 mg clofentezine/kg bodyweight/day. By far the greatest [^{14}C] residue was found in fat (3.0 mg/kg equivalents). The majority of each daily dose of clofentezine (71 – 79%) was excreted by hens during the subsequent 24 h period. The majority of the identified component of the residue found in all tissue samples, was the parent clofentezine (fat: 70%, muscle: 34%, unlaid developing eggs: 32% and skin: 7.0%), with varying quantities of both 3 and 4-OH clofentezine. The remaining residue most likely consisted of conjugates of the 3 and 4-OH clofentezine metabolites.

Plant metabolism

The Meeting received plant metabolism studies with clofentezine on lemon, apple, peach and grapes.

In plants, parent clofentezine was the major component of the residue at shorter and longer intervals with lower and higher application rates. Levels of metabolites were usually much lower than parent clofentezine. Residues were mostly found as a surface residue.

The metabolism of [^{14}C]clofentezine (applied at 0.3 kg ai/ha) on lemon leaves was studied. Parent clofentezine was the main residue component and 2-chlorobenzonitrile was also present, at levels of 88% TRR and 8.1% TRR 25 days post-treatment, and 77% TRR and 6.8% TRR 103 days post-treatment, respectively.

The metabolism of [^{14}C]clofentezine in apple foliage was studied following application at a nominal dosage of 0.5 kg ai/ha. The main component of the residue found 25 days after treatment was parent clofentezine present amounting to 87% of TRR. Levels of parent clofentezine decreased over time to a level of 66% of TRR, with an increase of fibre bound residue. The metabolite NC 22505 (3, 6-bis(2-chlorophenyl)-1,2-dehydro -1,2,4,5-tetrazine) was present 10 and 100 days post-treatment but was not found at intermediate time points. Other single metabolites appeared in concentrations less than 1% of TRR.

The metabolism of [^{14}C]clofentezine was investigated in apples treated at a field spray concentration of 0.03 kg ai/hL and the exaggerated spray concentration of 0.76 kg ai/hL. Residues in mature apple fruit 72 days post-treatment consisted predominantly of parent clofentezine and peel fibre bound residues. However, both residues were at levels of 0.012 mg/kg or less at a rate of 0.03 kg ai/hL. At the exaggerated spray concentration, the same components were present, however the level of the parent clofentezine was much higher, i.e., 82% of TRR (0.81 mg/kg).

Further investigation was made into the components of the fibre bound residue, with apples being treated at spray concentrations of 0.06 kg ai/hL and 0.48 kg ai/hL. Samples were taken at 25 and 64 days post-treatment. Only limited quantities (approximately 0.01 mg/kg) of fibre bound radioactivity was recovered in this trial. Base and enzyme hydrolysis revealed that approximately 50% of bound residue was likely to be unchanged clofentezine, with the remainder consisting of 2-chlorobenzoic acid, 2-chlorobenzylacohol and 2-chlorobenzaldehyde.

Peach trees in a glasshouse were treated with [^{14}C]clofentezine at spray concentrations of 0.01 and 0.1 kg ai/hL and peaches were harvested for analysis 62 days post-treatment. Following the application at 0.01 kg ai/hL the overall TRR was only 0.047 mg/kg, of which 0.036 mg/kg was parent clofentezine. At the higher treatment rate, the TRR was 0.70 mg/kg consisting of 0.63 mg/kg parent clofentezine and 0.038 mg/kg 2-chlorobenzonitrile.

Grape vines were treated in a glasshouse with [^{14}C]clofentezine at spray concentrations of 0.01 and 0.1 kg ai/hL (equivalent to application rates of 0.1 and 1.0 kg ai/ha). Grape samples were collected for analysis 25 and 46 days post-treatment. At 25 days post-treatment, the total radioactive residue found at the 0.1 kg ai/ha rate was 0.38 mg/kg and 2.5 mg/kg at the 1.0 kg ai/ha rate. At the lower rate, the majority of the residue was found to be parent clofentezine (0.29 mg/kg) followed by 2-chlorobenzonitrile (0.04 mg/kg), the remainder of the residue comprised of polar materials (< 0.01 mg/kg). At 46 DAT the overall residue levels were much lower (0.11 mg/kg at 0.1 kg ai/ha and 0.45 mg/kg at 1.0 kg ai/ha), and the parent clofentezine remained the prevalent component (69% of TRR or 0.31 mg/kg).

Environmental fate in soil

The Meeting received information on the environmental fate of clofentezine in soil, including studies on aerobic soil metabolism, field dissipation and crop rotational studies.

The environmental fate of clofentezine was investigated in a number of laboratory studies using either unlabelled or [^{14}C] tetrazine ring labelled material under aerobic conditions for various durations. The degradation rates were not strongly affected by soil organic carbon content, but greatly influenced by the soil pH, with the faster degradation at higher soil pH. The aerobic soil metabolism half-lives for clofentezine ranged from approximately 2 to 12 weeks. After one year, in the loamy sand, clay and clay loam, approximately 56%, 38% and 25% of the applied radioactivity respectively had been mineralized to [^{14}C]O_2, and 30 – 40% of the initial dose was extractable residue in the loamy sand, clay and clay loam respectively.

During the study period, the maximum concentrations of the major metabolites, expressed as the percentage of the initial dose, were: 13% of AEC 593600 [2-chlorobenzoic (2-chlorobenzylidene) hydrazide], 1.6% of AEC 512898 [N, N'-bis-(2-chlorobenzoyl)-hydrazine], 0.8% of AEF 092117 [2-chlorobenzamide] and 6.2% of AEC 500233 [2-chlorobenzoic acid].

Very little of the applied clofentezine moved below the top 15 cm of the soil during field dissipation trials of up to 8 months' duration in several different soils. Clofentezine concentrations declined to half of their initial values within 14 days to approximately 6 months. In orchard soil residue decline trials, quantifiable residues of clofentezine were mostly detected in the top soil layer and declined below the limit of determination within 60 days.

The low water solubility and relatively high octanol/water partition coefficient of clofentezine lessen the uptake of clofentezine residues from soil into following crops. A crop uptake study with orange and apple trees grown under glasshouse conditions indicated that the potential for uptake of clofentezine residues from the previously treated soil was low.

Methods of analysis

The Meeting received analytical methods descriptions and validation data for residues of clofentezine in a number of crops, and residues of clofentezine or 4-OH clofentezine or total residues of all compounds containing the 2-chlorobenzoyl moiety in animal tissues, milk and eggs.

Methods rely on HPLC-UV, HPLC-DAD, GC-ECD and GC-MSD for analysis of clofentezine or 4-OH clofentezine and all compounds containing the 2-chlorobenzoyl moiety in the various matrices. Several multi-residue methods with HPLC-DAD and GC-MSD were suitable for enforcement for plant and animal commodities (LOQ values 0.01 – 0.05 mg/kg).

In summary, numerous recovery data on a wide range of substrates showed that the methods for data collection and enforcement were valid over the relevant concentration ranges.

Stability of residues in stored analytical samples

The Meeting received information on the freezer storage stability of residues of clofentezine in apple, peach, almond (hulls and nutmeats), muscle, liver, fat and milk.

Residues were stable in apple, almond/nutmeat and peach samples for a period of at least one year when stored frozen.

After 6 months storage, the mean percentage of clofentezine fortified had fallen to 38% (muscle), 72% (liver), 50% peritoneal fat), and 50% (milk). The percentage of clofentezine-derived residues (all metabolites containing the 2-chlorobenzoyl moiety) determined by derivation to 2-chlorobenzoic acid was more than 90% of the original residue in muscle, liver and fat, and approximately 84% in milk after 15 months storage. Parent clofentezine is relatively unstable in products of animal origin, but the total residue of all metabolites containing the 2-chlorobenzoyl moiety was stable in animal products for at least 15 months.

Definition of the residue

The main residues in fruit crops were the parent clofentezine, and metabolite 2-chlorobenzonitrile. The levels of 2-chlorobenzonitrile found were < 0.05 mg/kg, which was approximately a tenth of those of the parent residue. Other metabolites identified were present only at low levels and these metabolites were not considered to be of toxicological significance. Therefore the parent compound is only included in the residue definition for plant matrices.

The metabolism data submitted for clofentezine in animal products showed the vast majority of the residue in cattle and goat tissues is 4-OH clofentezine. Poultry studies however, showed more significant quantities of parent clofentezine, in addition to 3 and 4-OH clofentezine. Quantities of 3 and 4-OH clofentezine were not separated in the poultry study, but quantities of 3-OH clofentezine are much smaller than the combined totals of parent and 4-OH clofentezine.

In the cow metabolism study, the TRRs in subcutaneous fat and muscle were about 0.02 mg/kg, but TRR in renal fat was about 16 times as high as that in muscle. The main component of the residue in poultry commodities is the parent clofentezine and the TRR in fat was approximately 22 times higher than in the muscle. Based on the above results and the octanol-water partition coefficient of clofentezine (log P_{OW}=4.1), clofentezine is considered as fat-soluble.

Based on the available comparative animal and plant metabolism studies, the Meeting recommended a residue definition for clofentezine as follows:

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

Definition of the residue (for compliance with the MRL and estimation of dietary intake) for animal commodities: *sum of clofentezine, and* a*ll metabolites containing the 2-chlorobenzoyl moiety, expressed as clofentezine.*

The Meeting decided that residue is fat-soluble.

Results of supervised residue trials on crops

The Meeting received supervised trial data for clofentezine uses on orange, lemon, mandarin, apple, pear, peach, apricot, cherry, nectarine, plum, grapes, strawberry, currant, gherkin, cucumber, melon, tomato, walnut and almond. Residue data were also provided on almond hulls.

Labels (or translations of labels) were available to the Meeting from Argentina, Australia, Belgium, Canada, France, Germany, Greece, Italy, the Netherlands, Portugal, South Africa, Spain, Switzerland, UK, and USA describing the relevant GAP for evaluation of clofentezine.

Citrus fruits

Supervised trials were conducted on orange trees in Greece (citrus GAP 0.015 kg ai/hL, one application, 30 day PHI), Italy (citrus GAP 0.015 – 0.02 kg/hL, one application, 30 day PHI) and Spain (citrus GAP 0.005 – 0.01 kg/hL, 21 day PHI) in 1984, 1990 and 2001.

In two orange trials from Greece with application conditions in line with GAP, clofentezine residues were 0.10 and 0.18 mg/kg, with residues in flesh at 0.02 and 0.03 mg/kg.

In six orange trials from Spain and two trials from Italy with application conditions matching Spanish GAP, clofentezine residues were: 0.06, 0.07, 0.09(3), 0.10, 0.12 and 0.14 mg/kg, with some residues in flesh at < 0.01, 0.01, 0.02(3) and 0.03 mg/kg.

In one trial on lemons from Greece with application conditions in line with GAP, clofentezine residue was 0.15 mg/kg, with residue in flesh at 0.03 mg/kg.

In one trial on lemons from Italy matching Spanish GAP, clofentezine residue in lemon was 0.09 mg/kg, with residue in flesh at 0.02 mg/kg.

In one trial on tangerines from Italy matching Spanish GAP, clofentezine residue was 0.24 mg/kg, with residue in flesh at 0.03 mg/kg.

In six mandarin trials from Spain with application conditions in line with GAP, clofentezine residues were 0.08(3), 0.15, 0.17 and 0.18 mg/kg, with residues in flesh for two trials at 0.02 and 0.17 mg/kg.

The Meeting noted that the residue data populations for orange, lemon, tangerine and mandarin were from similar populations and can be combined. The residues in ranked order on citrus fruits were: 0.06, 0.07, 0.08(3), 0.09(4), <u>0.10</u> (2), 0.12, 0.14, 0.15(2), 0.17, 0.18(2) and 0.24 mg/kg (n=19). A similar situation exists for the flesh and the ranked order of concentrations in flesh was: < 0.01, 0.01, <u>0.02</u>(5), 0.03(3) and 0.17 mg/kg (n=11).

The Meeting estimated a maximum residue level for citrus fruits and an STMR value for flesh of 0.5 and 0.02 mg/kg respectively. The Meeting also estimated an STMR value for clofentezine in whole citrus fruits of 0.10 mg/kg (for estimating STMR-R value in orange juice). The recommendation for a maximum residue level of 0.5 mg/kg for citrus fruits confirms the previous recommendation of 0.5 mg/kg.

Pome fruits

Supervised trials were conducted on apple trees in Australia (pome fruit GAP 0.015 kg/hL, one application, 21 day PHI), Canada (GAP 0.15 – 0.30 kg ai/ha, one application, 45 day PHI), UK (GAP 0.2 kg ai/ha, one application, 28 day PHI), France (GAP 0.02 kg ai/hL, one application, 42 day PHI), Greece (GAP 0.015 kg ai/hL, one application, 45 day PHI), Germany (pome fruit GAP 0.02 kg ai/hL, one application per year, 35 day PHI), Belgium (GAP 0.13 kg ai/ha, one application, no PHI), South Africa (GAP 0.02 kg ai/hL, 30 day PHI), USA (GAP 0.12 – 0.24 kg ai/ha, one application, 45 day PHI) annually from 1980 to 1987 and then 1992 and 1993.

In one trial from Canada with application conditions in line with GAP, the residue of clofentezine found in apple was 0.04 mg/kg.

In fifteen apple trials from France with application conditions in line with GAP, clofentezine residues were < 0.01(3), 0.01, 0.03, 0.05(2), 0.06, 0.07(3), 0.08, 0.10, 0.11 and 0.22 mg/kg.

In one apple trial from Greece with application conditions in line with GAP, clofentezine residue was 0.04 mg/kg.

In two apple trials from South Africa with application conditions in line with GAP, clofentezine residues were 0.09(2) mg/kg.

In sixteen apple trials from USA with application conditions in line with GAP, clofentezine residues were 0.01(2), 0.02(3), 0.04(3), 0.05(3), 0.07(2), 0.11 and 0.12(2) mg/kg.

In five apple trials from Germany with application conditions in line with GAP, clofentezine residues were 0.02, 0.03, 0.04, 0.05 and 0.09 mg/kg.

In four apple trials from UK with application conditions matching French GAP, clofentezine residues were 0.10, 0.16, 0.17 and 0.24 mg/kg.

The Meeting noted that the residues in apples from the above countries were from similar populations and could be combined. The ranked order of residues were: < 0.01(3), 0.01(3), 0.02(4), 0.03(2), 0.04(6), 0.05(6), 0.06, 0.07(5), 0.08, 0.09(3), 0.10(2), 0.11(2), 0.12(2), 0.16, 0.17, 0.22 and 0.24 mg/kg (n=44).

Supervised trials were conducted on pear trees in Australia (pome fruit GAP 0.015 kg/hL, one application, 21 day PHI), Canada (GAP 0.15-0.30 kg ai/ha, one application, 21 day PHI), Italy (GAP 0.02 kg ai/hL, one application, 30 day PHI), South Africa (GAP 0.02 kg ai/hL, 30 day PHI), USA (GAP 0.12 – 0.24 kg ai/ha, one application, 21 day PHI) in 1982, 1984, 1985, 1986, 1987, 1988 and 1993.

In one pear trial from Australia with application conditions in line with GAP, clofentezine residue was 0.02 mg/kg.

In four pear trials from Canada with application conditions in line with GAP, clofentezine residues were 0.12, 0.13, 0.14 and 0.19 mg/kg.

In one pear trial from Italy with application conditions in line with GAP, clofentezine residue was 0.04 mg/kg.

In two pear trials from South Africa with application conditions in line with GAP, clofentezine residues were 0.20 and 0.22 mg/kg.

In thirteen pear trials from USA with application conditions in line with GAP, clofentezine residues were 0.04, 0.05, 0.06(3), 0.08(2), 0.09(3), 0.15, 0.18 and 0.20 mg/kg.

The Meeting noted that the residues in pears from the above countries were from similar populations and could be combined. The residues in ranked order were: 0.02, 0.04(2), 0.05, 0.06(3), 0.08(2), 0.09(3), 0.12, 0.13, 0.14, 0.15, 0.18, 0.19, 0.20(2) and 0.22 mg/kg.

The Mann-Whitney test indicated the residue data populations for apple and pear were not significantly different and could be combined to support a pome fruit MRL. The ranked order of concentrations, median underlined, were < 0.01(3), 0.01(3),0.02(5), 0.03(2), 0.04(8), <u>0.05</u>(7), 0.06(4), 0.07(5), 0.08(3), 0.09(6), 0.10(2), 0.11(2), 0.12(3), 0.13, 0.14, 0.15, 0.16, 0.17, 0.18, 0.19, 0.20(2), 0.22(2) and 0.24 mg/kg (n=65). The Meeting estimated a maximum residue level and an STMR value for clofentezine in pome fruits of 0.5 and 0.05 mg/kg respectively. The recommendation for a maximum residue level of 0.5 mg/kg for pome fruits confirms the previous recommendation.

Stone fruits

Supervised trials were conducted on peach in Australia (stone fruit GAP 0.015 kg ai/hL, one application, 21 day PHI), in Italy (no GAP), and USA (GAP 0.059 – 0.24 kg ai/ha, one application, 21 day PHI) in 1984, 1986, 1987, 1988, 1991, 1993 and 2002.

In two peach trials from Australia with application conditions in line with GAP, clofentezine residues in flesh were 0.06 and 0.13 mg/kg.

In eighteen peach trials conducted at GAP in USA, clofentezine residues were 0.02, 0.03, 0.04, 0.05, 0.06, 0.08(2), 0.09(2), 0.11, 0.12, 0.13, 0.14(2), 0.18(2), 0.24 and 0.35 mg/kg.

The Meeting noted that the residue data populations from Australia and USA for peach were from similar populations and should be combined. The residues in ranked order were: 0.02, 0.03, 0.04, 0.05, 0.06(2), 0.08(2), 0.09(2), 0.11, 0.12, 0.13(2), 0.14(2), 0.18(2), 0.24 and 0.35 mg/kg.

Supervised trials were conducted on apricot in Greece (GAP 0.015 kg ai/hL, one application, 45 day PHI) and USA (GAP 0.059-0.24 kg ai/ha, one application, 21-day PHI) in 1987, 1989 and 1993.

In one apricot trial conducted at GAP in Greece, clofentezine residue was 0.16 mg/kg.

In two apricot trials conducted at GAP in USA, clofentezine residues were 0.13 and 0.14 mg/kg.

Combined residues from Greek and USA apricot trials were 0.13, 0.14 and 0.16 mg/kg.

Supervised trials were conducted on cherries in UK (0.2 kg ai/ha, one application, 56-day PHI) in 1983.

In two cherry trials conducted in accordance with British GAP, clofentezine residues were 0.01 and 0.02 mg/kg.

Supervised trials were conducted on nectarines in USA (GAP 0.059 – 0.24 kg ai/ha, one application, 21 day PHI) in 1986 and 1987.

In three nectarine trials conducted in accordance with USA GAP, clofentezine residues were 0.04, 0.11 and 0.17 mg/kg.

Supervised trials were conducted on plums in Germany (GAP 0.02 kg ai/hL, one application limited by growth stage of the crop, no PHI) in 1985 and 1986. The last application was made during fruit development, which is not in accordance with German GAP, as a result the residue data could not be used for the evaluation.

The Meeting combined the residue data in peach, apricot, cherry and nectarine. The residues in ranked order were: 0.01, 0.02(2), 0.03, 0.04(2), 0.05, 0.06(2), 0.08(2), 0.09(2), 0.11(2), 0.12, 0.13(3), 0.14(3), 0.16, 0.17, 0.18(2), 0.24 and 0.35 mg/kg (n=28).

The Meeting estimated a maximum residue level and an STMR value for clofentezine in stone fruits of 0.5 and 0.11 mg/kg respectively. The recommendation for a maximum residue level of 0.5 mg/kg for stone fruits replaces the previous recommendation of 0.2 mg/kg.

Grapes

Supervised trials were conducted on grapes in France (GAP 0.2 kg ai/ha, one application, 42-day PHI), in Greece (no GAP, use that of Spain), in Germany (GAP 0.06 – 0.24 kg ai/ha, one application, 35 day PHI), in Italy (GAP 0.01-0.015 kg ai/hL, one application, 30 day PHI) and in Spain (GAP 0.01 – 0.03 kg ai/hL, 30 day PHI) in 1984, 1985, 1986, 1987, 1991, 1992 and 2001.

In twelve trials conducted in line with German GAP, clofentezine residues were 0.09, 0.12, 0.14, 0.20, 0.22, 0.23, 0.39, 0.61, 0.69, 0.73, 0.79 and 0.89 mg/kg.

In one trial conducted in accordance with Italian GAP, clofentezine residue was 0.35 mg/kg.

In two trials conducted in accordance with Spanish GAP, clofentezine residues were 0.25 and 0.27 mg/kg.

In one trial in Greece conducted in accordance with Spanish GAP, clofentezine residue was 0.67 mg/kg.

In one trial in Italy conducted in accordance with Spanish GAP, clofentezine residue was 0.12 mg/kg.

In two trials in France conducted in accordance with Spanish GAP, clofentezine residues were 0.09 and 0.11 mg/kg.

The Meeting noted that the residues in grapes were from similar populations and could be combined. The residues in ranked order were: 0.09(2), 0.11, 0.12(2), 0.14, 0.20, 0.22, 0.23, 0.25, 0.27, 0.35, 0.39, 0.61, 0.67, 0.69, 0.73, 0.79 and 0.89 mg/kg (n=19).

The Meeting estimated a maximum residue level and an STMR value for clofentezine in grapes of 2 and 0.25 mg/kg respectively. The recommendation for a maximum residue level of 2 mg/kg for grapes replaces the previous recommendation of 1 mg/kg.

Strawberries

Supervised trials were conducted on strawberries in France (GAP 0.2 kg ai/ha, 3-day PHI), in Germany (GAP 0.3 kg ai/ha, one application, no PHI, growth-stage restriction), in the Netherlands (GAP 0.075 – 0.15 kg ai/ha, one to two applications, no PHI, growth-stage restriction) and in Spain (GAP 0.01 – 0.02 kg ai/hL, 3-day PHI) in 1989, 1990, 1992, 2000 and 2001.

In eight outdoor trials in France conducted in line with French GAP, clofentezine residues were 0.08(2), 0.09, 0.13, 0.19, 0.20 and 0.24(2) mg/kg.

In two outdoor trials in the Netherlands conducted in accordance with Dutch GAP, clofentezine residues were 0.08 and 0.13 mg/kg.

In nine outdoor trials in Spain conducted in accordance with Spanish GAP, clofentezine residues were 0.50, 0.56, 0.60, 0.70, 0.72, 0.73, 0.75, 0.81 and 1.10 mg/kg.

In five outdoor trials in Germany conducted in line with French GAP, clofentezine residues were 0.09(2), 0.16, 0.18 and 0.23 mg/kg.

The data populations from France and those from the Netherlands and Germany were not significantly different and should be combined. The data from Spain were significantly different from those from France, Germany and the Netherlands in strawberries on a Mann-Whitney test.

Based on data from Spain, the Meeting estimated a maximum residue level and an STMR value for clofentezine in strawberries of 2 and 0.72 mg/kg respectively. The recommendation for a maximum residue level of 2 mg/kg for strawberry confirms the previous recommendation of 2 mg/kg.

Black, red and white Currants

Four supervised trials were conducted on blackcurrants in France (maximum GAP: 0.2 kg ai/ha, 45 day PHI) in 2001. The residues in ranked order on currants were: < 0.04(3) and 0.09 mg/kg.

The Meeting agreed to extrapolate from blackcurrants to red and white currants and estimated a maximum residue level and an STMR value for clofentezine in currants of 0.2 and 0.04 mg/kg respectively. The recommendation for a maximum residue level of 0.2 mg/kg for currants replaces the previous recommendation of 0.05 mg/kg.

Cucurbits

Supervised trials were conducted on cucumbers in France (cucurbits GAP 0.2 kg ai/ha, 3-day PHI), in Greece (cucumber GAP 0.015 kg ai/hL, one application, 4 day PHI) and in Switzerland (cucumber GAP 0.02 kg ai/hL, one application, 14 day PHI) in 1985, 1987, 1988 and 1991.

In one trial in France conducted on cucumber in accordance with French GAP, clofentezine residue was 0.07 mg/kg.

In four trials in Greece conducted on cucumber in accordance with Greek GAP, clofentezine residues were 0.12(2), 0.14 and 0.16 mg/kg.

In one trial in Switzerland conducted on cucumber in accordance with French GAP, clofentezine residue was 0.13 mg/kg.

The Meeting noted that the residue data in cucumber were from similar populations and could be combined. The residues in ranked order were: 0.07, 0.12(2), 0.13, 0.14 and 0.16 mg/kg (n=6).

The Meeting estimated a maximum residue level and an STMR value for clofentezine in cucumber of 0.5and 0.125 mg/kg respectively. The recommendation for a maximum residue level of 0.5 mg/kg for cucumber replaces the previous recommendation of 1 mg/kg.

Melons

Supervised trials were conducted on melons in France (GAP 0.2 kg ai/ha, 3-day PHI), in Greece (no GAP, use that of France), in Italy (GAP 0.015 – 0.02 kg ai/hL, one application, 15-day PHI), in Portugal (no GAP, use that of France) and in Spain (GAP 0.01–0.02 kg ai/hL, 3-day PHI) in 1999 and 2000.

In two trials in France conducted in line with French GAP, clofentezine residues were < 0.05 and 0.05 mg/kg, with no detectable residue in pulp.

In one trial in Greece conducted in accordance with Greek GAP, clofentezine residue was 0.03 mg/kg, with no detectable residue in pulp.

In two trials in Portugal conducted in accordance with Portuguese GAP, clofentezine residues were < 0.05 mg/kg, with no detectable residue in pulp.

In two trials in Spain conducted in accordance with Spanish GAP, clofentezine residues were < 0.01 and < 0.05 mg/kg, with no detectable residue in pulp.

In two trials in Italy conducted in accordance with French GAP, clofentezine residues were 0.03 and 0.06 mg/kg, with no detectable residue in pulp.

The Meeting noted that the residues in melons were from similar populations and could be combined. The residues in ranked order were: < 0.01, 0.03(2), < 0.05(4), 0.05 and 0.06 mg/kg (n=9). The residues in all pulp samples were below the limit of quantification (n=9).

The Meeting estimated a maximum residue level of 0.1 mg/kg. Taking into account that the parent compound practically did not translocate in plants, the Meeting estimated an STMR value of 0 mg/kg for clofentezine in melons.

Tomato

Supervised trials were conducted on tomato in France (no GAP, use that of the Netherlands), in Germany (no GAP, use that of the Netherlands), in Greece (no GAP, use that of the Netherlands), in Italy (GAP 0.02 – 0.03 kg ai/hL, one application, 15 day PHI), in the Netherlands (GAP 0.075 – 0.23 kg ai/ha, 1 – 2 applications, 3 day PHI) and in Spain (0.01 – 0.02 kg ai/hL, one application, 3 day PHI) in 1986, 1992, 1999, 2000 and 2005.

In seven trials in Italy conducted in accordance with Italian GAP, clofentezine residues were 0.01, 0.03, 0.05(2), 0.06, 0.07 and 0.10 mg/kg.

In seven glasshouse trials in the Netherlands conducted in accordance with Dutch GAP, clofentezine residues were < 0.05, 0.09, 0.10, 0.11, 0.12, 0.16 and 0.18 mg/kg.

In four trials in France conducted matching Dutch GAP, clofentezine residues were < 0.05, 0.05, 0.06 and 0.09 mg/kg.

In three trials in Germany conducted matching Dutch GAP, clofentezine residues were < 0.05, 0.06 and 0.11 mg/kg.

In one trial in Greece conducted matching Dutch GAP, clofentezine residue was < 0.05 mg/kg.

In one trial in Italy conducted matching Dutch GAP, clofentezine residue was < 0.05 mg/kg.

In one trial in Spain conducted matching Dutch GAP, clofentezine residue was 0.09 mg/kg.

The Meeting noted that the residues from France, Germany and the Netherlands, in line with Dutch GAP, were from similar populations and could be combined. The data populations from France, Germany and the Netherlands and from Greece, Italy and Spain were not from similar

populations based on the Mann-Whitney test, and could not be combined. The residues in ranked order based on France, Germany and the Netherlands were: < 0.05(3), 0.05, 0.06(2), 0.09(2), 0.10, 0.11(2), 0.12, 0.16 and 0.18 mg/kg (n=14).

The Meeting estimated a maximum residue level and an STMR value for clofentezine in tomato of 0.5 and 0.09 mg/kg respectively.

Tree nuts

Eight supervised trials were conducted on walnut in USA (maximum GAP: 0.24 kg ai/ha, one application, 30 day PHI) in 1987 and 1988. The ranked order of concentrations on walnut was: < 0.02(8) mg/kg.

Thirty four supervised trials were conducted on almond in USA (maximum GAP: 0.24 kg ai/ha, 30 day PHI) in 1985, 1987, 1993, 1998 and 2002. The ranked order of concentrations on almond was: < 0.01(9), < 0.02(3), < 0.05(13), 0.10(3), 0.20(2), and 0.30(4) mg/kg.

The residue data for walnut and almond were from similar populations and could be combined. The residues in ranked order on tree nuts were: < 0.01(9), < 0.02(11), < 0.05(13), 0.10(3), 0.20(2), and 0.30(4) mg/kg (n=42).

The Meeting estimated a maximum residue level and an STMR value for clofentezine in tree nuts of 0.5 and 0.05 mg/kg respectively.

Animal feedstuffs

Almond hull

In 34 supervised trials on almond compliant with US GAP, residues of clofentezine in almond hulls in rank order, median and highest residue underlined, were: 0.06, 0.10, 0.11, 0.12(2), 0.20(2), 0.30, 0.40(2), 0.50(2), 0.60(2), 0.70(2), 0.90, 0.91, 1.00, 1.10(2), 1.20, 1.40(2), 1.50(2), 1.60, 1.70, 1.80, 2.00, 2.20, 2.30, 2.50 and 2.70 mg/kg (fresh weight) (n=34).

The meeting estimated an STMR value of 0.91 mg/kg and a highest residue of 2.70 mg/kg for clofentezine in almond hulls (fresh weight).

Allowing for the standard 90% dry matter for almond hulls (*FAO Manual*, p. 147) the residues in almond hulls were: 0.07, 0.11, 0.12, 0.13(2), 0.22(2), 0.33, 0.44(2), 0.56(2), 0.67(2), 0.78(2), 1.00, 1.01, 1.11, 1.22(2), 1.33, 1.56(2), 1.67(2), 1.78, 1.89, 2.00, 2.22, 2.44, 2.56, 2.78 and 3.00 mg/kg. The Meeting estimated a maximum residue level of 5 mg/kg and an STMR of 1.01 mg/kg for almond hulls (dry weight). A highest residue level of 3.00 mg/kg was estimated for calculating the dietary burden of farm animals.

Fate of residues during processing

The Meeting received information on the fate of clofentezine residues during aqueous hydrolysis under conditions representing pasteurisation, baking, brewing, boiling and sterilisation. Information was also provided on the fate of clofentezine residues during the food processing of citrus, apples, grapes and strawberries.

Clofentezine was stable at pH 4 for 20 minutes at 90 °C with no degradation products formed, and moderately stable at pH 5 at 100 °C for 60 minutes. In this latter instance, clofentezine degraded slightly (by approximately 10%) to form metabolite 2-chlorobenzoic (2-chlorobenzylidene) hydrazide (AEC 593600). The parent clofentezine was rapidly hydrolysed at pH 6 and 120 °C and it was not detected after 20 minutes. The major reaction products as a percentage of the applied radioactivity were 2-chlorobenzoic (2-chlorobenzylidene) hydrazide (78%), 2-chlorobenzonitrile (4.9%) and 2-chlorobenzamide (17%).

The processing was carried out to produce apple sauce from samples spiked with clofentezine in the laboratory. The time of cooking and pasteurizing was approximately 15 minutes at a temperature of over 97 °C. The treated apples, the apple sauce and the processing by-products

(washed apple, apple cores, peeled apples, wash water), were analysed for the potential degradation products of clofentezine (AEC 593600, 2-chlorobenzonitrile and 2-chlorobenzamide), but none of the compounds was present in quantifiable concentration in any of the samples. One uncooked apple peel sample contained 0.02 mg/kg for 2-chlorobenzonitrile. The clofentezine residue in the pasteurized apple sauce was reduced from 0.40 mg/kg in the apple in one study, and from 0.41 mg/kg to < 0.02 mg/kg in three samples in a follow up test, where the three degradation products were not detectable. The Meeting noted that the 3 degradation products found in the above hydrolysis study, performed at pH 6 and 120 °C, were not present in apple sauce prepared following a normal processing procedure.

Processing studies for the conversion of oranges, apples, grapes and strawberries to various processed products were reported from Germany, Italy, Spain and USA. In most cases, the raw agricultural commodities had quantifiable field incurred clofentezine residue. Calculated processing factors and the mean or best estimate for the processing factors are summarized in the following table.

Raw agricultural commodity (RAC)	Processed commodity	Calculated processing factors. [a]	Median or best estimate
Orange	Juice	< 0.08, < 0.11, < 0.14, 0.14, < 0.17(3), < 0.20, < 0.25(2), < 0.33(2)	0.14
Apples	Wet pomace	< 0.50, 1.20, 1.50 (2), 2.00 (4), 2.11, 2.40, 3.00, 3.44, 5.50, 5.69, 5.79, 6.00	2.06
	Juice	0.016, 0.11, 0.20, < 0.5 (3)	0.11
Grapes	Raisins	0.22, 0.28, 0.64, < 0.67, 1.09, 1.12, 1.70, 2.33, 2.92	1.11
	Juice	nd	0
	Wet pomace	1.88, 1.89	1.89
	White wine making	< 0.042, < 0.50 (2)	< 0.042

a - 'Less-than' (<) values are derived from cases where residues were not detected in the processed commodity. The 'less-than' processing factor is then calculated from the LOQ of the analyte in the processed commodity and the residue in the raw agricultural commodity.

The processing factor for orange juice (0.14) was applied to the estimated STMR for orange (0.10 mg/kg) to produce STMR-P values for orange juice (0.014 mg/kg).

The processing factors for wet apple pomace (2.06) and apple juice (0.11) were applied to the estimated STMR for apple (0.05 mg/kg) to produce STMR-P values for wet apple pomace (0.103 mg/kg) and apple juice (0.0055 mg/kg).

The processing factors for grapes to raisins (1.11), grape juice (0), white wine (0.042) and wet pomace (1.89) were applied to the estimated STMR for grapes (0.25 mg/kg) to produce an STMR-P value for dried grapes (0.28 mg/kg), grape juice (0), white wine (0.011 mg/kg) and wet pomace (0.47 mg/kg). The processing factors for raisins (1.09) were applied to the grape residue data (highest value 0.89 mg/kg) to produce estimated highest values for dried grapes (0.99 mg/kg).

The Meeting estimated a maximum residue level for clofentezine in dried grapes of 2 mg/kg.

Residues in animal commodities

Farm animal feeding studies

The Meeting received feeding studies on lactating dairy cows, calves and laying hens, which provided information on likely residues appearing in animal tissues, milk and eggs from residues in the animals' diet.

Lactating Friesian cows were dosed with clofentezine at the equivalent of 10 (1), 30 (3×) and 100 (10×) ppm in the dry-weight diet using an average feed consumption of 20 kg for 28 consecutive days. Milk was collected twice daily for analysis. Animals were sacrificed at 1 or 3 days after the final dosing.

No residues (total clofentezine=clofentezine and metabolites hydrolysable to 2-chlorbenzoic acid and expressed as clofentezine) were detected in milk samples taken from the control and the 1× dose groups. From day 7, total residues between < 0.05 and 0.14 mg/kg were detected in the milk of cows from the 3× dose group. In the 10× dose group, residues occurred regularly from day 7 in the concentration range of 0.11 to 0.27 mg/kg.

The total residue was below the LOQ (0.05 mg/kg) in all tissue samples except liver from the treatment rate of 1×. No residue was detectable in heart, muscle and fat samples at any dose level. The average residues were present in liver (0.26, 1.15, 2.20 mg/kg) and in kidney (< 0.05, 0.18, 0.40 mg/kg) at the dose rates of 1×, 3× and 10×, respectively.

Calves were dosed at a rate equivalent to 0.5 ppm feed (dry weight) using an average feed consumption of 3.5 kg for 28 days. Animals were sacrificed at 19 h after the final dosing. The total clofentezine residues were below the limit of quantification (0.05 mg/kg clofentezine equivalents) in the liver and kidney samples.

Laying hens were fed clofentezine at 0.05, 0.15, 0.50 and 6.0 ppm in the diet (dry weight) for 28 days. Egg samples were taken daily during the study and kept frozen. Birds were sacrificed 1 day after the final dosing. Only one egg sample obtained from the highest dose rate group contained quantifiable residues (0.06 mg/kg). From the dose groups of 0.05, 0.15, 0.50 ppm, no residue was present above the LOQ (0.05 mg/kg) in any tissue samples. Quantifiable residues were only present at the exaggerated 6.0 ppm dosage rate: liver (0.08 mg/kg), kidney (0.06 mg/kg) and abdominal and subcutaneous fat (0.13, 0.09 mg/kg).

Livestock dietary burden

The Meeting estimated the dietary burden of clofentezine in farm animals on the basis of the diets listed in the Annex 6 of the 2006 JMPR Report. Calculation from highest residue 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.

Estimated maximum and mean dietary burdens of farm animals

Dietary burden calculations for beef cattle, dairy cattle and turkey are provided in Annex 6. The calculations were made according to the animal diets from USA, Canada, EU and Australia in the OECD Table (Annex 6 of the 2006 JMPR Report).

The calculations are then summarized and the highest dietary burdens (underlined) are selected for MRL and STMR estimates on animal commodities.

	Animal dietary burden, clofentezine, ppm of dry matter diet					
	USA-Canada		EU		Australia	
	max	mean	max	mean	max	mean
Beef cattle	0.35	0.15	0.05	0.05	<u>0.98</u> [a/]	<u>0.78</u> [b/]
Dairy cattle	0.33	0.13	0.03	0.03	<u>0.95</u> [c/]	<u>0.75</u> [d/]
Poultry - layer	0	0	0	0	0	0
Poultry - layer	0	0	0	0	0	0

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

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

c - Highest maximum dairy cattle dietary burden suitable for MRL estimates for milk.

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

The dietary burden for both beef and dairy cattle was 0.93 mg/kg, below the lowest feeding level (10 ppm in the feed). Therefore, the resulting residues in milk and cattle tissues were calculated by applying the respective transfer factors (transfer factor=residue level in tissue or milk ÷ residue level in feed) to the estimated dietary burden. In the feeding study the highest residue levels in tissues were used to calculate the highest likely mammal commodity residue levels and mean residue levels

in milk and tissues were used to estimate the mammal commodity STMRs. In the table below, dietary burdens and the corresponding estimated residues in brackets are indicated with Italic fonts.

	Feeding level (mg/kg) actual	Clofentezine residues, mg/kg									
		Milk		Muscle		Fat		Liver		Kidney	
		Highest	Mean	Highest	Mean	Highest	Mean	Highest	Mean	Highest	Mean
MRL beef cattle	*0.98*			*(< 0.005)*		*(< 0.005)*		*(0.031)*		*(< 0.005)*	
	10			< 0.05		< 0.05		0.33		< 0.05	
MRL dairy cow	*0.95*		*(< 0.005)*								
	10		< 0.05								
STMR beef cattle	*0.78*				*(< 0.004)*		*(< 0.004)*		*(0.019)*		*(< 0.004)*
	10				< 0.05		< 0.05		0.26		< 0.05
STMR dairy cow	*0.75*		*(< 0.004)*								
	10		< 0.05								

The dietary burden for laying hens was 0 mg/kg, therefore the table of calculation of MRLs and STMRs for poultry meat and eggs is not necessary.

The Meeting estimated maximum residue levels of 0.05 (*) mg/kg for mammalian meat (fat), mammalian edible offal, and milks to replace the present recommendations of 0.05 (*) mg/kg for cattle meat, 0.01 (*) mg/kg and 0.1 mg/kg for edible offal of cattle. The Meeting also estimated the following STMR values: muscle 0 mg/kg, fat 0 mg/kg, edible offal 0.05 mg/kg, and whole milk 0 mg/kg.

The Meeting estimated maximum residue levels of 0.05 (*) mg/kg for eggs, poultry meat (fat), and poultry edible offal, based on the limit of quantification for poultry commodities to confirm the present recommendation of 0.05 (*) mg/kg. Also estimated were STMRs of 0 mg/kg for eggs, meat, and edible offal of poultry.

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.

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

Definition of the residue (for compliance with MRL and estimation of dietary intake): *sum of clofentezine, and all metabolites containing the 2-chlorobenzoyl moiety, expressed as clofentezine.*

The residue is fat soluble.

Commodity		MRL, mg/kg		STMR/STMR-P mg/kg
CCN	Name	New	Previous	
AM 0660	Almond hulls	5		1.01
JF 0226	Apple juice			0.0055
AB 0226	Apple pomace, dry			0.26
MM 0812	Cattle meat	W [a]	0.05 *	
ML 0812	Cattle milk	W [a]	0.01 *	
MO 0812	Cattle, Edible offal of	W [a]	0.1	
FC 0001	Citrus fruits	0.5	0.5	0.10 (flesh 0.02)
VC 0424	Cucumber	0.5	1	0.125

Commodity		MRL, mg/kg		STMR/STMR-P mg/kg
CCN	Name	New	Previous	
FB 0021	Currants, Black, Red, White	0.2	0.05	0.04
MO 0105	Edible offal (mammalian)	0.05 *		0.05 *
PE 0112	Eggs	0.05 * [b]	0.05 *	0
JF 0269	Grape juice			0
FB 0269	Grapes	2	1	0.25
DF 0269	Grapes, dried (=currants, raisins and sultanas)	2		0.28
-	Grape pomace, wet			0.47
MM 0095	Meat from mammals other than marine mammals	0.05 * (fat)		0
VC 0046	Melons, expect watermelon	0.1		0
ML 0106	Milks	0.05 *		0
JF 0004	Orange juice			0.014
FP 0009	Pome fruit	0.5	0.5	0.05
PM 0110	Poultry meat	0.05 * (fat) [b]	0.05 *	0
PO 0111	Poultry, edible offal of	0.05 * [b]	0.05 *	0
FS 0012	Stone fruits	0.5	0.2	0.11
FB 0275	Strawberry	2	2	0.72
VO 0448	Tomato	0.5		0.09
TN 0085	Tree nuts	0.5		0.05
-	Wine			0.011

* - at or about the limit of quantification

W - The previous recommendation is withdrawn.

a - Replaced by a group MRL.

b - Residues are not expected because the dietary burden of poultry is zero.

DIETARY RISK ASSESSMENT

Long-term intake

The International Estimated Daily Intakes (IEDIs) of clofentezine, based on the STMRs estimated for sixteen commodities, were 0 – 3% of the maximum ADI of 0.02 mg/kg bw for the thirteen GEMS/Food regional diets (see Annex 3 of the Report of the 2007 JMPR). The Meeting concluded that the long-term intake of residues of clofentezine resulting from its uses that have been considered by JMPR is unlikely to present a public health concern.

Short-term intake

The 2005 JMPR decided that an ARfD was unnecessary. The Meeting therefore concluded that the short-term intake of clofentezine residues is unlikely to present a public health concern.

REFERENCES

Code	Author	Year	Title, Institute, Report reference
[1]	Anonymous	2001	Estonian national monitoring programme of pesticide residues 2000 – Pesticides in fruit and vegetables. Agricultural Research Centre, Saku, Estland http://www.agri.ee/toiduohutus/seire_1_old.html accessed on 27.09.2006.
[10]	Anonymous	2006	Report of pesticide residue monitoring results of the Netherlands for 2005. Food and Consumer Safety Authority, Netherlands http://www2.vwa.nl/CDL/files/15/1004/12467%20Monitoring_G_en_F.pdf accessed on 27.09.2006.
[11]	Andersson A	2001	The Swedish monitoring of pesticide residues in food of plant origin: 2000. National Food Administration, Sweden http://www.slv.se/upload/dokument/Rapporter/Bekampningsmedel/slvrapp16_2001/slvrapp16_

2001_pesticide_residues.pdf.

[12]	Andersson A	2002	The Swedish monitoring of pesticide residues in food of plant origin: 2001. National Food Administration, Sweden http://www.slv.se/upload/dokument/Rapporter/Bekampningsmedel/slvrapp15_2002/Rapport2001_bekampningsmedel.pdf.
[13]	Andersson A	2003	The Swedish monitoring of pesticide residues in food of plant origin: 2002. National Food Administration, Sweden http://www.slv.se/upload/dokument/Rapporter/Bekampningsmedel/2003/rapp_eng_2002_slutversion_a.pdf accessed on 27.09.2006.
[14]	Andersson A	2004	The Swedish monitoring of pesticide residues in food of plant origin: 2003. National Food Administration, Sweden http://www.slv.se/upload/dokument/Rapporter/Bekampningsmedel/2004/rapp_12_2004___%20eng.pdf accessed on 27.09.2006.
[15]	Andersson A	2005	The Swedish monitoring of pesticide residues in food of plant origin: 2004. National Food Administration, Sweden http://www.slv.se/upload/dokument/Rapporter/Bekampningsmedel/2005/2005_17eng_Livsmedelsverket.pdf accessed on 27.09.2006.
[16]	United States Department of Agriculture	2005	Pesticide Data program – Annual summary Calendar Year 2003. http://www.ams.usda.gov/Science/pdp/Summary2003.pdf accessed on 27.09.2006.
[17]	United States Department of Agriculture	2006	Pesticide Data program – Annual summary Calendar Year 2004. http://www.ams.usda.gov/Science/pdp/Summary2004.pdf accessed on 27.09.2006.
[2]	Toome	2003	Estonian national monitoring programme of pesticide residues 2002 – Pesticides in fruit and vegetables. Agricultural Research Centre, Saku, Estland www.agri.ee/toiduohutus/seire_1.html accessed on 27.09.2006.
[3]	Anonymous	2005	Estonian national monitoring programme of pesticide residues 2004 – Pesticides in fruit and vegetables. Agricultural Research Centre, Saku, Estland http://pmk.agri.ee/est/ettekanded/JSL_kogumik_2005.pdf accessed on 27.09.2006.
[4]	van der Schee	2000	Report of pesticide residue monitoring results of the Netherlands for 1999. Food and Consumer Safety Authority, Netherlands http://www2.vwa.nl/CDL/files/15/1004/10390%20rap99.pdf accessed on 27.09.2006.
[5]	van der Schee	2001	Report of pesticide residue monitoring results of the Netherlands for 2000. Food and Consumer Safety Authority, Netherlands http://www2. vwa.nl/CDL/files/15/1004/10389%20011025_residuen_bestrijdingsmiddelen_2001.pdf accessed on 27.09.2006.
[6]	van der Schee	2002	Report of pesticide residue monitoring results of the Netherlands for 2001. Food and Consumer Safety Authority, Netherlands http://www2. vwa.nl/CDL /files/15/1004/10387%20020924_monitoring_ bestrijdingsmiddelen.pdf accessed on 27.09.2006.
[7]	Anonymous	2003	Report of pesticide residue monitoring results of the Netherlands for 2002. Food and Consumer Safety Authority, Netherlands http://www2.vwa.nl/CDL/files/15/1004/10386%20NL_monitoring_2002.pdf accessed on 27.09.2006.
[8]	van der Schee	2004	Report of pesticide residue monitoring results of the Netherlands for 2003. Food and Consumer Safety Authority, Netherlands http://www2.vwa.nl/CDL/files/15/1004/10384%2020041021_pesticide_residue_monitoring_2003.pdf accessed on 27.09.2006.
[9]	Anonymous	2005	Report of pesticide residue monitoring results of the Netherlands for 2004. Food and Consumer Safety Authority, Netherlands http://www2.vwa.nl/CDL/files/15/1004/10381%20200509_bestrijdingsmiddelen_jaarrapportage_2004.pdf accessed on 27.09.2006.
DHD-03/01	Heimann S	2001	Clofentezine: Calculation of Henry's law constant. Regulatory Affairs of Agricultural Chemicals, Report No. DHD-03/01, unpublished, issued 04.06.2001.
R-12513	Bright A A S	1988	Clofentezine: Solubility of Clofentezine in dimethyl sulfoxide at 25°C. Schering Agrochemicals Ltd., Report No. CHEM/87/114, Aventis No. A80930=C98, IRV No. R-12513, unpublished, issued 14.01.1988.
R-12514A	Bright A A S	1987	Clofentezine: Solubility of Clofentezine in organic solvents at 25°C. Schering Agrochemicals Ltd., Report No. CHEM/87/37, Aventis No. A80936=C104, IRV No. R-12514A, unpublished, issued 12.03.1987.
R-12514B	Bright A A S	1987	Clofentezine: Determination of solubility in organic solvents - the analytical method. Schering Agrochemicals Ltd., Report No. CHEM/87/37, Aventis No.

			A80936A=C104A, IRV No.R-12514B, unpublished, issued 12.03.1987.
R-12515	Bright A A S	1987	Clofentezine: Colour, physical state and odour of Clofentezine technical. Schering Agrochemicals Ltd., Report No. CHEM/87/28, Aventis No. A80938=C105, IRV No. R-12515, unpublished, issued 12.03.1987.
R-12516	Johnson M K	1987	Clofentezine: Determination of the density of technical Clofentezine. Schering Agrochemicals Ltd., Report No. AD 22/87, Aventis No. A80939=C106, IRV No. R-12516, unpublished, issued 25.03.1987.
R-12519	Bright A A S and Stalker A M	1990	Clofentezine: Determination of the partition coefficient between N-octanol and water at 25°C. Schering Agrochemicals Ltd., Report No. CHR/90/010, Aventis No. A80944=C111, IRV No. R-12519, unpublished, issued 22.03.1990.
R-12521	Kelly I D	1985	Clofentezine: The photodegradation of [^{14}C]-Clofentezine in water under natural sunlight conditions. FBC Ltd., Report No. METAB/84/15, Aventis No. A82520=W46, IRV No. R-12521, unpublished, issued 29.04.1985.
R-12523	Smith S and Kelly I D	1985	Clofentezine: Solubility of Clofentezine in aqueous solution under acid, neutral and basic conditions. FBC Ltd., Report No. METAB/85/30, Aventis No. A82532=W58, IRV No. R-12523, unpublished, issued 19.12.1985.
R-12541	Campbell J K Needham D	1983	Clofentezine: Residues of [^{14}C]-NC21314 in the milk and tissues of a goat given a single oral dose of 22 mg NC21314. FBC Ltd., Report No. METAB/83/13, Aventis No. A82017=M25, IRV No. R-12541, unpublished, issued 19.04.1983.
R-12545	Campbell J K and Needham D	1983	Clofentezine: Residues of [^{14}C]-NC21314 in the milk and tissues of a cow dosed orally with NC21314 for 5 days. FBC Ltd., Report No. METAB/83/29, Aventis No. A82021=M33, IRV No. R-12545, unpublished, issued 07.07.1983.
R-12549	Needham D and Challis I R	1985	Clofentezine: An investigation into the nature of the residues present in the liver of the rat, goat and calf following the oral administration of Clofentezine. FBC Ltd., Report No. METAB/85/8, Aventis No. A82025=M37, IRV No. R-12549, unpublished, issued 30.08.1985.
R-12550	Challis I R	1985	Clofentezine: A comparison of the metabolism of Clofentezine in rat, mouse, rabbit, calf, dog and baboon. FBC Ltd., Report No. METAB/85/9, Aventis No. A82026=M38, IRV No. R-12550, unpublished, issued 09.12.1985.
R-12555	Campbell J K	1987	Clofentezine: Residues of [^{14}C]-Clofentezine in the milk of a goat dosed at 2.2 mg/kg for 7 days. Schering Agrochemicals Ltd., Report No. ENVIR/87/33, Aventis No. A82035=M47, IRV No. R-12555, unpublished, 05.11.1987.
R-12557	Creedy C L and Challis I R	1988	Clofentezine: The excretion and tissue residues of Clofentezine in the hen following the oral administration of 17 mg/bird/day for three days. Schering Agrochemicals Ltd., Report No. ENVIR/88/22, Aventis No. A82037=M49, IRV No. R-12557, unpublished, 06.07.1988.
R-12559	Phillips M W A and Swalwell L	1988	Clofentezine: Characterisation of [^{14}C]-residues in milk and liver of a cow following oral dosing with [^{14}C]-Clofentezine for 3 days at 2.2 mg/kg/day. Schering Agrochemicals Ltd. Report No. ENVIR/88/50, Aventis No. A82040=M52, IRV No. R-12559, unpublished, issued 05.12.1988.
R-12560	Phillips M W A and Swalwell L	1989	Clofentezine: Comparison and characterization of [^{14}C]-residues in milk of the cow and goat following oral dosing with [^{14}C]-Clofentezine (2.2 mg/kg/day). Schering Agrochemicals Ltd., Report No. ENVIR/89/15, Aventis No. A82041=M53, IRV No. R-12560, unpublished, issued 05.05.1989.
R-12561	Phillips M W A and Swalwell L	1989	Clofentezine: Characterisation of [^{14}C]-residues of a cow following oral administration of [^{14}C]-Clofentezine (2.2 mg/kg/day) for 3 days. Schering Agrochemicals Ltd., Report No. ENVIR/89/19, Aventis No. A82042=M54, IRV No. R-12561, unpublished, issued 05.05.1989.
R-12690	Snowdon P J	1982	Clofentezine: Residue study in soil following application of NC 21314 in bare plots in Texas, 1980. FBC Ltd., Study number 073/04/002, Report No. RESID/82/19, Aventis No. A82487=W12, IRV No. R-12690, unpublished, issued 22.03.1982.
R-12691	Snowdon P J	1982	Clofentezine: Laboratory study of NC21314 degradation in two standard soils from West Germany. FBC Ltd., Report No. RESID/82/28, Aventis No. A82488=W13, IRV No. R-12691, unpublished, issued 02.04.1982.
R-12693	Leake C R and Arnold D J	1983	Clofentezine: The degradation of NC21314 in a clay loam and loamy sand soil at 15°C. FBC Ltd., Report No. METAB/83/12, Aventis No. A82506=W29, IRV No. R-12693, unpublished, issued 25.04.1983.
R-12697	Manley J D and Snowdon P J	1983	Clofentezine: Residues of NC21314 in soil from apple grower trials treated with 50W formulation in the UK, 1982. FBC Ltd., Report No. RESID/83/49, Aventis No. A82515=W40, MAK No. R-12697, unpublished, issued 07.06.1983.
R-12698	Leake C R and	1983	Clofentezine: The degradation of NC 21314 in three soil types under aerobic,

	Arnold D J		sterile and anaerobic conditions. FBC Ltd., Report No. METAB/83/31, Aventis No. A82517=W43, IRV No. R-12698, unpublished, issued 15.08.1983.
R-12705	Snowdon P J	1986	Clofentezine: Decline of Clofentezine residues in soil following orchard treatment with the 50 SC formulation in New York, 1984. FBC Ltd., Report No. RESID/85/64, Aventis No. A82529=W55, IRV No. R-12705, unpublished, issued 26.11.1986.
R-12708	Manley J D and Snowdon P J	1985	Clofentezine: Decline of Clofentezine residues in soil following orchard treatment with 50 SC formulation in Washington, U.S.A., 1985. FBC Ltd., Report No. RESID/85/114, Aventis No. A82538=W63, IRV No. R-12708, unpublished, 23.12.2985.
R-12709	Bardalaye P	1988	Clofentezine: Dissipation of Clofentezine SC 50% w/w in soil following maximum use rates in Canada. NOR-AM Chemical Company, Report No. 15025, Aventis No. A82554=W75, IRV No. R-12709, unpublished, issued 01.02.1988.
R-12711	Godfrey T L and Peatman M H	1993	Clofentezine: Residues of Clofentezine in soil following application of a 50 SC formulation in Italy 1990. Schering Agrochemicals Ltd., Report No. RESID/91/82, Aventis No. A825700=W91, MAK No. R-12711, unpublished, issued 29.01.1993.
R-12714	Manley J D and Snowdon P J	1986	Clofentezine: Analytical method for residues of Clofentezine in miscellaneous fruit crops. FBC Ltd., Report No. RESID/86/48, Aventis No. A82149=R111, IRV No. R-12714, unpublished, issued 04.08.1986.
R-12715	Bardalaye P and Kelly I D	1988	Clofentezine: Clofentezine through EPA multiresidue protocols I and III. NOR-AM Chemical Company, Report No. 15028, Aventis No. A82206=R169, IRV No. R-12715, unpublished, issued 25.03.1988.
R-12716	Peatman M H and Snowdon P J	1989	Clofentezine: Analytical method for the determination of residues of 4-hydroxy Clofentezine in milk and animal fat by high performance liquid chromatography. 2nd edition. Schering Agrochemicals Ltd., Report No. RESID/89/49, Aventis No. A82219=R182-2, IRV No. R-12716, unpublished, issued 05.05.1989.
R-12723	Warner P A	1981	Clofentezine: The metabolism of [^{14}C]-NC21314 in apple foliage. FBC Ltd., Report No. METAB/81/8, Aventis No. A82001=M4, IRV No. R-12723, unpublished, issued 22.07.1981.
R-12726	Kelly I D	1985	Clofentezine: The metabolism of Clofentezine in apple fruit. Final Report. FBC Ltd., Report No. METAB/85/35, Aventis No. A82032=M43, IRV No. R-12726, unpublished, issued 23.12.1985.
R-12727	Smith S and Kelly I D	1986	Clofentezine: Metabolism of Clofentezine on lemon foliage. FBC Ltd., Report No. METAB/85/37, Aventis No. A82033=M44, IRV No. R-12727, unpublished, issued 14.01.1986.
R-12728	Edwards, R.	1987	Clofentezine: Investigation of the fibre-bound residue following treatment of apple fruit with [^{14}C]-Clofentezine. Schering Agrochemicals Ltd., Report No. ENVIR/87/32, Aventis No. A82034=M46, IRV No. R-12728, unpublished, issued 26.10.1987.
R-12729	Edwards R	1988	Clofentezine: Metabolism of Clofentezine in peach fruit. Schering Agrochemicals Ltd., Report No. ENVIR/88/25, Aventis No. A82039=M51, IRV No. R-12729, unpublished, issued 24.06.1988.
R-12730	Campbell J K	1989	Clofentezine: The metabolism of Clofentezine in grapes. Schering Agrochemicals Ltd., Report No. ENVIR/89/32, Aventis No. A82043=M55, IRV No. R-12730, unpublished, issued 18.08.1989.
R-12731	Snowdon P J	1983	Clofentezine: Residues of NC21314 in apple juice, sauce, cider and pomace following processing of treated apples in the USA, 1981. FBC Ltd., Report No. RESID/83/30, Aventis No. A82074=R37, IRV No. R-12731, unpublished, issued 19.04.1983.
R-12732	Peatman M H and Snowdon P J	1985	Clofentezine: Residues of Clofentezine in apples, apple juice, mash and pomace following the processing of treated apples in the USA 1985. FBC Ltd., Report No. RESID/85/121, Aventis No. A82137=R99, IRV No. R-12732, unpublished, issued 24.12.1985.
R-12733	Snowdon P J	1985	Clofentezine: Residues of Clofentezine and metabolites in the tissues and milk of cattle following a 28-day feeding study in the UK, 1984. FBC Ltd., Report No. RESID/85/122, Aventis No. A82138=R100, IRV No. R-12733, unpublished, issued 24.12.1985.
R-12734	Peatman M H and Snowdon P J	1986	Clofentezine: Combined residues of Clofentezine and metabolites in animal tissues following a 28-day calf feeding study in the UK 1986. Schering Agrochemicals Ltd., Report No. RESID/86/107, Aventis No. A82162=R123, IRV No. R-12734, unpublished, issued 17.11.1986.
R-12735	Peatman M H and Snowdon	1987	Clofentezine: Residues of Clofentezine and metabolites in the tissues and eggs of laying hens following a 28 day feeding study in the UK 1986. Schering

	P J		Agrochemicals Ltd., Report No. RESID/87/30, Aventis No. A82180=R143, IRV No. R-12735, unpublished, issued 26.05.1987.
R-12736	Castro L	1987	Clofentezine: Residues of Clofentezine in mature grapes and raisins in trials conducted in the USA in 1984 and 1985. NOR-AM Chemical Company, Report No. 15014, Aventis No. A82190=R153, IRV No. R-12736, unpublished, issued 20.05.1987.
R-12737	Castro L	1987	Clofentezine: Residues of Clofentezine in grapes and its processed commodities following mid-season application in trials conducted in the USA in 1986. NOR-AM Chemical Company, Report No. 15017, Aventis No. A82192=R155, IRV No. R-12737, unpublished, issued 04.11.1987.
R-12738	Kelly I D	1987	Clofentezine: Residues of Clofentezine in oranges and its processed commodities following application to mature fruit in the USA in 1987. NOR-AM Chemical Company, USA, Report No. 15018, Aventis No. A82195=R158, IRV No. R-12738, unpublished, issued 04.11.1987.
R-12739	Snowdon P J and Godfrey T L	1991	Clofentezine: Residues of Clofentezine in grapes, must and wine following treatment with a 50 SC formulation in Italy 1987. Schering Agrochemicals Ltd., Report No. RESID/90/36, Aventis No. A82256=R221, IRV No. R-12739, unpublished, issued 01.02.1991.
R-12740	Castro L	1992	Clofentezine: Residues of Clofentezine in or on fresh fruit and dry pomace derived from apple trees treated with Apollo SC at different rates and growth stages – USA 1991, including Amendments 1 and 2. NOR-AM Chemical Company, Report No. J-91R-01, Aventis No. A82287=R256 + Amendments A82288 & A82289, IRV No. R-12740, unpublished, issued 19.02.1992.
R-12741	Scott Brady S	1992	Clofentezine: Clofentezine-derived residues in or on grapes and grape commodities following a single application of Apollo SC, 35 days PHI – USA, 1991. NOR-AM Chemical Company, Report No. J-91R-02, Aventis No. A82293=R260, IRV No. R-12741, unpublished, issued 05.08.1992.
R-12742	Scott Brady S	1995	Clofentezine: At-harvest Clofentezine residues in or on whole grapes and processed grape commodities following a single application of Apollo SC at maximum and exaggerated rates, 21 days PHI – USA 1993. AgrEvo USA Company, Report No. J-93R-02, Aventis No. A89222=R281, IRV No. R-12742, unpublished, issued 07.12.1995.
R-12789	Kelly I D	1985	Clofentezine: The uptake of Clofentezine residues by miniature orange trees (*Citrus mitis*) and apple seedlings from Clofentezine treated soil under glasshouse conditions. FBC Ltd., Report No. METAB/85/7, Aventis No. W52, IRV No. R-12789, unpublished, issued 29.04.1985.
R-12799	Peatman M H	1992	Clofentezine: The decline of Clofentezine residues in soil following application of a 50 SC formulation to bar soil in Germany 1991. Schering Agrochemicals Ltd., Report No. RESID/92/87, Aventis No. A82573=W94 , IRV No. R-12799, unpublished, issued 26.11.1992.
R-12800	Castro L	1991	Clofentezine: Dissipation of Clofentezine in soil following application of Apollo to bare soil, USA 1990. NOR-AM Chemical Company, Report No. R152.03.90, Aventis No. A82871=W92, IRV No. R-12800, unpublished, issued 13.08.1991.
R-12802	Patmore I R, Godfrey T L and Peatman M H	1996	Clofentezine: Clofentezine suspo emulsion + oil 6%, code CR19707 Decline of ai residues following application on bare soil, Germany; 1993/94. AgrEvo, Report No. RESID/95/10, Aventis No. A84775=W100, IRV No. R-12802, unpublished, 26.03.1996.
R-12803	Scott Brady S	1995	Clofentezine: Comparison of at-harvest Clofentezine residues in or on peaches following a single application of Apollo SE with those from a single application of Apollo SC, 21 days prior to harvest, USA, 1993. AgrEvo, Report No. J-93R-04, Aventis No. A89186=R276, IRV No. R-12803, unpublished, issued 30.10.1995.
R-12804	Nohl-Weiler C	1995	Clofentezine: Clofentezine - Report on the field data from the residue-decline-study A93RB01 with CR19707 in soil in Germany 1993. (= field data from Patmore et al., 1996). AgrEvo, Report No. A93RB01, Aventis No. A89365=W101, IRV No. R-12804, unpublished, issued 21.11.1995.
R-12805	Castro, L.	1987	Clofentezine: Dissipation of Clofentezine in soil following both early and mid-season application in trials conducted in Canada in 1986. NOR-AM Chemical Company Report No. 15023, Aventis No. A89384=W66, IRV No. R-12805, unpublished, issued 11.06.1987.
R-12853	Allen R	1997	Clofentezine: Calculation of the potential residues in follow-on crops after the use of Clofentezine in ornamentals and strawberries. AgrEvo UK Ltd., Report No. ENVIR/97/004, Aventis No. A81684=R284-1, IRV No. R-12853, unpublished, issued 03.03.1997.
R-12959	Pires A	2000	Clofentezine: Independent laboratory validation of an analytical method for determination of residues of Clofentezine in miscellaneous fruit crops. IBET -

			Instituto de Biologia Experimental e Tecnologica, Report No. LA-REL-V-23/2000-AVE, Aventis No. C011470, IRV No. R-12959, unpublished, issued 03.12.2000.
R-12997	Peatman M H and Snowdon P J	1989	Clofentezine: Residues of Clofentezine in tomatoes following application of a 50 SC formulation in Italy 1986. Schering Agrochemicals Ltd., Report No. RESID/89/6, Aventis No. R187, IRV No. R-12997, unpublished, issued 20.04.1989.
R-12999	Manley J D and Snowdon P J	1989	Clofentezine: Residues of Clofentezine in gherkins following application of a 50 SC formulation in the Netherlands, 1988. Schering Agrochemicals Ltd., Report No. RESID/89/8, Aventis No. A82226=R189, IRV No. R-12999, unpublished, issued 13.03.1989.
R-13002	Scott Brady S	1989	Clofentezine: Residues of Clofentezine in walnuts following application of Apollo SC, USA 1988. NOR-AM Chemical Company, Report No. R152.04.88, Aventis No. A82230=R193, IRV No. R-13002, unpublished, issued 11.07.1989.
R-13003	Scott Brady S	1989	Clofentezine: Residues of Clofentezine in almonds following application of Apollo SC at late season, USA 1988. NOR-AM Chemical Company, Report No. R152.01.88, Aventis No. A82231=R194, IRV No. R-13003, unpublished, issued 19.06.1989.
R-13007	Godfrey T L, Peatman M H and Snowdon P J	1989	Clofentezine: Decline of Clofentezine residues in apples following treatment with a 50 SC formulation in Argentina 1987. Schering Agrochemicals Ltd., Report No. RESID/89/95, Aventis No. A82236=R199, IRV No. R-13007, unpublished, issued 09.11.1989.
R-13011	Peatman M H and Snowdon P J	1990	Clofentezine: Residues of Clofentezine in cucumbers following application of a 50 SC formulation in Greece, 1988. Schering Agrochemicals Ltd., Report No. RESID/89/112, Aventis No. A82241=R205, IRV No. R-13011, unpublished, issued 17.05.1990.
R-13017	Peatman M H and Snowdon P J	1990	Clofentezine: Residues of Clofentezine in peaches following application of a 50 SC formulation in Australia 1988. Schering Agrochemicals Ltd., Report No. RESID/89/120, Aventis No. A82247=R211, IRV No. R-13017, unpublished, issued 06.07.1990.
R-13018	Peatman M H	1990	Clofentezine: The decline of Clofentezine residues in strawberries following application of a 50SC formulation in France 1989. Schering Agrochemicals Ltd., Report No. RESID/90/20, Aventis No. A82250=R214, IRV No. R-13018, unpublished, issued 19.04.1990.
R-13024	Peatman M H and Snowdon P J	1990	Clofentezine: The decline of Clofentezine residues in plums treated with a 50 SC formulation in the Federal Republic of Germany, 1986. Schering Agrochemicals Ltd., Report No. RESID/90/42, Aventis No. A82259=R224, IRV No. R-13024, unpublished, issued 21.05.1990.
R-13031	Peatman M H and Godfrey T L	1991	Clofentezine: Residues of Clofentezine in apricots following application of a 50SC formulation in Greece 1989. Schering Agrochemicals Ltd., Report No. RESID/91/6, Aventis No. A82268=R235, IRV No. R-13031, unpublished, 17.05.1991.
R-13055	Peatman M H	1991	Clofentezine: Residues of Clofentezine in strawberries following application of a 50 SC formulation in the Netherlands, 1990. Schering Agrochemicals Ltd., Report No. RESID/91/13, Aventis No. A82270=R237, IRV No. R-13055, unpublished, 03.05.1991.
R-13057	Godfrey T L and Peatman M H	1991	Clofentezine: Residues of Clofentezine in strawberries following treatment with a 50 SC formulation in France, 1990. Schering Agrochemicals Ltd., Report No. RESID/91/85, Aventis No. A82274=R241, IRV No. R-13057, unpublished, issued 18.12.1991.
R-13062	Scott Brady S	1992	Clofentezine: At-harvest residues of Clofentezine in peaches at 14 and 21 days following a single treatment with Apollo 21 days prior to normal harvest. USA 1988. NOR-AM Chemical Company, Report No. R152.02.88, Aventis No. A82280=R248, IRV No. R-13062, unpublished, issued 20.05.1992.
R-13063	Peatman M H	1992	Clofentezine: Residues of Clofentezine in cucumbers following application of a 50 SC formulation in Greece, 1991. Schering Agrochemicals Ltd., Report No. RESID/91/114, Aventis No. A82281=R249, IRV No. R-13063, unpublished, issued 07.02.1992.
R-13064	Godfrey T L and Peatman M H	1992	Clofentezine: Residues of Clofentezine in oranges and lemons following application of a 50 SC formulation in Greece in 1990. Schering Agrochemicals Ltd., Report No. RESID/91/120, Aventis No. A82282=R250, IRV No. R-13064, unpublished, issued 14.02.1992.
R-13067	Godfrey T L and Peatman M H	1992	Clofentezine: Residues of Clofentezine in tomatoes following application of a 50 SC formulation in France 1992. Schering Agrochemicals Ltd., Report No. RESID/92/81, Aventis No. RA82294=R261, IRV No. R-13067, unpublished, issued 30.11.1992.

R-13068	Godfrey T L and Peatman M H	1993	Clofentezine: Decline of residues of Clofentezine in strawberries following application of a 50 SC formulation in Spain, 1992. Schering Agrochemicals Ltd., Report No. RESID/92/68, Aventis No. A82295=R263, IRV No. R-13068, unpublished, issued 04.03.1993.
R-13072	Peatman M H and Wright P	1993	Clofentezine: Decline of Clofentezine residues in apples following application of a Clofentezine plus oil formulation (CR 19707) in Germany, 1992. Schering Agrochemicals Ltd., Report No. RESID/93/26, Aventis No. A82299=R267, IRV No. R-13072, unpublished, issued 11.10.1993.
R-13075	Peatman M H	1994	Clofentezine: Clofentezine suspo-emulsion (SE) 6% + oil: suspension concentrate (SC) 50% (Code: CR19707:CR18390). Apollo: Apples - decline of a.i. residues, Belgium 1993. Schering Agrochemicals Ltd., Report No. RESID/94/19, Aventis No. A82307=R275, IRV No. R-13075, unpublished, issued 24.08.1994.
R-13076	Snowdon P J	1991	Clofentezine: A summary of Clofentezine residues in soil (0-10 cm) following application of the 50SC formulation in the Federal Republic of Germany, 1989. Abschlußbericht. Schering Agrochemicals Ltd., Report No. RESID/90/101, Aventis No. A82569=W90=R229, IRV No. R-13076, unpublished, issued 13.03.1991
R-13077	Godfrey T L and Peatman M H	1993	Clofentezine: Residues of Clofentezine in peaches following application of a 50SC formulation in Italy, 1991. Schering Agrochemicals Ltd., Report No. RESID/92/11, Aventis No. A89388=R254, IRV No. R-13077, unpublished, issued 29.01.1993.
R-13081	Scott Brady S	1995	Clofentezine: Comparison of at-harvest Clofentezine residues in or on apricots following a single application of Apollo SE with those from a single application of Apollo SC, 21 days prior to harvest, USA, 1993. AgrEvo, Report No. J-93R-05, Aventis No. A549486=R277, IRV No. R-13081, unpublished, issued 29.09.1995.
R-13082	Scott Brady S	1995	Clofentezine: Comparison of at-harvest Clofentezine residues in or on almonds following a single application of Apollo SE with those from a single application of Apollo SC, 30 days prior to harvest, USA, 1993. AgrEvo, Report No. J-93R-06, Aventis No. R278, IRV No. R-13082, unpublished, issued 14.12.1995.
R-13083	Cole M G	1996	Clofentezine: At-Harvest Clofentezine residues in or on whole apples and apple pomace following a single application of Apollo at two exaggerated rates and seven growth stages, USA, 1993. AgrEvo USA Company, Report No. J-93R-01, Aventis No. A80974, IRV No. R-13083, unpublished, issued 29.07.1996.
R-13090	Snowdon P J	1981	Clofentezine: Residues in apples following the application of NC 21314, 50W, in South Africa 1980/81. FBC Ltd., Report No. RESID/81/77, Aventis No. A82055=R13, IRV No. R-13090, unpublished, issued 10.02.1982.
R-13102	Snowdon P J	1982	Clofentezine: Residue of NC21314 in mature apples and pears following mid- and late season application of the 80W formulation in Australia, 1981/82. FBC Ltd., Report No., RESID/82/105, Aventis No. A82068=R31, IRV No. R-13102, unpublished, issued 21.12.1982.
R-13112	Manley J D and Snowdon P J	1983	Clofentezine: Residues of NC 21314 in cherries treated with the 50SC formulation in the UK, 1983. FBC Ltd., Report No. RESID/83/81, Aventis No. A82082R45, IRV No. R-13112, unpublished, issued 24.10.1983.
R-13113	Manley J D and Snowdon P J	1983	Clofentezine: Residues of NC21314 in apples following application of the 50 SC formulation in France, 1983. Schering Agrochemicals Ltd., Report No. RESID/83/85, Aventis No. A82083=R46, IRV No. R-13113, unpublished, issued 04.10.1983.
R-13121	Peatman M H and Snowdon P J	1989	Clofentezine: Analytical method for the determination of free Clofentezine residues in milk and animal fat by high performance liquid chromatography. Schering Agrochemicals Ltd., Report No. RESID/89/50, Aventis No. A82091=R54-3, IRV No. R-13121, unpublished, issued 06.05.1989.
R-13126	Snowdon P J	1984	Clofentezine: Residues of Clofentezine in mature apples from trials with the 50 SC formulation in the UK, 1983. Schering Agrochemicals Ltd., Report No. RESID/84/58, Aventis No. A82098=R60, IRV No. R-13126, unpublished, issued 14.09.1984.
R-13127	Snowdon P J	1984	Clofentezine: Stability of Clofentezine residues in apples during deep freeze storage. Schering Agrochemicals Ltd., Report No. RESID/84/50, Aventis No. A82099=R61, IRV No. R-13127, unpublished, issued 12.09.1984.
R-13131	Snowdon P J	1985	Clofentezine: Decline of Clofentezine residues in apples following treatment with the 50 SC formulation in West Germany, 1984. Schering Agrochemicals Ltd., Report No. RESID/85/9, Aventis No. A82105=R67, IRV No. R-13131, unpublished, issued 09.04.1985.
R-13133	Manley J D	1985	Clofentezine: Residues of NC21314 in apples treated the 50 SC formulation in

	and Snowdon, P J		France, 1984. Schering Agrochemicals Ltd., Report No. RESID/85/12, Aventis No. A82107=R69, IRV No. R-13133, unpublished, issued 25.02.1985.
R-13137	Manley J D and Snowdon P J	1985	Clofentezine: Residues of Clofentezine in peaches treated with the 50SC formulation in Italy, 1984. FBC Ltd., Report No. RESID/85/46, Aventis No. A82114=R76, IRV No. R-13137, unpublished, issued 21.05.1985.
R-13144	Manley JD and Snowdon P J	1985	Clofentezine: Residues of Clofentezine in pears treated with the 50 SC formulation in Italy, 1984. Schering Agrochemicals Ltd., Report No. RESID/85/47, Aventis No. A82115=R77, IRV No. R-13144, unpublished, issued 13.05.1985.
R-13146	Manley J D and Snowdon P J	1985	Clofentezine: Residues of Clofentezine in citrus fruit treated with the 50 SC formulation in Italy, 1984. Schering Agrochemicals Ltd., Report No. RESID/85/57, Aventis No. A82118=R80, IRV No. R-13146, unpublished, issued 31.07.1985.
R-13148	Manley J D and Snowdon P J	1989	Clofentezine: Analytical method for residues of Clofentezine in citrus fruit. FBC Ltd., Report No. RESID/85/59, Aventis No. A82120, IRV No. R-13148, unpublished, issued 31.07.1985.
R-13159	Peatman M H and Snowdon P J	1986	Clofentezine: The decline of residues in apples and pears treated with the 50SC formulation in the Republic of South Africa, 1984/85. FBC Ltd., Report No. RESID/85/102, Aventis No. A82131=R93, IRV No. R-13159, unpublished, issued 12.05.1986.
R-13160	Manley J D and Snowdon P J	1985	Clofentezine: Analytical method for residues of NC21314 in apples. (2nd edition.). FBC Ltd., Report No. RESID/85/112, Aventis No. R94, IRV No. R-13160, unpublished, issued 20.12.2985.
R-13160A	Snowdon P J and Crofts M	1981	Clofentezine: Analytical method for residues of NC21314 in apples. FBC Ltd., Report No. RESID/81/33, Aventis No. R6=C8, IRV No. R-13160A, unpublished, issued 14.07.1981.
R-13162	Manley J D and Snowdon P J	1986	Clofentezine: Residues of Clofentezine in apples treated during fruit development with the 50SC formulation in the USA, 1985. FBC Ltd., Report No. RESID/85/117, Aventis No. A82134=R96, IRV No. R-13162, unpublished, issued 16.07.1986.
R-13163	Manley J D and Snowdon P J	1986	Clofentezine: Decline of Clofentezine residues in apples treated with the 50SC formulation in the USA, 1985. FBC Ltd., Report No. RESID/85/117, Aventis No. A82135=R97, IRV No. R-13163, unpublished, issued 16.07.1986.
R-13164	Peatman M H and Snowdon P J	1985	Clofentezine: Stability of Clofentezine residues in animal tissues and milk during deep freeze storage. Schering Agrochemicals Ltd., UK, Report No. RESID/85/120, Aventis No. A82136=R98, IRV No. R-13164, unpublished, issued 20.12.1985.
R-13167	Manley J D and Snowdon P J	1986	Clofentezine: Decline of Clofentezine residues in apples following treatment with the 50 SC formulation in the Federal Republic of Germany, 1985. Schering Agrochemicals Ltd., Report No. RESID/86/9, Aventis No. A82141=R103, IRV No. R-13167, unpublished, issued 28.02.1986.
R-13169	Castro L	1985	Clofentezine: Residues of Clofentezine on apples from study R15.03.85-JCA-01. NOR-AM Chemical Company, Aventis No. A82143=R105, IRV No. R-13169, unpublished, issued 07.02.1986.
R-13170	Drew M	1986	Clofentezine: Residues of Clofentezine in pears and peaches following application of Apollo, Australia, 1984-85. Schering Pty Ltd., Report No. A/V 1-83, I/HO 1-84, Aventis No. A82175=R137, IRV No. R-13170, unpublished, 20.11.1986.
R-13184	Castro L and Powley C	1986	Clofentezine: Residue of Clofentezine in almond hulls and nutmeats following application of Apollo 50 SC at early and mid-season. NOR-AM Chemical Company, Report No. 15006, Aventis No. A82158=R119, IRV No. R-13184, unpublished, issued 30.07.1986.
R-13187	Manley J D and Snowdon P J	1986	Clofentezine: Residues of Clofentezine in apples treated with the 50 SC formulation in Greece, 1986. Schering Agrochemicals Ltd., Report No. RESID/86/104, Aventis No. A82161=R122, IRV No. R-13187, unpublished, issued 28.10.1986.
R-13197	Hanni R	1985	Clofentezine: Residues in cucumbers treated with Apollo 50 SC in Switzerland, 1985. MAAG Ltd., Report No.6123-85102, Aventis No. A82177=R139, IRV No. R-13197, unpublished, issued 05.12.1985.
R-13200	Castro L	1987	Clofentezine: Residues of Clofentezine in peaches following both early and mid-season application in trials conducted in the USA in 1986. NOR-AM Chemical Company, Report No. 15022, Aventis No. A82181R144, IRV No. R-13200, unpublished, 19.03.1987.
R-13201	Castro L	1987	Clofentezine: Residues of Clofentezine in apples at harvest following early season and both early and mid-season application in trials conducted in Canada in 1986. NOR-AM Chemical Company, Report No. 15021, Aventis

			No. A82182=R145, IRV No. R-13201, unpublished, issued 05.03.1987.
R-13202	Castro L	1987	Clofentezine: Residues of Clofentezine in peaches and nectarines at harvest following early season and both early and mid-season application in trials conducted in the USA in 1986. NOR-AM Chemical Company, Report No. 15010, Aventis No. A82183=R146, IRV No. R-13202, unpublished, issued 05.03.1987.
R-13225	Snowdon P J and Peatman M H	1988	Clofentezine: Residues of Clofentezine in cucumbers treated with the 50 SC formulation in France, 1987. Schering Agrochemicals Ltd., Report No. RESID/88/24, Aventis No. A82205=R168, IRV No. R-13225, unpublished, 06.05.1988.
R-13228	Castro L	1987	Clofentizine: Residues of Clofentezine in apples at harvest following both early and mid-season application in trials conducted in the USA in 1986. NOR-AM Chemical Company, Report No. 15020, Aventis No. A82187=R150, IRV No. R-13228, unpublished, issued 07.04.1987.
R-13235	Peatman M H and Snowdon P J	1988	Clofentezine: The decline of Clofentezine residues in plums treated with the 50 SC formulation in the Federal Republic of Germany, 1985. Schering Agrochemicals Ltd., Report No. RESID/87/105, Aventis No. A82198=R161, IRV No. R-13235, unpublished, issued 25.10.1988.
R-13238	Bardalaye P	1988	Clofentezine: Residues of Clofentezine in apples at harvest following both early and late season treatment with Apollo SC, Canada 1987. NOR-AM Chemical Company, Report No. 15031, Aventis No. A82208=R171, IRV No. R-13238, unpublished, issued 17.05.1988.
R-13239	Bardalaye P	1988	Clofentezine: Residues of Clofentezine in peaches and nectarines at harvest following two later season applications of Apollo SC, USA 1987. NOR-AM Chemical Company, Report No. 15030, Aventis No. A82210=R173, IRV No. R-13239, unpublished, issued 16.08.1988.
R-13240	Castro L	1988	Clofentezine: Stability of Clofentezine in almond hulls and nutmeats under freezer storage for a period of two years. NOR-AM Chemical Company, Report No. 15002, Aventis No. A82211=R174, IRV No. R-13240, unpublished, issued 29.09.1988.
R-13241	Bardalaye P C	1988	Clofentezine: Residues of Clofentezine in plums, apricots and cherries at harvest following treatment with Apollo SC, USA 1987. NOR-AM Chemicals Company, Report No. 15035, Aventis No. A82212=R175, IRV No. R-13241, unpublished, issued 30.08.1988.
R-13242	Castro L	1988	Clofentezine: Stability of Clofentezine in peaches under freezer storage for a period of two years. NOR-AM Chemical Company, Report No. 15004, Aventis No. A82213=R176, IRV No. R-13242, unpublished, issued 29.08.1988.
R-13243	Kelly I D	1988	Clofentezine: Residues of Clofentezine in walnuts at harvest following two applications of Apollo SC, USA 1987. NOR-AM Chemical Company, Report No. 15034, Aventis No. A82214=R177, IRV No. R-13243, unpublished, issued 29.08.1988.
R-13244	Kelly I D	1988	Clofentezine: Residues of Clofentezine in almond nutmeat and hulls at harvest following two applications of Apollo SC, USA 1987. NOR-AM Chemical Company, Report No. 15033, Aventis No. A82216=R179, IRV No. R-13244, unpublished, issued 14. 10.1988.
R-13279	Sonder K	2000	Clofentezine: Residues at harvest in unprotected tomatoes. European Union (northern zone) 1999. Aventis CropScience GmbH, Report No. ER99ECN703, Aventis No. C009212, IRV No. R-13279, unpublished, issued 08.09.2000.
R-13283	Smeykal H	2000	Clofentezine: Melting point, melting range, boiling point, boiling range, thermal stability. Clofentezine substance, pure. Axiva, Report No. SI093-00, Aventis No. C010335, IRV No. R-13283, unpublished, issued 17.10.2000.
R-13284	Smeykal H	2000	Clofentezine: Relative density. Clofentezine substance, pure. Axiva, Report No. SI094-00, Aventis No. C010336, IRV No. R-13284, unpublished, issued 17.10.2000.
R-13287	Sonder K	2000	Clofentezine: Clofentezine 500 g/L SC: Residues at harvest in musk melon. European Union (southern zone) 1999. Aventis CropScience GmbH, Report No. ER 99 ECS 701, Aventis No. C009215, IRV No. R-13287, unpublished, issued 03.11.2000.
R-13288	Sonder K	2000	Clofentezine: Residues at harvest in unprotected tomatoes. European Union (southern zone) 1999. Aventis CropScience GmbH, Report No. ER99ECS702, Aventis No. C009216, IRV No. R-13288, unpublished, issued 10.11.2000.
R-13314	Mühlberger B	2001	Clofentezine: AEB 084866: Partition coefficient N-octanol/water. Aventis, Report No. PA00/037, IRV No. R-13314, unpublished, issued 18.05.2001
R-13315	Mühlberger B	2001	Clofentezine: AEB 084866: Solubility in organic solvents at 20°C. Aventis, Report No. PA00/036, IRV No. R-13315, unpublished, issued 18.05.2001.

R-13317	van der Gaauw A	2001	Clofentezine: [^{14}C]-Clofentezine simulated processing. RCC Ltd., Report No. 795194, IRV No. R-13317, unpublished, issued 14.11.2001.
R-13318	van der Gaauw A	2001	Clofentezine: [^{14}C]-Clofentezine: Hydrolysis at three different pH values. RCC Ltd., Report No. 793124, IRV No. R-13318, unpublished, issued 14.11.2001
R-13464	Pollmann B	2002	Clofentezine: Determination of residues of Clofentezine after application of Apollo 50 SC in grapes at 6 sites in southern Europe, 2001 Arbeitsgemeinschaft GAB & IFU, Report No. 20011152/E1-FPVI, IRV No. R-13464, unpublished, issued 24.04.2002.
R-13465	Pollmann B	2002	Clofentezine: Determination of residues of Clofentezine after application of Apollo 50 SC in strawberries at 6 sites in Northern Europe, 2001 Arbeitsgemeinschaft GAB & IFU, Report No. 20011152/E1-FPST, IRV No. R-13465, unpublished, 23.04.2002.
R-14208	Wende S	2001	Clofentezine: Residue analysis of Clofentezine in strawberries and apples. Method validation. GAB & IFU, Report No. 20011171/01-RVP, IRV No. R-14208, unpublished, issued 29.11.2001.
R-14209	Pollmann B	2002	Clofentezine: Generation of processed goods from strawberries treated with Apollo 50 SC and determination of residues of Clofentezine in the processed goods. Arbeitsgemeinschaft GAB & IFU, Report No. 20011152/E1-FPPR, IRV No. R-14209, unpublished, issued 23.04.2002.
R-14527	Sonder K	2001	Clofentezine: Clofentezine 500 g/L SC: Residues at harvest in strawberries. European Union (northern zone) 2000. Aventis CropScience GmbH, Report No. DR 00 EUN 230, Aventis No. C013148, IRV No. R-14527, unpublished, issued 07.08.2001.
R-14529	Sonder K	2001	Clofentezine: Clofentezine 500 g/L SC: Residues at harvest in melon. European Union (southern zone) 2000. Aventis CropScience GmbH, Report No. DR 00 EUS 231, Aventis No. C013505, IRV No. R-14529, unpublished, issued 27.06.2001.
R-14625	Sonder K	2001	Clofentezine: Residues at harvest in glasshouse tomatoes. European Union (northern zone) 1999. Aventis CropScience GmbH, Report No. ER99ECN704, Aventis No. C009214, IRV No. R-14625, unpublished, issued 13.11.2001.
R-15401	Scott-Brady S	1993	Clofentezine: At-harvest residues of Clofentezine in pears treated with Apollo at 21 days PHI. Canada 1988. NOR-AM Chemical Company, Report No. R152.08.88, Aventis No. 82290, IRV No. R-15401, unpublished, issued 08.04.1993.
R-15402	Bardalaye, P.	1988	Clofentezine: Residues of Clofentezine in pears at harvest following both early and late season treatment with Apollo SC, Canada 1987. NOR-AM Chemical Company, Report No. 15029, Aventis No. A82209, IRV No. R-15402, unpublished, issued 02.06.1988.
R-15403	Cole M G	1995	Clofentezine: Comparison of at-harvest Clofentezine residues in or on pears following a single application of Apollo SE with those from a single application of Apollo SC, 21 days prior to harvest, USA, 1993. AgrEvo, Report No. J-93R-03, Aventis No. R279, IRV No. R-15403, unpublished, issued 30.10.1995.
R-15405	Kelly I D and Castro L	1987	Clofentezine: Residues of Clofentezine in pears at harvest following early season and both early and mid-season application in trials conducted in the USA in 1986. NOR-AM Chemical Company, Report No. 15016, Aventis No. A82176=R138, IRV No. R-15405, unpublished, 09.02.1987.
R-15521	Heintze A	2002	Clofentezine: Water solubility and abiotic degradation of Clofentezine. Statement. Arbeitsgemeinschaft GAB & IFU, IRV No. R-15521, unpublished, issued 08.05.2002.
R-15918	Pollmann B	2003	Clofentezine: Generation and analysis of processed goods from apples treated with Clofentezine for determination of Clofentezine and its metabolites. GAB & IFU, Report No. 20031162/G1-FPPR, IRV No. R-15918, unpublished, issued 11.09.2003.
R-15921	Serano C	2002	Clofentezine: Determination of residues of Clofentezine in citrus fruit (Oranges and Mandarines), following a single application with Apollo 20 SC during 2001-2002 in Spain. TrialCamp SLL, Report no. TRC01-7, IRV No. R-15921A, R-15921B and R-15921C, unpublished, issued 18.10.2002.
R-16387	Geffke T	2003	Clofentezine: Residue analytical method for the determination of Clofentezine technical (dried) in cores of sunflower (rich fat) and wheat grains (low fat). Dr. U. Noack Laboratorium, Report No. CRA90081, IRV No. R-16387, unpublished, issued 26.09.2003.
R-16523	Heintze A	2003	Clofentezine: Dissociation Constant of Clofentezine. Statement. GAB & IFU, IRV No. R-16523, unpublished, 29.09.2003.
R-17532	Witte A	2004	Clofentezine: Validation of an analytical method for the determination of

			Clofentezine and metabolites in animal tissue. Independent Laboratory Validation. GAB Biotechnologie GmbH, GAB Analytic GmbH Report No. 20041042/01-RVAT, IRV No. R-17532, unpublished, issued 22.06.2004.
R-18416	Manley J D and Snowdon P J	1985	Clofentezine: Residues of Clofentezine in grapes treated with the 50 SC formulation in Italy, 1984. Schering Agrochemicals Ltd., Report No. RESID/85/45, Aventis No. A82113=R75, IRV No. R-18416, unpublished, issued 20.05.1985.
R-18417	Peatman M H and Snowdon P J	1989	Clofentezine: The decline of Clofentezine residues in grapes treated with a co-formulation of Clofentezine and Cyhexatin in the Federal Republic of Germany, 1986. Schering Agrochemicals Ltd., Report No. RESID/88/9, Aventis No. A82201=R164, IRV No. R-18417, unpublished, issued 21.04.1989.
R-18418	Godfrey T L and Peatman M H	1992	Clofentezine: Residues of Clofentezine in grapes following application either alone or as a co-formulation with Bifenthrin in France 1991. Schering Agrochemicals Ltd., Report No. RESID/92/13, Aventis No. A82286=R255, IRV No. R-18418, unpublished, issued 22.07.1992.
R-18419	Peatman M H	1993	Clofentezine: Clofentezine: 6% SE plus oil, CR 19707): Vines, Grapes): Decline of ai residues Germany 1992. Schering Agrochemicals Ltd., Report No. RESID/93/39, Aventis No. A82303=R270, IRV No. R-18419, unpublished, issued 21.10.1993.
R-18736	Snowdon P J	1983	Clofentezine: Analytical method for residues of NC21314 in apples and pears. (Improved method.) FBC Ltd., Report No. RESID/82/102, Aventis No. R29, IRV No. R-18736, unpublished, issued 07.01.1983.
R-18909	Chambers J G	2006	Clofentezine: Independent laboratory validation for the determination of clofentezine residues in strawberries and apple based on the DFG-S19 multiresidue enforcement methods. Synergy Laboratories Ltd., Report No. SYN/0802, IRV No. R-18909, unpublished, issued 16.03.2006.
R-18971	Malet J C and Allard A	2003	Clofentezine: Mesure du niveau de résidus de Clofentezine sur cassis – Residues of Clofentezine in black currant. Ministère de l'Agriculture et de la Pêche, Bureau Biovigilance et Expérimentation, Report No. RAPF49701, IRV No. R-18971, unpublished, issued 27.03.2003.
R-20184	Malet J C	2006	Clofentezine: Mesure du niveau de résidus de Clofentezine après 1 application de la préparation Apollo 50 SC sur cassis dans le cadre d'une extension d'usage sur la culture – Residues of Clofentezine after 1 application of Apollo 50 SC in black currant in support of the registration. Ministère de l'Agriculture et de la Pêche, Bureau Biovigilance et Expérimentation, Report No. RAPF22904, IRV No. R-20184, unpublished, issued 08.03.2006.
R-20408	Chambers A	2006	Clofentezine: Independent Laboratory Validation of an analytical method for the determination of Clofentezine and its metabolites in animal tissues. Synergy Laboratories Ltd., Report No.SYN/0801, IRV No. R-20408, unpublished, issued 30.05.2006.
R-21165	Peatman M H and Snowdon P J	1988	Clofentezine: The decline of Clofentezine residues in Grapes treated with a co-formulation of Clofentezine and Cyhexatin in the federal Republic of Germany, 1985. Schering Agrochemicals Ltd., Report No. RESID/86/140, Aventis No. A82169=R131, IRV No. R-21165, unpublished, issued 20.09.1988.
R-21166	Peatman M H and Snowdon P J	1989	Clofentezine: The decline of Clofentezine residues in grapes treated with the 50 SC formulation in the Federal Republic of Germany, 1986. Schering Agrochemicals Ltd., Report No. RESID/88/8, Aventis No. R163, IRV No. R-21166, unpublished, 21.04.1989.
R-21211A	Manley J D, Peatman M H and Snowdon P J	1985	Clofentezine: Analytical method for residues of Clofentezine and metabolites in animal tissues and milk. Schering Agrochemicals Ltd., Report No. RESID/85/32, Aventis No. A83895=R72, IRV No. R-21211A, unpublished, issued 24.12.1985.
R-21211B	Ferreira E M	1997	Clofentezine: Addendum to Report RESID/85/32: Analytical method for residues of Clofentezine and metabolites in milk, AgrEvo No. A81686 and Amendment to the Addendum, AgrEvo No. 81687. Report No. RESID/97/34, IRV No. R-21211B, unpublished, issued 04.06.1997.
R-21212	Peatman M H and Snowdon P J	1988	Clofentezine: Analytical method for the determination of residues of Clofentezine and metabolites in animal tissues and milk. 2nd edition. Schering Agrochemicals Ltd., Report No. RESID/87/124, Aventis No. A83896=R72-2, IRV No. R-21212, unpublished, issued 04.01.1988.
R-21213	Peatman M H and Snowdon P J	1989	Clofentezine: Analytical method for the determination of residues of Clofentezine and 4-hydroxy-Clofentezine in animal tissues by gas chromatography. 3rd edition. FBC Ltd., Report No. RESID/89/51, Aventis No. A82110=R72-3, IRV No. R-21213, unpublished, issued 05.05.1989.
R-21214	Cooley T A	2003	Clofentezine: Magnitude of Residues of Apollo SC (Clofentezine) in almond

			and peach raw agricultural commodities. Excel Research Services Inc., Report No. ERS22084, IRV No. R-21214, unpublished, issued 12.08.2003.
R-21215	Baravelli P L	2006	Clofentezine: Field and Laboratory Phase in the determination of Clofentezine residues in tomato – 2005 Italy. Agriparadigma SRL, Report No. AGRI 031/05 GLP DEC, IRV No. R-21215, unpublished, issued 14.12.2005.