CYFLUTHRIN (157)/BETA-CYFLUTHRIN (228)

First draft was prepared by Dr Dugald MacLachlan, Australian Quarantine and Inspection Service, Canberra

EXPLANATION

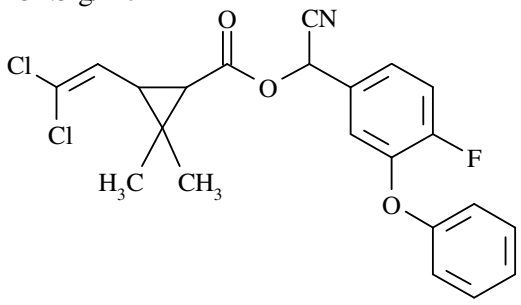
Cyfluthrin has been evaluated several times since the initial evaluation in 1986, the latest in 1992 for residues and in 2006 for toxicology. Cyfluthrin was identified as a priority compound under the Periodic Re-evaluation Programme at the 37th Session of the CCPR and scheduled for the 2007 JMPR.

Beta-cyfluthrin is an enriched isomeric form of the two biologically active diastereoisomeric pairs of isomers of cyfluthrin. It is considered a new compound and is evaluated together with cyfluthrin as data generated with cyfluthrin are used in support of beta-cyfluthrin and *vice versa*.

Data to support the existing CXLs for cyfluthrin and new MRLs for beta-cyfluthrin and other critical data required for the estimation of MRLs have been provided by the company.

The governments of Australia and The Netherlands have submitted national GAP information.

IDENTITY

Common name	Cyfluthrin, Beta-cyfluthrin
Manufacturers code number	FCR 1272 (Cyfluthrin), FCR 4545 (Beta-cyfluthrin)
Chemical name	(cyfluthrin and beta-cyfluthrin)
IUPAC (cyfluthrin):	(*R,S*)-α- cyano-4-fluoro-3-phenoxybenzyl (1*RS*,3*RS*;1*RS*,3*SR*)-3-(2,2-dichlorovinyl)-2,2-dimethylcyclopropane carboxylate
IUPAC (beta-cyfluthrin):	3-(2,2-dichloro-vinyl)-2,2-dimethyl-cyclopropane-carboxylic acid cyano-(4-fluoro-3-phenoxy-phenyl)-methyl ester (unstated stereochemistry)
CAS:	cyclopropanecarboxylic acid, 3-(2,2-dichloroethenyl)-2,2-dimethyl-cyano, (4-fluoro-3-phenoxyphenyl)methyl ester (unstated stereochemistry)
CAS number	68359-37-5 (unstated stereochemistry) 86560-92-1 (diastereoisomer I) 86560-93-2 (diastereoisomer II) 86560-94-3 (diastereoisomer III) 86560-95-4 (diastereoisomer IV)
Molecular formula:	$C_{22}H_{18}Cl_2FNO_3$
Molecular weight:	434.3 g/mol
Structural formula:	

Isomer compositions of cyfluthrin and beta-cyfluthrin (FAO specifications)

Isomers	Cyfluthrin	Beta-cyfluthrin
Diastereoisomer I (1*R*,3*R*,1*R* + 1*S*,3*S*,1*S* = 1:1; *cis*)	23 – 27%	≤ 2%
Diastereoisomer II (1*R*,3*R*,1*S* + 1*S*,3*S*,1*R* = 1:1; *cis*)	17 – 21%	30 – 40%
Diastereoisomer III (1*R*,3*S*,1*R* + 1*S*,3*R*,1*S* = 1:1; *trans*)	32 – 36%	≤ 3%
Diastereoisomer IV (1*R*,3*S*,1*S* + 1*S*,3*R*,1*R* = 1:1; *trans*)	21 – 25%	57 – 67%

PHYSICAL AND CHEMICAL PROPERTIES

Pure active ingredient

Property	Description or results	Reference
Physical state, colour	all isomers, pure: colourless crystals active substance as manufactured: brown, oily, viscous mass with crystalline parts	
Odour	all isomers, pure: none active substance as manufactured: like amygdaline	
Melting point	Diastereoisomer I (98.7%) 64.4 °C Diastereoisomer II (99.2%) 80.7 °C Diastereoisomer III (98.1%) 65.0 °C Diastereoisomer IV (99.8%) 106.2 °C	Krohn, 1984 PC 180
Boiling point	Not measurable, decomposition above 220 °C	Padberg, 1981 PC 181
Relative density	1.281 g/cm^3 at 20 °C (cyfluthrin, 97.2%) 1.346 g/cm^3 at 22 °C (beta-cyfluthrin, 97.5%)	Padberg, 1981 PC 181 Weber, 1988 PC 124
Vapour pressure	Diastereoisomer I (98.8%): 9.6×10^{-7} Pa at 20 °C Diastereoisomer II (97.4%): 1.4×10^{-8} Pa at 20 °C Diastereoisomer III (97.8%): 2.1×10^{-8} Pa at 20 °C Diastereoisomer IV (98.9%): 8.5×10^{-8} Pa at 20 °C (extrapolation)	Sewekow, 1981a PC 100 Sewekow, 1981b PC 101 Sewekow, 1981c PC 102 Sewekow, 1981d PC 103
Volatility	Henry's law constant at 20 °C (calculated) Diastereoisomer I: 1.9×10^{-1} Pa.m^3.mol^{-1} Diastereoisomer II: 3.2×10^{-3} Pa.m^3.mol^{-1} Diastereoisomer III: 4.2×10^{-3} Pa.m^3.mol^{-1} Diastereoisomer IV: 1.3×10^{-2} Pa.m^3.mol^{-1}	Krohn, 1987a PC 182
Solubility in water including effect of pH	at pH 3: Diastereoisomer I 2.5 µg/L Diastereoisomer II 2.1 µg/L Diastereoisomer III 3.2 µg/L Diastereoisomer IV 4.3 µg/L at pH 7: Diastereoisomer I 2.2 µg/L	Krohn, 1987b PC 109

Property	Description or results	Reference
	Diastereoisomer II 1.9 µg/L	
	Diastereoisomer III 2.2 µg/L	
	Diastereoisomer IV 2.9 µg/L	
	Measurements at 20 °C. Due to hydrolytic instability measurements under alkaline conditions were not possible (mixture 4 diastereomeric enantiomer pairs 94.2%; I 25.1%, II 18.8%, III 31.6%, and IV 21.1%)	
Solubility in organic solvents	Diastereoisomer I (> 98%)	Krohn, 1981 PC 362
	toluene > 200 g/L at 20 °C	
	n-hexane 10 – 20 g/L at 20 °C	
	2-propanol 20 – 50 g/L at 20 °C	
	dichloromethane > 200 g/L at 20 °C	
	Diastereoisomer II (> 98%)	
	toluene > 200 g/L at 20 °C	
	n-hexane 10 – 20 g/L at 20 °C	
	2-propanol 5 – 10 g/L at 20 °C	
	dichloromethane > 200 g/L at 20 °C	
	Diastereoisomer III (> 98%)	
	toluene > 200 g/L at 20 °C	
	n-hexane 10 – 20 g/L at 20 °C	
	2-propanol 10 – 20 g/L at 20 °C	
	dichloromethane > 200 g/L at 20 °C	
	> 200 g/L at 20 °C	
	Diastereoisomer IV (> 98%)	
	toluene 100 – 200 g/L at 20 °C	
	n-hexane 1 – 2 g/L at 20 °C	
	2-propanol 2 – 5 g/L at 20 °C	
	dichloromethane > 200 g/L at 20 °C	
Dissociation constant	Not applicable; the substance does not have acid or alkaline properties.	Krohn, 1988 PC 108
Partition coefficient n-octanol/water	Diastereoisomer I log Pow = 6.0 at 20 °C	Krohn, 1987c M7120
	Diastereoisomer II log Pow = 5.9 at 20 °C	
	Diastereoisomer III log Pow = 6.0 at 20 °C	
	Diastereoisomer IV log Pow = 5.9 at 20 °C	
	pH value not reported	
	(mixture 4 diastereomeric enantiomer pairs 94.2%; I 23.6%, II 17.2%, III 31.6%, IV 21.1%)	

Hydrolysis of cyfluthrin

The hydrolysis of cyfluthrin was studied by Sandie (1983 MR86051) using 0.02 mg/L [phenyl-UL-^{14}C]cyfluthrin in sterile phosphate buffers of pH 5, 7 and 9 containing 1% acetonitrile (25 °C). The rate of hydrolysis proved to be pH dependent. Cyfluthrin was stable at pH 5 with no degradation

observed after 35 days. Linear regression analysis of the data for pH 7 and 9 provided DT_{50} value estimates of 193 days (extrapolated) and < two days, respectively.

Other than cyfluthrin only one major component (FPBald) was identified which accounted for 89% of the radioactivity at pH 9 (day 21), and 11% of the applied radioactivity at pH 7 (day 35). Two minor unidentified hydrolysis products were in the range of 3% or less. The material balance ranged from 97 to 102% (except for the 35 days, pH 7 sample which was 95%).

Krohn (1983 M1590) investigated the hydrolytic stability of cyfluthrin at pH 4, 7 and 9 (sterile citrate, phosphate and borate buffers, respectively and at temperatures from 30 to 80 °C) and analysed for the 4 diastereoisomers. The major hydrolysis products identified by HPLC were 3-(2,2-dichloroethenyl)-2,2-dimethylcyclopropanecarboxylic acid (DCVA) and 3-phenoxy-4-fluorobenzaldehyde (FPBald) (see section on Metabolism and environmental fate for chemical structure).

A hydrolysis study was carried out with cyfluthrin and beta-cyfluthrin (Krohn, 1997a 145000926). The hydrolysis of cyfluthrin and beta-cyfluthrin (4 µg/L) was performed in 0.005 M aqueous buffer solutions adjusted to pH 4, 7 and 9. Under all conditions tested, reversible conversion (epimerisation) of cyfluthrin diastereomer II into diastereomer I and of diastereomer IV into III occurs before degradation by hydrolysis becomes significant. Therefore it is only possible to specify half-lives for the degradation of the sum of diastereomers I and II on the one hand and of diastereomers III and IV on the other hand. The values for 20 °C and 25 °C were calculated by extrapolation from values measured at higher temperatures (50 °C for pH 4; 50 °C, 60 °C and 70 °C for pH 7; 40 °C and 50 °C for pH 9). The resulting half-lives for cyfluthrin/beta-cyfluthrin ranged from greater than one year (pH 4) to 156 days (pH 7, mean value) and greater than two days (pH 9).

Half-lives for hydrolysis of cyfluthrin / beta-cyfluthrin

	Substance	Half-life days		
		pH 4	pH 7	pH 9
40 °C	diastereomer I+II			0.14
	diastereomer III+IV			0.10
50 °C	diastereomer I+II	> 128	3.2	0.048
	diastereomer III+IV	> 84	2.3	0.036
60 °C	diastereomer I+II		0.94	
	diastereomer III+IV		0.65	
70 °C	diastereomer I+II		0.27	
	diastereomer III+IV		0.19	

The linear relationship between the logarithm of the rate constant *k* and the reciprocal value of the absolute temperature T was used to calculate half-lives (t½) for ambient temperatures by linear regression:

$\log k = a - b/T$; $t½ = 0.693/k$

At pH 4 where only measurements at 50 °C had been performed, an estimation under the presumption of a doubling of the half-life for each decrease of the temperature by 10 °C indicated half-lives greater than one year for all diastereomers of cyfluthrin and beta-cyfluthrin.

Results of hydrolysis of cyfluthrin/beta-cyfluthrin

Report	Substance	half-lives (days)		
		pH 4	pH 7	pH 9
Sandie (1983 MR86051) 25 °C	cyfluthrin	Stable (pH 5)	193	< 2
Krohn (1997a 145000926)	diastereomer I+II	> 365	270	< 2
20 °C	diastereomer III+IV	> 365	160	1
Krohn (1997a 145000926)	diastereomer I+II	> 365	120	< 1
25 °C	diastereomer III+IV	> 365	75	< 1

Studies with permethrin (Sharom and Solomon, 1981) show that DCVA is formed as the only relevant degradate of the acid component during hydrolysis. A hydrolysis study starting with DCVA was conducted (Krohn, 1997b 145000921). The hydrolysis of *cis*- and *trans*-DCVA was performed in 0.01 M aqueous buffer solutions adjusted to pH 4, 7 and 9. DCVA was found to be stable at 50 °C at pH 4, 7 and 9, corresponding to a half-life greater than one year at 25 °C.

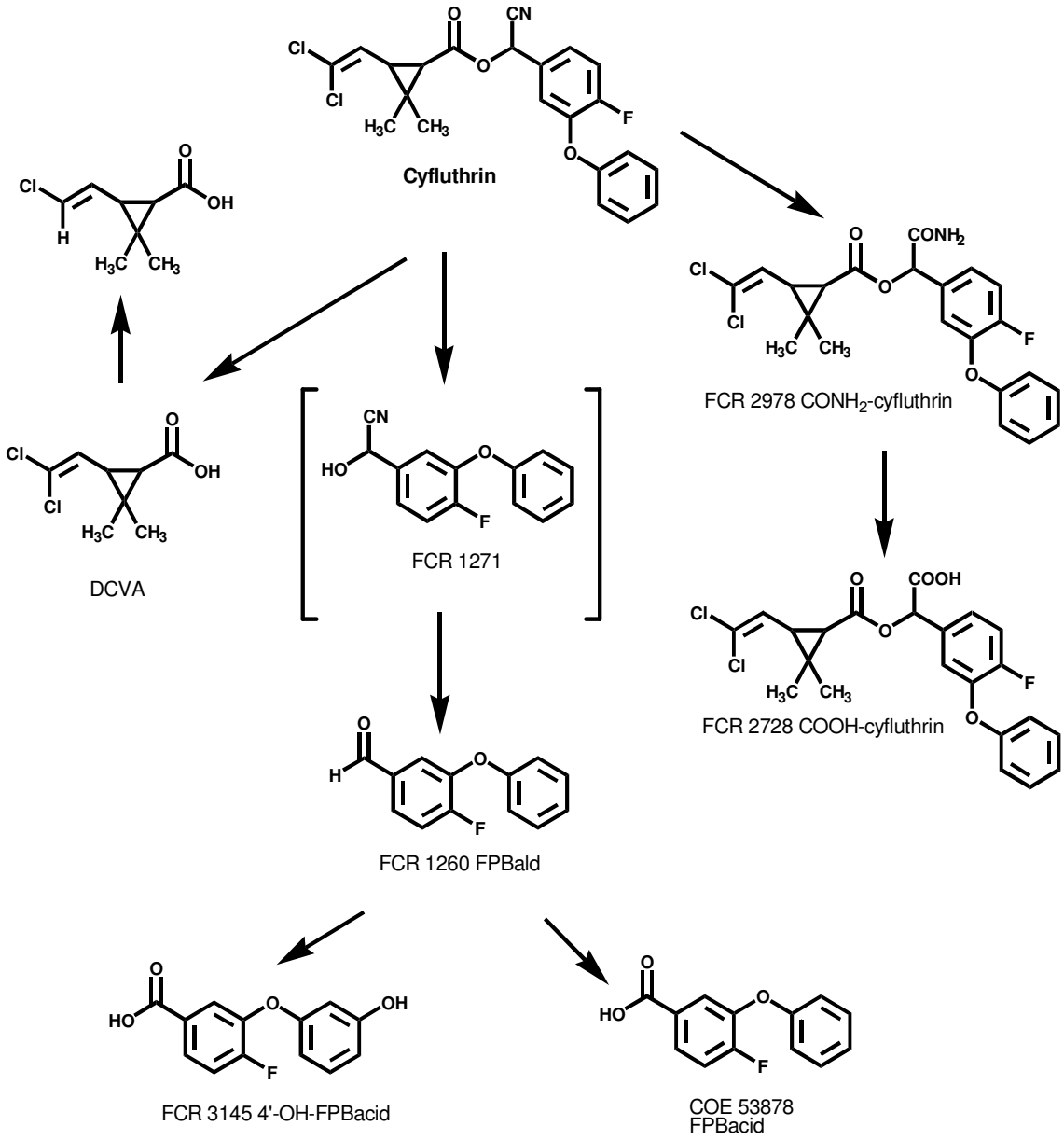


Figure 1. Proposed hydrolysis pathway for cyfluthrin.

Photolysis of cyfluthrin

The photolysis of cyfluthrin in aqueous solutions was studied in the laboratory using a medium-pressure mercury vapour lamp as the light source (Hellpointner 1991 PF-3555). The quantum yield was 0.0052. Puhl *et al.,* (1983 86182) determined the half-life for degradation of 12 days for [phenyl-

^{14}C]cyfluthrin on irradiation of aqueous solutions using a medium-pressure mercury vapour lamp (6700 μW/cm at the sample surface).

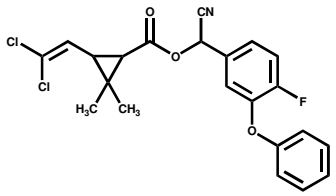
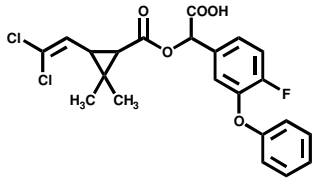
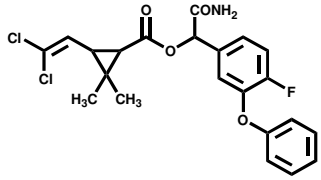
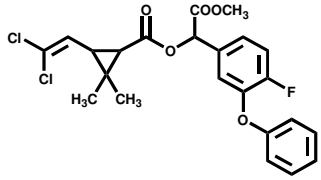
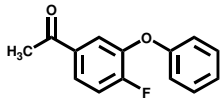
Gronberg (1984, 88598) studied the photolysis of [phenyl-UL-^{14}C]cyfluthrin by natural sunlight (August/September, Kansas USA, 38°49' north). Degradation occurred by cleavage of the ester bond and formation of FPBald and 4-fluoro-3-phenoxybenzoic acid (FPBacid) as the major photoproducts. The half-life for degradation by natural sunlight was biphasic with an initial fast rate, t½ < 1 day followed by slower declines in residues (t½ approximately7 days).

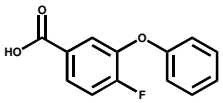
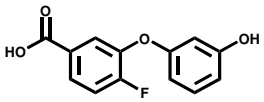
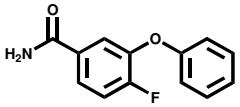
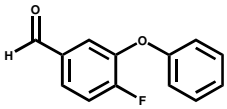
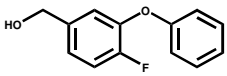
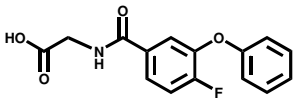
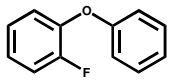
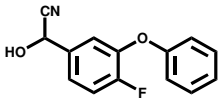
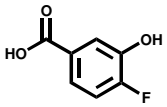
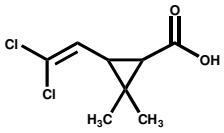
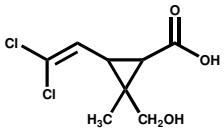
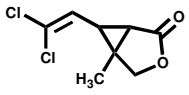
FORMULATION

Code	Description	Active Ingredient Content (g/L)
EC	Emulsifiable concentrate	Cyfluthrin 50 g/L
EC	Emulsifiable concentrate	Cyfluthrin 100 g/L
EC	Emulsifiable concentrate	Beta-cyfluthrin 25 g/L
EC	Emulsifiable concentrate	Beta-cyfluthrin 50 g/L
SC	Suspension concentrate	Beta-cyfluthrin 125 g/L

METABOLISM AND ENVIRONMENTAL FATE

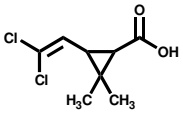
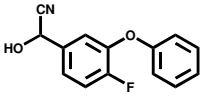
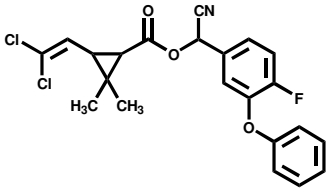
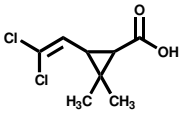
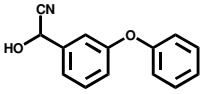
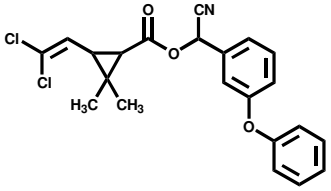
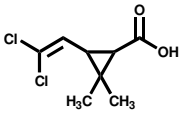
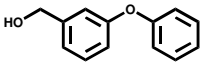
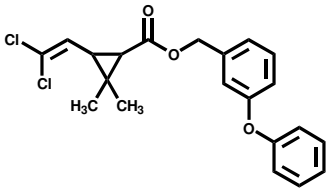
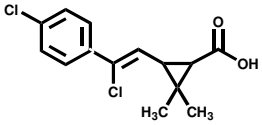
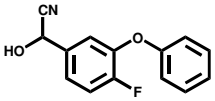
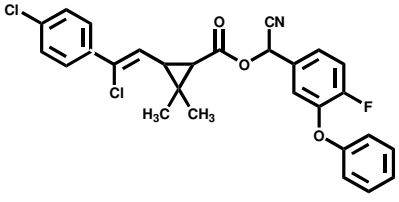
Metabolites are given various abbreviations and code numbers in the studies. Structures and abbreviations and codes are shown below.

Common Name	Code	Designation	Structural formula
cyfluthrin	FCR 1272	cyfluthrin	
α-[[[3-(2,2-dichloroethenyl)-2,2-dimethylcyclopropyl]carbonyl]oxy]-4-fluoro-3-phenoxybenzeneacetic acid	FCR 2728	Acid-cyfluthrin, COOH-cyfluthrin	
2-amino-l-(4-fluoro-3-phenoxyphenyl)-2-oxoethyl 3-(2,2-dichloroethenyl)-2,2-dimethylcyclopropanecarboxylate	FCR 2978	$CONH_2$-cyfluthrin	
Methyl-α-[[[3-(2,2-dichloroethenyl)-2,2-dimethylcyclopropyl]carbonyl]oxy]-4-fluoro-3-phenoxybenzeneacetic acetate	FCR 2956	$COOCH_3$-cyfluthrin Methyl-cyfluthrin	
Methyl 4-fluoro-3-phenoxybenzoate	COE 263/78	Methyl-FPBacid	

Common Name	Code	Designation	Structural formula
4-Fluoro-3-phenoxybenzoic acid or 3-phenoxy-4-fluorobenzoic acid	COE 538/78	FPBacid	
3-(4'-hydroxyphenoxy)-4-fluorobenzoic acid	FCR 3145	OH-FPBacid	
4-Fluoro-3-phenoxybenzamide	FCR 2947	FPBamide	
4-fluoro-3-phenoxybenzaldehyde or 3-phenoxy-4-fluorobenzaldehyde	FCR 1260	FPBald	
3-phenoxy-4-fluorobenzyl alcohol	FCR 1261	FPBalc	
	FCR 3343	Hippuric acid	
1-fluoro-2-phenoxybenzene	FCR 3030	FPB	
4-fluoro-α-hydroxy-3-phenoxybenzene acetonitrile or α -cyano-3-phenoxy-4-fluorobenzyl alcohol	FCR 1271	αOH-FPB-ACN	
3-hydroxy-4-fluorobenzoic acid	FCR 4209	OH-FB	
3-(2,2-dichloroethenyl)-2,2-dimethylcyclopropanecarboxylic acid	DCVA	DCVA; dv-chrysanthemic acid; permethric acid	
cis-3-(2,2-dichlorovinyl)-*trans*-2-hydroxymethyl-*cis*-2-methylcyclopropane-1-carboxylic acid	FCR 4088	OH-DCVA	
trans-3-(2,2-dichlorovinyl)-*cis*-2-hydroxymethyl-*trans*-2-methylcyclopropane-1-carboxylic acid lactone	FCR 4093	OH-DCVA-lactone	

Synthetic pyrethroids can be considered to result from the esterification of an appropriate alcohol with an appropriate acid. Cyfluthrin is a mixture of eight stereoisomeric esters derived from esterification of the dichloroanalogue of chrysanthemic acid, 2,2-dimethyl-3-(2,2-dichlorovinyl)

cyclopropanecarboxylic acid (DCVA), with α-hydroxy-(4-fluoro-3-phenoxyphenyl)acetonitrile (FCR 1271). The same acid component of the ester (DCVA) is common to both permethrin and cypermethrin. In fact the latter differs from cyfluthrin only in the absence of a fluorine substitution on the phenyl ring of the alcohol component: α-hydroxy-(4-fluoro-3-phenoxyphenyl) acetonitrile for cyfluthrin and α-hydroxy-(3-phenoxyphenyl) acetonitrile for cypermethrin. The pyrethroid esters cyfluthrin and flumethrin share a common alcohol component (FCR 1271 α-hydroxy-(4-fluoro-3-phenoxyphenyl) acetonitrile).

Acid component	Alcohol component	Pyrethroid
DCVA 	α -cyano-3-phenoxy-4-fluorobenzyl alcohol 	cyfluthrin 
DCVA 	A -cyano-3-phenoxy-benzyl alcohol 	cypermethrin 
DCVA 	α-hydroxy-3-phenoxy-benzene 	permethrin 
3-(β,4-dichlorostyryl)-2,2-dimethylcyclopropanecarboxylic acid (flumethrin acid, BNF 5533A) 	α -cyano-3-phenoxy-4-fluorobenzyl alcohol 	flumethrin 

Metabolism of pyrethroids proceeds by the hydrolysis of the ester bond to give the constituent acid and alcohol components. Once these acid and alcohol components are formed their metabolism and degradation is independent of the pyrethroid from which they are derived. Consequently the results from metabolism studies on appropriately labelled cypermethrin and permethrin (for the acid component, DCVA) and flumethrin (for the alcohol component, FCR 1271 α -cyano-3-phenoxy-4-

fluorobenzyl alcohol) can be used to support results from studies conducted with radiolabelled cyfluthrin.

Animal metabolism

The Meeting received studies on the metabolism of cyfluthrin in rats, dairy cows and laying hens. The studies on the metabolism of cyfluthrin in animals using radiolabelled material were conducted with the phenyl-UL-^{14}C- and fluorophenyl-UL-^{14}C labelled parent compound. The studies on rats were evaluated by the WHO Core Assessment Group (JMPR 2006).

The studies on rats showed cyfluthrin is well absorbed and rapidly eliminated from the body. More than 97% of the orally administered dose was eliminated after two days, one third in bile within two days. Part of the radioactivity eliminated in the bile was subject to enterohepatic circulation. The highest levels of radioactivity were found in fat, kidney and liver. Major metabolites identified in faeces and urine were a conjugate of 3-(4'-hydroxyphenoxy)-4-fluorobenzoic acid (OH-FPBacid) (35 – 52% of recovered radioactivity), its free form (3.0 – 11%) and FPBacid (8.3 – 24%). The first step in the process of biotransformation is the cleavage of the ester bond and oxidation to FPBacid, which then undergoes further hydroxylation and conjugation to glycine with formation of the relevant hippuric acid derivatives. Depending upon the dose groups, unchanged parent compound and metabolites account for 65 – 82% of the recovered radioactivity with 4 – 8% of the radioactivity remaining unextracted.

Lactating cow

Shaw *et al.,* (1983 MR86043) dosed orally, by gelatine capsule, a lactating dairy cow (Holstein, 484 kg bw) with [phenyl-UL-^{14}C]cyfluthrin at 0.5 mg/kg bw/day for five consecutive days. Assuming feed consumption of between 3.5 and 4% bodyweight this corresponds to 12 – 17 ppm in the diet. Milk was collected twice daily. The animal was slaughtered approximately 16 hours after the last dose and tissue samples collected (brain, heart, liver, kidney, omental fat, subcutaneous fat, renal fat, round muscle, flank muscle and loin muscle). Radioactivity in all samples was quantified and residues characterized by TLC and HPLC. Additionally, extracts from liver were subjected to analysis by GC-MS. Milk samples were extracted with acetone/chloroform (2:1), the extract evaporated to dryness and the residue taken up in hexane and partitioned with acetonitrile for analysis by TLC and HPLC. Tissue samples were homogenised and extracted with acetone/chloroform (2:1) (2 ml HCl added to liver and kidney samples), filtered, the filtrate evaporated to dryness and the residue dissolved in acetonitrile for assay. Fat samples were mixed with sodium sulphate, homogenised with hexane, filtered and the filtrate evaporated to dryness and redissolved in acetonitrile for assay. Identification of metabolites was by comparison of retention times against those of authentic standards.

Radiocarbon content in various tissues was highest in liver (0.62 mg/kg), fat (0.20 mg/kg) and kidney (0.19 mg/kg) and lowest (< 0.1 mg/kg cyfluthrin equivalents) in other tissues. The majority of radioactive residues were extracted with the organic solvents used, with the parent compound accounting for ≥ 93% of the extracted residue in muscle, fat and milk. In addition to the parent compound, 29 and 43% of the radioactivity in heart and kidney respectively was identified as FPBalc. Residues in liver were identified as FPBald (14%) and unchanged parent compound (86%).

Table 1. Distribution of total radioactive residue (cyfluthrin equivalents) and metabolites identification in different organs, tissues and milk after oral dosing of [phenyl-UL-^{14}C]cyfluthrin to dairy cows

		Distribution of solvent extracted radioactivity (%TRR)			
Organ / Tissue	TRR (mg/kg)	Percent Extracted	cyfluthrin	FPBald	FPBalc
Muscle, round	0.022	99	99	ND	ND
Muscle, should.	0.021	98	98	ND	ND
Muscle, loin	0.028	100	100	ND	ND
Fat, renal	0.23	100	100	ND	ND
Fat, subcutaneous	0.12	93	93	ND	ND

Organ / Tissue	TRR (mg/kg)	Distribution of solvent extracted radioactivity (%TRR)			
		Percent Extracted	cyfluthrin	FPBald	FPBalc
Fat, omental	0.23	96	96	ND	ND
Heart	0.040	100	71	ND	29
Kidney	0.19	99	56	ND	43
Liver	0.62	100	86	14	ND
Brain	0.015	-	-	-	-
Milk	0.039 – 0.079	98	98	ND	ND

Laying hen

White Leghorn laying hens (1.3 kg) were orally dosed with 5 mg/kg bw of phenyl-UL-^{14}C-cyfluthrin/hen/day by gelatine capsules for three consecutive days (Chopade *et al.,* 1983 MR86044). Assuming a daily feed intake of 5 – 7% of bodyweight, the daily dose would be equivalent to 71 – 115 ppm in the diet. Eggs were collected daily. The animals were sacrificed two hours after the last dose and tissues collected (liver, heart, kidney, gizzard, fat (renal, omental, and subcutaneous), breast muscle, leg and thigh muscle, and skin). Radioactivity in eggs and tissues was quantified and characterized by TLC, GC and GC/MS.

Samples (except fat) were homogenised and extracted with acetone/chloroform (2:1) and 2 ml HCl, filtered, the filtrate evaporated to dryness, the residue extracted with acetonitrile/hexane (1:1) and the acetonitrile and hexane extracts analysed separately.

The solids remaining after solvent extraction were refluxed with 6 N HCl for two hours. Fat samples were processed as in the dairy cow metabolism study.

TRR in eggs collected at 24 h intervals after commencement of dosing were < 0.01, 0.01, 0.02 and 0.05 mg/kg, expressed in cyfluthrin equivalents.

Table 2. Distribution of total radioactive residue (cyfluthrin equivalents) and identification of metabolites in different organs, tissues and eggs after oral dosing of [phenyl-UL-^{14}C]cyfluthrin to laying hens

Organ / Tissue	TRR (mg/kg)	Distribution of solvent extracted radioactivity (%TRR)							%TRR
		CH_3CN	hexane	cyfluthrin	FPBacid	OH-FPBacid	COOH-cyfluthrin	Unknown [a]	Unextracted [b]
Liver	3.0	53	7	12	12	10	1	25	40
Kidney	4.7	54	7	9	11	12	1	28	39
Gizzard	1.6	85	1	40	13	11	0	22	14
Muscle [c]	0.2	81	0	39	15	11	0	16	19
Muscle [d]	0.3	82	0	21	21	20	0	20	18
Skin	0.4	76	3	28	19	13	0	19	21
Heart	0.4	79	2	16	26	19	0	20	19
Fat	0.1 – 0.2	80	3	75	3	0	2	3	17
Eggs (96 h)	0.05	60	15	56	4	7	6	2	25

a - Unknown radioactivity consists of two metabolites (U1 and U2) and TLC origin; only one metabolite reached a level of 12% (kidney), usually levels were ≤ 7% of the total radioactivity, in eggs U1 and U2 were not detected

b - acid hydrolysis increased the extracted radioactivity by 6 – 12%, identification increased by 2 – 4%, mainly FPBacid and OH-FPBacid

c - breast

d - leg + thigh

No conjugates were identified after enzyme hydrolysis (β-glucuronidase, arylsulfatase and protease) of polar residues remaining at the origin of the TLC plate.

In addition to unchanged parent compound which accounted for 9 – 75% of the total radioactivity depending on the tissue, FPBacid and OH-FPBacid were identified as main metabolites. The highest levels of these metabolites were found in muscles, gizzard, skin and heart. COOH-cyfluthrin was found in eggs (6%) and also in trace amounts in liver, kidney and fat. Of the solvent extracted ^{14}C, except for U1 in kidney (12%) no other single unidentified metabolite was present at > 7% of the total radioactive residue. Up to 40% of the total radioactivity was not extracted with organic solvents. Acid hydrolysis released radioactivity which was mainly attributed to FPBacid and OH-FPBacid, presumably present as conjugates.

In a further study the structure of metabolites in the excreta of three White Leghorn hens (1.3 kg) following a single oral administration of unlabelled cyfluthrin at 3000 mg/kg body weight in Cremophor®/EL/water was elucidated by mass and NMR spectra (Eben *et al*., 1987, 15849). The isomer ratio of cyfluthrin eliminated in faeces did not change appreciably compared to that of the oral dose. In addition to the parent compound (26 – 40%), more than 20 metabolites whose structures could be elucidated were found in the excreta collected in the 14-day period following dosing. OH-FPBacid (FCR 3145), a corresponding dihydroxylated compound and 3-hydroxy-4-fluorobenzoic acid (FCR 4209) were detected as the main metabolites of the fluorophenyl fraction of the cyfluthrin molecule. All three main metabolites occurred in both free and conjugated (glucuronide and sulphate) forms. No conjugates with amino acids were found. DCVA, the corresponding acid amide and the permethric acid derivatives (oxidised at the methyl groups of the C2 of the cyclopropane ring to the corresponding alcohol or acid) were identified as main metabolites of the DCVA fraction.

Summary of metabolism of cyfluthrin in animals

Although metabolites identified in different animal species differ to some extent, the basic metabolic steps are the same. Following absorption, cyfluthrin is widely and rapidly distributed to most tissues, particularly those with high lipid contents. The first step in the process of biotransformation of cyfluthrin in animals is the cleavage of the ester bond and the formation of the acid (DCVA) and presumably the alcohol component (α -cyano-3-phenoxy-4-fluorobenzyl alcohol FCR 1271), a postulated intermediate.

Oxidation of the alcohol component gives FPBald which may be hydroxylated to yield FPBalc or FPBacid (COE 538/78), which then undergoes further hydroxylation to OH-FPBacid and conjugation or combines with glycine to form hippuric acid derivatives (FCR 3343). In addition "COOH-cyfluthrin" (FCR 2728) is formed in laying hens.

The alcohol component of cyfluthrin is also found in the related pyrethroid flumethrin. The results on the metabolism of the alcohol component are consistent with those observed for flumethrin as reported by the 1996 JMPR. The metabolism of this component in flumethrin yields FPBacid (COE 538/78) which is oxidized to OH-FPBacid (both of which are conjugated with glycine).

The metabolic fate of the substituted cyclopropanecarboxylic acid component (DCVA) of cyfluthrin was not specifically studied except in excreta of laying hens that received a single high dose. DCVA, the corresponding acid amide and the permethric acid derivatives oxidised at methyl groups of the C2 of the cyclopropane ring (to the corresponding alcohol or acid) were identified as main metabolites of laying hens.

DCVA is also released on hydrolysis of the ester bond of the related pyrethroids, permethrin and cypermethrin, and studies on the metabolic fate of the acid component of these compounds in mouse, rat, cow and hen are relevant to cyfluthrin. In the related pyrethroids, DCVA undergoes hydroxylation at the methyl groups which may also form various conjugates (glucuronide, glycine and taurine) (Casida *et al.,* 1978; Miyamoto *et al.,* 1981, Gaughan *et al.,* 1977). The lactone of the *cis*-HO-DCVA, *cis*-HO-DCVA-lactone, and its glucuronide were tentatively identified (Gaughan *et al*., 1978, Hutson *et al.,* 1981).

A proposed metabolic pathway in animals is given in Figure 2.

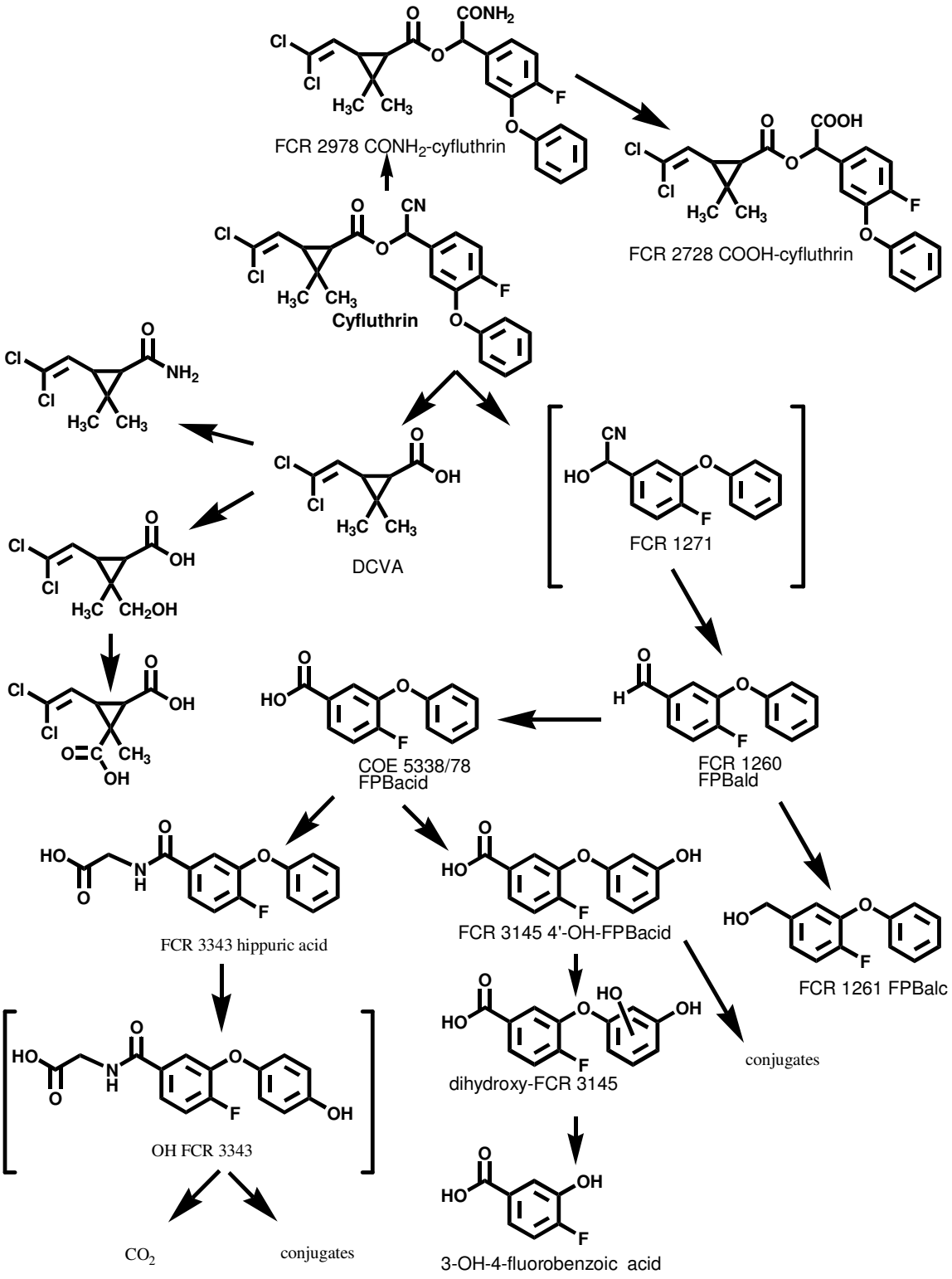


Figure 2 Proposed metabolic pathway for cyfluthrin in animals

Plant metabolism

Metabolism studies on cotton, wheat, soya bean, potato and apple were made available to the Meeting. With the exception of wheat where both labels in both parts of the molecule were applied ([cyclopropyl-1-^{14}C]cyfluthrin and [phenyl-UL-^{14}C]cyfluthrin), studies used cyfluthrin labelled in one of the phenyl rings.

Apples

Individual apple fruit were treated one month before harvest with [phenyl-UL-^{14}C]cyfluthrin (240 EC) at a concentration of 0.3 g ai/l which corresponded approximately to a tenfold field rate (Minor & Freeseman, 1985, 88833). A wetting agent (Ortho X-77) was included in the spray mix to assist application. A canopy of polyethylene sheeting was used to protect the apple tree (*Pyrus malus*, Red Rome Beauty) from direct rainfall. Ten apples were collected at days 0, 7, 14, 21 and 28 after treatment; sample weights ranged from 790 – 1160 g.

After sampling, the apples were individually rinsed by swirling each apple with methanol/water (4:1). The rinses were then concentrated, water added and the residue extracted with chloroform/acetone (1:2). The rinsed apples were separated into peel and pulp and the peel extracted with chloroform/acetone. The rinse and peel extracts were subjected to analysis by TLC.

Table 3. Distribution of radioactivity in apples harvested from trees with fruit treated with [phenyl-UL-^{14}C]cyfluthrin

	%TRR				
DAA	0 days	7 days	14 days	21 days	28 days
MeOH/water rinse	96	44	23	22	16
Peel	2	52	73	74	80
Pulp	2	4	4	4	4
Total	100	100	100	100	100

DAA=days after application

Table 4. Identification of radioactivity in apples harvested from trees with fruit treated with [phenyl-UL-^{14}C]cyfluthrin

	%TRR at different days after last application				
Component	0 days	7 days	14 days	21 days	28 days
MeOH extract (rinse)	96	44	23	22	16
cyfluthrin	89	35	16	16	11
FPBald	2	2	2	1	2
FPBacid	0	< 1	< 1	< 1	< 1
Water soluble	2	1	1	1	1
Unidentified	2	6	4	4	2
Peel	2	52	73	74	80
cyfluthrin	2	48	67	67	73
Unidentified	0	4	6	7	7
Pulp [a]	2	4	4	4	4
Total	100	100	100	100	100

a - The radioactivity in the pulp was not characterized because of the low levels of radioactivity.

The analysis of the total radioactive residue showed that there was a decrease in the radioactivity which was found in the surface rinse with time (96% at day 0, 16% at day 28) while the radioactivity in the peel increased steadily from 2 to 80%. All of the radioactivity in the peel could be extracted. The radioactivity in the apple pulp was 4% or less throughout the study. This may be a result of contamination from the peel during sample preparation as there was no increase in the radioactivity in the pulp during the study. Due to the low levels of radioactivity in pulp it was not characterized further.

The degradation of cyfluthrin on apples progressed at a slow rate. Of the radioactivity present in the apples (surface rinse + peel) at 28 days after application, 84% was still identified as unmetabolized cyfluthrin and 2% as FPBald. Several other minor metabolites were formed of which only FPBacid could be identified. The unidentified radioactivity comprised only 6 to 16% TRR.

Tomato

In a greenhouse study, leaves and fruit of a tomato plant were treated with [[fluorophenyl-UL-14C]cyfluthrin (42% cis, 58% trans). Samples of leaves were collected 14, 28 and 35 days after application and fruit at 1, 5, 7, 9, 14, 21, 28 and 35 days after application. Tomatoes and leaves were individually rinsed with acetone. The rinses were then concentrated and radioactivity measured. Radioactivity in washed fruit and leaves was extracted with acetone, water added and the solution partitioned against CH2Cl2. Radioactivity was measured in both the aqueous and CH2Cl2 phases. The rinse and fruit/leaf extracts were subjected to analysis by TLC.

The majority of the applied radioactivity was recovered on the acetone rinse (92.6%).

Table 5. Characterisation of radioactivity in tomato leaves and fruit (%TRR) sampled from plants treated with [fluorophenyl-UL-^{14}C]cyfluthrin (Wagner and Neitzel 1986)

	DALA											Total[a]
	1	5	7	9	14		21	28		35		
Sample	Fruit	Fruit	Fruit	Fruit	Fruit	Leaves	Fruit	Fruit	Leaves	Fruit	Leaves	
Acetone	94.9	86.5	96.3	89.0	79.4	93.3	94.1	96.4	94.5	85.8	96.8	92.6
CH_2Cl_2	4.35	7.30	2.74	8.54	16.2	3.33	4.90	1.19	2.45	7.80	0.768	2.21
Aqueous	0.725	0.562	0.457	1.22	1.47	0.833	0.980	1.79	0.613	2.31	0.377	0.70
Unextracted	< 0.72	5.62	0.457	1.22	2.94	2.5	< 1	0.595	0.613	0.289	0.782	2.27
Total	100	100	100	100	100	100	100	100	100	100	100	97.8

a - total = percentage of applied radioactivity

Unchanged cyfluthrin accounted for > 91% of the TRR present in acetone rinses of fruit and leaf samples from 1 to 35 days after application and was the only component in the CH_2Cl_2 extracts. The aqueous extract of the leaves revealed the presence of a metabolite (45%; not further identified), but which in total only accounted for 0.12% of the applied radioactivity.

Potato

In a greenhouse study potato plants were treated 60 days after planting (beginning of blooming) with [phenyl-UL-^{14}C]cyfluthrin (200 EC) at a rate of approximately 0.1 kg ai/ha (40 g ai/ac). The soil surrounding the plants was covered to prevent uptake of radioactivity from soil (Minor and Ernst, 1983, MR86053). Samples of foliage and tubers were taken at 0, 42, 52, 80 and 98 days post-treatment.

TRR increased from 7.0 mg/kg (expressed as cyfluthrin equivalents) to 26 mg/kg during the experiment due to natural drying of the foliage. At all sampling times the tubers contained residues of 0.01 mg/kg or less.

Table 6. Total radioactive residue in potato foliage and tubers (expressed as mg/kg cyfluthrin equivalents) following application of [phenyl-UL-^{14}C]cyfluthrin

Crop	Crop Part	Days after Application				
		0 days	42 days	52 days	80 days	98 days
Potato	Foliage (mg/kg)	7.0	9.0	11	12	26
	Tubers (mg/kg)	< 0.01	< 0.01	0.01	< 0.01	< 0.01

Table 7. Identity of radioactive residue in potato foliage (expressed as %TRR) following application of [phenyl-UL-^{14}C]cyfluthrin

Component	Days after application				
	0 days [a]	42 days [a]	52 days [a]	80 days [b]	98 days [b]
cyfluthrin	95	86	83	80	70
FPBald	0	1	1	1	2
FPBalc	0	1	1	4 (2)	4 (2)
FPBacid	0	1	1	2 (1)	1
4'-OH-FPBacid	0	0	0	4 (3)	3 (2)
FPB	0	0	0	1 (1)	3 (2)
Unidentified	5	10	12	8	12
Unextracted	0	1	2	0	4
Total	100	100	100	100	99

a - Extraction with methanol/water followed by direct TLC of the concentrated extract.

b - Extraction with methanol/water followed by acid hydrolysis of polar material isolated (6 N HCl) followed by chloroform/acetone and water partitioning and TLC.

Figures in brackets are percent of total identified as conjugated.

At day 98 after application 95% of the radioactive residue was extracted from leaves. Besides unmetabolized cyfluthrin (70%) the following metabolites were identified: FPBalc, FPBacid, OH-FPBacid and FPB (all < 5% TRR, present as free and conjugated forms). A total of 12% of the radioactive residue at day 98 was not identified.

Soya bean

In a greenhouse study, soya beans (*Glycine max. (L.) Merr.*) were treated with [phenyl-UL-^{14}C]cyfluthrin (200 EC 40:60 *cis:trans* ratio) at a rate of approximately 0.1 kg ai/ha (40 g ai/ac) at the beginning of blooming (*ca.* 40 days after planting). Whole plants were harvested 4, 19, 33, 48, 62, 84 days after application (Minor & Ernst, 1983, MR86049). At the final sampling date (88 days post-treatment) plants were divided into leaves, stalks, pods and seeds. Samples were analysed for radioactivity using scintillation detection and methanol extracts by TLC and HPLC. Characterisation of radioactivity was by comparison of retention times with authentic standards.

TRR (expressed as cyfluthrin equivalents) was 61, 2.5, 0.22 and 0.04 mg/kg, respectively in leaves, stalks, pods and seeds. At least 85% of the radioactivity in the leaves and stalks and 67% in the pods could be extracted with methanol: water (4:1).

Table 8. Identity of radioactive residues in soya foliage and pods (expressed as mg/kg cyfluthrin equivalents) following application of [phenyl-UL-^{14}C]cyfluthrin

	Days after last application								
	4	19	33	48	62	84	88	88	88
	Whole plant	Whole plant	Whole plant	Whole plant	Whole plant	Whole plant	Leaves	Stalks	Pods Without Seeds
TRR (mg/kg)							61	2.5	0.22
					%TRR				
Cyfluthrin	92	81	76	73	59	61	43	51	55
FPBald	1	4	3	4	5	4	1	1	0
4'-OH-FPBacid[a]		< 1	< 1	< 1	2	2	3	2 (1)	0
FPBacid[a]		3	5	5	7	8	4	10 [b] (4)	0

	Days after last application								
	4	19	33	48	62	84	88	88	88
	Whole plant	Whole plant	Whole plant	Whole plant	Whole plant	Whole plant	Leaves	Stalks	Pods Without Seeds
FPBalc[a]		7	8	8	10	9	4		0
Me-FPBacids[a]		1	1	1	1	1	5	0	0
FPBamide		0	0	0	0	0	3 (3)	2	0
FPB		0	0	0	0	0	1	1	0
COOH-cyfluthrin		0	0	0	0	0	0	4 (0)	0
Unidentified		2	2	4	8	3	10	15	11
Unextracted		2	5	5	8	12	17	14	34
Total		100	100	100	100	100	100	100	100

NA = polar material not analysed.

Figures in brackets are percent of total identified as conjugated.

a - Polar material released after HCl hydrolysis. Small amounts of cyfluthrin and FPBald were released by hydrolysis and are likely to be occluded residues

b - Not able to differentiate between FPBacid and FPBalc.

Unchanged cyfluthrin was the major component of the radioactivity, accounting for 43% of the methanol extracted radioactivity in the leaves, 51% in the stalks and 55% in the pods at 88 days after application. The amount of parent cyfluthrin decreased from 92% of methanol extracted radioactivity (day 4) to 61% (day 84). Several minor metabolites were formed and most of them were present mainly as conjugates. They include: FPBald, FPBalc, FPBacid, OH-FPBacid, COOH-cyfluthrin, methyl-FPBacid, FPB and FPBamide. No single metabolite exceeded 10% of the extracted radioactivity. The low TRR in pods were assumed to arise from contamination by surrounding treated foliage.

The major degradation products identified in plants from the glass house experiments were also identified in separate experiments with soya bean tissue cultures. Analysis of the tissue suspensions after incubation for 19 days with ^{14}C-cyfluthrin indicated that the majority of the radioactivity was associated with the soya bean cells with < 10% of the applied radioactivity remaining in the broth after centrifugation of the cell suspension. Major components in the broth were cyfluthrin and unconjugated FPBacid. Methanol/water extracts of the soya bean tissue showed that the major component of the ^{14}C residue was cyfluthrin with several polar components. Acid hydrolysis of the polar material yielded three main metabolites, FPBalc, FPBacid and OH-FPBacid. Control solutions showed little or no degradation of cyfluthrin.

Cotton

In a greenhouse study, cotton plants (*Gossypium hirustum* variety Coker 310) were treated with [phenyl-UL-^{14}C]cyfluthrin (200 EC) at a rate of approximately 0.1 kg ai/ha (40 g ai/ac) (Minor & Ernst, 1983, MR86048). Leaves were collected at 0, 7, 14, 21, 35, 49 and 63 days after application. A second set of cotton plants were treated and removed from the glasshouse daily to expose plants to natural sunlight. Leaves from these plants were sampled at 7, 22 and 31 days after treatment.

At harvest cotton bolls were separated into gin trash, lint and seeds. Samples were homogenised and extracted with methanol and the extracts analysed for radioactivity. Components were identified using TLC and comparison of retention times with authentic standards. Lint samples were subject to Soxhlet extraction with chloroform/methanol.

Polar extracts were hydrolysed with 6 N HCl

Degradation of cyfluthrin was relatively slow and cyfluthrin was the major compound that could be identified in leaves. For plants raised in the greenhouse, the relative contribution of cyfluthrin to the ^{14}C residue decreased to 64% by 63 days after application. The decomposition of cyfluthrin was accelerated when treated plants were exposed to natural sunlight, with only 61% of the

radioactivity identified as parent compound 37 days post-treatment, compared to 84% on day 35 in plants from the greenhouse plants. No single metabolite exceeded 10% of the total radioactive residue.

Table 9. Characterisation and identification of TRR in cotton leaves harvested at different times after application of [phenyl-UL-^{14}C]cyfluthrin to cotton plants

	%TRR at days after last application [a]									
	Greenhouse Conditions					Outdoor [b]				
	0 days	7 days	14 days	21 days	35 days	49 days	63 days	7 days	22 days	37 days
MeOH extract	99	96	91	85	87	76	70	88	81	68
cyfluthrin	99	96	91	82	84	69	64	88	75	61
FPBald	NA	NA	NA	3	3	7	6	NA	6	7
Polar [c]	NA	NA	NA	6	9	14	17	NA	11	17
FPBalc				4	5	8	10		6	10
FPBacid				1	2	4	5		3	5
Me-FPBacid				1	1	1	1		1	1
4'-OH-FPBacid				0	1	1	1		1	1
Unidentified	NA	NA	NA	4	2	8	10	NA	6	11
Total				95	98	98	97		98	96

a - Extraction with methanol/water blending followed by acid hydrolysis of polar material isolated from origin after running thin-layer chromatography plates

b - Plants were taken out of the greenhouse daily and exposed to natural sunlight.

c - Released from polar material (TLC origin material) by acid hydrolysis. Small amounts of cyfluthrin and FPBald were released by hydrolysis and are likely to be occluded residues

NA = not analysed.

In addition the remaining bolls of ten one metre high cotton plants, for which outer bolls were removed, were treated with labelled cyfluthrin. Plants were allowed to mature in the glasshouse. At harvest, 53 days after application, TRRs (expressed as cyfluthrin equivalents) were 52, 0.1 and 0.03 mg/kg in gin trash, lint and seeds, respectively. More than 69% of the radioactivity could be extracted by homogenisation with methanol, including all of the cyfluthrin present. Parent cyfluthrin was also the major component accounting for 83% of the recovered radioactivity in gin trash and 68% in cotton lint. The presence of the parent compound in the cotton lint could possibly be the result of a contamination from the surrounding gin trash or from a limited amount of translocation.

For three one metre high cotton plants, in which all bolls were removed, leaves were treated with labelled cyfluthrin. TRR of leaves sampled 85 days post-treatment comprised 73% parent compound.

In addition to unchanged cyfluthrin, several minor metabolites were formed (all individually < 10% TRR): FPBald, FPBalc, FPBacid, OH-FPBacid, methyl-FPBacid in both leaves and bolls and lint. Except FPBald they were found only as conjugates.

Table 10. Characterisation and identification of TRR in cotton leaves, trash and lint harvested at different times after application of [phenyl-UL-^{14}C]cyfluthrin to cotton plants

	Leaves (85 days)	Leaves (85 days)	Cotton bolls 53 days	
	A [a]	B [b]	Gin Trash	Lint
TRR (mg/kg)			52	0.1
		%TRR		
MeOH extract	79	75	85	69
Cyfluthrin	73	70	83	68
FPBald	6	1	2	1
FPBalc		2 [c]		
FPBacid		2 [c]		

	Leaves (85 days)	Leaves (85 days)	Cotton bolls 53 days	
	A [a]	B [b]	Gin Trash	Lint
Polar	8	6	**7**	NA
FPBalc	3	3	3	NA
FPBacid	3	1	3	NA
Me-FPBacid	1	1	< 1	NA
4'-OH-FPBacid	1	1	1	NA
Unidentified	8	13	5	NA
TOTAL	95	94	97	

a - Extraction with methanol/water followed by acid hydrolysis of polar material isolated from the origin of thin-layer chromatography plates

b - Extraction with methanol/water followed by acid hydrolysis (6 N HCl) of polar material isolated with acetone/chloroform/water partitioning.

c - This percentage of the total was released without acid hydrolysis and was thus not conjugated.

NA = polar material not analysed.

In a translocation experiment a number of individual leaves of an immature cotton plant were treated with labelled cyfluthrin and untreated leaves sampled after 14 days. There was no translocation of radioactivity to new growth or cotton boll components (gin trash, lint, seeds).

It can be concluded that the degradation of cyfluthrin on cotton is quite slow but the rate is increased after exposure to natural sunlight. There is no translocation of cyfluthrin or its metabolites from the treated area to new growth or boll components.

Analysis of radioactivity in samples that were stored frozen showed no apparent decomposition of cyfluthrin in cotton gin trash or cotton leaves after 105 or 234 days of frozen storage respectively.

Wheat

Two glasshouse experiments were conducted to investigate the metabolism of cyfluthrin in wheat (Minor *et al.,* 1985, MR88832). In the first experiment [phenyl-UL-^{14}C]cyfluthrin (*cis*:*trans* 40:60, 200 EC, 0.099 kg ai/ha was applied once to spring wheat (*Triticum aestivum* Era) at the 2 – 4 leaf stage, and half the plants harvested when at the 4 – 6 leaf stage. The remaining plants received two further applications; one at the 4 – 6 leaf stage and one 21 days before harvest.

Wheat samples were homogenized and extracted with methanol/water, the solvent evaporated and the residue dissolved and partitioned against chloroform/acetone (1:2). The chloroform/acetone phase was separated from the aqueous phase, radioassayed, concentrated and subjected to TLC. The aqueous phase was radioassayed, combined with the filter cake and subjected to a 6 N hydrochloric acid reflux for one hour. The hydrolysate was filtered, and the filtrate was partitioned against chloroform/acetone (1:2). Separation of polar radioactive residues was by Florisil column eluted using solvents of increasing polarity, (I) methylene chloride, (II) methylene chloride/acetone (1:1), and finally (III) methanol. Fractions containing sufficient radioactivity were concentrated, radioassayed and subjected to TLC.

At harvest the immature wheat forage contained TRR (expressed as cyfluthrin equivalents) of 1.4 mg/kg. Radioactive residues were highest in the wheat that received three treatments. In these mature samples, the TRR was 5.1 mg/kg in the wheat heads and 21 mg/kg in the mature wheat forage. With the exception of the wheat heads (11% solids), all radioactivity could be extracted.

The major component present in all wheat tissues was the parent compound. It comprised 65% of the radioactivity recovered from the immature wheat forage and 51% to 69% in the mature wheat heads and straw, respectively. Small quantities of conjugated metabolites were detected (FPBacid, OH-FPBacid, COOH-cyfluthrin) Methyl-FPBacid and $COOCH_3$-cyfluthrin may be artefacts formed during the extraction process. Although 22% to 25% of the recovered radioactivity could not be identified, no single unidentified metabolite exceeded 4% of the radioactivity.

Table 11. Characterisation of radioactivity in wheat treated with [phenyl-UL-^{14}C] cyfluthrin

	Immature wheat forage (4 – 6 leaf stage, 1 application)	Mature wheat straw (3 applications)	Mature wheat heads (3 applications)
TRR (mg/kg)	1.4	5.1	21
		%TRR	
Organosoluble	76	78	53
Aqueous phase	15	9	22
Solids	9	13	25
Total	100	100	100
Hydrolysis of combined aqueous and solid fractions with 6N HCl			
Organosoluble	19	17	28
Aqueous	5	5	8
Solid	0	0	11

Table 12. Identification of radioactivity (%TRR) in wheat treated with [phenyl-UL-^{14}C] cyfluthrin

	Immature	Mature	
Component	Forage	Straw	Heads
MeOH extract			
cyfluthrin	65 (1) [a]	69 (1)	51 (4)
COOH-cyfluthrin	(trace)	(trace)	1 (1)
Polar			
Me-cyfluthrin [b]	1 (1)	1 (1)	3 (3)
Me-FPBacid [b]	1 (1)	1 (1)	2 (2)
FPBacid	3 (3)	3 (2)	4 (4)
4'-OH-FPBacid	(5)	4 (4)	5 (5)
Unknown 1	1(1)	1(1)	1(1)
Unknown 2	3 (3)	3 (3)	3 (2)
Unknown 3	4 (1)	3 (1)	3 (3)
Other Organosoluble [c]	12 (3)	10 (3)	8 (3)
Non-Extracted [d]	5	5	19 [e]
Total	100	100	100

a - Value in parenthesis is percent of total radioactivity identified as conjugated or bound material.

b - Considered to be an artefact of acid hydrolysis.

c - Percent comprised of streaking, origin material and radioactive zones less than 1.

d - Radioactivity retained in the solids and/or aqueous fraction after acid hydrolysis.

e - Includes 11% of the radioactivity that was not extracted from the solids.

In a second study four applications of [phenyl-UL-^{14}C]cyfluthrin (*cis*:*trans* 40:60, 200 EC, 0.099 kg ai/ha) or [cyclopropyl-1-^{14}C]cyfluthrin (50:50 *cis*:*trans*, 200 EC, 0.099 kg ai/ha) were made to wheat heads of glasshouse grown plants. Applications were made at intervals of seven days with harvest of wheat heads and straw one day after the last application.

At harvest, one day after the last of four applications, radioactive residues were in the range of 16 to 27 mg/kg (expressed as cyfluthrin equivalents).

Table 13. Characterisation of metabolites (% of recovered radioactivity) in wheat 1 day after the last of 4 applications of [cyclopropyl-1-^{14}C]cyfluthrin or [phenyl-UL-^{14}C]cyfluthrin to wheat

	Wheat straw		Wheat heads	
	[cyclopropyl-1-^{14}C]	[phenyl-UL-^{14}C]	[cyclopropyl-1-^{14}C]	[phenyl-UL-^{14}C]
TRR (mg/kg)	22	16	27	27
	%TRR			
Organosoluble	86	84	74	72
Non-organosoluble	9	12	18	16
Solid	5	4	10	12
Total	100	100		100
Hydrolysis of combined non-organosoluble and solid fractions with 6N HCl				
Organosoluble	12	14	23	25
Aqueous	2	2	5	3
Solid	0	0	0	0

Table 14. Identification of metabolites (% of recovered radioactivity) in wheat 1 day after the last of 4 applications of [cyclopropyl-1-^{14}C]cyfluthrin or [phenyl-UL-^{14}C]cyfluthrin to wheat

Compound	[Cyclopropyl-1-^{14}C]		[Phenyl-UL-^{14}C]	
	Straw	Heads	Straw	Heads
Cyfluthrin	86 (6 [a])	74 (6 [a])	84 (9 [a])	77 (13 [a])
COOH-cyfluthrin (FCR 2728)	0	5 (5)	1 (1)	1 (1)
$COOCH_3$-cyfluthrin (FCR 2956) [a]	1 (1)	1 (1)	1 (1)	1 (1)
DCVA	4 (2)	7 (6)	-	-
FPBald (FCR 1260)	-	-	1 (1)	1
FPBalc (FCR 1261)	-	-	Trace	Trace
FPBacid (COE 538/78)	-	-	1 (1)	1 (1)
4'-OH-FPBacid (FCR 3145)	-	-	1 (1)	3 (3)
Unknown	9 (6) [b]	13 (5) [b]	11 (2) [b]	16 (6) [b]
not extracted	0	0	0	0
Total %	100	100	100	100

( ): % Compound liberated by acid hydrolysis and assumed to be present as conjugates.

a - May be an artefact or due to occluded residues rather than a conjugate.

b - Large number of metabolites, each < 1% of total radioactivity.

As in the first study, cyfluthrin was the major component found in the extracts and comprised between 74 and 86% of the recovered radioactivity. The (conjugated) metabolites, which all appeared in small amounts (< 7% TRR), were also the same as in the first study with [phenyl-UL-^{14}C]cyfluthrin. It is possible that $COOCH_3$-cyfluthrin is an artefact of the acid hydrolysis. It is supposed that the polar material contained occluded (not conjugated) cyfluthrin that survived acid hydrolysis. The major metabolites detected after application of the [cyclopropyl-1-^{14}C] label were COOH-cyfluthrin and DCVA. They were mainly detected after acid hydrolysis and were considered to be derived from conjugates. Similar metabolites have been described in the literature in plant metabolism studies with structurally similar synthetic pyrethroids cypermethrin and permethrin.

In both wheat experiments several unknown products were detected, but no unidentified metabolite exceeded 5% of the total radioactivity. The same metabolic pattern was found after application of cyfluthrin regardless of the growth stage or application rate. Consistent with other structurally related pyrethroids (permethrin and cypermethrin) DCVA was the main metabolite after application of [cyclopropyl-1-^{14}C]cyfluthrin. DCVA did not exceed 7% of the total radioactivity.

In a separate study the degradation of cyfluthrin was studied on stored grain (Linke and Heukamp 1986). Samples of wheat grain (1 kg, 12.9% moisture content) were treated with [fluorophenyl-UL-^{14}C]cyfluthrin solutions at application rates of 0.4 and 0.3 mg/kg for an EW formulation and 0.8 and 0.3 mg/kg when applied as an acetone solution and stored at 24 -27 °C in the dark. Samples were collected for analysis on the day of application and at 1.5, 3, 4.5, 6 and 9 months of storage. Wheat grain was rinsed with methanol:water 4:1 and the radioactivity released measured to determine surface residues. The rinsed grain was ground and subject to Soxhlet extraction with methanol: water. Radioactivity in the rinses and extracts was characterized by TLC.

The majority of the radioactivity was located on the surface and released into the rinse solutions. Unchanged cyfluthrin was the major component identified in both rinse solutions and Soxhlet extracts. After 9 months of storage, 79% of the TRR was cyfluthrin with a further 1.9% identified as FPBald and 0.8% as FPBalc.

A proposed metabolic pathway is given in Figure 3.

The metabolism of cyfluthrin in plants appears to involve an initial hydrolysis of the ester linkage to yield DCVA and FPBald, the later can then undergo reduction to FPBalc or oxidation to FPBacid. This metabolite in turn may be further hydroxylated to OH-FPBacid, methylated to methyl-FPBacid or converted to FPB or FPBamide. Cyfluthrin may also be converted to COOH-cyfluthrin.

All metabolites except FPBald appear mainly in their conjugated forms.

If cyfluthrin is applied to plants it predominantly remains on the plant surface (fruit, leaves) or is absorbed into the cuticle. Cyfluthrin is not translocated within the plant. The rate of degradation of cyfluthrin on (or in) plants in greenhouse experiments is quite low. Under outdoor conditions (natural sunlight) the rate of degradation is increased.

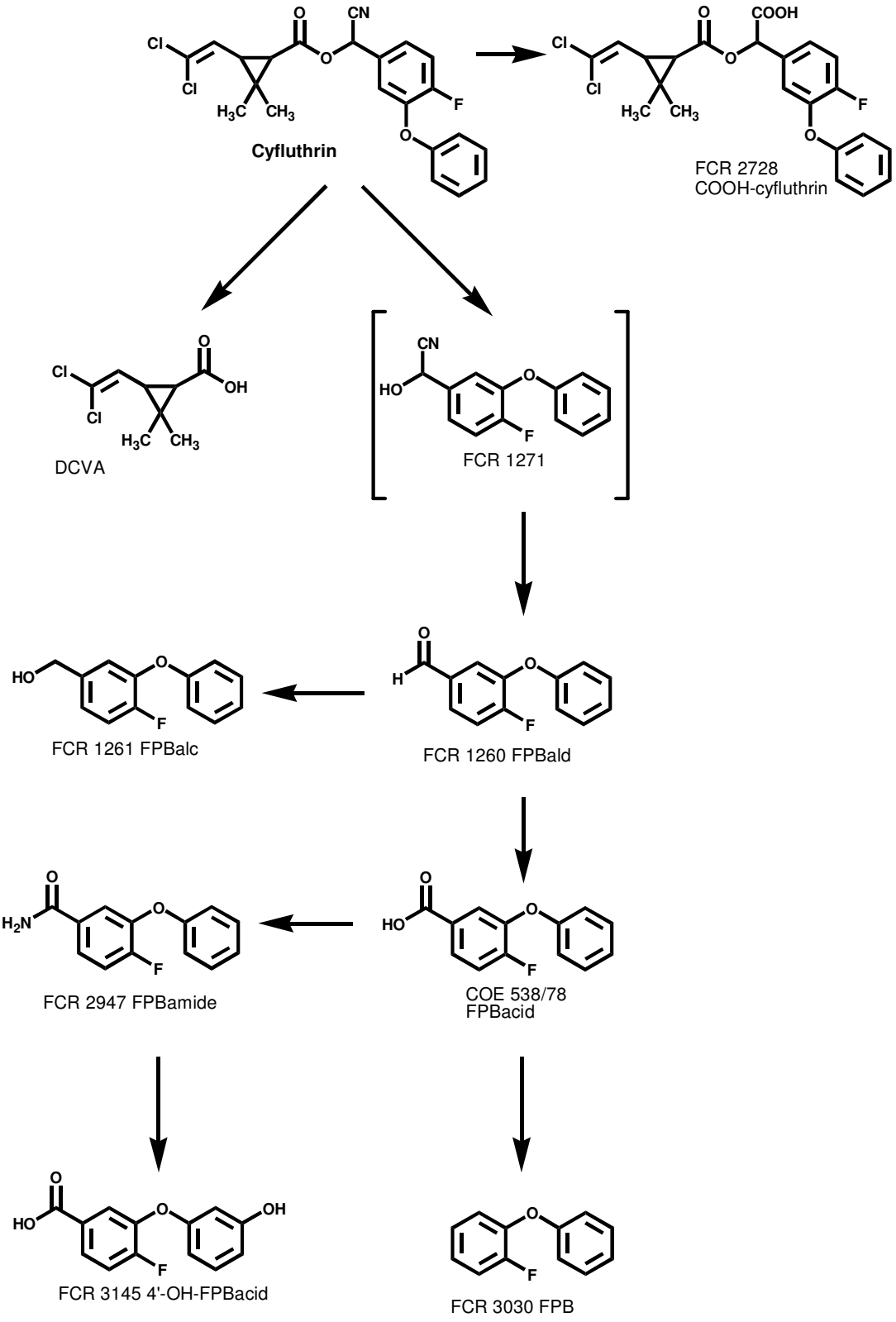


Figure 3. Proposed metabolic pathway of cyfluthrin in plants, conjugates not indicated

Environmental Fate in soil

The Meeting received information on the confined rotational crops, field crop rotation, aerobic and anaerobic soil metabolism and soil photolysis. The fate and behaviour of cyfluthrin in soils was investigated with [fluorophenyl-UL-^{14}C]- or [phenyl-UL-^{14}C]-labelled cyfluthrin. The fate of the acid component of cyfluthrin (DCVA) was extrapolated from data on structurally similar pyrethroids like cypermethrin and permethrin reported in the literature.

Confined crop rotation studies

Information on the fate of radiolabelled cyfluthrin in a confined crop rotation study was made available to the Meeting.

The metabolism of cyfluthrin in confined rotational crops was investigated by Minor and Ernst (1983 MR86050) using [phenyl-U-^{14}C]cyfluthrin (200 EC, *cis:trans* 40:60) in a greenhouse experiment. Cyfluthrin was applied directly to a sandy loam soil (62% sand, 25% silt, 13% clay, % OM 2.6, pH 5.0, CEC 18 meq/100 g, bulk density 2.6 g/cm^3) in a metal trough (0.6 × 2.4 × 0.6 m, soil depth 38 cm) at a rate equivalent to 988 g ai/ha. Following tilling the soil to a depth of 15 cm, rotational crops (kale, red beet and wheat) were planted at 36, 121 and 285 days after soil application. Crops planted at 36 and 121 days were maintained in a greenhouse to prevent frost damage while those planted at 285 days after soil application of ^{14}C-cyfluthrin were maintained outdoors. Table 15 gives the results of the analysis for TRR (expressed as mg cyfluthrin equivalents) in the harvested mature crops.

Of the rotational crops planted at 36 days after soil application of ^{14}C-cyfluthrin, wheat contained the highest residues; 0.35 mg equiv/kg in wheat heads and 0.16 mg equiv/kg in wheat stalks. Radioactive residues in mature crops decreased with each planting. All of the mature crops harvested from planting 285 days after application of ^{14}C-cyfluthrin to soil contained radioactive residues of < 0.01 mg/kg.

TRR in soil declined from the initial value of 0.72 mg/kg after application to be 0.24 mg/kg at 36 days, 0.12 mg/kg at 121 days, 0.15 mg/kg at 285 days and 0.10 mg/kg at 359 days.

Table 15. Total radioactive residue of cyfluthrin (expressed as mg cyfluthrin equivalents/ kg) in succeeding crops after soil application of [phenyl-UL-^{14}C]cyfluthrin

Rotational crop	Planting days after application to soil					
	36 days		121 days		285 days [a]	
	Harvest (days after planting)	TRR (mg/kg)	Harvest (days after planting)	TRR (mg/kg)	Harvest (days after planting	TRR (mg/kg)
Kale	70	0.030	59	0.014	44	0.003
Wheat:	89		83		79	
- Immature Stalks		-		0.056		0.006
- Stalks		0.16		0.16		0078
- Heads		0.35		0.19		0.020
Red Beet:	117		112		57	
- Foliage		0.025		0.027		0.004
- Bulb		0.053		0.019		0.003

a - The final crop was allowed to mature outside

The only crop with sufficient radioactivity for characterisation was the mature wheat sample from crop planted 36 days after application to soil. In the stalks and heads only 31% and 14% of the radioactivity was organosoluble, even after acid hydrolysis. Thin-layer chromatography revealed continuous undefined vertical bands (smears) of radioactivity that could not be related structurally to known cyfluthrin metabolites. The nature of the radioactivity in the acid hydrolysed aqueous fraction (27% in stalks and 54% in heads) and solids (42% in stalks and 32% in heads) was not further characterized.

Field crop rotation studies

A field crop rotation study on cereals was conducted (Leslie, 1988 MR98429; Leslie, 1989 MR98429-1). Following the last of 10 applications of cyfluthrin (240 EC) to silty clay loam (Kansas, plot1 3.7 by 15 m; clay 38%, silt 51%, sand 11%, %OM 4.6, pH 5.3, CEC 43 meq/100 g, bulk density 2.6 g/cm^3, plot 2: clay 34%, silt 58%, sand 8%, %OM 4.4, pH 5.7, CEC 46 meq/100 g, bulk density 2.6 g/cm^3) and silty loam (Mississippi, plot 3.9 by 46 m; clay 26%, silt 61%, sand 13%, %OM 1.4, pH 5.6, CEC 28 meq/100 g, bulk density 2.6 g/cm^3) soils at 28 g ai/ha, winter wheat was sown at 38 and 105 or 135 days after the last application to soil. Samples (mature wheat plus soil) were taken at various intervals after the last application and planting of the crops. Rainfall during the study was reported to be 144 cm for the Kansas site and 143 cm for the Mississippi site.

No residues above the limit of detection were found at the 38, 105 and 135 days plant-back intervals in the mature wheat crop components (green forage, threshed grain and straw). Residues in soil samples (0 – 15 cm depth) taken at the time of sowing were < 0.03 mg cyfluthrin/kg soil. At harvest, soil residues were < 0.01 mg cyfluthrin/kg soil.

Table 16. Residues in rotational crops

Experiment No. (Location) Crop	Sample Type	Plant back interval (days)	Planting to harvest interval (days)	Cyfluthrin (mg/kg)
STF-BD055-86R Wheat (Kansas)	Soil (0 days)			0.06
	Soil (38 days)			0.03
	Forage	38	48	< 0.01
	Grain	38	255	< 0.01
	Straw	38	255	< 0.01
	Soil (harvest)	38		< 0.01
BMS-BD054-86R Wheat (Mississippi)	Soil (0 days)			0.36
	Soil (135 days)			< 0.01
	Grain	135	195	< 0.01
	Straw	135	195	< 0.01
	Soil (harvest)			< 0.01
STF-BD056-86R Wheat (Kansas)	Soil (0 days)			0.24
	Soil (105 days)			0.02
	Forage	105	45	< 0.01
	Grain	105	241	< 0.01
	Straw	120	241	< 0.01
	Soil (harvest)			< 0.01

Soil degradation

Aerobic degradation

The aerobic metabolism of [fluorophenyl-UL-^{14}C]cyfluthrin (*cis/trans* 40:60) in two soils (a loam and a sandy loam) under different conditions was investigated by Wagner *et al.,* (1983, RA-87/83). The soils were taken from the field and their water content was measured and maintained during the studies. [Fluorophenyl-UL-^{14}C]cyfluthrin was thoroughly mixed into soil at a rate of 1 mg/kg, the soil was incubated in the greenhouse in the dark at 18 – 22 °C. In two additional trials the metabolism of [fluorophenyl-UL-^{14}C]cyfluthrin under anaerobic and sterile conditions in the same loam and sandy loam soils was also investigated.

Tables 17 and 18 show the distribution of radioactivity and metabolites on different sampling dates. [Fluorophenyl-UL-^{14}C]cyfluthrin was rapidly metabolized in both soils. Its mineralization rate showed a possible dependence on soil moisture. In soils maintained at 13 – 17% moisture content, 32 to 36% of the applied radioactivity was released as $^{14}CO_2$ at day 190, whereas in the same soils maintained at 8.6 – 11% moisture content only 18% was released as $^{14}CO_2$ by day 365. The formation

of bound or unextracted residues was also much slower in soils with lower moisture content compared to those with higher moisture contents. Cyfluthrin ranged from 15 to 86% of the applied radioactivity for high moisture content soils and 18 – 30% for low moisture content soils. In all cases around 30% of the applied radioactivity was not extracted. The degradates FPBacid, $CONH_2$-cyfluthrin, COOH-cyfluthrin and FPBamide were formed. In the sterile samples no bound residues were formed and essentially all of the radioactivity could be extracted 118 days after application.

Table 17. Recovery of radioactivity and distribution of metabolites after application of [fluorophenyl-UL-^{14}C]cyfluthrin to higher moisture soil (1 mg/kg) and incubation in the greenhouse at 18 – 22 °C

Soil	Water content		% applied ^{14}C				
			14 days	28 days	56 days	84 days	190 days
Laacherhof B (loam)	17%	cyfluthrin	86	60	51	33	15
Clay 23%		FPBald	Traces	ND	ND	ND	ND
Silt 37%		$CONH_2$-cyfluthrin	1	< 1	< 1	2	1
Sand 40%		COOH-cyfluthrin	< 1	< 1	< 1	< 1	< 1
pH 6.2		FPBamide	< 1	< 1	< 1	< 1	< 1
%organic matter 0.95		FPBacid	7	5	3	2	1
CEC 14 meq/100 g		$^{14}CO_2$	6	17	25	20	32
Bulk density 2.6 g/cm^3		Unknown	0	< 1	< 2	< 2	< 2
		unextracted		17	18	34	32
		Total	100	99	97	91	81
Laacherhof C (sandy loam)	13%	cyfluthrin	84	69	47	36	22
Clay 19%		FPBald	Traces	ND	ND	ND	ND
Silt 25%		$CONH_2$-cyfluthrin	1	4	< 1	< 1	1
Sand 56%		COOH-cyfluthrin	< 1	< 1	< 1	< 1	< 1
pH 5.9		FPBamide	< 1	< 1	< 1	< 1	< 1
%organic matter 0.95		FPBacid	7	10	3	5	< 1
CEC 14 meq/100 g		$^{14}CO_2$	5	6	1	23	36
Bulk density 2.6 g/cm^3		Unknown	< 2	0	< 2	< 4	< 1
		unextracted	-	14	21	27	31
		Total	97	103	92	91	89

Table 18. Recovery of radioactivity and distribution of metabolites after application of [fluorophenyl-UL-^{14}C]cyfluthrin to "dry" soil (1 mg/kg) and incubation in the greenhouse at 18 – 22 °C

Soil	Water content		% applied ^{14}C		
			118 days	265 days	365 days
Laacherhof B (loam)	11.2%	cyfluthrin	23	18	19
Clay 23%		FPBald	ND	ND	ND
Silt 37%		$CONH_2$-cyfluthrin	15	7	3
Sand 40%		COOH-cyfluthrin	7	3	< 1
pH 6.2		FPBamide	14	7	< 1
%organic matter 0.95		FPBacid	29	18	3
CEC 14 meq/100 g		$^{14}CO_2$	3	4	18
Bulk density 2.6 g/cm^3		Unknown	6	15	< 2
		unextracted	3	8	29
		Total	100	80	69
Laacherhof C (sandy loam)	8.6%	cyfluthrin	30	21	18
Clay 19%		FPBald	ND	ND	ND
Silt 25%		$CONH_2$-cyfluthrin	22	11	6
Sand 56%		COOH-cyfluthrin	7	4	1
pH 5.9		FPBamide	5	9	2

	Water			% applied ^{14}C	
Soil	content		118 days	265 days	365 days
%organic matter 0.95		FPBacid	31	25	4
CEC 14 meq/100 g		$^{14}CO_2$	1	2	18
Bulk density 2.6 g/cm^3		Unknown	2	19	1
		unextracted	2	9	33
		Total	100	100	83

The half-lives of cyfluthrin for both soils under higher moisture conditions were calculated to be 54 – 63 days. Cyfluthrin was also degraded in soil which was sterilized prior to the beginning of the experiment, although to a lesser extent. At 118 days after application 70% of the applied radioactivity was recovered as unchanged parent compound in the sterile soil experiment.

Imposing anaerobic conditions to samples of the sandy loam, which had been pre-incubated aerobically for 30 days, changed the metabolic pattern with higher amounts of unextracted or bound residues were formed. FPBacid was the only metabolite measured in significant amounts in anaerobic samples (19% at day 30).

Minor (1986, 91816) investigated the metabolism of [phenyl-UL-^{14}C]cyfluthrin (200 EC) in a sandy loam. Soil samples were taken from the confined crop rotation study described above (Minor & Ernst, 1983 MR86050) 36 days after the application of cyfluthrin (988 g ai/ha) to the soil surface (clay 13%, silt 25%, sand 62%, %OC 1.4, pH 5.0). The radioactivity recovered was assumed to be 100% and the further metabolism of [fluorophenyl-UL-^{14}C]cyfluthrin was followed for another 189 days after mixing the soil. The samples were kept outside (Kansas City, USA, Sept. 1982 to Feb. 1983) and watered periodically.

Table 19 gives a summary of the results. A 33% loss of radioactivity occurred within 189 days and was attributed to $^{14}CO_2$. The amount of bound residues increased from 25% at the time of incorporation (day 0) to 42% of the recovered radioactivity (day 189). The amount of unchanged cyfluthrin decreased from 55 to 15% while small amounts of metabolites (FPBacid and $CONH_2$-cyfluthrin) were formed however neither exceeded 8% of the total radioactivity.

Table 19. Distribution of metabolites after application of [phenyl-UL-^{14}C]cyfluthrin (200 EC) to soil (988 g ai/ha), incubation outside

		%radioactivity recovered 36 days after application		
Soil		0 days	70 days	189 days
Sandy loam (from confined rotational crop study)	cyfluthrin	55	35	15
	$CONH_2$-cyfluthrin	4	4	2
	FPBacid	8	3	2
	$^{14}CO_2$ [a]	-	36	33
	Unknown	8	10	6
	unextracted	25	12	42
	Total	100	100	100

a - Decreases in total radioactive residue were assumed to be $^{14}CO_2$ losses

From these data Minor (1986, 91816) calculated the half-life of cyfluthrin to be 105 days.

The fate of the acid component of cyfluthrin (DCVA) in soils can be extrapolated from results of experiments with cypermethrin (Roberts & Standen, 1977 and 1981; Sakata *et al.,* 1986) and permethrin (Kaufman *et al.,* 1977; Kaneko *et al.,* 1978; Jordan *et al.,* 1982).

The degradation of cypermethrin in soils was investigated by Roberts and Standen (1977) who demonstrated that 23.7% of the applied radioactivity ([cyclopropyl-^{14}C]cypermethrin) was evolved as $^{14}CO_2$ within 22 weeks. In a further study on the metabolism of *cis*-and *trans*-[cyclopropyl-^{14}C]cypermethrin in soil (Roberts and Standen, 1981) it was shown that DCVA, the primary metabolite resulting from ester cleavage can undergo further oxidation at one of the methyl groups

yielding the dicarboxylic acid. The intermediate OH-DCVA was only detected in metabolism studies of labelled DCVA when ^{14}C-cypermethrin was applied at very high rates (Roberts and Standen, 1981). Similar results were obtained by Sakata *et al.,* (1986) following application of ^{14}C-cypermethrin to two soils stored at 25 °C and 40% maximum water holding capacity.

The degradation of permethrin was investigated by Kaneko *et al.,* (1978) on a clay soil and a sandy soil and by Jordan *et al.,* (1982) on a sandy loam. Significant amounts of $^{14}CO_2$ were formed after incubation of carbonyl- or cyclopropyl-labelled permethrin implying that opening of the cyclopropyl-ring must have occurred. However, in all of the above mentioned studies DCVA was the only metabolite resulting from this label that was detected in significant amounts (> 10%). Depending upon the compound, isomer, soil type and sampling date DCVA accounted for up to 51% of the applied radioactivity, but typically its concentration was much lower.

The degradation behaviour of DCVA in two soils was followed by Sakata *et al.,* (1992, MO-04-003278). Depending upon the isomer applied the DT50 values in silty loam are in a range of 12 – 23 days and in clay loam in a range of 16 – 62 days. The *trans*-isomer proved to be more stable than the *cis*-isomer under the conditions of the study.

With respect to the degradation rates of *trans*-isomers of DCVA, the 1R isomers degraded faster than the 1*S* isomers in both soils. In the case of *cis*-isomers, there was no significant difference in the degradation rate between 1*R* and 1*S* isomers.

Table 20. Half-lives of four isomers of DCVA in aerobic soils (Sakata *et al.,* 1992 MO-04-003278)

Soil type	DT-50 (days)			
	1*R*,*trans*	1*S*,*trans*	1*R*,*cis*	1*S*,*cis*
Ushiku silty loam [a]	12	23	14	16
Noichi clay loam [b]	31	62	16	16

a - Ushiku silty loam: 43% sand, 47% silt, 10% clay, pH 7.0, 7.6% OM.

b - Noichi clay loam: 55% sand, 26% silt, 19% clay, pH 7.0, 3.3% OM.

Incubation conditions: 25 °C, 40% maximum water holding capacity.

The distribution of unextracted residues within soil organic matter fractions was studied by Kaufman *et al.,* (1977) using carbonyl- and methylene-labelled permethrin in different soils. Irrespective of the soil type and label only small amounts of radioactivity were associated with the humic acid fraction (< 16%). Addition of Na-azide increased the amounts found in the fulvic acid and decreased those in the humin fraction. Microbial activity is important for incorporation of the radioactivity into the humin fraction.

Photolysis on Soil

Puhl *et al.,* (1983 81862) irradiated [phenyl-UL-^{14}C]cyfluthrin on a sandy loam soil (Merrill Oregon, clay 13%, silt 14%, sand 73%, pH 6.6, %OM 2.8, CEC 12.6 meq/100 g, particle density 2.6 g/cm^3) with light from a medium pressure mercury vapour lamp (6000 W/cm^2, filter wavelengths below 290 nm to simulate natural sunlight). Microscope slides with a soil layer (about 0.5 g of soil each) were fortified with cyfluthrin corresponding to 8 mg/kg soil and were irradiated for up to nine days.

Table 21 gives the results of the distribution of photo products and the balance of radioactivity. The degradation of cyfluthrin proved to be biphasic with estimated half-lives for decline of two days and 16 days. Unextracted radioactivity increased during the duration of the experiment and was 7.8% after nine days irradiation. In dark control samples essentially all of the radiocarbon was extracted suggesting that unextracted residues form as a consequence of the photolytic degradation of cyfluthrin. The only photoproducts identified were FPBald and FPBacid which both reached a maximum after two days (10% and 7.0% of applied ^{14}C, respectively). In a separate experiment to identify volatile degradation products using [phenyl-UL-^{14}C]cyfluthrin and [fluorophenyl-UL-^{14}C]cyfluthrin, trace amounts of ^{14}C was identified as phenol for the fluorophenyl label with 3.5% for the phenyl label. Sodium hydroxide trapped more ^{14}C from the phenyl label (13%)

than from the fluorophenyl label (8.4%). The appearance of phenol indicates the ether bond between the two phenyl rings may be cleaved.

Table 21. Balance of radioactivity and distribution of degradation products after irradiation of [phenyl-UL-^{14}C]cyfluthrin on soil

Compound	%applied radioactivity							
	0 days	0.125 days	0.5 days	1 day	2 days	3 days	6 days	9 days
Cyfluthrin	97	82	72	58	54	46	41	36
FPBald	1.4	7.0	2.7	10	10	5.2	5.9	2.1
FPBacid	0.4	2.4	4.7	5.4	7.0	6.6	5.2	4.4
Unknown	1.2	5.0	10	12	15	14	13	12
Unextracted	0	1.2	2.8	6.2	4.6	6.2	7.2	7.8
Total	100	98	92	87	80	78	73	62

Recoveries from control samples were always close to 100%

The photo-decomposition of cyfluthrin on soil by natural sunlight was studied by Chopade (1986 ,88981). Microscope slides with thin layers of a sandy loam (Johnson, Kansas, clay 9%, silt 25%, sand 66%, pH 5.4, %OM 2.2, CEC 16 meq/100 g, particle density 2.6 g/cm^3) were fortified with [phenyl-UL-^{14}C]cyfluthrin at a concentration of 37 mg/kg soil and exposed to natural sunlight (Kansas City, USA, August/September 1985) for up to six days. The half-life of cyfluthrin biphasic in irradiated soil and was 2.1 days and 6.6 days with negligible decomposition occurring in dark controls. Essentially all the radioactivity from control samples was extracted with methanol and consisted mainly of unchanged parent compound (Table 22.). In contrast in the irradiated samples unextracted radioactivity increased steadily to 14% after six days.

Table 22. Soil photolysis of [phenyl-UL-^{14}C]cyfluthrin

	%TRR						
	0 days	1 days	2 days	3 days	4 days	5 days	6 days
Cyfluthrin	99	70	66	58	49	46	43
FPBald	< 1	0	12	15	17	18	18
$CONH_2$-cyfluthrin	-	3	4	6	6	7	7
FPBacid	-	3	3	5	5	6	6
COOH-cyfluthrin	-	2	2	2	3	3	3
FPBalc	-	2	2	2	1	1	1
OH-FPBacid	-	-	< 1	-	1	< 1	1
Unknown	< 1	1	1	2	3	2	2
Unextracted	-	7	10	10	10	12	14
Total	100	98	100	100	95	95	95

The major degradation product was FPBald, increasing steadily to 18% of the applied radioactivity at day 6. In addition $CONH_2$-cyfluthrin, FPBacid, COOH-cyfluthrin, FPBalc and OH-FPBacid were identified, each accounting for ≤ 7% of the applied radioactivity or less (Table 22.).

The soil photolysis of the acid component of cyfluthrin (DCVA) can be extrapolated from studies on permethrin (Holmstead *et al.,* 1978) and cypermethrin (Takahashi *et al.,* 1985). In both studies the compounds were exposed to natural sunlight. In both cases DCVA was the only metabolite resulting from the cyclopropyl moiety that could be identified. However, the levels were quite low (< 0.1 – 1.5% of applied radioactivity). Depending on soil type and compound up to 20% of the applied radioactivity could not be identified and a maximum of 47% was not extracted from soil. In addition, losses (11 – 28% compared to dark controls) occurred. Though not monitored it is assumed that these resulted from the formation of volatile compounds. It is concluded that cyfluthrin adsorbed to soil will be readily degraded if exposed to sunlight.

A proposed metabolic pathway for cyfluthrin in soil is given in figure 4. Besides $^{14}CO_2$, which appears to be the principal aerobic degradation product, the major degradation product is FPBacid. Measurable amounts of FPBald, which is postulated as an intermediate, are only detected in photolysis studies. Only traces are detected shortly after the start of the experiment. In addition, small amounts of $CONH_2$-cyfluthrin, COOH-cyfluthrin, FPBamide, FPBalc and OH-FPBacid and probably phenol are also formed.

Based on results with cypermethrin and permethrin, it is concluded that the primary metabolite resulting from ester hydrolysis will be DCVA, which can be further metabolized via OH-DCVA and the corresponding dicarboxylic acid. Ring opening occurs and finally $^{14}CO_2$ is formed.

The half-life for degradation of cyfluthrin in soil is estimated to be approximately three months. Photodegradation on soil surfaces is fast with half-lives for degradation of cyfluthrin that are < 20 days. Hydrolysis in water is pH dependent. Cyfluthrin is considered stable at pH 4 and 7 but is rapidly hydrolysed at pH 9 with a half-life of < 2 days.

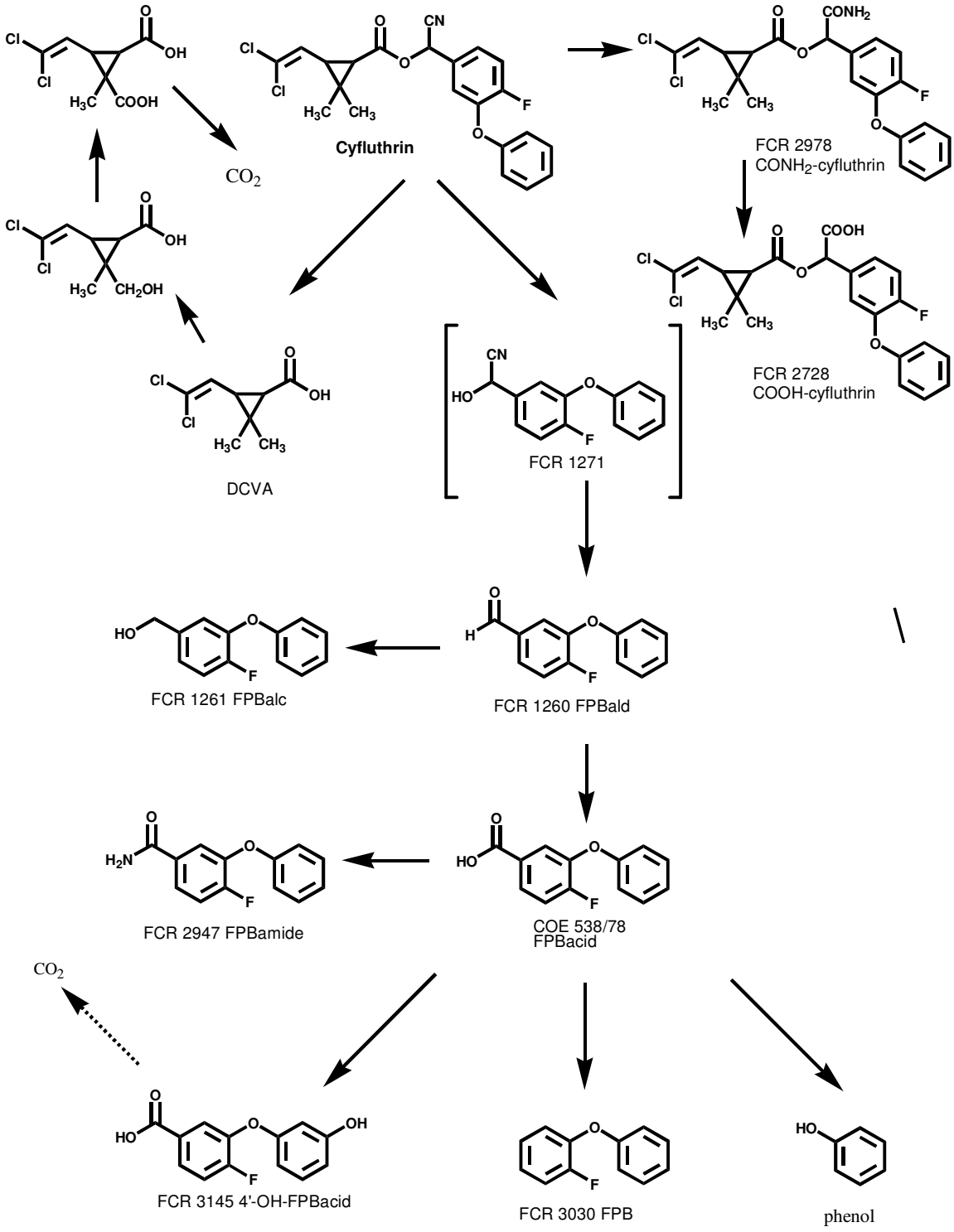


Figure 4. Proposed degradation pathway of cyfluthrin in the soil

METHODS OF RESIDUE ANALYSIS

A number of different analytical methods have been reported for the analysis of cyfluthrin (and beta-cyfluthrin) in plant and animal matrices. All of these are essentially based upon similar principles with variations derived from the introduction of improved techniques and technologies over the years. The

basic approach employs extraction by homogenisation with an organic solvent mixture incorporating varying proportions of polar and non-polar solvents depending primarily upon the nature of the matrix being extracted and its water content. In general, a primary liquid – liquid partition follows extraction to transfer cyfluthrin residues to less polar solvents prior to column clean-up. Residues are determined by gas chromatography (GC) with an electron capture detector (ECD) or mass spectra detection (MS). The limit of quantitation was usually between 0.01 and 0.05 mg/kg. The table below provides a summary of some methods for cyfluthrin analysis of crops and animal tissues and milk. Details of recoveries reported as part of method validation are reported in tables that follow for the major methods used for determination of cyfluthrin and beta-cyfluthrin in the residue trials. The majority of the methods are not stereospecific; thus no distinction is made between residues of cyfluthrin (all eight stereoisomers) and beta-cyfluthrin.

Table 23. Summary of major analytical methods used for the determination of cyfluthrin, including beta-cyfluthrin, in various matrices

Method/reference	Matrix	Extraction	Clean-up	Detection, % recovery + fortification levels	LOQ (mg/kg)
00086 (DFG Sammelmethode S19, Nolting et al., 1991	Cyfluthrin Beta-cyfluthrin Crops	1) acetone:water 2:1 2) partition CH_2Cl_2 (+NaCl)	1) Gel chromatography (Bio-Beads SX3) 2) silica gel Elute cyclohexane/ethyl acetate	GC-ECD Cyfluthrin: 71 – 105% at 0.01 – 0.54 mg/kg. Beta-cyfluthrin: 80 – 120% at 0.01 – 0.25 mg/kg.	0.01
M-016057-03-1 (method 00002) Brennecke 1984 00002; 1988 MO-02-006135; Blass 1985 00002/M007, M007/E011, M007/E012, M007/E016, M007/E017, M007/E018, M007/E019, Jaczyk 1982 00002/E010	Cyfluthrin Crops, soil, water	1) acetone or acetone:water 2) partition CH_2Cl_2 (+NaCl)	1) silica gel 2) polystrole gel Bio Bead S-X3 Elute cyclohexane	GC-ECD 74 – 117% at 0.02 – 1.0 mg/kg.	0.02
M-007384-02-1 (method 00010) Wagner 1980 00010, 1981 00010/E006, M001, M004, 1982 00010/M006, M007, 1983 00010/E001, E007, 1984 00010/M013, Blass 1985 00010/E011, E013, E016, 1987 00010/E021; 1988 00010/E023; Burger 1988 00010/M019	cyfluthrin Apples	1) acetone followed by H_3PO_4/NH_4Cl to precipitate solids or none. Alternative solvents e.g., acetone/n-hexane 2) partition CH_2Cl_2	1) silica gel or Florisil or none Elute cyclohexane	GC-ECD 70 – 125% at 0.01 – 1.0 mg/kg.	0.01 – 0.05
M-007653-01-2 (method 00015) Blass 1987-1991 00015; 00015/E002,	Cyfluthrin and beta-cyfluthrin Crops, beer, sugar	1) acetone or acetone:water or CH_3CN 2) partition CH_2Cl_2	silica gel or Florisil column or Extrelut or C_{18} cartridge or combinations	GC-ECD Cyfluthrin 61 – 127% at 0.01 – 2.0 mg/kg.	0.01 – 0.4

Method/reference	Matrix	Extraction	Clean-up	Detection, % recovery + fortification levels	LOQ (mg/kg)
E003, E004, E005, E009, M007, M009, Ohs 1989 00015/M005, E007, Seym 1993 00015/M012, M013		or n-hexane/CH_3CN	Elute cyclohexane	beta-cyfluthrin (only for 00015/M009 and M0013) 74 – 119% at 0.02 – 2 mg/kg	0.01 – 0.05
M009396-01-1 (method 00033) Harbin *et al.,* 1985 00033; Anon 1984-1986, 00033/M001, M002, M003	FPBacid FPBald FPBalc Crops:	1) MeOH:water 2:1 2) partition CH_2Cl_2 3) acid reflux to release conjugates 4) for oily crops partition hexane/CH_3CN	Gel permeation chromatography Followed by oxidation with MnO_4^- to FPBacid, methylation and bromination of the methyl ester	GC-ECD 50 – 140% at 0.01 – 0.5 mg/kg (most 70 – 120%)	0.01 – 0.05
M-008277-01-2 (method 00047) Blass 1985 00047; Blass 1985 – 1989; 00047/M001, M001/E012, E001, E002, E004, E007, E010	Cyfluthrin Hops, wheat and corn	1) acetone:hexane or acetone:water 2) partition CH_2Cl_2 or CH_3CN saturated with n-hexane	Florisil column	GC-ECD 74 – 125% at 0.01 to 1.0 mg/kg	0.01 – 0.2
M-008534-01-1 (method 00223) Harbin *et al.,* 1983 00223; Anon. 1983 – 1985, M037/E006, E033, E034, E010, E012, M038/E013, M038/E015, E016, E022, Leslie 1988 00223/M003, M005, M014, M031, M037, M038, 1989 00223/M001, M034, 1991 00223/M018, M019, M021, M023, M043, Leslie and Bailey 1988 00223/M011, Wiedmann, and Jablonski 1990 00223/M030, M042	Cyfluthrin Crops	1) MeOH:water 2) partition acetone:CH_3Cl	Florisil column	GC-ECD 65 – 130% at 0.01 – 2.5 mg/kg	0.01 – 1.0
M-009335-02-1 (method 00255) Ohs 1992 00255, Seym 1996 00255/E003, E004, E005, M001	Cyfluthrin Crops	1) acetone or acetone:water 2) partition CH_2Cl_2 3) an additional partition for oily extracts was with CH_3CN:n-hexane		LC-GC-ECD 89 – 126% at 0.01 to 0.1 mg/kg for cyfluthrin 95 – 117% at 0.01 to 0.1 mg/kg for beta-cyfluthrin	0.01 – 0.04
M-022605-01-2 (method I385)	Cyfluthrin Cotton seed	1) acetone:n-hexane	Silica gel	GC-ECD 83 – 90% at 0.05	0.03

Method/reference	Matrix	Extraction	Clean-up	Detection, % recovery + fortification levels	LOQ (mg/kg)
Wagner 1981 I385		2) wash 10% NaCl 3) partition CH_3CN (saturated n-hexane) 4) phosphate precipitation 5) partition CH_2Cl_2		to 0.1 mg/kg	
M-059522-01-1 (method I471) Ishii and Ueyama 1983 86066	Cyfluthrin Crops and soil	1) acetone 2) partition CH_2Cl_2	Silica gel	GC-FID 86 – 98% at 0.1 to 1.0 mg/kg	0.1
M-059615-01-2 (method I478) Wagner 1983 I478	Cyfluthrin Rape seed	1) acetone:CH_3CN	Florisil column	GC-ECD 89 – 97% at 0.05 to 0.2 mg/kg.	0.05
M-064455-01-1 (method I07839) Sandie and Gronberg 1997 107839	Cyfluthrin Crops	1) MeOH:water 2) acidify, partition n-hexane	1) Florisil column, eluted n-hexane/acetone 2) reverse phase SPE column, eluted acetone	GC-MS in selected ion monitoring mode, m/z 226 70 – 116% at 0.05 – 2.5mg/kg	0.05
M-059050-01-1 (method I08139) Sandie and Gronberg 1998 108139; Moore *et al.,* 2002 108139-1; Bajzik 1998 108451	Cyfluthrin Beta-cyfluthrin Crops	1) MeOH:1.2 M HCl 2) partition n-hexane 3) for some crops, partition acetone:CH_2Cl_2	1) Florisil column, eluted n-hexane/acetone 2) reverse phase SPE column, eluted acetone	GC-MS selected ion m/z 207 or 226, d6-cyfluthrin internal standard for method 108139 78 – 121% at 0.01 – 7.5 mg/kg	0.01
M-0066143-01-1 (method I476) Shaw *et al.,* 1983 I476; Anon 1983 – 1985, 85983, 85982, 85981, 90392, 90388, 86038, 86220, 87216, 87217; Seym 1995 MR-303/95	Cyfluthrin Animal commodities	Tissues: 1) Extract with acetone:$CHCl_3$ 2:1 except fat and skin for which n-hexane was employed. For liver, kidney add HCl. Evaporate to dryness. Milk: 1) Add HCl and extract with acetone:$CHCl_3$ 2:1 Eggs: 1) homogenize eggs with Florisil to obtain a free flowing powder and add to a Florisil column prepared using hexane: $CHCl_3$ saturated with CH_3CN	1) dissolve MeOH:H_2O, partition against ethyl acetate. Evaporate to dryness 2) partition with CH_3CN and hexane Fat, skin, milk only: Partition against hexane acetone:H_2O Eggs: 1) partition hexane/5% NaCl 2) partition hexane/ acetonitrile 3) Florisil column Other tissues and milk: Silica gel column	GC-ECD	0.05 tissues and eggs 0.02 milk

Method/reference	Matrix	Extraction	Clean-up	Detection, % recovery + fortification levels	LOQ (mg/kg)
		2) Elute with hexane: $CHCl_3$ saturated with CH_3CN , remove solvent, redissolve in acetone			
M-066384-01-1 (method I488) Shaw *et al.,* 1983 I488	FPBacid FPBald FPBalc Animal commodities:	1) Extract with acetone:CH_2Cl_2 2:1. For liver, kidney add HCl. Evaporate to dryness. 2) Dissolve in MeOH:H_2O and partition with ethyl acetate	Gel permeation chromatography a) acid-cyfluthrin: column chromatography, methylation, bromination b) other metabolites: column chromatography, oxidation using MnO_4^-, partitioning, methylation	HPLC-UV (230 nm) 72 – 98% at 0.05 mg/kg	0.05
M-066520-01-1 (method I578) Shaw *et al.,* 1985 I578	DCVA in bovine, poultry tissues and eggs	1) MeOH:0.1 N NaOH and CH_3CN 2) partition ethyl acetate: diethyl ether NaOH 3) separate free DCVA from conjugated DCVA by partition using acid CH_2Cl_2:acetone 4) hydrolyse conjugated DCVA with acid and combine with free DCVA	1) silica gel 2) HCO_3^- extraction 3) derivatise with 1-propanol in acid solution to form the propyl ester of DCVA 4) partition with isooctane:0.1N NaOH 5) Florisil column	GC-Hall detector (halogen) Tissues: 70 – 105% at 0.05 mg/kg Eggs: 74 – 76% at 0.05 mg/kg	0.05
M-066719-01-1 (method I675) Gronberg and Pfankuche 1986 I657	DCVA in milk	1) add NaOH to make milk 0.05 N, extract with acetone:n-hexane 2) acidify the aqueous extract and reflux to hydrolyse conjugates 3) partition with CH_2Cl_2 under acid, base and then acid conditions 4) methylation of free DCVA 5) partition n-hexane: NaOH		GC-MS, selected ions, m/z DCVA 163 60 and 70% at 0.01 mg/kg, 88 – 108% at 0.02 – 0.1 mg/kg	0.02

Method/reference	Matrix	Extraction	Clean-up	Detection, % recovery + fortification levels	LOQ (mg/kg)
M-12515-01-1 (method 00553) Maasfield and Schöning 1989 – 2001 00553	Cyfluthrin Bovine and poultry tissues and eggs and milk	Tissues: 1) extract with CH_3CN 2) partition against n-hexane 3) add CH_2Cl_2, dry over Na_2SO_4, remove solvent, redissolve in n-hexane Milk: 1) extract with CH_3CN:water 2) partition against CH_2Cl_2:NaCl 3) remove solvent, redissolve in CH_3CN 4) partition against n-hexane, remove solvent, redissolve in n-hexane	Silica gel and reverse phase Sep Pak C_{18} columns	GC-ECD Tissues: (bovine) 74 – 103% at 0.01 and 0.05 mg/kg. (poultry) 71 – 111% 0.01 – 1.0 mg/kg Milk: 69 – 89% at 0.005 to 0.05 mg/kg Eggs:75 – 97% at 0.01 – 0.1 mg/kg	Bovine tissues: 0.05 Milk: 0.005 Poultry tissues and eggs: 0.01
M-066095 (method I369) Wagner 1981 I369	Cyfluthrin Milk	1) extract using CH_2Cl_2, and the extract dried using Na_2SO_4 and the solvent removed. 2) redissolve in hexane and partition against CH_3CN (saturated n-hexane) 3) dry the CH_3CN over Na_2SO_4 and remove the solvent	Silica gel, eluted with hexane/ethyl acetate, the solvent removed and the residue dissolved in acetone	GC-ECD 80 – 83% at 0.005 and 0.01 mg/kg	0.005
M-066102-01-2 (method I395) Wagner 1982 I395	Cyfluthrin Pig tissues	1) add Na_2SO_4 and Soxhlet extract using acetone/hexane 2) partition against CH_3CN (saturated n-hexane) 3) dry over Na_2SO_4	Silica gel, eluted with hexane/ethyl acetate, the solvent removed and the residue dissolved in acetone	GC-ECD 68 – 87% at 0.05 – 0.1 mg/kg	0.05
M-066133-01-2 (method I415) Wagner 1982 I415	Cyfluthrin Eggs	1) homogenize eggs with Florisil to obtain a free flowing powder and add to a Florisil column prepared using CH_3CN: CH_2Cl_2		GC-ECD 73 – 83% at 0.1 mg/kg	0.1

Method/reference	Matrix	Extraction	Clean-up	Detection, % recovery + fortification levels	LOQ (mg/kg)
		saturated with hexane 2) Elute with CH_3CN: CH_2Cl_2 saturated with hexane, remove solvent, redissolve in acetone			
M-014250-01-1 (method 00111) Minor and Freeseman 1988 00111	FPBacid and 4'OH-FPBacid Bovine liver and kidney	1) reflux with conc. HCl to hydrolyse residues 2) partition with CH_3Cl 3) partition against HCO_3^- and then acid	1) methylation using basic dimethyl sulphate 2) Florisil column	GC-MS Selected ions (m/z) 1) FPBacid 215, 246 2) 4'OH-FPBacid 261, 276 FPBacid: Liver 70 – 116% at 0.01 – 0.05 mg/kg Kidney: 90 – 130% 4'OH-FPBacid: Liver 60 – 124% Kidney 90 – 120%	0.01
M-027565-01-1 (method 00614) Krebber 1999	Cyfluthrin Bovine tissues and milk	Tissues: 1) extract with CH_3CN and centrifuge 2) partition against CH_3CN-saturated n-heptane 3) add CH_2Cl_2 and dry over Na_2SO_4 Milk: 1) extract with acetone 2) silica gel was added, solvent removed and the gel added to a column and eluted with CH_2Cl_2 3) remove solvent, redissolve in CH_3CN 4) partition against n-heptane	1) Silica gel 2) C_{18} column (not for milk)	GC-MS SIM, m/z= 163, 206 and 226 Tissues: (bovine) 53 – 85% at 0.01 and 0.04 mg/kg. Milk: 76 – 110% at 0.01 to 0.04 mg/kg	Bovine tissues and milk 0.01

Method 00223 (Harbin *et al.,* 1983 00223): cyfluthrin is extracted from homogenised samples with methanol/water (4:1) and the filtered extract concentrated to the aqueous phase which is partitioned against acetone/chloroform (1:2). Clean-up of the concentrated acetone/chloroform is on a Florisil column prior to quantitation of residues using GC-ECD. The results of recovery experiments support a method LOQ of 0.01 to 0.05 mg/kg for a wide range of crops.

Table 24. Analytical recoveries for various matrices fortified with cyfluthrin

Crop	Fortification level (mg/kg)	N	Mean recovery (%)	Range (%)	Reference
Apple dry pomace	001 – 0.4	5	92	70 – 110	00223/M030
Apple juice	0.4	1		107	00223/M030
Apple wet pomace	04 – 2.5	2	106	100 – 113	00223/M030
Apples	0.01 – 0.1	26	98	84 – 110	00223/M032/E018 00223/E020 00223/M030
Broccoli	0.05 – 5	6	80	71 – 97	00223/M038/E013
Cabbage heads	0.05 – 0.5	8	91	70 – 108	00223/M038/E015
Cauliflower	0.05 – 1	5	87	73 – 107	00223/E016
Cottonseed	0.01 – 1	13	83	70 – 92	00223 00223/M034
Cottonseed hulls	0.05 – 0.5	2	76	70 – 82	00223
Maize dry cobs	0.02 – 0.05	4	78	74 – 80	00223/M011/E026
Maize dry husks	0.02 – 0.05	4	76	70 – 82	00223/M011/E026
Maize fodder dry	0.01 – 0.1	8	84	75 – 100	00223/M011/E026 00223/M018
Maize forage green	0.02 – 0.05	4	82	75 – 90	00223/M011/E026
Maize kernels dry	0.01 – 0.05	9	77	70 – 90	00223/M011/E026 00223/M018
Oranges	0.01 – 0.05	4	84	80 – 96	00223/M022
Peanut dry vines	0.05	1		84	00223
Peanut meat	0.05	1		74	00223
Peanut shells	0.05	1		78	00223
Pears	0.05 – 0.1	12	96	84 – 110	00223/E017
Peppers	0.01 – 0.05	4	89	70 – 100	00223/M031
Potato dry	0.05	1		78	00223/M037/E006
Potato tubers	0.05	4	86	84 – 90	00223/M037/E006
Rape seed	0.05	3	92	88 – 100	00223/E010
Rapeseed meal	0.05	2	97	92 – 102	00223/E012
Rapeseed oil	0.05	4	80	76 – 82	00223/E012
Sorghum	0.01 – 0.05	4	95	65 – 120	00223/M017
Soya bean dry vines	0.05 – 0.1	8	83	80 – 85	00223 00223/M017
Soya bean forage	0.01 – 0.5	16	100	75 – 140	00223 00223/M017
Soya bean hay	0.05 – 0.1	7	94	76 – 110	00223/M017
Soya beans	0.05 – 0.1	6	82	80 – 85	00223 00223/M017
Sunflower fodder dry	0.01 – 0.1	6	88	70 – 108	00223/E022
Sunflower seed	0.01 – 0.1	6	94	80 – 110	00223/E022
Sweet corn cob	0.01 – 0.1	5	84	70 – 110	00223/M021 00223/M043
Sweet corn forage green	0.01 – 0.1	5	105	79 – 135	00223/M021 00223/M043
Sweet corn, kernel	0.01 – 0.1	5	81	70 – 90	00223/M021 00223/M043

Method 107839 (Sandie and Gronberg 1997 107839): cyfluthrin residues are extracted from homogenised sample material with methanol/water. The filtered extract is concentrated to the aqueous phase, acidified and partitioned against n-hexane. Clean-up of the hexane is by Florisil column with elution of cyfluthrin residues using hexane/acetone. Additional clean-up is with a reverse phase SPE column with elution using acetone. The concentrated extract is diluted with cyclohexane and residues determined using GC-MS. Data are acquired for ions of m/z 226, 206 and 163. Quantitation is based on the 226 m/z ion only

Modifications to the method included extraction of residues from crops with methanol/aqueous 1.2 M HCl and the addition of an additional clean-up step involving partitioning of the aqueous extract against acetone/CH_2Cl_2 prior to column clean-up.

Table 25. Analytical recoveries reported for analysis of cyfluthrin using Method 107839 on a range of crop matrices

Crop	Fortification level (mg/kg)	N	Mean recovery (%)	Range (%)
Alfalfa forage	0.1 – 1	7	90	77 – 110
Alfalfa hay	0.5 – 2.5	8	91	83 – 102
Maize fodder	0.5 – 4	12	94	70 – 119
Maize forage	0.05 – 4	6	91	75 – 108
Maize grain	0.01 – 0.05	9	101	90 – 112
Orange	0.05 – 0.2	11	94	79 – 116
Sugarcane	0.05 – 1	8	82	70 – 90
Sweet corn fodder	2 – 6	7	98	91 – 108
Sweet corn forage	0.25 – 2.5	10	100	91 – 111
Sweet corn kernel cob	0.05	4	88	78 – 98
Tomato	0.05 – 0.1	7	89	79 – 102

Table 26. Analytical recoveries reported for analysis of beta-cyfluthrin using Method 107839 on a range of crop matrices

Crop	Fortification level (mg/kg)	N	Mean recovery (%)	Range (%)
Alfalfa forage	0.1 – 1	8	95	89 – 104
Alfalfa hay	0.5 – 1.5	7	81	74 – 90
Orange	0.05 – 0.1	7	89	73 – 110
Sugarcane	0.05 – 0.3	7	82	72 – 105
Sweet corn fodder	2 – 6	7	102	99 – 106
Sweet corn forage	0.25 – 2.0	7	93	81 – 102
Sweet corn kernel cob	0.05	4	82	76 – 90
Tomato	0.05 – 0.1	7	97	92 – 110

Method 108139: Sandie and Gronberg (1998 108139) described an improved GC-MS method for crops. Extraction of residues from crops is with methanol/water. The filtered extract is concentrated to an aqueous phase, acidified and partitioned against hexane. The hexane phase is applied to a Florisil column, and the cyfluthrin or beta-cyfluthrin is eluted with hexane/acetone. The eluted cyfluthrin or beta-cyfluthrin is further purified on a C_{18} reverse phase (SPE) column and the residue eluted with acetone. The acetone is replaced with cyclohexane, and the cyclohexane solution is analysed by GC-MS (electron ionisation). Data are acquired for ions of m/z 226, 206, and 163 with the ion at m/z 226 used for quantitation.

In a modification by Moore *et al.,* (2002 108139-1) detection was by GC-MS with chemical ionisation and quantification using a known amount of deuterated internal standard added to the samples after the extraction. Chemical ionisation in negative mode is more sensitive and selective than electron ionisation in positive mode. The m/z 207 ion is used as the quantitation ion.

Table 27. Analytical recoveries reported for analysis of cyfluthrin using Method 108139 on a range of crop matrices (Moore *et al.,* 2002 108139-1)

Crop	Fortification level (mg/kg)	N	Mean recovery (%)	Range (%)
Apple	0.01 – 0.05	12	105	88 – 121
Bean seed dry	0.01 – 0.15	10	88	74 – 98
Broccoli	0.01 – 0.75	8	97	92 – 101
Cabbage (with wrappers)	0.01 – 3.5	8	100	93 – 105
Cabbage (without wrappers)	0.01 – 0.02	6	102	99 – 107
Carrot roots	0.01 – 0.05	8	96	92 – 102
Celery, trimmed leaf stalk	0.01 – 1	13	100	93 – 103
Celery, untrimmed leaf stalks	0.01 – 3.5	10	98	80 – 103
Cherry	0.01 – 0.5	15	97	91 – 103
Cucumber	0.01 – 0.07	9	97	91 – 100
Grape	0.01 – 1	16	97	93 – 101
Grapefruit	0.01 – 0.1	9	96	90 – 99
Lemon	0.01 – 0.15	11	96	88 – 101
Lettuce (with wrapper)	0.01 – 2	8	99	95 – 105
Lettuce (without wrapper)	0.01 – 0.05	7	97	93 – 98
Lettuce leaves	0.01 – 3.5	10	97	94 – 99
Melon cantaloupe	0.01 – 0.05	10	95	90 – 98
Mustard green leaves	0.01 – 5.5	11	95	82 – 100
Orange	0.01 – 0.1	10	98	94 – 102
Pea seed dry	0.01 – 0.15	10	86	78 – 92
Peach	0.01 – 0.4	21	95	88 – 101
Peanut hay	0.01 – 6.5	12	75	68 – 87
Peanut meat	0.01	8	89	76 – 97
Pear	0.01 – 0.05	14	88	78 – 96
Pepper	0.01 – 0.4	10	94	86 – 104
Plum	0.01 – 0.1	16	97	86 – 109
Potato	0.01	6	95	90 – 100
Radish roots	0.01 – 0.025	9	97	91 – 108
Radish tops	0.01 – 5	7	85	80 – 90
Spinach leaves	0.01 – 6	10	96	91 – 102
Tomato	0.01 – 0.5	8	93	91 – 95
Zucchini (squash)	0.01 – 0.08	8	98	96 – 99

Method I476. A method used for the determination of residues of cyfluthrin in bovine tissues and milk in residue trials was reported by Shaw *et al.,* (1983 I476). Tissue samples are extracted with acetone: $CHCl_3$ 2:1, except fat and skin for which n-hexane is employed. For liver or kidney a small amount of HCl is added. The solvent is removed and the residue dissolved in methanol:H_2O and partitioned against ethyl acetate, solvent removed and the residue partitioned with CH_3CN and hexane or in the case of fat, skin, milk partition against hexane acetone:H_2O. Further clean-up of extracts is by silica gel column.

In the case of eggs, samples are homogenized with Florisil to obtain a free flowing powder and added to a Florisil column eluted with CH_3CN:$CHCl_3$, the solvent removed and the residue redissolved in acetone, partition against hexane/5% NaCl followed by hexane/ CH_3CN and clean-up on a Florisil column. Quantitation was by GC-ECD.

Table 28. Analytical recoveries reported for analysis of cyfluthrin using Method I476 on a range of animal matrices

Crop	Fortification level (mg/kg)	N	Mean recovery (%)	Range (%)	Reference
Bovine					
Milk	0.02	3 3	112 98	100 – 125 (normal) 90 – 110 (confirmatory)	I476
Muscle	0.05	1		85	I476
Fat	0.05	2	72	67 – 78	I476
Kidney	0.05	1		78	I476
Liver	0.05	1		76	I476
Milk	0.01 – 0.1	12	111	86 – 161 (alternative column)	90392
Milk	0.05	5	100	84 – 112 (standard column)	90392
Liver	0.01 – 0.1	7	86	80 – 100	90388
Kidney	0.01 – 0.1	7	89	80 – 105	90388
Muscle	0.01 – 0.1	7	99	80 – 145	90388
Fat	0.01 – 0.1	6	82	60 – 110	90388
Chicken					
Eggs	0.05	3 3	73 69	70 – 78 (normal) 62 – 80 (confirmatory)	I476
Muscle	0.05	1		94	I476
Fat	0.05	1		84	I476
Skin	0.05	1		88	I476
Liver	0.05	1		76	I476
Kidney	0.05	1		74	I476

Method 00553: a routine method for the determination of residues of cyfluthrin in bovine tissues and milk has been reported (Maasfeld 1989 00553). Samples are extracted with acetonitrile and partitioned against hexane to remove lipids. The residue remaining after solvent evaporation is further cleaned on a silica column or on Sep-Pak-C18 cartridge. Following solvent evaporation of the fraction containing cyfluthrin the residue is dissolved in ethyl acetate with quantitative determination by GC-ECD. The limit of quantification is in the order of 0.01 mg/kg for all tissues and 0.005 mg/kg for milk.

Table 29. Analytical recoveries reported for analysis of cyfluthrin using Method 00553 on a range of animal matrices

Sample	Fortification level (mg/kg)	N	Recovery (%)	Mean (%)	Reference
Bovine					
Fat	0.01 – 0.05	10	74 – 96	87	00553
Kidney	0.01 – 0.05	10	77 – 99	87	00553
Liver	0.01 – 0.05	10	74 – 103	89	00553
Muscle	0.01 – 0.05	10	74 – 86	80	00553
Milk	0.005	10	69 – 85	76	00553
Liver	0.01 – 0.04	15	59 – 84	71	00553/M001
Kidney	0.01 – 0.04	20	49 – 80	64	00553/M001
Muscle	0.01 – 0.04	14	65 – 85	74	00553/M001
Fat	0.025 – 0.25	20	57 – 87	77	00553/M001
Chicken					
Muscle	0.01 – 1.0	6	71 – 85	79	MR-871/98
Fat	0.01 – 1.0	6	82 – 111	92	MR-871/98
Egg	0.01 – 1.0	6	75 – 93	86	MR-871/98
Egg	0.01 – 0.1	10	75 – 97	88	MR-355/99

Stability of Residues in Stored Analytical Samples

Lenz and Lemke (1996 103821-3) studied the freezer storage stability of twenty-four crops and processing fractions that were fortified with 1 mg/kg of cyfluthrin (100 mg/kg for wheat grain dust) and stored in a freezer at -18 °C. Sample aliquots were fortified individually and were contained in glass jars with Teflon-lined caps. Aliquots were analysed at day 0 and for periods of up to 38 months.

The whole potato sample used in the study was also used as a source of potato peels for the wet and dry peel samples. Wet peels were dried overnight at 77 °C to obtain dry peels. Wheat, rice and corn grains were ground in a Straub Mill with dry ice and the dry ice allowed to sublime before sampling. Apple, cantaloupe, cucumbers, oranges, potatoes and tomatoes were chopped with dry ice in a Hobart Food Chopper. Peanut shells were ground up with dry ice in a Waring blender. All samples were homogenized before sampling for the study.

Cyfluthrin was stable in homogenized samples stored frozen for at least 1118 days for apple, 1145 days for cantaloupe, 1130 days for corn, 1145 days for corn oil, 1125 days for corn starch, 1145 days for cucumber, 739 days for oranges, 1145 days for orange juice, 739 days for orange pulp, 1145 days for peanut shells, 1126 days for potatoes, 1130 days for potato chips, 1126 days for potato granules, 95 days for potato peel (wet), 1146 days for potato peel (dry), 102 days for rice, 207 days for rice hulls, 1155 for sugar cane stalks, 1125 days for molasses, 1151 days for tomatoes, 1130 days for wheat and for wheat bran, 1118 days for wheat flour and 1126 days for wheat dust.

Table 30. Storage stability for cyfluthrin residues in various crops fortified at nominal concentrations of 1 mg/kg and stored frozen

Days storage	Concentration (mg/kg)	Procedural recovery (%)	Days storage	Concentration (mg/kg)	Procedural recovery (%)
Apple			Cantaloupe		
0	0.93 0.99 0.78	106	0	0.95 1.06 1.12	68 81
38	1.05 1.00	71	52	0.88 0.96	87
103	0.71 0.79	70	101	0.73 0.99	82
207	0.90 0.98	100	213	0.90 0.88	98
396	0.93 0.95	94	402	1.04 0.92	103
572	0.34 0.39	80	579	0.67 0.67	78
746	0.71 0.73	101	732	0.76 0.84	98
1118	0.84 0.79	92	1145	0.72 0.80	77
Corn			Corn oil		
0	0.91 1.01 0.92	74 79	0	0.81 0.88 0.85	108 115
35	0.82 0.89	94	39	0.87 0.87	127
98	0.84 0.92	89	61	0.58 0.61	73
201	0.93 0.85	99	102	0.50 0.73	66
392	0.87 0.77	99	206	0.69 0.67	84
579	0.57 0.61	71	396	0.75 0.86	95
742	0.57 0.57	79	580	0.60 0.78	75
1130	0.77 0.75	87	745	0.69 0.65	81
			1145	0.78 0.75	80
Corn starch			Cucumbers		
0	1.05 1.12 1.10	117 121	0	0.89 0.86 0.90	72
32	0.80 0.74	91	52	0.69 0.71	88
101	0.76 0.51	82	101	0.74 0.73	95
202	0.85 0.86	103	213	0.73 0.77	84
390	0.81 0.80	91	402	0.96 0.92	103
567	0.31 0.51	86	579	0.64 0.61	72
742	0.65 0.66	95	749	0.61 0.64	71
1125	0.78 0.81	87	1145	0.63 0.60	76

Days storage	Concentration (mg/kg)	Procedural recovery (%)	Days storage	Concentration (mg/kg)	Procedural recovery (%)
Orange			Orange juice		
0	1.00 1.02 1.10	96	0	0.89 0.83 0.85	104
33	1.14 1.07	122	40	0.81 0.89	98
98	1.04 1.00	108	102	0.73 0.88	72
201	0.77 0.71	80	206	0.65 0.72	79
395	0.92 0.90	100	396	0.54 0.61	91
580	0.76 0.77	72	579	0.46 0.40	79
739	0.86 0.80	95	745	0.67 0.81	97
1126	0.61 0.60	77	1145	0.62 0.64	78
Orange pulp, dry			Peanut shells		
0	1.23 1.15 1.12	101	0	0.65 0.75 0.78	76 93
33	1.04 1.10	114	45	0.81 0.78	74
98	0.86 0.97	91	98	0.72 0.74	72
201	0.80 0.93	87	208	0.73 0.76	85
395	0.93 0.75	91	402	0.94 0.85	95
580	0.81 0.76	102	587	0.87 0.94	95
739	0.82 0.86	100	741	0.66 0.67	78
1124	0.66	80	1145	0.59 0.43	77
Potato			Potato chips		
0	1.01 0.81 0.93	100	0	0.75 0.67 0.82	71
39	0.72 0.71	75	35	0.82 0.81	86
96	0.62 0.64	93	98	0.86 0.80	100
202	0.66 0.66	83	201	0.86 0.89	89
391	0.59 0.61	81	392	0.79 0.79	80
595	0.66 0.68	86	579	0.63 0.63	80
746	0.62 0.63	95	742	0.56 0.57	73
1126	0.62 0.64	85	1130	0.83 0.79	87
Potato granules			Potato wet peel		
0	0.83 0.70 0.81	71	0	0.92 0.90 0.85	97
39	0.85 0.82	96	55	0.69 0.76	70
92	0.62 0.63	66	95	0.77 0.77	78
202	0.67 0.66	88	209	0.59 0.61	73
391	0.84 0.84	91	392	0.52 0.47	73
575	0.50 0.73	83	578	0.51 0.52	74
746	0.57 0.58	87	748	0.59 0.61	86
1126	0.90 0.74	94	1146	0.60 0.52	67
Potato dry peel			Rice		
0	0.78 0.71 0.75	87 91	0	0.93 0.84 0.74	94 81
55	0.75 0.61	71	34	0.93 0.80	87
95	0.65 0.68	73	102	0.74 0.57	68
209	0.66 0.68	82	207	0.56 0.52	66
392	0.69 0.71	91			
578	0.67 0.53	67			
748	0.68 0.72	83			
1146	0.57 0.63	75			
Rice hulls			Sugarcane		
0	0.86 0.80 0.89	79 76	0	0.78 0.77 0.79	90
34	0.85 0.68	67	52	0.70 0.76	67
106	0.52 0.51	67	105	0.72 0.69	69
207	0.71 0.76	89	213	0.68 0.79	70
			398	0.90 0.98	85

Days storage	Concentration (mg/kg)	Procedural recovery (%)	Days storage	Concentration (mg/kg)	Procedural recovery (%)
			592	0.69 0.72	81
			732	0.66 0.66	80
			1155	0.84 0.81	80
Molasses			Tomato		
0	0.94 0.76 1.15	123	0	1.02 0.99 1.04	106 109
66	0.51 0.57	92	52	0.67 0.67	72
101	0.77 0.40	86	102	0.86 1.02	106
202	0.28 0.43	85	213	0.97 0.87	95
390	0.64 0.79	97	398	0.95 1.16	103
567	0.27 0.29	85	581	0.83 0.81	92
742	0.51 0.62	84	747	0.50 0.67	94
977	0.83 0.77	83	1151	0.68 0.80	80
984	0.81 0.81	73			
1125	0.82 0.83	90			
Wheat			Wheat bran		
0	0.94 0.88 0.83	77 84	0	0.84 0.95 0.87	77 88
38	0.74 0.77	90	38	0.83 0.74	81
103	0.79 0.64	85	103	0.75 0.77	78
203	0.54 0.63	85	203	0.70 0.72	93
397	0.70 0.70	73	397	0.60 0.65	73
605	0.84 0.85	95	605	0.79 0.79	91
741	0.84	94	741	0.71 0.64	92
749	0.73 0.75	95	1130	0.78 0.73	88
1130	0.75 0.77	83			
Wheat flour			Wheat dust		
0	0.75 0.91 0.87	118 123	0	120 105 98	102
38	0.95 1.04	93	32	105 116	90
103	0.86 0.97	86	122	116 117	106
207	0.93 0.90	92	215	111 110	102
396	0.95 0.99	95	398	98.3 99.2	101
606	0.54 0.52	77	594	95.8 96.4	118
746	0.61 0.50	87	747	99.8 101.1	111
1118	0.82 0.79	80	1126	101.2 101.5	99

In addition, ungrounded wheat grain was fortified and stored at room temperature (21 °C) and at 28 °C. Nominal analysis times for these samples were 1, 2, 3, 7 and 9 months for the 28 °C samples and 1, 2 and 3 months for the 21 °C samples. Both of these samples lost 29 or 31% of the cyfluthrin in the first month. The room temperature wheat did not have additional losses. The 28 °C wheat had only 50% of the cyfluthrin left after 293 days.

Table 31. Storage stability of wheat containing residues of cyfluthrin (Lenz and Lemke 1996 103821-3)

Days storage	Concentration (mg/kg)	Procedural recovery (%)	Days storage	Concentration (mg/kg)	Procedural recovery (%)
Wheat (28 °C)			Wheat (21 °C)		
0	1.00 1.03 1.03		0	1.00 1.03 1.03	
34	0.79 0.64	91	34	0.61 0.79	98
66	0.55 0.60	89	74	0.53 0.47	72
117	0.59 0.52	77	117	0.74 0.71	83
203	0.40 0.27	82			
293	0.53 0.48	81 81			

Delk (1988 98334) studied the stability of samples of potato, tomato, corn dry forage and soya bean green forage held in frozen storage for up to 35 months. Finely chopped portions of each crop were weighed into glass bottles with Teflon lined caps. Each bottle was then individually spiked with a standard solution of cyfluthrin, FPB acid, FPB alcohol, FPB aldehyde or DCVA in acetone for a fortification level of 1.0 mg/kg. All samples were then stored in a -7 °C freezer until extraction and analysis.

Table 32. Storage stability of cyfluthrin and selected metabolites fortified at nominal concentrations of 1 mg/kg in tomatoes, potatoes, corn forage (dry) and soya bean forage (green)

Compound	Cherry tomato		Potato		Corn forage dry		Soya bean forage, green	
	Days	Residue (mg/kg)	Days	Residue (mg/kg)	Days	Residue (mg/kg)	Days	Residue (mg/kg)
Cyfluthrin	0	0.87 0.79		0.96 0.96				
	63	0.70 0.71	63	0.96 0.79				
	133	0.87 0.77	133	0.38 0.41				
	246	0.54 0.86	246	0.47 0.58				
	1107	0.54 0.42 0.44	1111	0.32 0.28 0.35				
DCVA	0	1.08 0.71	0	1.05 0.80	0	1.14 0.83	0	0.87 0.74
	63	0.74 0.65	57	0.92 0.76	60	0.82 0.72	63	0.51 0.68
	131	0.60 0.68	25	0.76 0.68	122	0.94 0.68	139	0.50 0.92
	251	0.84 0.67	251	0.83 0.91	246	0.61 0.64	246	0.75 0.60
	1148	0.27 0.47 0.38	1146	1.65 0.64 0.87				
FPBacid	0	0.85 1.13	0	0.92 0.93	0	1.0 0.86	0	1.18 0.76
	68	0.26 0.72	-	-	60	0.64 1.12	63	1.07 1.04
	229	0.77 0.66	229	0.47 0.54	122	0.59 0.64	139	0.55 0.24
	1149	0.84 0.82 0.88	1141	1.00 0.99 1.00	246	0.32 0.52	251	0.64
FPBalc	0	0.91 0.84	0	0.98 0.82	0	0.95 0.96	0	1.13 1.01
	68	1.03 0.74	68	0.69 0.81	60	1.15 0.94	63	0.80 0.54
	229	0.790	229	0.927 0.846	122	0.485 0.180	139	0.71 0.35
	1149	0.49 0.64 0.41	1141	0.72 0.93 0.76	246	0.59 0.31	251	0.490 0.307
FPBald	0	1.07 0.85	0	1.12 0.79	0	0.79 0.79	0	0.77 0.92
	68	0.28 0.80	68	0.58 0.49	60	0.87 0.83	63	0.87 0.94
	229	0.32 0.33	229	0.59 0.74	122	0.088 0.47	139	1.22 0.79
	1149	0.24 0.16 0.19	1141	0.72 1.02 0.66	246	0.81 0.61	251	0.75 0.40

Recoveries for samples fortified at 1 mg/kg were 107 100 88 and 85% for potato and 82 89 98 and 82% for tomato. For FPBald the recoveries were 94 and 72% for potato, 74 and 84% for tomato, 85 71 and 82% for corn dry forage and 77 81 and 94% for soya bean forage (green). For DCVA recoveries were 86 and 71% for potato, 83 and 88% for tomato, 70 and 94% for corn forage (dry) and 71 and 70% for soya bean forage (green).

The measured residues of cyfluthrin and metabolites were variable. The concurrent recoveries conducted within the study ranged from 70 – 107%. Residues of cyfluthrin, DCVA, FPBacid, FPBalc and FPBald were stable in tomatoes for 246, 251, 1149, 229 and < 68 days respectively. The stability in potatoes of residues of cyfluthrin, DCVA, FPBacid, FPBalc and FPBald was for 63, 1146, 1141, 1141 and 1141 days respectively. Residues of DCVA, FPBacid, FPBalc and FPBald were stable in

corn forage for 122, 60, 60 and 246 days respectively and for soya bean forage for 246, 63, < 63 and 139 days.

The storage stability results of Delk (1988 98334) differ from those of Lenz and Lemke (1996 103821-3) who determined that residues in samples of homogenized tomato and potato fortified with cyfluthrin were stable (< 30% decline) for 1126 and 1151 days frozen storage respectively. The stability of residues of cyfluthrin and metabolites is also supported by studies that follow suggesting the apparent 'instability' indicated by the results of Delk (1988 98334) are due to difficulties with the analysis (variable recoveries) rather than a real effect.

Minor and Freeseman (1989 99631) studied the freezer storage stability of soya beans, soya bean dry vines, and cotton seeds fortified at 1 mg/kg with [^{14}C] cyfluthrin and maintained under freezer conditions for 1895, 1890, and 1888 days, respectively.

Additionally, samples from the [^{14}C]cyfluthrin apple and potato metabolism studies were re-analysed after 1726 days and 2436 days freezer storage when compared to the original analysis at time of harvest. The greatest amount of decomposition was seen in soya bean leaves in which cyfluthrin showed an apparent 19% decomposition at 164 days and 28% decomposition after 2512 days of freezer storage.

Table 33. Effect of Frozen Storage at -18 to -23 °C on cyfluthrin residues (Minor and Freeseman 1989 99631)

Matrix	Storage interval (days)	Residue (mg/kg)
Soya beans [a]	0	1.06 1.03 1.01
Beans	96	0.98 1.03
	187	1.02 1.06
	1895	0.92 0.97
Dry Vines	0	1.10 1.06 1.09
	91	0.99 1.11
	182	0.97 0.94
	1890	1.00 0.93
Cotton[a,b]	0	1.11 1.08 1.00
Seeds	89	1.04 1.07
	180	1.06 1.10
	1888	1.11 1.24

a - Data obtained from a [14C]Baythroid Stability Study (anon. 1984 87026)

b - Data obtained from a [14C]Baythroid Stability Study (anon. 1984 87027)

Cyfluthrin showed no apparent decomposition in cotton seeds after 1888 days under freezer conditions. Cyfluthrin appeared stable in soya beans and dry vines with only 8% and 11% decomposition apparent after 1895 and 1890 days, respectively.

In another study individual samples of cotton seed were fortified with 10 mg/kg ^{14}C-FPBalc, 9.5 mg/kg ^{14}C-FPBald, 9.0 mg/kg ^{14}C-FPBacid or 0.95 mg/kg ^{14}C-DCVA (Anon 1985 87103) and stored at -18 to -23 ºC for periods of up to 189 days. The distribution of radioactivity in the TLC patterns was determined by liquid scintillation spectroscopy. The percentage decomposition was calculated based on the theoretical amount of compound added to each sample as determined by radio assaying the spiking solution.

Table 34. Stability of cyfluthrin metabolites on cotton seed stored at -18 to -23 ºC (Anon 1985 87103)

Metabolite	Interval (days)	Residue (mg/kg)	Average residue
FPBalc	0	9.8 10 11	10
	95	8.6 9.0	8.8
	189	8.4 8.6	8.5
FPBald	0	9.1 9.5 9.1	9.2
	95	8.0 7.9	8.0
	189	7.6 7.7	7.6
FPBacid	0	9.0 8.7 9.0	8.9
	91	8.3 8.2	8.2
	185	8.4 9.3	8.8
DCVA	0	0.88 0.80 0.84	0.84
	132	0.93 1.04	0.99
	187	0.94 0.93	0.94

Residues of cyfluthrin metabolites (DCVA, FPBalc, FPBald and FPBacid) in cotton seeds were stable for at least 185 – 189 days storage under freezer conditions.

The freezer storage stability of DCVA was studied (Anon 1985 87104). Samples of soya bean were fortified with approximately 1 mg/kg of ^{14}C-DCVA.

Table 35. Stability of DCVA on soya beans and dry vines on storage at -18 to -23 ºC (Anon 1985 87104)

Metabolite	Interval (days)	Residue (mg/kg)	average
Bean	0	0.77 0.78 0.79	0.78
	139	0.90 0.85	0.88
	194	0.83 0.76	0.80
Dry Vines	0	0.79 0.89 0.76	0.81
	134	0.97 1.02	1.0
	189	0.89 0.92	0.91

DCVA showed no apparent decomposition in soya bean seeds and dry vines after 189 – 194 days under freezer conditions.

Krebber (1999 MR-697/99) studied the frozen storage stability of cyfluthrin in samples of bovine liver, kidney, muscle and fat fortified with a mixture of cyfluthrin. Tissue samples were cut into 10 g pieces, placed into glass bottles and fortified with cyfluthrin. The fortification resulted in nominal concentrations of 0.095 mg/kg in liver, kidney and muscle samples and 0.238 mg/kg in fat samples. The bottles were stored in a freezer at -18 °C or below until analysis. Samples were analysed after approximately two months of storage. Control samples and concurrent recovery samples were analysed along with the storage samples. Method 00553/M001 (MR-492/99) was used for the analyses.

Table 36. Percentage decomposition of cyfluthrin in fortified cattle tissues (Krebber 1999 MR-697/99)

Sample Material	Storage Period (days)	Concentration (mg/kg)			Procedural Recovery %[a]		
		Sample 1	Sample 2	Mean	1	2	Mean
Liver	54	0.0513	0.0522	0.0518	93	69	81
Kidney	55	0.0532	0.0513	0.0522	60	62	61
Muscle	57	0.0542	0.0560	0.0551	64	-	-
Fat	54	0.155	0.155	0.155	81	70	76

a - fortification level for concurrent recoveries not specified

The analytical recoveries were low for procedural recovery samples. It is not clear that fortification of tissue pieces rather than homogenates would reflect the stability of incurred residues in tissues.

Shaw (1983, 86041) evaluated the stability of cyfluthrin residues in bovine tissues. Samples from the lactating cow metabolism study were analysed by HPLC, TLC, autoradiography and radioassay after various intervals of freezer storage. The tissues were ground with dry ice and maintained in a freezer (-18 to -23 °C) until analysed.

Table 37 Storage stability of incurred and fortified residues of cyfluthrin in bovine tissues and milk on freezer storage (Shaw 1983, 86041)

Sample	Cyfluthrin (%TRR)	Fortification level (mg/kg)	Storage interval (days)	Residue (mg/kg)
Cyfluthrin				
Milk	98		0	0.055
	98		315	0.055
	98		342	0.055
Liver	93	2	0	1.86
	91		21	1.82
Muscle	99		144 [a]	0.021
Fat	96		158 [a]	0.221
Kidney	82		0	0.154
	57		43	0.107
	17		189	0.032
COOH-cyfluthrin				
Liver			0	0.609
			35	0.566
			76	0.535
			222	0.386

a - Storage interval is the time from sacrifice. Residues of cyfluthrin in muscle reported in the metabolism study were 0.022 mg/kg and in fat 0.23 mg/kg.

Incurred residues of cyfluthrin in milk, muscle and liver are stable when stored frozen for at least 342, 144 and 158 days respectively. Residues in fortified liver samples stored frozen are stable for at least 21 days.

The stability of fortified liver samples (10 g) was studied further by Lemke (1987 94303). Residues in liver samples fortified at 1 mg/kg with cyfluthrin were stable for at least 350 days storage. Residues measured at 11, 42, 154 and 350 days were 0.93, 0.88, 1.02 and 1.05 mg/kg.

USE PATTERNS

Cyfluthrin and beta-cyfluthrin are insecticides from the group of pyrethroids. Action is mainly as a contact insecticide. Cyfluthrin and beta-cyfluthrin have a broad spectrum of activity against numerous species of insects, in particular lepidopterous insects, e.g., *Heliothis spp., Spodoptera spp., Earias insulana* etc. They also give control of some beetle species and their larvae. In the group of sucking insects, cyfluthrin is registered for control of *Psylla piri* and acceptable control of aphids and spider mites.

Formulations containing beta-cyfluthrin/cyfluthrin, alone or co-formulated with other compounds, are registered for use on a wide variety of crops in over 50 countries. Registered uses include as a foliar spray on vegetables, citrus, tree fruits, cotton, soya bean, and other oilseed crops as well as seed treatments.

Table 38. Registered uses of cyfluthrin on citrus

	Country	Application							
		F/G [a]	FL	Method	N	Rate g ai/ha	Conc g ai/hL	Spay vol L/ha	PHI days
Citrus	Japan	F	EW	Spray	5	0.175	3.5	7000	14
Citrus	USA	F	WP/EC	Spray [b]	4	28 – 112 max 112/season max 112/7 days		Min 234	0

a - Field or Greenhouse

b - Application method aerial or ground

Table 39. Registered uses of beta-cyfluthrin on citrus

	Country	Application							
		F/G	FL	Method	N	Rate g ai/ha	Conc g ai/hL	Spray vol L/ha	PHI days
Citrus	Cyprus	F	EC	Spray			1.2		14
Citrus	Indonesia	F	EC	Spray			1.2 – 2.5		15
Citrus	Korea	F	EC	Spray	1 – 5		1.2		14
Citrus	USA	F	EC	Spray [a]	4	14 – 56 max 56/season max 56/7 days		Min 234	0

Table 40. Registered uses of cyfluthrin on pome fruits

	Country	Application							
		F/G	FL	Method	N	Rate g ai/ha	Conc g ai/hL	Spray vol L/ha	PHI days
Apple	Belgium	F	EC	Spray	3	15 – 20			7
Apple	Greece	F	EC	Spray			0.25		14
Apple	Italy	F	EW	Spray			2.5 – 5		3
Apple	Japan	F	EW	Spray	2	0.03 – 0.18	0.66 – 3.5	2000 – 7000	14
Apple	Korea	F	WP/EC	Spray	1 – 6		2.5		7
Apple	Portugal	F	EC	Spray			2.5	1000	7
Apple	Spain	F	SL	Spray		20 – 35	(0.05 – 0.08%)		15
Apple	USA	F	EC/WP	Spray		25 – 49 max 49/season max 49/14 days		Min 936 Min 234 [a]	7
Japanese Pear	Japan	F	EW	Spray	2	0.18	3.5	7000	7
Pear	Belgium	F	EC	Spray	3	15 – 20			7
Pear	Greece	F	EC	Spray			0.25		14
Pear	Italy	F	EW/EC	Spray			2.5 – 5		21
Pear	Portugal	F	EC	Spray			2.5	1000	7
Pear	Spain	F	SL	Spray		20 – 35	(0.05 – 0.08%)		15
Pear	USA	F	EC/WP	Spray		25 – 49 max 49/season max 49/14 days		Min 936 Min 234 [a]	7

a - Application method aerial or ground

Table 41. Registered uses of beta-cyfluthrin in pome fruits

	Country	Application							
		F/G	FL	Method	N	Rate g ai/ha	Conc. g ai/hL	Spray vol. L/ha	PHI days
Apple	Cyprus	F	SC	Spray			1.2		7
Apple	France	F	EC	Spray		0.75			14
Apple	Korea	F	EC	Spray			1.2		7
Apple	Poland	F	EC	Spray		19		500 – 750	7
	Portugal	F	SC	Spray			1.2	1000	7
Apple	Spain	F	CS	Spray		10 – 18	(0.05 – 0.08%)		15
Apple	Sweden	F	SC	Spray			(0.05%)	200 – 400	
Apple	USA	F	EC	Spray	2	12 – 25 max 24.6/season max 25/14 days		Min 936 Min 234 [a]	7
Pear	Cyprus	F	SC	Spray			1.2		7
Pear	France	F	EC	Spray		0.75			14
Pear	Poland	F	EC	Spray		19		500 – 750	7
Pear	Portugal	F	SC	Spray			1.2	1000	7
Pear	Spain	F	CS	Spray		10 – 18	(0.05 – 0.08%)		15
	Sweden	F	SC	Spray			(0.05%)	200 – 400	
Pear	USA	F	EC	Spray	2	12 – 25 max 24.6/season max 25/14 days		Min 936 Min 234 [a]	7

a - Application method aerial or ground

NA = not applicable

Table 42. Registered uses of cyfluthrin on mangoes

	Country	Application							
		F/G	FL	Method	N	Rate g ai/ha	Conc. g ai/hL	Spray Vol. L/ha	PHI days
Mango	Philippines	F	EC [a]	Spray		18 – 50	2.3 – 2.5	800 – 2000	14
Mango	Philippines	F	EC	Spray		12 – 30	2.5 – 3.8	500 – 800	7

a - EC (+imidacloprid)

Table 43. Registered uses of beta-cyfluthrin on mangoes

	Country	Application							
		F/G	FL	Method	N	Rate g ai/ha	Conc g ai/hL	Spray vol L/ha	PHI days
Mango	Philippines	F	EC [a]	Spray		3.6 – 8	1.2 – 1.6	300 – 500	14
Mango	Philippines	F	EC	Spray		38 – 250	4.7 – 10	800 – 2500	28
Mango	Taiwan	F	EC	Spray	7	29 – 35			6

a - EC (+chlorpyrifos)

Table 44. Registered uses of cyfluthrin on brassica (cole) leafy vegetables

	Country	Application							
		F/G	FL	Method	N	Rate g ai/ha	Conc. g ai/hL	Spray vol. L/ha	PHI days
Brassicas	Belgium	F	EC	Spray	2	25			14
Brassicas	Italy	F	EC/EW	Spray	5	25	2.5 – 5		3
Brassicas	Spain	F	SL	Spray		20 – 35	(0.05 – 0.08%)		7
Brassicas	USA	F	WP	Spray [b]	4	15 – 56 max 224/season max 56/7 days			0
Cabbage	China	F	EC	Spray		20 – 25			7
Cabbage	Japan	F	AL	Spray	1 – 4		5		7
Cabbage	Japan	F	EC	Spray	1 – 4		5	1500	7
Cabbage	Portugal	F	EC	Spray			3.1 – 3.8	800	2
Chinese Cabbage	Italy	F	EC/EW	Spray	5		2.5 – 5		7
Chinese Cabbage	Japan	F	EC	Spray	1 – 4		5	1500	7
Chinese Cabbage	Korea	F	G [a]	Spray	1	0.2			n.a.

a - Granule (+ tebuprimifos)

b - Application method aerial or ground

Table 45. Registered uses of beta-cyfluthrin on brassica (cole) leafy vegetables

	Country	Application							
		F/G	FL	Method	N	Rate g ai/ha	Conc g ai/hL	Spray vol L/ha	PHI days
Brassicas	Australia	F	EC	Spray		7.5 – 15	1 – 2	100 – 1000	1 (3 broccoli)
Brassicas	Cyprus	F	SC	Spray			1.2		7
Brassicas	Germany	F	EC	Spray	3	7.74			7
Brassicas	Poland	F	EC	Spray		5 – 10		200 – 600	7
Brassicas	Spain	F	CS	Spray		10 – 18	(0.05 – 0.08%)		7
Brassicas	USA	F	EC	Spray [a]	1 – 4	7.3 – 28 max 112/season max 28/7 days			0
Cabbage	China	F	SC	Spray		10 – 12			7
Cabbage	France	F	EC	Spray		7.5			7
Cabbage	Indonesia	F	EC	Spray			1.2 – 2.5		15
Cabbage	Portugal	F	SC	Spray			0.75 – 1.2	800	2
Cabbage	Slovenia	F	EC	Spray	2	8.0 – 13			7
Cabbage	Sweden	F	SC	Spray		10		200 – 400	7

a - Application method aerial or ground

Table 46. Registered uses of cyfluthrin on aubergines, peppers and tomatoes

		Application							
	Country	F/G	FL	Method	N	Rate g ai/ha	Conc g ai/hL	Spray vol L/ha	PHI days
Aubergine	USA	F	EC/WP	Spray [a]	6	28 – 49 max 295/season max 49/7 days			7
Pepper	Italy	F	EC/EW	Spray	5	max 25	2.5 – 5		3
Pepper	Spain	F	SL	Spray		20 – 35	0.4 – 0.7 (0.05 – 0.08%)		3
Pepper	USA	F	EC/WP	Spray [a]	6	28 – 49 max 295/season max 49/7 days			7
Tomato	Italy	F	EC/EW	Spray			2.5 – 5		3
Tomato	Philippines	F	EC	Spray		22 – 25		500 – 800	7
Tomato	Portugal	F	EC	Spray			3.1 – 3.8	800	2
Tomato	Spain	F	SL	Spray		20 – 35	(0.05 – 0.08%)		3
Tomato	Turkey	F	EC	Spray		25	2.5		14
Tomato	USA	F	EC/WP	Spray [a]	6	28 – 49 max 295/season max 49/7 days		15 3 – 7.7 [a]	0

a - Application method aerial or ground

NA = not applicable

Table 47. Registered uses of beta-cyfluthrin on aubergine, peppers and tomatoes

		Application							
	Country	F/G	FL	Method	N	Rate g ai/ha	Conc. g ai/hL	Spray vol. L/ha	PHI days
Aubergine	Cyprus	F	SC	Spray			1.2		3
Aubergine	Philippines	F	EC [a]	Spray			0.62 – 0.81	300 – 500	3
Aubergine	Philippines	F	EC	Spray			0.38 – 0.5	400	3
Aubergine	Thailand	F	EC	Spray			10		14
Aubergine	USA	F	EC	Spray [b]	6	14 – 25 max 147/season max 25/7 days			7
Pepper	Cyprus	F	SC	Spray			2.5		3
Pepper	Indonesia	F	EC	Spray		12 – 25			15
Pepper	Korea	F	EC	Spray	1 – 6		1.2		7
Pepper	Slovenia	F	EC	Spray	2	8 – 13			3
Pepper	Spain	F	CS	Spray		10 – 18	(0.05 – 0.08%)		3
Pepper	USA	F	EC	Spray [b]	6	14 – 25 max 147/season max 25/7 days			7
Tomato	Australia	F	EC	Spray		7.5 – 15	1 – 2	100 – 1000	1
Tomato	Cyprus	F	SC	Spray			12		3

	Country	Application							
		F/G	FL	Method	N	Rate g ai/ha	Conc. g ai/hL	Spray vol. L/ha	PHI days
Tomato	Indonesia	F	EC	Spray			1.2 – 1.5		15
Tomato	Poland	F	EC	Spray		7.5		200 – 600	7
Tomato	Portugal	F	SC	Spray			1.6	800 – 1000	2
Tomato	Spain	F	CS	Spray		10 – 18	(0.05 – 0.08%)		3
Tomato	USA	F	EC	Spray [b]	6	14 – 25 max 147/season max 25/7 days			0

a - EC (+ chlorpyrifos)

b - Application method aerial or ground

NA = not applicable

Table 48. Registered uses of cyfluthrin on sweet corn

	Country	Application							
		F/G	FL	Method	N	Rate g ai/ha	Conc. g ai/hL	Spray vol L/ha	PHI days
Corn	Philippines	F	EC				0.63 – 1.2	300 – 500	13
Sweet Corn	USA	F	EC/WP	Spray [b]	1 – 10	15 – 49 max 493/season max 49/2 days			0
Sweet Corn	USA	G	EC	Soil at planting	1 – 10	35 – 49			0

Table 49. Registered uses of beta-cyfluthrin on sweet corn

	Country	Application							
		F/G	FL	Method	N	Rate g ai/ha	Conc. g ai/hL	Spray vol L/ha	PHI days
Sweet Corn	USA	F	EC	Spray [a]	10	7.3 – 25 max 246/season max 25/2 days			0

a - Application method aerial or ground

Table 50. Registered uses of cyfluthrin on potatoes

	Country	Application							
		F/G	FL	Method	N	Rate g ai/ha	Conc g ai/hL	Spray vol L/ha	PHI days
Potato	Italy	F	EW	Spray			1.5 – 2.5		35
Potato	Portugal	F	EC	Spray			3.1	800	14
Potato	Spain	F	SL	Spray		20 – 35	(0.05 – 0.08%)		15
Potato	Turkey	F	EC	Spray		25			14

	Country	Application							
		F/G	FL	Method	N	Rate g ai/ha	Conc g ai/hL	Spray vol L/ha	PHI days
Potato	USA	F	EC/WP	Spray [b]	6	15 – 49 max 295 season max 49/5 days			0 [a]
Potato	USA	F	SE	Spray [b]		26 – 33 max 131/season max 33/7 days			7

a - If more than 48 g ai/ha allow at least 14 days between last application and grazing

b - Application method aerial or ground

Table 51. Registered uses of beta-cyfluthrin on potatoes

	Country	Application							
		F/G	FL	Method	N	Rate g ai/ha	Conc g ai/hL	Spray vol L/ha	PHI days
Potato	Cyprus	F	SC	Spray			1.25		3
Potato	Germany	F	EC	Spray	1	7.7			28
Potato	Indonesia	F	EC	Spray			1.2 – 2.5		15
Potato	Poland	F	EC	Spray		5 – 7.5			7
Potato	Portugal	F	SC	Spray			1.2	800 – 1000	14
Potato	Slovenia	F	EC	Spray	2	13			7
Potato	Spain	F	CS	Spray		10 – 18	(0.05 – 0.08%)		15
Potato	Sweden	F		Spray		10 – 12		200 – 400	28
Potato	USA	F	EC	Spray [b]	5	7.3 – 25 max 147 season max 25/5 days			0 [a]

a - If more than 48 g ai/ha allow at least 14 days between last application and grazing.

b - Application method aerial or ground

Table 52. Registered uses of cyfluthrin on oilseeds

	Country	Application							
		F/G	FL	Method	N	Rate g ai/ha	Conc. g ai/hL	Spray vol. L/ha	PHI days
Cotton	China	F	EC	Spray		24 – 38			
Cotton	Greece	F	EC	Spray			0.25		21
Cotton	Philippines	F	EC	Spray		22 – 25		500 – 800	7
Cotton	Spain	F	SL	Spray		20 – 35	(0.05 – 0.08%)		15
Cotton	Turkey	F	EC	Spray		75 – 100			14
Cotton	USA	F	EC	Spray [e]	10	15 – 49 max 560/season, max 56/3 days			0 [a]
Cotton	USA	F	SE	Spray [e]	10	26 – 33 max 197/season max 33/7 days			14
Rape	Belgium	F	EC	Spray	2	15			[b]

	Country	Application							
		F/G	FL	Method	N	Rate g ai/ha	Conc. g ai/hL	Spray vol. L/ha	PHI days
Soy bean	USA	F	EC	Spray [e]	1 – 4	15 – 49 max 196/season, max 49/7 days			45[c]
Sunflower	USA	F	EC	Spray [e]	1 – 5 1 – 4 [e]	15 – 49 max 147/season, max 49/7 days		15 3 [e]	30 [d]

a - Do not graze treated fields.

b - Application according to growth stage, maximum 2 applications per crop, one spray at seed to 3 leaf BBCH 10 – 13, one at bud development BBCH 50 – 59 and one at pod development BBCH 70 – 75.

c - Feeding of dry vines 45 days, green forage may be fed 15 days after last application.

d - Pre-grazing or foraging interval 30 days

e - Aerial application method

Table 53. Registered uses of beta-cyfluthrin on oilseeds

	Country	Application							
		F/G	FL	Method	N	Rate g ai/ha	Conc. g ai/hL	Spray vol. L/ha	PHI days
Cotton	Australia	F	EC/UL	spray		5 – 20			28
Cotton	China	F	EC	Spray		9.4 – 13			
Cotton	India	F	SC	Spray		12 – 18		500 – 1000	20
Cotton	Indonesia	F	EC	Spray		12 – 50			15
Cotton	Spain	F	CS	Spray		10 – 18	(0.05 – 0.08%)		15
Cotton	Thailand	F	EC	Spray			3.8		14
Cotton	USA	F	EC	Spray [e]	1 – 10	7.3 – 28 max 280/season max 28/3 days			0 [a]
Rape	Australia	F	EC	Spray		5 – 10			14
Rape	France	F	EC	spray		7.5			30
Rape	Germany	F	EC	spray	3	5.2 – 7.7	0.2 – 0.3		56
Rape	Slovenia	F	EC	spray	2	8.0 – 13			30
Soya bean	Brazil	F	SC	[e] spray		2.5 – 10			20
Soya bean	Brazil	F	SC [d]	[e] spray	2	5 – 12.5			21
Soya bean	Indonesia	F	EC	spray	1 – 5	6.2 – 25	0.94 – 3.8		15
Soya bean	Thailand	F	EC	spray			5		14
Soya bean	USA	F	EC	Spray [e]	1 – 4	7.3 – 28 max 98/season max 28/3 days			45 [b]
Sunflower	USA	F	EC	Spray [e]	1 – 6	7.3 – 28 max 73/season max 28/3 days			30 [c]

a - Do not graze treated fields.

b - Feeding of dry vines 45 days, green forage may be fed 15 days after last application.

c - Pre-grazing or foraging interval 30 days.

d - Formulation includes imidacloprid.

e - Application method aerial or ground

RESIDUES RESULTING FROM SUPERVISED TRIALS

The Meeting received information on supervised field trials for cyfluthrin and beta-cyfluthrin uses on the following crops:

Crop group	Compound	Commodity	Table
Citrus fruits	Cyfluthrin	Grapefruit	Table 54
	Cyfluthrin	Lemon	Table 55
	Cyfluthrin	Orange	Table 56
Pome fruit	Cyfluthrin	Apples	Table 57
	Cyfluthrin	Pears	Table 58
Assorted tropical fruits, inedible peel	Cyfluthrin	Mango	Table 59
	Beta-cyfluthrin	Mango	Table 60
Brassica and Cole vegetables	Cyfluthrin	Broccoli	Table 61
	Cyfluthrin	Brussels sprouts,	Table 62
	Cyfluthrin	Cabbage including Chinese cabbage	Table 63
	Cyfluthrin	Cauliflower	Table 64
Fruiting vegetables	Cyfluthrin	Tomato	Table 65
	Cyfluthrin	Peppers	Table 66
	Cyfluthrin	Sweet corn	Table 67
Root and tuber vegetables	Cyfluthrin	Potato	Table 68
Oilseeds	Cyfluthrin	Cotton	Table 69
	Beta-cyfluthrin	Cotton	Table 70
	Cyfluthrin	Soya beans	Table 71
	Beta-cyfluthrin	Soya beans	Table 72
	Cyfluthrin	Rape	Table 73
	Beta-cyfluthrin	Rape	Table 74
	Cyfluthrin	Sunflowers	Table 75
Animal feed	Cyfluthrin	Sweet corn feed items	Table 76
	Cyfluthrin	Cotton gin-trash	Table 77
	Beta-cyfluthrin	Cotton gin-trash	Table 78
	Cyfluthrin	Soya bean feed items	Table 79
	Cyfluthrin	Rape feed items	Table 80
	Beta-cyfluthrin	Rape feed items	Table 81
	Cyfluthrin	Sunflower fodder	Table 82

Where duplicate field samples from an unreplicated plot were taken at each sampling time and were analysed separately, the mean of the two analytical results was taken as the best estimate of the residues in the plot and the means are recorded in the tables. When residues were not detected they are shown as below the LOQ (e.g., < 0.01 mg/kg). Residues, application rates and spray concentrations have generally been rounded to two significant figures or, for residues near the LOQ, to one significant figure. Residue values from the trials conducted according to maximum GAP have been used for the estimation of maximum residue levels. Those results included in the evaluation are underlined.

Conditions of the supervised residue trials were generally well reported in detailed field reports. Most trial designs used non-replicated plots. Most field reports provided data on the sprayers used, plot size, field sample size and sampling date. Some studies on sweet corn and soya beans were based on analytical data from Craven Laboratories and are invalidated and could not be used.

Citrus fruit

Duah and Lemke (2002 111025) studied residues in citrus after single applications of EC and WP sprays to trees using both dilute and concentrated sprays. Plot sizes were 133 – 357 m^2 with applications made using air-blast sprayers. Control samples fortified with cyfluthrin were extracted and analysed with the treated samples. The method used in the trials conducted in 2000/2001 was detailed in report no. 108139-1 (Moore *et al.,* 2001 108139-1) and is based on quantification using GC-MS (SIM m/z 207). Recovery of cyfluthrin from grapefruits fortified at 0.01 mg/kg ranged from 90% to 98%, while the recovery from grapefruits fortified at 0.10 mg/kg was 98%. Recoveries of cyfluthrin from lemons fortified at 0.01, 0.10, and 0.15 mg/kg ranged from 88% to 100%, from 92% to 99%, and from 94% to 101%, respectively. Recoveries of cyfluthrin from oranges fortified at 0.01 and 0.10 mg/kg ranged from 93% to 102% and from 97% to 98%, respectively. The citrus fruit were held in frozen storage for a maximum of 522 days prior to extraction.

Trials on grapefruit, oranges and lemons conducted in the USA in 1993 were reported by Burger (1993 106243) with amendments reported by Woodward (1999 106243). Trial plots ranged from four trees to one or two rows of 37 m length. Application was by air-blast or mist sprayers. The analytical method used was Harbin *et al.,* 1983 (85823). Concurrent recoveries for cyfluthrin ranged from 73% to 110%. Citrus samples were held in frozen storage for a maximum period of 305 days.

Burger (1992 102618) reported results for trials conducted in 1991 on grapefruit, lemons and oranges. Plot sizes were three to four trees. Application was by air-blast or mist sprayers. The analytical method used was Harbin *et al.,* 1983 (85823). Concurrent recoveries ranged from 68% to 123%. Processed orange samples and orange, grapefruit and lemon samples were held in frozen storage for a maximum period of 199 days (date of harvest to date of analysis).

Table 54. Residues for cyfluthrin in grapefruit

Country	Application					Residues			Reference
	FL	N	g ai/ha	L/ha	Growth stage at last application	Sample	PHI (days)	Cyfluthrin (mg/kg)	
Calipatria, California, USA, 2001, Ruby Red	EC	1	118	4482	83	fruit	0	0.04	111025
	EC	1	114	2012	83	fruit	0	0.04	
	WP	1	118	4452	83	fruit	0	0.03	
	WP	1	112	1974	83	fruit	0	0.04	
Sacaton, Arizona, USA, 2000, Ruby Red	EC	1	114	4505	89	fruit	0	0.03	111025
	EC	1	110	2306	89	fruit	0	0.02	
	WP	1	107	4311	89	fruit	0	0.04	
	WP	1	108	2265	89	fruit	0	0.02	
Nipomo, California, USA, 2000, Star Ruby	EC	1	112	4821	83	fruit	0 7 14 21	0.06 0.07 < 0.01 0.05	111025
	EC	1	112	1971	83	fruit	0 7 14 21	0.01 0.02 0.03 0.02	
	WP	1	112	4667	83	fruit	0 7 14 21	0.01 0.01 0.02 < 0.01	
	WP	1	112	1967	83	fruit	0 7 14 21	< 0.01 < 0.01 < 0.01 < 0.01	

Country	Application					Residues			Reference
	FL	N	g ai/ha	L/ha	Growth stage at last application	Sample	PHI (days)	Cyfluthrin (mg/kg)	
Arizona, USA 1992, Marsh	EC	1	112	654		fruit	0 3 7 14	0.05 0.11 0.06 0.03	106243
Fresno, California USA 1992 Marsh	EC	1	112	2356		fruit	0 3 7 14	0.04 0.02 0.02 0.02	106243
Corona, California, USA 1991, Star Ruby	EC	1	26 (3 g ai/hL)	866	bloom	fruit	0 3 7 14 31	0.02 0.01 < 0.01 < 0.01 < 0.01	102618
Yuma, Arizona, USA, 1991, Pink	EC	1	50 (3 g ai/hL)	1737	Mature fruit	fruit	0 3 7 14 31	< 0.01 < 0.01 0.02 0.02 0.02	102618

Table 55. Residues for cyfluthrin in oranges

Country	Application					Residues			Reference
	FL	N	g ai/ha	L/ha	Growth stage at last application	sample	PHI (days)	Cyfluthrin (mg/kg)	
Calipatria, California, USA, 2001, Valencia	EC	1	112	2007	83	fruit	0 7 14 21	0.05 0.03 0.01 0.01	111025
	EC	1	113	4526	83	fruit	0 7 14 21	0.04 0.02 < 0.01 0.01	
	WP	1	112	2013	83	fruit	0 7 14 21	0.03 0.01 0.01 0.01	
	WP	1	119	4485	83	fruit	0 7 14 21	0.02 0.01 < 0.01 < 0.01	
Sacaton, Arizona, USA, 2000, Washington Navel	EC	1	112	2078	89	fruit	0	0.06	111025
	EC	1	114	4142	89	fruit	0	0.06	
	WP	1	114	2167	89	fruit	0	0.06	
	WP	1	113	4106	89	fruit	0	0.05	
Fresno, California, USA, 2000, Navel	EC	1	114	2372	83	fruit	0	0.06	111025
	EC	1	113	4464	83	fruit	0	0.04	
	WP	1	113	2357	83	fruit	0	0.06	
	WP	1	114	4481	83	fruit	0	0.03	
	WP	1	114	4479	83	Fruit [a]	0	0.03	
						Fruit cooked		< 0.01	
						Flesh		0.003	
						Peel		0.08	
Fresno, California, USA, 1992, Navel	EC	1	112	2356		fruit	0 3 7 14	0.05 0.04 0.03 0.03	106243

Country	Application					Residues			Reference
	FL	N	g ai/ha	L/ha	Growth stage at last application	sample	PHI (days)	Cyfluthrin (mg/kg)	
Fresno, California, USA, 1992, Valencia	EC	1	112	2356		fruit	0 3 7 14	0.05 0.05 0.04 0.05	106243
Yuma, Arizona, USA, 1992, Navel	EC	1	112	654		fruit	0 3 7 14	0.03 0.02 0.01 0.02	106243
Ivanhoe, California, USA 1991, Navel	EC	1	70 (10 g ai/hL)	683	mature	fruit	0 3 7 13	0.20 0.10 0.08 0.07	102618
Yuma, Arizona, USA 1991 Valencia	EC	1	21 (3 g ai/hL)	701	mature	fruit	0 3 7 14 31	< 0.01 0.01 0.01 < 0.01 < 0.01	102618
Corona, California, USA, 1991, Olinda Valencia	EC	1	26 (3 g ai/hL)	866	bloom	fruit	0 3 7 14 31	0.02 < 0.01 < 0.01 < 0.01 < 0.01	102618
Exeter, California, USA, 1990, Atwood	EC	1	113	1870	petal fall	fruit	196 196 196 196 196 196	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	100345

a - A subsample of the oranges was peeled, the peeled fruits were quartered, and the combined peels and fruit pieces (with juice) were cooked. Water and baking soda were added to the peels, and the peel was cooked for 20 to 25 min after reaching a boil. The quartered peeled fruits (with juice) were added to the cooked peel, and the peeled fruit (with juice) and cooked peel mixture was simmered for 10 to 15 min after reaching a boil. Three to six cups of the mixture were measured into a container, sugar was added, and the mixture was heated to a boil. Pectin was added, and the sample was allowed to boil for 1 min. The sample was removed from heat, foam was skimmed off, and the cooked sample was transferred into 3-pint jars. The samples in the jars were sequentially processed in a boiling-water canner for 5 min, allowed to cool, transferred into plastic bags, and placed in frozen storage. A second subsample of the oranges was peeled, the peeled fruits were quartered, and the peels and peeled fruits were individually frozen.

Table 56. Residues for cyfluthrin in lemons

Country	Application					Residues			Reference
	FL	N	g ai/ha	L/ha	Growth stage at last application	sample	PHI (days)	Cyfluthrin (mg/kg)	
Terra Bella, California, USA, 2000, Lisbon	EC	1	110	2548	89	fruit	0	0.08	111025
	EC	1	110	3992	89	fruit	0	0.06	
	WP	1	109	2524	89	fruit	0	0.05	
	WP	1	111	4000	89	fruit	0	0.05	
Yuma, Arizona, USA, 2000, Lisbon	EC	1	105	1993	83	fruit	0 7 14 21	0.08 0.06 0.05 0.05	111025
	EC	1	112	4667	83	fruit	0 7 14 21	0.11 0.10 0.06 0.08	

Country	Application					Residues			Reference
	FL	N	g ai/ha	L/ha	Growth stage at last application	sample	PHI (days)	Cyfluthrin (mg/kg)	
	WP	1	106	1975	83	fruit	0 7 14 21	0.08 0.07 0.05 0.06	
	WP	1	112	4649	83	fruit	0 7 14 21	0.10 0.07 0.07 0.06	
Fallbrook, California, USA, 2000, Eureka	EC	1	110	2092	83	fruit	0	0.08	111025
	EC	1	110	4583	83	fruit	0	0.07	
	WP	1	115	2163	83	fruit	0	0.05	
	WP	1	111	4606	83	fruit	0	0.06	
Fresno, California, USA, 1992, Lisbon	EC	1	112	2356		fruit	0 3 7 14	0.08 0.10 0.10 0.08	106243
Yuma, Arizona, USA, 1992 Lisbon	EC	1	112	654		fruit	0 3 7 14	0.05 0.02 0.10 < 0.01	106243
Corona, California, USA, 1991, Eureka	EC	1	26 (3 g ai/hL)	866	bloom	fruit	0 3 7 14 31	0.06 0.04 0.03 0.02 0.02	102618
Yuma, Arizona, USA, 1991, Lisbon	EC	1	70 (3 g ai/hL)	2337	fruiting	fruit	0 3 7 14 28	0.04 0.02 0.03 0.01 0.02	102618

Apples and Pears

Harbin (2002 110031) reported residue trials conducted in the USA on apples and pears. Plot sizes were 67 – 268 m^2. Application was by air-blast and mist blowers. A minimum of 24 fruits, total at least 2.3 kg, were collected for each apple and pear sample. Analyses were by GC/MS (Moore 2001 108139-1). Recoveries of cyfluthrin from apples and pears fortified at 0.01 mg/kg ranged from 97% to 103% and from 78% to 96%, respectively. Recoveries of cyfluthrin from apples and pears fortified at 0.05 ppm ranged from 86% to 121%. The pome fruit samples analysed were held in frozen storage for a maximum of 17 months (508 days).

Table 57. Residues for Cyfluthrin in apples

Country	Application					Residues			Reference
	FL	N	g ai/ha	L/ha	Growth stage at last application	sample	PHI (days)	Cyfluthrin (mg/kg)	
Lyons, New York, USA, 1999, Northern Spys	EC	1	49	748	Beginning of ripening	fruit	0 7 14 21 28	0.04 0.02 0.02 0.01 0.01	110031
	EC	1	49	2243	Beginning of ripening	fruit	0 7 14 21 28	0.02 0.02 0.01 0.01 < 0.01	

Country	Application					Residues			Reference
	FL	N	g ai/ha	L/ha	Growth stage at last application	sample	PHI (days)	Cyfluthrin (mg/kg)	
	WP	1	49	747	Beginning of ripening	fruit	0 7 14 21 28	0.05 0.01 0.01 < 0.01 0.01	
	WP	1	49	2243	Beginning of ripening	fruit	0 7 14 21 28	0.04 0.03 0.01 0.01 0.01	
Parkdale, Oregon, USA, 1999, Red Delicious	EC	1	50	799	Advanced ripening	fruit	7 14	0.02 0.02	110031
	EC	1	49	2461	Advanced ripening	fruit	7 14	0.04 0.03	
	WP	1	49	789	Advanced ripening	fruit	7 14	0.02 0.02	
	WP	1	52	2565	Advanced ripening	fruit	7 14	0.02 0.02	
Dundee, New York, USA, 1999, McIntosh	WP	1	49	938	Beginning of ripening	fruit	7 14	0.01 < 0.01	110031
	WP	1	50	1918	Beginning of ripening	fruit	7 14	0.02 0.01	
Barto, Pennsylvania, USA, 1999, Law Rome/MM	WP	1	49	818	Beginning of ripening	fruit	6 13	0.02 0.01	110031
	WP	1	49	2522	Beginning of ripening	fruit	6 13	0.02 < 0.01	
Knightdale, North Carolina, USA, 1999, Red Delicious	WP	1	49	927	Beginning of ripening	fruit	7 14	< 0.01 < 0.01	110031
	WP	1	49	1880	Beginning of ripening	fruit	7 14	0.02 0.01	
Conklin, Michigan, USA, 1999, Golden Delicious	WP	1	49	725	Fruit about 90% final size	fruit	7 14	0.03 0.02	110031
	WP	1	49	2092	Fruit about 90% final size	fruit	7 14	0.03 0.03	
Beckemeyer, Illinois, USA, 1999, Red Delicious	WP	1	49	822	Beginning of ripening	fruit	7 13	0.02 0.02	110031
	WP	1	49	2621	Beginning of ripening	fruit	7 13	0.02 0.01	
Bonita, Arizona, USA, 1999, Granny Smith	WP	1	49	750	Advanced ripening	fruit	7 14	0.01 0.02	110031
	WP	1	48	2567	Advanced ripening	fruit	7 14	0.02 0.02	
San Luis Obispo, California, USA, 1999, Winesap	WP	1	50	713	Fruit ripe for picking	fruit	7 14	0.04 0.02	110031
	WP	1	49	2352	Fruit ripe for picking	fruit	7 14	0.06 0.04	
Hood River, Oregon, USA, 1999, Jonagold	WP	1	49	697	Beginning of ripening	fruit	0 8 15 21 27	0.03 0.01 0.01 < 0.01 < 0.01	110031
	WP	1	49	2393	Beginning of ripening	fruit	0 8 15 21 27	0.02 0.01 < 0.01 < 0.01 < 0.01	

Country	Application					Residues			Reference
	FL	N	g ai/ha	L/ha	Growth stage at last application	sample	PHI (days)	Cyfluthrin (mg/kg)	
Ephrata, Washington, USA, 1999, Red Delicious	WP	1	49	709	Beginning of ripening	fruit	7 14	< 0.01 < 0.01	110031
	WP	1	49	2569	Beginning of ripening	fruit	7 14	< 0.01 0.01	
White Salmon, Washington, USA, 1999, Red Delicious	WP	1	49	855	Fruit about 90% final size	fruit	7 14	0.03 0.02	110031
	WP	1	49	2231	Fruit about 90% final size	fruit	7 14	0.02 < 0.01	

Table 58. Residues for Cyfluthrin in pear

Country	Application					Residues			Reference
	FL	N	g ai/ha	L/ha	Growth stage at last application	sample	PHI (days)	Cyfluthrin (mg/kg)	
Lyons, New York, USA, 1999, Clapps Favorit	WP	1	49	468	Beginning of ripening	fruit	7 14	0.02 0.01	110031
	WP	1	49	1860	Beginning of ripening	fruit	7 14	0.02 0.02	
LaGrange, California, USA, 1999, Bartlett	WP	1	56	792	Beginning of ripening	fruit	7 14	0.05 0.03	110031
	WP	1	49	1896	Beginning of ripening	fruit	7 14	0.03 0.02	
Sacramento, California, USA 1999, Bartlett	EC	1	52	701	Beginning of ripening	fruit	6 14	0.02 0.02	110031
	EC	1	53	1990	Beginning of ripening	fruit	6 14	0.02 0.01	
	WP	1	49	694	Beginning of ripening	fruit	7 14	0.02 0.01	
	WP	1	50	1996	Beginning of ripening	fruit	7 14	0.02 < 0.01	
White Salmon, Washington, USA, 1999, Red Bartlett	WP	1	49	870	Fruit about 90% final size	fruit	0 7 14 22 30	0.05 0.04 0.03 0.03 0.02	110031
	WP	1	50	3590	Fruit about 90% final size	fruit	0 7 14 22 30	0.03 0.03 0.03 0.02 0.02	
Parkdale, Oregon, USA, 1999, Red Anjou	WP	1	47	701	Fruit ripe for picking	fruit	7 14	0.02 0.02	110031
	WP	1	50	2173	Fruit ripe for picking	fruit	7 14	0.02 0.01	
Greenleaf, Idaho, USA, 1999, Bartlett	WP	1	49	614	Advanced ripening	fruit	7 14	0.01 0.01	110031
	WP	1	49	2716	Advanced ripening	fruit	7 14	0.02 0.02	

Mango

Results of residue trials on mangoes conducted with EC formulations of cyfluthrin and beta-cyfluthrin in 1999 – 2003 were reported. Plot sizes were one to three trees. In the 2003 trials, samples were stored frozen for one month prior to analysis. Method 00200 was used for the 1999 trial.

Table 59. Residues for beta-cyfluthrin and cyfluthrin

	Application					Residues			Reference
Country	FL	N	g ai/hL	L/ha	Growth stage at last application	sample	PHI (days)	cyfluthrin (mg/kg)	
Beta-cyfluthrin									
Dasmarinas Cavite, Philippines, 2001, mango	EC	2	1.6		Fruit maturation	fruit	0 3 7 14 28	0.03 0.03 0.02 0.03 0.02	M-278812-01-1
Kabacan, Cotabato, Philippines, 2003, Carabao Manila Supermango	EC	4	10	(25 L/tree)		fruit	0 3 7 14 28	0.09 0.06 0.03 < 0.01 < 0.01	M-281540-01-1 M-281539-01-1
Cyfluthrin									
Laguna Philippines, 1999, Carabao Manila Supermango	EC	5	2.5	800 – 1000	99 days after Flower induction	fruit	0 3 7 14	0.09 0.08 0.02 0.02	M-283514-01-1
						Peel	14	0.03	
						Pulp	14	< 0.01	

Brassica (Cole) vegetables

Field trials were reported from the USA for trials conducted in 1997 (Grace 1999 109119) and 2000/2001 (Fischer 2002 110339) in which EC and/or WG formulations were applied to broccoli and cabbages. Broccoli samples were collected as heads and stems. Cabbage heads were collected with the wrapper leaves intact, discarding any decomposed leaves from the wrapper as necessary. Sample sizes were ≥1.1 kg for broccoli and 12 heads for cabbage.

For the 2000/2001 trials, plot sizes were 34 – 241 m^2 and application was with knapsack or motorised boom sprayers. The broccoli and cabbage samples analysed in the USA 2000/2001 study were held in frozen storage for a maximum of 10 months (298 days) and 11 months (327 days) respectively, prior to extraction. Control samples fortified with cyfluthrin were extracted and analysed with the treated samples. The method used in the trials conducted in 2000/2001 was detailed in report no. 108139-1 (Moore *et al.,* 2001 108139-1) and is based on quantification using GC-MS (SIM m/z 207). Recoveries of cyfluthrin from broccoli fortified at 0.01 mg/kg and 0.75 mg/kg ranged from 92% to 101%. Recoveries of cyfluthrin from cabbage fortified at 0.01 mg/kg and 3.5 mg/kg ranged from 93% to 105%.

For the 1997 trials, plot sizes were 93 – 279 m^2 and application was with knapsack or motorised boom sprayers. The broccoli and cabbage analysed in the USA 1997 study were held in frozen storage for a maximum of 482 days prior to extraction. The analytical method used was Sandie and Gronberg (1998 108139). Cyfluthrin recoveries from broccoli and cabbage ranged from 72% to 111% when fortified at 0.01 mg/kg, from 77% to 111% when fortified at 0.05 mg/kg, 102% when fortified at 0.10 mg/kg, from 73% to 107% when fortified at 0.40 mg/kg, and from 82% to 88% when fortified at 2.0 mg/kg.

Trials were also reported for cauliflower and cabbage from Canada. The plot size in the 1983 Canada trials was 18 m^2, application by backpack sprayer. Method 085823 was used for analyses.

Leslie (1988 98426) reported residue trials on brassica vegetables conducted in the USA in 1987/88. Plot sizes were 5.6 – 290 m^2. Samples were held in frozen storage for a maximum of 379 days prior to analysis. The samples were analysed using the analytical procedure described in Mobay Report No. 85823. Recoveries for cyfluthrin in broccoli heads fortified at 0.05, 0.5, and 5.0 mg/kg ranged from 72% to 97%. Recoveries for Brussels sprouts fortified at 0.05, 0.10, and 0.5 mg/kg ranged from 72% to 82%. Recoveries for cabbage heads fortified at 0.05, 0.10, and 0.5 mg/kg ranged from 70% to 108%. For cauliflower heads recoveries for samples fortified at 0.05, 0.10, 0.50, and 1.0 mg/kg were 73% to 107%.

Seym and Walz-Tylla (1993 RA-2074/92) reported trials on cabbage from Germany. The size of the treated plots ranged from 15 to 30 m^2. Procedural recoveries were 106 – 113% for samples fortified at 0.01 – 1.0 mg/kg.

Cyfluthrin residues were reported for cabbage and Chinese cabbage trials conducted in Japan in 1983. Plot sizes were 7 – 16 m^2 for the Japan trials. Recoveries for samples fortified at 0.1 mg/kg were 83 – 96% and 94% for samples fortified at 0.2 mg/kg. Samples were analysed within seven months of collection.

Table 60. Residues for Cyfluthrin in Brussels sprouts

Country	Application					Residues			Reference
	FL	N	g ai/ha	L/ha	Growth stage at last application	Sample	PHI (days)	Cyfluthrin (mg/kg)	
Half Moon Bay, California, USA, 1987, Jade Cross	EC	12	50	327	harvest	Heads	1 2 4 8	0.44 0.32 0.11 0.11 0.01[u]	98426
Half Moon Bay, California, USA, 1987, Jade Cross	EC	12	50	327	harvest	Heads	1 2 4 8	0.39 0.21 0.05 0.07	98426

u - Sample from control (untreated) plot.

Table 61. Residues for cyfluthrin in broccoli

Country	Application					Residues			Reference
	FL	N	g ai/ha	L/ha	Growth stage at last application	Sample	PHI (days)	Cyfluthrin (mg/kg)	
Fresno, California, USA, 2001 Green Comet	EC	4	48 50 48 48	179 179 179 172	Main inflorescence visible, inflorescence beginning elongation	curd	0 7 12 17	0.26 0.05 0.02 < 0.01	110339
	WP	4	48 49 48 49	177 176 180 175	Main inflorescence visible, inflorescence beginning elongation	curd	0 7 12 17	0.16 0.03 0.02 < 0.01	
Live Oak, California, USA, 2000, Laguna	EC	4	49 49 49 49	189 190 187 189	9 or more side shoots		0	0.46	110339
	WP	4	49 49 49 49	190 189 189 189	9 or more side shoots		0	0.30	

Country	Application					Residues			Reference
	FL	N	g ai/ha	L/ha	Growth stage at last application	Sample	PHI (days)	Cyfluthrin (mg/kg)	
Hillsboro, Oregon, USA, 2000, Packman	EC	4	48 49 50 49	286 228 290 233	Harvest		0	0.28	110339
	WP	4	52 48 47 50	295 222 265 240	Harvest		0	0.26	
	WP	4	52 48 47 50	295 222 265 240	Harvest	Curd Washed cooked	0 [a]	0.18 0.14 0.39	
Fresno, California, USA 1997, Green Sprouting	EC	4	57 56 56 56	143 141 141 141	mature	curd	0 3 7 14	0.19 0.15 0.12 0.03	109119
Corvallis, Oregon, USA 1997, Gem	EC	4	56 55 56 56	162 165 167 167	harvest	curd	0	0.19	109119
Porterville, California, USA, 1997, Acadia	EC	4	56 56 56 56	140 140 141 139	mature	curd	0	0.19	109119
King City, California, USA, 1997, Green Belt	EC	4	56 56 56 56	140 139 140 140	mature	curd	0	0.04	109119
Guadalupe, California, USA, 1997, Marathon	EC	4	56 57 56 56	188 193 187 187	mature	curd	0	0.30	109119
Uvalde, Texas, USA, 1997, Legacy	EC	4	56 56 56 56	140 140 140 140	mature	curd	0	0.19	109119
Beavercreek, Oregon, USA, 1983, Green Comet	EC	7	50	561	30 – 40% buds open	curd	0 1 3 7	1.5 1.1 0.28 0.14 0.01 [u]	98426
Greenfield, California, USA, 983, Topper 430	EC	7	50	468	harvest	curd	0 1 3 7	0.20 0.12 0.07 0.01 0.02 [u]	98426
Benoit, Mississippi, USA, 1983, Morses 4638	EC	7	375	85	curd development	curd	0 1 3 7	11 9.6 3.4 1.7	98426
Glendale, Arizona, USA, 1983, Gem	EC	7	50	309	earliest harvest	curd	0 1 3 7	0.05 0.02 0.03 0.03	98426
Harlingen, Texas, USA, 1983, Waltham 29	EC	7	50	486	curd development	curd	0 1 3 7	0.29 0.26 0.14 0.05	98426

a - Samples were prepared by rinsing all portions of the broccoli under a stream of tap water (water temperature was lukewarm to cool) for approximately 30 sec. The washed samples were allowed to dry for approximately 2 min on a paper towel. This procedure simulated the preparation of washed broccoli in the home environment. The broccoli

heads were halved or quartered, placed into plastic bags, and frozen for storage. The broccoli samples were washed and frozen on the same day that they were received at BRP. The control and treated cooked sub-samples were prepared by removing the outer leaves and tough parts of the stalk with a knife. The broccoli was cut into florets, and the florets were cooked with a small amount of boiling, salted water in a covered pan until the florets were crisp-tender. This procedure simulated the preparation of cooked broccoli in the home environment. The cooked broccoli was allowed to cool.

u - Sample from control (untreated) plot.

Table 62. Residues for Cyfluthrin in cauliflower

Country	Application					Residues			Reference
	FL	N	g ai/ha	L/ha	Growth stage at last application	Sample	PHI (days)	Cyfluthrin (mg/kg)	
Unionville, Ontario, Canada, 1983, Super Snowball	EC	7	50 50 50 50 50 50 50	199 199 248 248 248 248 248	36 – 41 cm height	curd	0 1 3 7	0.57 0.91 0.65 0.32	84689
Phelps, New York, USA, 1983, Imperial 10-6	EC	7	50	468	curds begin to form	curd	0 1 3 7	0.31 0.28 0.16 0.12	98426
Beavercreek, Oregon, USA, 1983, Snowball	EC	7	50	561	curd development	curd	0 1 3 7	0.30 0.32 0.17 0.02 0.01 [u]	98426
Greenfield, California, USA, 1983, Snowball-Y	EC	7	50	468	harvest	curd	0 1 3 7	0.17 0.10 0.08 < 0.01 0.01 [u]	98426
Santa Maria, California, USA, 1983, Snowball	EC	10	50	561	harvest	curd	0 1 3 7	0.10 0.11 0.02 < 0.01	98426
Glendale, Arizona, USA, 1983, Snowflower	EC	12	50	309	harvest	curd	0 1 3 6	< 0.01 < 0.01 < 0.01 < 0.01	98426

u - Sample from control (untreated) plot

Table 63. Residues for Cyfluthrin in cabbage

Country	Application					Residues			Reference
	FL	No	g ai/ha	L/ha	Growth stage at last application	Sample	PHI (days)	Cyfluthrin (mg/kg)	
Germansville, Pennsylvania, USA, 2000, Market Prize	EC	4	52 52 50 53	344 293 312 344	80% expected head size	Head WWL W/OWL	0 0	0.42 0.01	110339
	WP	4	50 50 52 50	332 289 322 336	80% expected head size	Head WWL W/OWL	0 0	0.30 0.01	
Tifton, Georgia, USA, 2001, Blue Thunder	EC	4	49 49 49 49	151 156 145 147	40% expected head size	Head WWL	0 7 14 21	2.1 0.76 0.22 0.26	110339

Country	Application					Residues			Reference
	FL	No	g ai/ha	L/ha	Growth stage at last application	Sample	PHI (days)	Cyfluthrin (mg/kg)	
	WP	4	49 49 49 49	151 156 145 147	40% expected head size	Head WWL	0 7 14 21	1.3 0.32 0.37 0.22	
Springfield, Nebraska, USA, 2000, Golden Acre	EC	4	49 49 49 49	160 161 160 163	80% expected head size	Head WWL W/OWL	0 0	0.07 0.006	110339
	WP	4	50 49 50 50	161 161 162 165	80% expected head size	Head WWL W/OWL	0 0	0.10 0.006	
Fresno, California USA 1997 Copenhagen Market	EC	4	57 57 55 55	150 140 174 174	Mature	head	0 3 7 14	0.02 0.08 0.04 0.10	109119
North Rose, New York USA 1997 Heads-up	EC	4	57 58 58 57	188 191 190 189	Mature	head	0	1.0	109119
Hawkinsville, Georgia, 1997, Bravo	EC	4	56 56 56 56	147 133 135 175	Mature	head	0	1.3	109119
Sanford, Florida, USA, 1997, Bravo	EC	4	55 57 57 57	139 168 168 168	mature	head	0	1.2	109119
Uvalde, Texas, USA, 1997, Vantage Point	EC	4	56 56 58 56	141 140 148 140	mature	head	0	0.24	109119
Delavan, Wisconsin, USA, 1997, Vantage Point	EC	4	55 56 56 55	168 170 168 169	mature	head	0	0.58	109119
Unionville, Ontario, Canada, 1983, Market Prize	EC	7	50 50 50 50 50 50 50	199 199 248 248 248 248 248	heads 25 – 30 cm	head	0 1 3 7	0.33 0.26 0.18 0.09	84679
Phelps, New York, USA, 1983, King Cole	EC	7	50	467	harvest	head	0 1 3 7	0.01 < 0.01 < 0.01 < 0.01	98426
Greenfield, California, USA, 1983, Express	EC	7	50	467	harvest	head	0 1 3 7	0.02 0.04 0.14 < 0.01 0.02 [u]	98426
Leeds, Wisconsin, USA, 1983, King Cole	EC	7	50	374 374 374 374 374 215 215	harvest	head	0 1 3 7	0.02 0.03 0.02 < 0.01	98426

Country	Application					Residues			Reference
	FL	No	g ai/ha	L/ha	Growth stage at last application	Sample	PHI (days)	Cyfluthrin (mg/kg)	
Howe, Indiana, USA, 1983, Golden Acre	EC	7	50	467	harvest	head	0 1 3 7	0.25 0.14 0.01 0.01	98426
Adams Gardens, Texas, USA, 1983, Sanibel	EC	7	50	486	harvest	head	0 1 3 7	< 0.01 0.01 < 0.01 0.03	98426
Vero Beach, Florida, USA, 1983, Market Prize	EC	7	50	421	head development	head	0 1 3 7	0.04 0.07 0.05 0.03	98426
Vero Beach, Florida, USA, 1983, Market Prize	EC	7	50	365	head development	head	0 1 3 7	0.04 0.04 0.06 0.04	98426
Santa Maria, California, USA, 1983, Headstart	EC	10	50	561	harvest	head	0 1 3 7	0.07 0.04 0.06 0.18	98426
Santa Maria, California, USA, 1983, Headstart	EC	10	50	561	harvest	head	0 1 3 7	0.37 0.33 0.62 0.39	98426
Nagano, Japan, 1983, Nagano Kohai chusei SE	SL	4	100	2000		Head	7 14 21	0.01 < 0.01 < 0.01	1028
	SL	6	100	2000		Head	14 21	< 0.01 < 0.01	
Japan, 1983, Suehiro	SL	4	100	2000		Head	7 14 21	0.04 < 0.01 0.01	1029
	SL	6	100	2000		Head	14 21	< 0.01 < 0.01	
Chinese Cabbage									
Nagano, Japan, 1983, Taibyo 60 nichi	SL	4	100	2000		Head	7 14 21	0.32 0.34 0.36	1030
Japan, 1983, Gyokuhai	SL	4	100	2000		Head	7 14 21	0.12 0.02 0.02	1031

u - Sample from control (untreated) plot

Table 64. Residues for beta-cyfluthrin in cabbage

Country	Application					Residues			Reference
	FL	N	g ai/ha	L/ha	Growth stage at last application	Sample	PHI (days)	Cyfluthrin (mg/kg)	
Kleinniedesheim, Germany, 1992, Juliwirsing	SC	3	10	600	30% of final size	Head	0 3 5 7 10	0.07 0.04 0.03 < 0.01 < 0.01	RA-2074/92
Worms – Heppenheim, Germany, 1992, Juliwirsing	SC	3	10	600	30% of final size	Head	0 3 5 7 10	0.03 0.03 0.02 < 0.01 < 0.01	RA-2074/92

Country	Application					Residues			Reference
	FL	N	g ai/ha	L/ha	Growth stage at last application	Sample	PHI (days)	Cyfluthrin (mg/kg)	
Monheim, Germany, 1992, Fischenicher Advent	SC	3	10	600	50% of final size	Head	0 3 5 7 10	0.19 0.13 0.09 <u>0.08</u> 0.08	RA-2074/92
Burscheid, Germany, 1992, Fischenicher Advent	SC	3	10	600	50% of final size	Head	0 3 5 7 10	0.10 0.07 0.06 <u>0.06</u> 0.04	RA-2074/92

Tomatoes and peppers

Field trials were reported from the USA for trials conducted in 2000/2001 (Lemke 2002 110981) and 1987/1988 (Leslie 1988 98336) in which EC and/or WG formulations were applied to <u>tomatoes</u> and <u>peppers</u>. The minimum sample size for peppers was 1.7 kg, and the minimum sample size for tomatoes was 2.3 kg.

For the 2000/2001 trials, plot sizes were 93 – 201 m^2 and application was with knapsack or motorised boom sprayers. The peppers and tomatoes analysed in this study were held in frozen storage for a maximum of 441 days prior to extraction. Control samples fortified with cyfluthrin were extracted and analysed with the treated samples. The method used in the trials conducted in 2000/2001 was detailed in report no. 108139-1 (Moore *et al.,* 2001 108139-1) and is based on quantification using GC-MS (SIM m/z 207). Recovery of cyfluthrin from peppers fortified at 0.01 mg/kg and 0.4 mg/kg ranged from 86% to 99% and from 101% to 104%, respectively. Recovery of cyfluthrin from tomatoes fortified at 0.01 and 0.5 mg/kg ranged from 91% to 95% and from 93% to 94%, respectively.

Tomato residue data in USA 1987/88 (Leslie 1988 98336) were obtained using the analytical procedure described in Mobay Report No. 85823. Recoveries for tomatoes fortified at 0.05, 0.10, and 0.50 mg/kg ranged from 72% to 112%. Recoveries for <u>cherry tomatoes</u> fortified at 0.01, 0.02, and 0.05 mg/kg ranged from 70% to 88%. Samples analysed were held in frozen storage for a maximum of 789 days. Plot sizes were 19 – 251 m^2.

Pepper (capsicum and chilli) residue data in USA 1985/1986 (Freeseman 1999 addendum to Leslie 1988 96775) were obtained using the analytical procedure described in Mobay Report No. 85823. Recoveries for peppers fortified at 0.05 and 0.10 mg/kg ranged from 62% to 120%. Samples analysed were held in frozen storage for a maximum of 830 days. Plot sizes were 6.5 – 334 m^2.

Table 65. Residues for cyfluthrin in tomatoes

Country	Application					Residues			Reference
	FL	N	g ai/ha	L/ha	Growth stage at last application	Sample	PHI (days)	Cyfluthrin (mg/kg)	
Fresno, California, USA, 2000, UC 82-1	EC	6	50 50 49 52 47 48	179 178 174 189 171 180	3rd fruit has reached typical size and form	fruit	0 7 14 21	<u>0.08</u> 0.05 0.04 0.03	110981

Country	Application					Residues			Reference
	FL	N	g ai/ha	L/ha	Growth stage at last application	Sample	PHI (days)	Cyfluthrin (mg/kg)	
	WP	6	48 50 49 52 49 49	172 180 180 186 180 182	3rd fruit has reached typical size and form	fruit	0 7 14 21	0.04 0.03 0.02 0.01	
	WP	6	48 50 49 52 49 49	172 180 180 186 180 182	3rd fruit has reached typical size and form	Fruit Fruit, cooked Fruit, dried	7 [a]	0.02 < 0.01 0.19	
Vero Beach, Florida, USA, 2000, Sunpar	EC	6	48 49 49 50 52 49	160 160 156 167 181 186	4th fruit has reached typical size and form	fruit	7	0.04	110981
	WP	6	52 49 48 50 50 50	169 157 153 167 181 188	4th fruit has reached typical size and form	fruit	7	0.02	
	WP	6	52 49 48 50 50 50	169 157 153 167 181 188	4th fruit has reached typical size and form	fruit fruit, washed	7 [a]	0.01 0.01	
Hughson, California, USA, 2000, Mt Fresh	EC	6	50 49 49 52 49 49	192 187 188 193 185 187	50% of fruits show typical fully-ripe colour	fruit	7	0.08	110981
	WP	6	49 49 49 49 50 49	187 187 185 188 191 185	50% of fruits show typical fully-ripe colour	fruit	7	0.06	
Rock Springs, Pennsylvania USA, 1986 Supersonic	EC	6	50	281		Mature Mature Mature Mature mature	0 1 3 7 14	0.07 0.06 <u>0.08</u> 0.04 0.02	98336
Elmer, New Jersey USA 1986 Pacesetter	EC	6	50	421		Earliest h Earliest h Mature Mature mature	0 1 3 7 14	<u>0.07</u> 0.02 0.03 0.02 0.01	98336
Davis, California USA 1986 Ace	EC	6	50	683		Mature Mature Mature Mature Slight over ripe	0 1 3 7 14	<u>0.10</u> 0.10 0.10 0.05 0.04	98336

Country	Application					Residues			Reference
	FL	N	g ai/ha	L/ha	Growth stage at last application	Sample	PHI (days)	Cyfluthrin (mg/kg)	
Holtville, California, USA 1986 Jackpot	EC	6	50	561		Green f Green f Green f Pink f Mature f	0 1 3 7 14	< 0.01 0.01 0.01 0.01 < 0.01	98336
Howe, Indiana, USA, 1983, Heinz 2653	EC	6	50		mature	Mature Mature Mature Mature mature	0 1 3 7 14	0.06 0.07 0.07 0.06 0.02	98336
Adams Garden, Texas, USA, 1983, Flora Dade	EC	6	50	374	fruit 5.08 – 7.62 cm diameter	Mature Mature Mature Mature mature	0 1 3 7 14	0.08 0.07 0.07 0.09 0.06	98336
Tifton, Georgia, USA, 1983, Floridate	EC	6	50	90	mature	Mature Mature Mature Mature mature	0 1 3 7 14	0.06 0.04 0.07 0.03 0.02	98336
Vero Beach, Florida, USA, 1983, Sunny	EC	6	50	421	fruit immature	Mature Mature Mature Mature mature	0 1 3 7 14	0.04 0.06 0.03 0.05 0.03	98336
La Feria, Texas, USA, 1983, Flora Dade	EC	6	50	561	fruit 2.54 – 5.08 cm diameter	fruit	0 1 3 7 14	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	98336
Vero Beach, Florida, USA, 1983, Sunny	EC	6	50	365	fruit immature, full	Green mature Green mature Green mature Green mature [b] Green immature [c]	0 1 3 7 14	0.06 0.07 0.07 0.03 0.02 0.01 [u]	98336
Napoleon, Ohio, USA, 1983, Easy Red	EC	7	50	654	fruit immature - colouring	Immature Immature Immature Mature mature	0 1 3 7 14	0.02 0.02 0.01 0.02 < 0.01	98336
Cherry tomatoes									
Adams Gardens, Texas, USA, 1986, Cherry	EC	6	50	561	10% red fruit	40% red 60% red	7 14	0.03 0.02	98336
Vero Beach, Florida, USA, 1986, Cherry Grande	EC	6	50	374	fruit mature	mature	7 14	0.05 0.03	98336
Howe, Indiana, USA, 1986, Large Cherry	EC	6	50	271	fruit mature	Mature mature	7 14	0.05 0.02	98336
Woodland, California USA 1986 NK 82-17	EC	6	59 51 50 49 56 51	281		Mixed maturity	7 14	0.04 0.02	98336

a - Samples were washed by placing individual tomatoes under lukewarm to cool running tap water for approximately 30 sec. The tomatoes were allowed to drain for at least 2 min. Samples for cooking were peeled, cut into quarters,

crushed with a wooden spoon, heated to boiling, and allowed to simmer for 5 min. The cooked tomatoes were transferred to canning jars and processed for 35 min using a boiling water canning method. Samples for drying were washed as previously described and then dehydrated by placing 1/4 inch slices of tomatoes into a dehydrator for 5 to 9 hrs or until the slices were leathery or brittle.

b - Green mature fruit, crop freeze damaged, vines completely wilted.

c - Green immature fruit from areas sheltered under vines, vines completely dry.

u - Sample from control (untreated) plot.

Table 66. Residues for cyfluthrin in peppers

Country	Application					Residues			Reference
	FL	No	g ai/ha	L/ha	Growth stage at last application	Sample	PHI (days)	Cyfluthrin (mg/kg)	
Fresno, California, USA, 2000, Green Jalapeno	EC	6	52 50 49 50 48 52	187 186 176 180 174 188	2nd fruit has reached typical size and form	fruit	7	<u>0.06</u>	110981
	WP	6	50 47 49 50 49 49	181 177 79 180 180 178	2nd fruit has reached typical size and form	fruit	7	0.05	
	WP	6	49 49 49 50 49 49	179 183 176 179 180 179	2nd fruit has reached typical size and form	Fruit Fruit washed	7 [a]	0.02 0.01	
Levelland, Texas, USA, 2000, Jalapeno M	EC	6	52 48 49 50 52 49	287 274 277 281 288 276		fruit	7	<u>0.08</u>	110981
	WP	6	49 49 50 50 50 49	283 277 285 287 288 283		fruit	7	0.05	
Uvalde, Texas, USA, 2000, Jalopeno-M	EC	6	49 49 49 49 49 50	277 180 235 185 190 199	2nd fruit has reached typical size and form	fruit	8	<u>0.08</u>	110981
	WP	6	50 50 52 49 48 48	279 183 241 184 188 184	2nd fruit has reached typical size and form	fruit	8	0.04	
Vero Beach, Florida, USA, 2000, Enterprize	EC	6	50 50 48 49 50 49	143 144 139 141 143 142	2nd fruit has reached typical size and form	fruit	0 7 14 22	0.16 <u>0.12</u> 0.10 0.05	110981

Country	Application					Residues			Reference
	FL	No	g ai/ha	L/ha	Growth stage at last application	Sample	PHI (days)	Cyfluthrin (mg/kg)	
	WP	6	50 50 48 49 50 49	143 144 139 141 143 142	2nd fruit has reached typical size and form	fruit	0 7 14 22	0.11 0.08 0.05 0.04	
Fresno, California, USA, 2000, Jupiter	EC	6	50 49 50 50 49 50	183 185 180 180 178 189	1st fruit has reached typical size and form	fruit	7	0.06	110981
	WP	6	49 49 49 50 49 49	e180 183 176 179 180 179	2nd fruit has reached typical size and form	fruit	7	0.02	
Adams Gardens, Texas, USA, 1986, Grande Rio 66	EC	6	50	561	red fruit	fruit	0 1 3 7	0.22 0.14 0.21 0.08 0.02 [u]	96775
Indio, California, USA, 1986, Keysone Resistant Giant	EC	6	55	411	fruiting	fruit	3 7	0.04 0.05	96775
Clayton, North Carolina, USA, 1985, California Wonder	EC	6	124	327	fruit	fruit	0 1 3 7	0.27 0.33 0.33 0.12	96775
Centerton, New Jersey, USA, 1985, Yolo Wonder	EC	6	50	832	ripe	fruit	0 1 3 7	0.04 0.04 0.02 0.01	96775
Adams Gardens, Texas, USA, 1985, Grande Rio 66	EC	6	50	561	full size red fruit	fruit	0 1 3 7	0.13 0.08 0.08 0.08 0.01 [u]	96775
Vero Beach, Florida, USA, 1985, Yolo Wonder	EC	6	50	94	fruit mature	fruit	0 1 3 7	0.10 0.08 0.05 0.03 0.01 [u]	96775
Vero Beach, Florida, USA, 1985, Yolo Wonder	EC	6	50	94	fruit mature	fruit	0 1 3 7	0.07 0.06 0.03 0.01	96775

a - 'Washed' samples were washed by placing individual peppers under lukewarm to cool running tap water for approximately 30 sec. The peppers were allowed to drain for at least 2 min. The stem, core, and seeds were removed and the inside of the pepper rinsed for 30 sec. The peppers were allowed to drain again for at least 2 min.

u - Sample from control (untreated) plot.

Sweet corn

Weidmann *et al.,* (1990 100248) reported trials from the USA in for trials conducted in 1989 in which EC and/or WG formulations were applied as 10 sprays to sweet corn at intervals of two to three days.

Leslie (1988 98313) reported results of trials conducted in 1983 where applications of a granular formulation combined with foliar sprays of an EC formulation were made to corn.

For the 1989 trials, plot sizes were 93 – 201 m^2 and application was by aircraft. The sweet corn commodities analysed in this study were held in frozen storage for a maximum of 298 days prior to extraction. Control samples fortified with cyfluthrin were extracted and analysed with the treated samples. Sweet corn residue data in USA 1989 (Weidman *et al.,* 1990 100248) were obtained using the analytical procedure described in Mobay Report No. 85823. Concurrent recoveries from green forage fortified at 0.1 mg/kg ranged from 97% to 106%. Recoveries from cobs fortified at 0.1 mg/kg ranged from 67% to 92%. Recoveries from husks fortified at 0.1 and 8.0 mg/kg ranged from 78% to 95%. Recoveries from kernels fortified at 0.1 mg/kg ranged from 70% to 101%.

For the USA 1983 trials the initial application was a band over row soil incorporated application of a granular cyfluthrin formulation at the rate of 0.85 g ai/305 m of row made at planting, followed by ten to fifteen foliar spray applications of an EC formulation at the rate of 50 g ai/ha/application. The initial application interval between planting and the first foliar spraying ranged from 43 to 74 days. The intervals between the multiple foliar spray applications ranged from one to seven days. Each of the applications was made utilizing ground spray equipment.

Residue data in USA 1983 trials (Leslie 1988 98313) were obtained using the analytical procedure described in Mobay Report No. 85823. Recoveries for green forage fortified at 0.05 and 0.10 mg/kg ranged from 85% to 100% (Craven Laboratories, invalidated data). Recoveries for corn kernels harvested at the milk stage and fortified at 0.05 and 0.1 mg/kg ranged from 88% to 98% and 85 – 102% for dry kernels fortified at the same levels. Recoveries for corn cobs (at milk stage & dry harvest) fortified at 0.05 and 0.10 mg/kg ranged from 94% to 104% for cobs collected at milk stage and 92% to 101% for cobs at dry harvest. Recoveries for corn husks (at milk stage & dry harvest) fortified at 0.05 and 0.10 mg/kg ranged from 84% to 101% for husks collected at milk stage and 89% to 100% for dry harvest samples. Recoveries for dry fodder (0.05 and 0.1 mg/kg) ranged from 86 to 102%.

Samples analysed were held in frozen storage for a maximum of 160 days. Plot sizes were 15 – 418 m^2.

Table 67 Residues for cyfluthrin in sweet corn

Country	Application					Residues			Reference
	FL	N	g ai/ha	L/ha	Growth stage at last application	Sample	PHI (days)	cyfluthrin (mg/kg)	
Lamberton, Minnesota, USA, 1989, Green Giant	EC	10	50 [b]	47	kernel filling	kernel	0	< 0.01	100248
							1	< 0.01	
							3	< 0.01	
							7	< 0.01	
							14	< 0.01	
						cob	0	< 0.01	
							1	< 0.01	
							3	< 0.01	
							7	< 0.01	
							14	< 0.01	
Dundee, Oregon, USA, 1989, Golden Jubilee	EC	10	50 [b]	47	ears mature	kernel	0	0.01	100248
							1	< 0.01	
							3	< 0.01	
							7	< 0.01	
							14	< 0.01	
						cob	0	< 0.01	
							1	< 0.01	
							3	< 0.01	
							7	< 0.01	
							14	< 0.01	

Country	Application					Residues			Reference
	FL	N	g ai/ha	L/ha	Growth stage at last application	Sample	PHI (days)	cyfluthrin (mg/kg)	
Sodus, New York, USA, 1989, Crusade	EC	10	50 [b]	52	fresh market ears	kernel cob	0 1 3 7 14 0 1 3 7 14	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	100248
Phelps, New York, USA, 1983, Jubilee	GR EC	1 10	50	238	grain milk stage	kernel cob K+CWHR	0 0 0	< 0.01 < 0.01 < 0.01	98313 [a]
Benson, Minnesota, USA, 1983, Golden Bantam	GR EC	1 10	50	123	milk stage	kernel cob K+CWHR	0 0 0	< 0.01 < 0.01 < 0.01	98313 [a]
Danville, Iowa, USA, 1983, Northrup King 199	GR EC	1 10	50	280	soft dough	kernel cob K+CWHR	0 0 0	< 0.01 < 0.01 < 0.01	98313 [a]
Vero Beach, Florida, USA, 1983, Guardian	GR EC	1 10	50 (+0.5 g/L X-77)	421	early milk stage	kernel cob K+CWHR	0 0 0	< 0.01 < 0.01 < 0.01	98313 [a]
Urbana, Illinois, USA, 1983, Silver Queen	GR EC	1 10	50	468	milk stage	kernel cob K+CWHR	0 0 0	< 0.01 0.01 0.01	98313 [a]
Marcellus, Michigan, USA, 1983, Seneca Star	GR EC	1 10	50	505	milk stage	kernel Cob K+CWHR	0 0 0	0.02 0.02 0.02	98313 [a]
Adams Gardens, Texas, USA, 1983, Silver Queen	GR EC	1 10	50	234	milk stage	kernel cob K+CWHR	0 0 0	< 0.01 < 0.01 < 0.01	98313 [a]
Stilwell, Kansas, USA, 1983, Iochief	GR EC	1 10	50	140	grain milk stage	kernel cob K+CWHR	0 0 0	< 0.01 < 0.01 < 0.01	98313 [a]
Tifton, Georgia, USA, 1983, Silver Queen	GR EC	1 10	50	64	milk stage	kernel cob K+CWHR	0 0 0	< 0.01 < 0.01 < 0.01	98313 [a]
Springfield, Nebraska, USA, 1983, Iochef Hybrid Earl May	GR EC	1 13	50	468	grain milk stage	kernel cob K+CWHR	0 0 0	< 0.01 < 0.01 < 0.01	98313 [a]
Howe, Indiana, USA, 1983, Silver Queen	GR EC	1 10	50	468	milk stage	kernel Cob K+CWHR	0 0 0	< 0.01 0.01 < 0.01	98313 [a]
Canby, Oregon, USA, 1983, Golden Jubilee	GR EC	1 15	50	374	milk stage	kernel cob K+CWHR	0 0 0	< 0.01 < 0.01 < 0.01	98313 [a]
Greenfield, California, USA, 1983, Tendersweet	GR EC	1 10	50	468	harvest	kernel cob K+CWHR	0 0 0	< 0.01 < 0.01 < 0.01	98313 [a]

a - Invalidated analytical data

b - Application method aerial

K+CWHR = kernels plus cob with husk removed

Potato

Field trials were reported from Canada and the USA in for trials conducted in 1983 and 2000 in which EC and WP formulations were applied to potatoes. Plot sizes were 2.8 – 254 m^2 and application was with knapsack or motorised sprayers. Sample sizes were ≥ 24 tubers. The storage interval between sampling and harvest was 319 – 386 days. Control samples fortified with cyfluthrin were extracted and analysed with the treated samples. The method used in the trials conducted in 2000 was detailed in report no. 108139-1 (Moore *et al.,* 2001 108139-1). Recoveries for samples fortified at 0.01 mg/kg were 94 – 100% and for samples fortified at 0.05 mg/kg 84 – 90%.

Table 68. Residues of cyfluthrin in potatoes

Location, year, variety	FL	N	g ai/ha	L/ha	Growth stage at last application	Sample	PHI (days)	Cyfluthrin (mg/kg)	Reference
Ephrata, Washington, USA, 2000, Russet Burback	EC	6	50 49 49 49 50 50	194 191 191 192 189 190	48	tuber	0	< 0.01	110988
	WP	6	6×49	192 191 189 194 190 190	48	tuber	0	< 0.01	
Jerome, Idaho, USA 2000, Russet Burback	EC	6	49 50 49 49 52 52	306 308 296 308 313 324	48	tuber	0 7 15 19	< 0.01 < 0.01 < 0.01 < 0.01	110988
	WP	6	49 50 49 48 50 50	305 309 297 306 311 318	48	tuber	0 7 15 19	< 0.01 < 0.01 < 0.01 < 0.01	110988
Marysville, Ohio, 2000, Kennebec	EC	6	47 49 47 49 49 49	228 229 226 235 236 233	48	tuber	0 7 14 21	< 0.01 < 0.01 < 0.01 < 0.01	110988
	WP	6	49 47 49 49 49 49	228 228 225 235 236 233	48	tuber	0 7 14 21	< 0.01 < 0.01 < 0.01 < 0.01	
Unionville, Ontario, Canada, 1983, Superior	EC	6	6×50	6×248	46 – 51 cm height	tuber	0 3	< 0.01 < 0.01	84396
Portage La Prairie, Manitoba, Canada, 1983, Norland	EC	7	7×50	7×152	46 – 51 cm height	tuber	0 3 7	< 0.01 < 0.01 < 0.01	84399
Presque Isle, Maine, USA, 1983, Yankee Chipper	EC	6	6×50	6×1216	41 – 46 cm height	tuber	0 3	< 0.01 < 0.01	98423

Location, year, variety	FL	N	g ai/ha	L/ha	Growth stage at last application	Sample	PHI (days)	Cyfluthrin (mg/kg)	Reference
Phelps, New York, USA, 1983, Superior	EC	6	6×50	6×468	56 – 61 cm height	tuber	0 3	<u>< 0.01</u> < 0.01	98423
Northwood, North Dakota, USA, 1983, Kennebec	EC	6	6×56	6×187	56 – 61 cm height	tuber	0 3	<u>< 0.01</u> < 0.01	98423
Benson, Minnesota, USA, 1983, Norchip	EC	6	6×56	6×123	fruiting	tuber	0 3	<u>< 0.01</u> < 0.01	98423
Yakima, Washington, USA, 1983, Russet Burbank	EC	6	6×50	6×206	56 – 61 cm height	tuber	0 3	<u>< 0.01</u> < 0.01	98423
Beavercreek, Oregon, USA, 1983, Red Lasoda	EC	6	6×50	6×374	earliest harvest	tuber	0 3	<u>< 0.01</u> < 0.01	98423
Parma, Idaho, USA, 1983, Russet Burbank	EC	6	6×50	6×281	harvest	tuber	0 3	<u>< 0.01</u> < 0.01	98423
Leeds, Wisconsin, USA, 1983, Superior	EC	6	6×50	6×215	earliest harvest	tuber	0 3	<u>< 0.01</u> < 0.01	98423
Howe, Indiana, USA, 1983, Katahdin	EC	6	6×50	6×468	fruiting	tuber	0 3	<u>< 0.01</u> < 0.01	98423
Vero Beach, Florida, USA, 1983, Red Lasoda	EC	6	6×50	6×421	mature tubers	tuber	0 3	<u>< 0.01</u> < 0.01	98423
Lasalle, Colorado, USA, 1983, Norgold Russet	EC	6	6×50	6×238	51 – 56 cm height	Tuber tuber, dried	0 3 0	<u>< 0.01</u> < 0.01 < 0.01	98423
Moxee, Washington, USA, 1989, Russet Burbank	EC	6	6×281	6×175	mid to late season green	granules	0	<u>< 0.01</u>	100201

Cotton

Lenz *et al.,* (1997 107539) studied residues of cyfluthrin in <u>cotton</u> following application of cyfluthrin EC or beta-cyfluthrin SC formulations as 10 foliar sprays at 56 or 28 g ai/ha respectively. Applications were made at two to five day intervals. Each application was made using a spray volume of approximately 94 L/ha. In two trials the cotton was harvested by hand to simulate machine picked cotton. This was accomplished by first removing the cotton bolls from the plant and then stripping the gin trash (burrs, leaves, stems, sticks, lint, immature seeds and dirt) by hand. The cotton bolls were ginned within 48 hours of harvest in an on-site, field-sized cotton gin. Cottonseed and gin trash samples taken following ginning were put in frozen storage.

In the third trial the cotton bolls were harvested by a commercial-scale, cotton plant stripper (John Deere tractor stripper). The machine stripped cotton plants were transported the next day to a nearby commercial gin. The plants were put through a burr extractor to remove the majority of the gin trash prior to ginning. Following ginning, the cottonseed and gin trash were put in frozen storage. Additional gin trash samples were also harvested by hand and stored frozen.

Residues were measured according to the method of Harbin *et al.,* 1983 (85823). Recoveries of cyfluthrin ranged from 72% to 106% in cottonseed and 71% to 103% in gin trash. Recoveries of

beta-cyfluthrin ranged from 72% to 114% in cottonseed and 73% to 120% in gin trash. Cottonseed samples were held in frozen storage for a maximum of 343 days and gin trash samples for a maximum of 564 days.

Leslie (1989 98405) studied residues in cotton commodities following applications to cotton. Samples were held in frozen storage for a maximum of 431 days prior to analysis. Concurrent recoveries at the 0.05 mg/kg fortification level were done with percent recoveries that ranged from 98% to 114%.

Table 69. Residues for Cyfluthrin on cotton seed

Country	Application					Residues			Reference
	FL	N	g ai/ha	L/ha	Growth stage at last application	Sample	PHI (days)	Cyfluthrin (mg/kg)	
Dill City, Oklahoma, USA, 1995, Paymaster HS-26	EC	10	56	96 – 110	Bolls ca. 75% open	seed	0	0.52	107539
Benoit, Mississippi, USA, 1995, DPL 50	EC	10	55 – 57	85 – 91	Bolls 60 – 70% Open	seed	0	< 0.1	107539
Fresno, California, USA, 1995, Maxxa	EC	10	55 – 56	88 – 94	Bolls 50 – 60% Open	seed	0	< 0.1	107539
Adams Gardens, Texas, USA, 1987, Stoneville 825	EC	9	56	150	60% of bolls open	seed	0	0.02	98405
Benoit, Mississippi, USA, 1987, Delta Pine 20	EC	9	56 [a]	47	pre harvest	seed	0	< 0.01	98405
Benoit, Mississippi, USA, 1987, Delta Pine 20	EC	9	56	48	pre harvest	seed	0	0.10	98405
Selma, California, USA, 1987, Acala SJ-2	EC	9	56	187	pre harvest	seed	0	0.03	98405
Holtville, California, USA 1982, Delta Pine 61	EC	8	100 [a]	2.3	majority of buds open	seed	0 7	< 0.01 < 0.01	84361
Holtville, California, USA, 1982, Delta Pine 61	EC	9	100	23	majority of buds open	seed	0 7	0.28 0.15	84362
Hollis, Oklahoma, USA, 1982, CAMD-E	EC	10	100	93	0 – 10% buds open	seed	0 7	0.04 0.01	84363
Bishopsville, South Carolina, USA, 1982, Coker 315	EC	10	100	56	fruiting	seed	0 7	0.09 0.04	84364
Benoit, Mississippi, USA, 1982, Stoneville 213	EC	10	100	41	after flowering	seed	0 7	0.11 0.09	84365
Tifton, Georgia, USA, 1982, Coker 310	EC	10	100	86	10 – 20% buds open	seed	0 7	0.55 0.54 0.01 [u]	84366

Country	Application					Residues			Reference
	FL	N	g ai/ha	L/ha	Growth stage at last application	Sample	PHI (days)	Cyfluthrin (mg/kg)	
Adams Gardens, Texas, USA, 1982, McNair 220	EC	10	5×500 [a] 5×100 [a]	19	Majority of buds open	seed	0 7	0.15 0.04 0.02 [u]	84367
Hollis, Oklahoma, USA, 1982, Westburn M	EC	10	100 [a]	2.9	20 – 30% buds open	seed	0 7	0.04 0.01	84369
Bishopsville, South Carolina, USA, 1982, Coker 315	EC	10	100 [a]	2.3	fruiting	seed	0 7	0.08 0.01	84370
Benoit, Mississippi, USA, 1982, Stoneville 213	240 EC	10	100 [a]	4.6	after flowering	seed	0 7	0.07 0.03	84371
Adams Gardens, Texas, USA, 1982, McNair 220	EC	10	5×500 [a] 5×100 [a]	2.3	majority of buds open	seed	0 7	0.12 0.03 0.02 [u]	84372
Eudora, Arkansas, USA, 1982, DPL 55	EC	10	100	41	after flowering	seed	0 7	0.06 0.13 0.02 [u]	84380

u - Sample from control (untreated) plot.

a - Application method aerial

Table 70 Residues for beta-cyfluthrin in cotton seed

Country	Application					Residues			Reference
	FL	N	g ai/ha	L/ha	Growth stage at last application	Sample	PHI (days)	Beta-cyfluthrin (mg/kg)	
Dill City, Oklahoma, USA, 1995, Paymaster HS-26	SC	10	25 – 28	96 – 110	Bolls ca. 75% open	seed	0	0.38	107539
Benoit, Mississippi, USA, 1995, DPL 50	SC	10	28 – 29	87 – 91	Bolls 60 – 70% Open	seed	0	< 0.1	107539
Fresno, California, USA, 1995, Maxxa	SC	10	28 – 29	88 – 94	Bolls 50 – 60% Open	seed	0	< 0.1	107539

Soya beans

Burger (1992 103823). Harbin *et al.,* 1983 (85823). Concurrent recoveries were conducted with each sample set at the 0.1, 0.5, 2.0 mg/kg and/or 5.0 mg/kg fortification levels. Concurrent recoveries for cyfluthrin ranged from 71% to 109%. Soya bean matrices were held in frozen storage for a maximum period of 440 days (date of harvest to date of analysis).

Leslie (1988 98398) reported results for trials conducted in 1982 (samples analysed by Craven laboratories, invalidated analytical data) and 1984 (McKenzie labs). Dry soya beans were not analysed in the 1984 trials.

Recovery data for cyfluthrin in threshed beans, generated at the 0.05 and 0.10 mg/kg fortification levels produced percent recoveries that ranged from 80% to 85% (Craven labs). Recovery data for cyfluthrin in soya bean hay generated at the 0.01, 0.02, 0.05, 0.10 mg/kg and 0.50 mg/kg

fortification levels produced percent recoveries that ranged from 76% to 110% (McKenzie labs). Recovery data for cyfluthrin in soya bean dry vines (straw), generated at the 0.05 and 0.10 mg/kg fortification levels produced percent recoveries that ranged from 80% to 85% (Craven Laboratories, invalidated analytical data). Samples were held in frozen storage for a maximum of 299 days (1982 samples) and 198 days (1984 samples).

Trials from Brazil using beta-cyfluthrin were made available to the Meeting. Plot sizes were 20 – 90m^2. Samples were stored frozen for up to nine months prior to analysis using method M0-03-010892. Recoveries for samples of seed fortified at 0.05 – 0.5 mg/kg were 82 – 119%.

Table 71 Residues for Cyfluthrin in soya beans

Country	Application					Residues			Reference
	FL	N	g ai/ha	L/ha	Growth stage at last application	sample	PHI (days)	Cyfluthrin (mg/kg)	
Danville, Iowa, USA, 1990, Washington VI	EC	4	49	42	full pod	seed, dry	49	< 0.01	103823
Danville, Iowa, USA, 1990, Washington VI	EC	4	49	187	full pod	seed, dry	49	< 0.01	103823
Benoit, Mississippi, USA, 1990, Young	EC	4	49	47	pod fill	seed, dry	52	< 0.01	103823
Stilwell, Kansas, USA, 1990, Williams 82	EC	4	49	180	pod filling	seed, dry	45	< 0.01	103823
Tifton, Georgia, USA, 1990, Braxton	EC	4	49	3.15	mid pod	seed, dry	54	< 0.01	103823
Springfield, Nebraska, USA, 1982, Williams	EC	4	50	243	early pod stage	seed, dry	61	< 0.01	98398 [a]
Urbana, Illinois, USA, 1982, SRF 350 P	EC	4	50	187	fruit development	seed, dry	48	0.02	98398 [a]
Jackson, Tennessee, USA, 1982, Forrest	EC	4	50	85	fruit development	seed, dry	31	< 0.01	98398 [a]
Eudora, Arkansas, USA, 1982, Macks	EC	4	50	102	Vegetative	seed, dry	59	< 0.01	98398 [a]
Benoit, Mississippi, USA, 1982, Bragg	EC	4	50	41	Vegetative	seed, dry	76	< 0.01	98398 [a]
Howe, Indiana, USA, 1982, Wells II	EC	4	50	234 234 252 252	pod fill	seed, dry	39	< 0.01	98398 [a]
Tifton, Georgia, USA, 1982, Bragg	EC	4	50	888	mid-pod fill	seed, dry	35	< 0.01	98398 [a]

a - Invalidated analytical data

Table 72. Residues for beta-cyfluthrin in soya beans

Country	Application					Residues			Reference
	FL	N	g ai/ha	L/ha	Growth stage at last application	sample	PHI (days)	Cyfluthrin (mg/kg)	
Rio Pardo/RS, Brazil, 2002, BR 16	SC +	2	12	200	Beginning of pod filling	seed	21	< 0.05	BRA-I-P633/02-S1
			25	200	Beginning of pod filling	seed	21	< 0.05	
Londrina-PR, Brazil, 2002, Embrapa 48	SC+	2	12	200	R 5.5	seed	21	< 0.05	BRA-I-P633/02-S2
			25	200	R 5.5	seed	21	< 0.05	
Porteirao-GO, Brazil, 2002, Conquista	SC+	2	12	200	85	seed	21	< 0.05	BRA-I-P633/02-S3
			25	200	85	seed	21	< 0.05	
Sidrolandia – MS, Brazil, 1989, Dourados	SC	2	7.5	400	begin of maturity	seed, dry seed + pod	10 20 0	< 0.01 < 0.01 3.5	42453-A
			15	400		seed, dry	20	< 0.01	
			30	400		seed, dry	20	< 0.01	

SC+ = formulation also contained imidacloprid

Rape seed

Trials of cyfluthrin on rape seed were made available from Germany (1984 and 1985), France (1982) and the UK (1984). For the German trials, plot sizes were 70 – 200 m^2. Samples were stored frozen for up to 16 months prior to analysis using method 00047. Recoveries for seed fortified at 0.05 mg/kg were 97%, pods at 0.1 mg/kg 86% and straw/green material at 0.02 mg/kg 105 – 109%. Plot sizes for the UK trials were 45 m^2. Samples were stored frozen for less than 4 months prior to analysis. Residues were determined using Bayer method MOA 365. Recoveries for samples of seed fortified at 0.05 and 1.0 mg/kg were 79 and 84%. The plot sizes for the trials in France were 30 – 85 m^2. Samples were stored frozen for 4 months prior to analysis using method I478. Recoveries for samples of seed fortified at 0.05 mg/kg were 97%.

Trials for beta-cyfluthrin on rape were made available from Germany. Plot sizes were 60 – 2500 m^2. Samples were held in frozen storage for a maximum period of 212 days (date of harvest to date of analysis) prior to analysis using method 00015.

Table 73. Residues for Cyfluthrin

Country	Application					Residues			Reference
	FL	N	g ai/ha	L/ha	Growth stage at last application	Sample	PHI (days)	Cyfluthrin (mg/kg)	
Burscheid, Versuchsgut Höfchen, Germany, 1985, Quinta	EC	2	15	600	end of flowering	seed	66	0.05 0.07 [u]	5600-85
Gröhnwohld, Germany, 1985, Jet Nuef	EC	2	15	600	end of flowering	seed	75	< 0.05	5601-85
Ensheim, Germany, 1985, Belinda	EC	2	15	600	end of flowering	seed	56	< 0.05	5602-85
Worms-Heppenheim, Germany, 1985, Petranova	EC	2	15	600	end of flowering	seed	35	< 0.05	5603-85

Country	Application					Residues			Reference
	FL	N	g ai/ha	L/ha	Growth stage at last application	Sample	PHI (days)	Cyfluthrin (mg/kg)	
Burscheid, Versuchsgut Höfchen, Germany, 1984, Quinta	EC	2	15	600	end of flowering	seed	72	< 0.05	5600-84
Gröhnwohld, Germany, 1984, Korina	EC	2	15	600	end of flowering	seed	73	< 0.05	5601-84
Göllheim, Germany, 1984, Elvira	EC	2	15	600	end of flowering	seed	70	0.05	5602-84
Gau-Odernheim, Germany, 1984, Loras	EC	2	15	600	end of flowering	seed	63	< 0.05	5603-84
Braintree, Essex, UK, 1984, Bienvenue	EC	1	12.5	300	first flower bud visible	seed	117	< 0.01	TCR 253
Bury St Edmunds, Suffolk, UK, 1984, Bienvenue	EC	1	12.5	300	early stem extension	seed	126	< 0.01	TCR 253
Baugy, France, 1982, Jet Nuef	EC	1	15	400	4 leaf stage	seed	241	< 0.01	5608-83
Deols, France, 1982, Jet Nuef	EC	2	15	400	5 – 6 leaf stage	seed	260	< 0.01	5609-83

u - Sample from control (untreated) plot.

The size of the plots was 2500, 125 and 60 m^2. Samples analysed in these studies were held in frozen storage for a maximum period of 212 days (date of harvest to date of analysis).

Table 74. Residues of beta-cyfluthrin in rape

Country	Application					Residues			Reference
	FL	N	g ai/ha	L/ha	Growth stage at last application	Sample	PHI (days)	Cyfluthrin (mg/kg)	
St. Alban, Hengstbacher Hof, Germany, 1992, Arabella	SC	3	10	424	End of flowering	seed	63	< 0.02	RA-2070/92
Grönwohld, Germany, 1992, Falcon	SC	3	10	300	End of flowering	seed	56 63	< 0.02 < 0.02	RA-2070/92
Burscheid, Versuchsgut Höfchen, Germany, 1992, Lirajet	SC	3	10	300	End of flowering	seed	64	< 0.02	RA-2070/92
Walksfelde, Germany, 1992, Falcon	SC	3	10	300	End of flowering	seed	56 63	< 0.02 < 0.02	RA-2070/92
Burscheid-Höfchen, Germany, 1988, Ceres	EC	3	7.5	300		seed	64	< 0.05	0369-88

Country	Application					Residues			Reference
	FL	N	g ai/ha	L/ha	Growth stage at last application	Sample	PHI (days)	Cyfluthrin (mg/kg)	
Ensheim, Germany, 1988, Arabella	EC	3	7.5	600	51 – 53	seed	56	< 0.05	0370-88
Grönwohld, Germany, 1988, Ceres	EC	3	7.5	600	70	seed	62	< 0.05 0.05 [u]	0371-88
Burscheid, Versuchsgut Höfchen, Germany, 1987, Jef Nuef	EC	3	7.5	600	pod development	seed	54	0.01	5700-87
Kirchlauter-Pettstadt, Germany, 1987, Lirabon	EC	3	7.5	600	full flowering	seed	77	< 0.01	5701-87
Grönwohld, Germany, 1987, Ceres	EC	3	7.5	600	end of flowering	seed	71	< 0.01	5702-87

u - Sample from control (untreated) plot.

Sunflower

Leslie (1988 98392) studied residues in sunflowers following applications of cyfluthrin foliar sprays. Samples were held in frozen storage for a maximum period of 479 days prior to analysis. The method of analysis was as described in Mobay Report No. 85823. Recoveries for samples of seed and fodder (dry) fortified at 0.01 to 0.1 mg/kg were 80 – 110% and 70 – 108% respectively.

Table 75. Residues for cyfluthrin in sunflower seeds

Country	Application					Residues			Reference
	FL	N	g ai/ha	L/ha	Growth stage at last application	Sample	PHI (days)	Cyfluthrin (mg/kg)	
High Bluff, Manitoba, Canada, 1984, HY 894	EC	3	50		bloom, post	seed	28 43	< 0.01 < 0.01	87205
Portage, Manitoba, Canada, 1984, HY 894	EC	3	50		bloom, post	seed	28 43	0.01 < 0.01	87206
East Grand Forks, Minnesota, USA, 1984, Sigco 448	EC	3	50	187	bloom, post	seed	28 42	< 0.01 < 0.01	98392
Northwood, North Dakota, USA, 1984, Sigco 448	EC	3	56	187	bloom post	seed	28 42	0.01 < 0.01	98392
Adams Gardens, Texas, USA, 1984, T 5198	EC	3	50	187	blooming	seed	30 44	< 0.01 < 0.01	98392

Table 76. Residues of DCVA + FPBacid in sunflower seeds

Country	FL	N	g ai/ha	Growth stage at last application	Sample	PHI (days)	DCVA (mg/kg)	FPBacid (mg/kg)	Reference
East Grand Forks, Minnesota, USA, 1984, Sigco 448	EC	3	58	bloom, post	seed	28 42	0.01 0.05 [u] -	< 0.01 < 0.01	87350
Northwood, North Dakota, USA, 1984, Sigco 448	EC	3	58	bloom post	seed	28	0.01	< 0.01	87351
Harlingen, Texas, USA, 1984, T 5198	EC	3	50	blooming	seed	30 44	< 0.01 -	< 0.01 < 0.01	87352
High Bluff Manitoba, Canada, 1984, HY 894	EC	3	50	bloom, post	seed	28	< 0.01	< 0.01	87353
Portage, Manitoba, Canada, 1984, HY 894	EC	3	50	bloom, post	seed	28	0.02 0.01 [u]	< 0.01	87354

u - Sample from control (untreated) plot.

Animal feeds

Table 77. Residues for cyfluthrin in sweet corn feed items (residues on an 'as received' basis)

Country	Application					Residues			Reference
	FL	N	g ai/ha	L/ha	Growth stage at last application	Sample	PHI (days)	Cyfluthrin (mg/kg)	
Lamberton, Minnesota, USA, 1989, Green Giant	EC	1 0	50[b]	47	kernel filling	forage husk Cannery waste (calculated)	0 1 3 7 14 0 1 3 7 14 0 1 3 7 14	3.0 3.4 3.2 <u>3.7</u> 2.5 0.02 [u] 0.42 0.36 0.37 0.41 0.31 <u>0.20</u> 0.17 0.16 0.18 0.14	100248

Country	Application					Residues			Reference
	FL	N	g ai/ha	L/ha	Growth stage at last application	Sample	PHI (days)	Cyfluthrin (mg/kg)	
Dundee, Oregon, USA, 1989, Golden Jubilee	EC	10	50 [b]	47	ears mature	forage	0	7.7	100248
							1	3.2	
							3	5.8	
							7	3.3	
							14	1.9	
								0.01 [u]	
						husk	0	1.8	
							1	1.2	
							3	0.93	
							7	0.75	
							14	0.79	
						Cannery waste (calculated)	0	0.90	
							1	0.54	
							3	0.44	
							7	0.36	
							14	0.34	
Sodus, New York, USA, 1989, Crusade	EC	10	50 [b]	52	fresh market ears	forage	0	3.0	100248
							1	2.8	
							3	3.7	
							7	1.0	
							14	0.97	
								0.02 [u]	
						husk	0	0.20	
							1	0.54	
							3	0.82	
							7	0.34	
							14	0.34	
						Cannery waste (calculated)	0	0.10	
							1	0.26	
							3	0.43	
							7	0.16	
							14	0.16	
Phelps, New York, USA, 1983, Jubilee	GR EC	1 10	50	238	grain milk stage	forage	0	6.5	98313 [a]
						kernel, dry	45	< 0.01	
						cob, dried	45	0.02	
						husk, dry	45	0.24	
						husk	0	0.37	
						fodder, dry	45	1.7	
						Cannery ws	0	0.16	
Benson, Minnesota, USA, 1983, Golden Bantam	GR EC	1 10	50	123	milk stage	husk	0	2.3	98313 [a]
						forage	0	12	
						Cannery ws	0	1.1	
Danville, Iowa, USA, 1983, Northrup King 199	GR EC	1 10	50	280	soft dough	husk	0	2.1	98313 [a]
						forage	0	9.9	
						Cannery ws	0	0.71	
						kernel, dry	44	< 0.01	
						cob, dried	44	< 0.01	
						husk, dry	44	1.7	
						fodder, dry	44	1.9	
Vero Beach, Florida, USA, 1983, Guardian	GR EC	1 10	50 (+0.5 g/L X-77)	421	early milk stage	husk	0	1.4	98313[a]
						forage	0	3.1	
						Cannery ws	0	0.70	
						kernel, dry	45	< 0.01	
						cob, dried	45	< 0.01	
						husk, dry	45	0.58	
						fodder, dry	45	5.6	

Country	Application					Residues			Reference
	FL	N	g ai/ha	L/ha	Growth stage at last application	Sample	PHI (days)	Cyfluthrin (mg/kg)	
Urbana, Illinois, USA, 1983, Silver Queen	GR EC	1 10	50	468	milk stage	husk forage Cannery ws kernel, dry cob, dried husk, dry fodder, dry	0 0 0 69 69 69 69	< 0.01 8.6 0.41 < 0.01 < 0.01 0.94 0.53	98313 [a]
Marcellus, Michigan, USA, 1983, Seneca Star	GR EC	1 10	50	505	milk stage	Husk forage Cannery ws kernel, dry cob, dried husk, dry fodder, dry	0 0 0 47 47 47 47	2.9 6.8, 0.01 [u] 1.5 < 0.01 < 0.01 3.9 3.6, 0.01 [u]	98313 [a]
Adams Gardens, Texas, USA, 1983, Silver Queen	GR EC	1 10	50	234	milk stage	husk forage Cannery ws kernel, dry cob, dried husk, dry fodder, dry	0 0 0 24 24 24 24	0.53 10, 0.01 [u] 0.28 0.01 < 0.01 1.2 22	98313 [a]
Stilwell, Kansas, USA, 1983, Iochief	GR EC	1 10	50	140	grain milk stage	husk forage Cannery ws kernel, dry cob, dried husk, dry fodder, dry	0 0 0 44 44 44 44	1.4, 0.01 [u] 7.7 c0.03 0.62 0.02 0.02 6.2 28	98313 [a]
Tifton, Georgia, USA, 1983, Silver Queen	GR EC	1 10	50	64	milk stage	husk forage Cannery ws kernel, dry cob, dried husk, dry fodder, dry	0 0 0 45 45 45 45	2.2, 0.01 [u] 53 c0.05 1.1 < 0.01 0.02 1.8 23	98313 [a]
Springfield, Nebraska, USA, 1983, Iochef Hybrid Earl May	GR EC	1 13	50	468	grain milk stage	forage Cannery ws kernel, dry cob, dried husk, dry fodder, dry	0 0 46 46 46 46	3.7, 0.01 [u] 0.21 < 0.01 < 0.01 0.59 2.1	98313 [a]
Howe, Indiana, USA, 1983, Silver Queen	GR EC	1 10	50	468	milk stage	forage Cannery ws kernels dry cob dried husk dry fodder dry	0 0 45 45 45 45	12 0.59 < 0.01 < 0.01 0.57 1.4	98313 [a]
Canby, Oregon, USA, 1983, Golden Jubilee	GR EC	1 15	50	374	milk stage	husk Forage Cannery ws	0 0 00	1.0, 0.04 [u] 14, 0.04 [u] 0.42	98313 [a]

Country	Application					Residues			Reference
	FL	N	g ai/ha	L/ha	Growth stage at last application	Sample	PHI (days)	Cyfluthrin (mg/kg)	
Greenfield, California, USA, 1983, Tendersweet	GR EC	1 10	50	468	harvest	forage Cannery ws kernel, dry cob, dried husk, dry fodder, dry	0 0 44 44 44 44	7.5, 0.02 [u] 1.3 < 0.01 < 0.01 n.a. 5.0	98313 [a]

Cannery waste (calculated) = cob plus husk with kernels removed = (cob (mg/kg)×cob weight(kg) + husk (mg/kg)×husk weight (kg))/(cob weight + husk weight)

a - Invalidated analytical data

b - Application method aerial

u - Sample from control (untreated) plot.

Table 78. Residues for cyfluthrin on cotton forage and gin trash (residues on an 'as received' basis)

Country	Application					Residues			Reference
	FL	N	g ai/ha	L/ha	Growth stage at last application	Sample	PHI (days)	Cyfluthrin (mg/kg)	
Dill City, Oklahoma, USA, 1995, Paymaster HS-26	EC	10	56	96 – 110	Bolls ca. 75% open	gin trash	0	2.4	107539
Benoit, Mississippi, USA, 1995, DPL 50	EC	10	55 – 57	85 – 91	Bolls 60 – 70% Open	gin trash	0	2.8	107539
Fresno, California, USA, 1995, Maxxa	EC	10	55 – 56	88 – 94	Bolls 50 – 60% Open	gin trash	0	9.2	107539
Benoit, Mississippi, USA, 1982, Stoneville 213	EC	10	100	41	after flowering	green material	0	11	84365
Tifton, Georgia, USA, 1982, Coker 310	EC	10	100	86	10 – 20% buds open	forage, green	0	14	84366
Eudora, Arkansas, USA, 1982, DPL 55	EC	10	100	41	after flowering	forage, green	0	15	84380

Table 79. Residues for beta-cyfluthrin in cotton gin trash (residues on an 'as received' basis)

Country	Application					Residues			Reference
	FL	N	g ai/ha	L/ha	Growth stage at last application	Portion analysed	PHI (days)	Beta-cyfluthrin (mg/kg)	
Dill City, Oklahoma, USA, 1995, Paymaster HS-26	SC	10	25 – 28	96 – 110	Bolls ca. 75% open	gin trash	0	2.3	107539

Country	Application					Residues			Reference
	FL	N	g ai/ha	L/ha	Growth stage at last application	Portion analysed	PHI (days)	Beta-cyfluthrin (mg/kg)	
Benoit, Mississippi, USA, 1995, DPL 50	SC	10	28 – 29	87 – 91	Bolls 60 – 70% Open	gin trash	0	2.6	107539
Fresno, California, USA, 1995, Maxxa	SC	10	28 – 29	88 – 94	Bolls 50 – 60% Open	gin trash	0	2.9	107539

Soya beans

Table 80. Residues for cyfluthrin in soya bean animal feeds (residues on an 'as received' basis)

Country	Application					Residues			Reference
	FL	N	g ai/ha	L/ha	Growth stage at last application	Portion analysed	PHI (days)	Cyfluthrin (mg/kg)	
Danville, Iowa, USA, 1990, Washington VI	EC	4	49	42	full pod	forage hay vines	15 15 49	0.26 0.36 0.31	103823
Danville, Iowa, USA, 1990, Washington VI	EC	4	49	187	full pod	forage hay vines	15 15 49	0.38 0.57 0.21, 0.02 [u]	103823
Benoit, Mississippi, USA, 1990, Young	EC	4	49	47	pod fill	forage hay vines	15 15 52	0.36, 0.05 [u] 1.4, 0.16 [u] 0.09	103823
Stilwell, Kansas, USA, 1990, Williams 82	EC	4	49	180	pod filling	forage hay vines	8 15 45	1.3, 0.10 [u] 3.2 2.7, 0.05 [u]	103823
Tifton, Georgia, USA, 1990, Braxton	EC	4	49	3.15	mid pod	forage hay vines	15 15 54	0.45 1.4 0.01	103823
Benoit, Mississippi, USA, 1984, Centennial	EC	4	50	78 78 71 71	height 51 – 56 cm	forage hay	15 15	0.34 1.0	98398
Howe, Indiana, USA, 1984, Pella	EC	4	50	234	pod fill	forage hay	15 15	0.10 0.63	98398
Springfield, Nebraska, USA, 1984, Century	EC	4	50	243	pod fill	forage hay	15 15	0.96 0.96	98398
Stilwell, Kansas, USA, 1984, Williams	EC	4	50	224	pod fill	forage hay	15 21	0.33 0.46	98398
Tifton, Georgia, USA, 1984, Bragg	EC	4	50	62	pod fill	forage hay	15 15	3.3 0.62	98398
Springfield, Nebraska, USA, 1982, Williams	EC	4	50	243	early pod stage	forage straw	15 61	1.3 0.19	98398 [a]
Urbana, Illinois, USA, 1982, SRF 350 P	EC	4	50	187	fruit development	forage straw	15 48	2.0 0.49	98398 [a]
Jackson, Tennessee, USA, 1982, Forrest	EC	4	50	85	fruit development	forage straw	14 31	1.3 0.34	98398 [a]

Country	Application					Residues			Reference
	FL	N	g ai/ha	L/ha	Growth stage at last application	Portion analysed	PHI (days)	Cyfluthrin (mg/kg)	
Eudora, Arkansas, USA, 1982, Macks	EC	4	50	102	Vegetative	forage straw	15 59	2.2 0.01 [u] 0.37	98398 [a]
Benoit, Mississippi, USA, 1982, Bragg	EC	4	50	41	Vegetative	forage straw	15 76	1.2 0.04, 0.04 [u]	98398 [a]
Howe, Indiana, USA, 1982, Wells II	EC	4	50	234 234 252 252	pod fill	forage straw	15 39	0.85 0.36, 0.02 [u]	98398 [a]
Tifton, Georgia, USA, 1982, Bragg	EC	4	50	888	mid-pod fill	forage straw	15 35	7.2 0.03, 0.01 [u]	98398 [a]

a - Invalidated analytical data

u - Sample from control (untreated) plot.

Rape seed

Table 81. Residues for cyfluthrin in rape forage and straw (residues on an 'as received' basis)

Country	Application					Residues			Reference
	FL	N	g ai/ha	L/ha	Growth stage at last application	Sample	PHI (days)	Cyfluthrin (mg/kg)	
Burscheid, Versuchsgut Höfchen, Germany, 1985, Quinta	EC	2	15	600	end of flowering	Forage straw	0 42 49 56	0.32 < 0.02 0.02 0.06	5600-85
Gröhnwohld, Germany, 1985, Jet Nuef	EC	2	15	600	end of flowering	forage	0 42 49 56	0.27 < 0.02 < 0.02 < 0.02	5601-85
Ensheim, Germany, 1985, Belinda	EC	2	15	600	end of flowering	Forage straw	0 42 49 56	0.21< 0.02 < 0.02 < 0.02	5602-85
Worms-Heppenheim, Germany, 1985, Petranova	EC	2	15	600	end of flowering	forage	0 21 28	0.18 0.06, 0.02 [u] 0.04	5603-85
Burscheid, Versuchsgut Höfchen, Germany, 1984, Quinta	EC	2	15	600	end of flowering	forage	0 42	0.13 0.02	5600-84
Gröhnwohld, Germany, 1984, Korina	EC	2	15	600	end of flowering	forage	0 42 49 56	0.15 < 0.02 < 0.02 < 0.02	5601-84
Göllheim, Germany, 1984, Elvira	EC	2	15	600	end of flowering	forage	0 42 49 56 63	0.20 0.03 0.04 0.02 < 0.02	5602-84

	Application					Residues			Reference
Country	FL	N	g ai/ha	L/ha	Growth stage at last application	Sample	PHI (days)	Cyfluthrin (mg/kg)	
Gau-Odernheim, Germany, 1984, Loras	EC	2	15	600	end of flowering	Forage straw	0 42 49 56 63	0.34 < 0.02 0.02 0.02 < 0.02	5603-84

u - Sample from control (untreated) plot.

Table 82. Residues of beta-cyfluthrin in rape forage and straw

	Application					Residues			Reference
Country	FL	N	g ai/ha	L/ha	Growth stage at last application	Sample	PHI (days)	Cyfluthrin (mg/kg)	
St. Alban, Hengstbacher Hof, Germany, 1992, Arabella	SC	3	10	424	End of flowering	forage straw	0 42 49 56 63	0.19 < 0.05 < 0.05 < 0.05 0.08	RA-2070/92
Grönwohld, Germany, 1992, Falcon	SC	3	10	300	End of flowering	Forage straw	0 42 49 56 63	0.27 < 0.05 < 0.05 < 0.05 0.07	RA-2070/92
Burscheid, Versuchsgut Höfchen, Germany, 1992, Lirajet	SC	3	10	300	End of flowering	Forage straw	0 42 49 56 64	0.17 < 0.05 < 0.05 < 0.05 < 0.05	RA-2070/92
Walksfelde, Germany, 1992, Falcon	SC	3	10	300	End of flowering	Forage straw	0 42 49 56 63	0.33 < 0.05 < 0.05 < 0.05 0.06	RA-2070/92
Burscheid-Höfchen, Germany, 1988, Ceres	EC	3	7.5	300		whole plant without roots	0 42 64	< 0.05 < 0.05 < 0.05	0369-88
Ensheim, Germany, 1988, Arabella	EC	3	7.5	600	51 – 53	whole plant without roots	0 42 49 56	0.08 < 0.05 < 0.05 < 0.05	0370-88
Grönwohld, Germany, 1988, Ceres	EC	3	7.5	600	70	whole plant without roots	0 42	0.24 < 0.05	0371-88
Burscheid, Versuchsgut Höfchen, Germany, 1987, Jef Nuef	EC	3	7.5	600	pod development	green material rest of plant	0 42 49	0.26 0.02 0.02	5700-87
Kirchlauter-Pettstadt, Germany, 1987, Lirabon	EC	3	7.5	600	full flowering	forage	0 42	0.10, 0.02 [u] < 0.01	5701-87

Country	Application					Residues			Reference
	FL	N	g ai/ha	L/ha	Growth stage at last application	Sample	PHI (days)	Cyfluthrin (mg/kg)	
Grönwohld, Germany, 1987, Ceres	EC	3	7.5	600	end of flowering	forage	0 42 49 56	0.16 0.03 0.03 0.02	5702-87

u - Sample from control (untreated) plot.

Sunflower

Table 83. Residues for Cyfluthrin in sunflower fodder

Country	Application					Residues			Reference
	FL	N	g ai/ha	L/ha	Growth stage at last application	Sample	PHI (days)	Cyfluthrin (mg/kg)	
High Bluff, Manitoba, Canada, 1984, HY 894	EC	3	50		bloom, post	fodder, dry	28 43	0.30 0.09	87205
Portage, Manitoba, Canada, 1984, HY 894	EC	3	50		bloom, post	fodder, dry	28 43	0.04 0.03	87206
East Grand Forks, Minnesota, USA, 1984, Sigco 448	EC	3	50	187	bloom, post	fodder, dry	28 42	0.22 0.33	98392
Northwood, North Dakota, USA, 1984, Sigco 448	EC	3	56	187	bloom post	fodder, dry	28 42	0.26 0.63	98392
Adams Gardens, Texas, USA, 1984, T 5198	EC	3	50	187	blooming	fodder, dry	30 44	0.13 0.08	98392

Table 84. Residues of DCVA + FPBacid in sunflower fodder

Country	FL	N	g ai/ha	Growth stage at last application	Sample	PHI (days)	DCVA (mg/kg)	FPBacid (mg/kg)	Reference
East Grand Forks, Minnesota, USA, 1984, Sigco 448	EC	3	58	bloom, post	fodder, dry	28	0.01 0.02 [u]	0.04	87350
Northwood, North Dakota, USA, 1984, Sigco 448	EC	3	e58	bloom post	fodder, dry	28	0.01	0.03	87351
Harlingen, Texas, USA, 1984, T 5198	EC	3	50	blooming	fodder, dry	30	0.22 0.01 [u]	< 0.01	87352
High Bluff Manitoba, Canada, 1984, HY 894	EC	3	50	bloom, post	fodder, dry	28	0.02 0.02 [u]	0.09	87353

Country	FL	N	g ai/ha	Growth stage at last application	Sample	PHI (days)	DCVA (mg/kg)	FPBacid (mg/kg)	Reference
Portage, Manitoba, Canada, 1984, HY 894	EC	3	50	bloom, post	fodder, dry	28	0.01 0.02 [u]	0.02	87354

u - Sample from control (untreated) plot.

Fate of residues in storage and processing

Residues after Processing

Processing studies are necessary according to the uses and the residues of beta-cyfluthrin/cyfluthrin on raw agricultural commodities. The fate of beta-cyfluthrin/cyfluthrin residues during processing of raw agricultural commodities was investigated in several major registered crops (oilseeds, fruits, cereals, and brassicas) using important processing procedures.

As a measure of the transfer of residues into processed products, a processing factor was used, which is defined as:

$$F = \frac{\text{Residue in processed product (mg/kg)}}{\text{Residue in raw agricultural commodity (mg/kg)}}$$

A concentration of residues takes place when PF > 1.

Residues in processed citrus fruit

A processing study was conducted to determine if residues would concentrate in orange processed products following treatment with cyfluthrin (Burger 1992 102618). Cyfluthrin was applied at a spray concentration of 15 g ai/hL in the orange processing study, at five times the exaggerated rate. Mature fruit was sampled from each tree. The processing procedure simulated typical commercial practices as closely as possible. Cyfluthrin residues in orange processed commodities were determined by GC-ECD.

No details were available on the methodology used in the processing process for the oranges.

All orange processed commodities, except for juice, showed a concentration of residues from the unprocessed whole fruit. For oranges the dried pulp, peel, oil and molasses had calculated concentration factors of 5.3, 1.2, 5.3 and 2.9, respectively.

Table 85. Results of processing of orange

Country	FL	N	g ai/hL	L/ha	Growth stage at last application	Sample	PHI (days)	Cyfluthrin (mg/kg)	PF	Reference
Vero Beach, Florida, USA, 1991, Valencia	EC	1	15		Maturity	fruit	14	0.2	-	Burger, R. N. 22.04.1992 M-044752-01-1
						pulp, dry	14	1.0	5	
						peel	14	0.23	1.2	
						oil	14	1.1	5	
						molasses	14	0.58	3	
						juice	14	< 0.01	< 0.05	

Recoveries for oranges and processed commodities fortified at 0.01 – 0.1 mg/kg were 73 – 123%.

Heinemann and Seym (1996 RA-3000/95) studied the effect of processing on residues of cyfluthrin in apples (washed fruit, dried fruit, juice, pomace-dried and sauce). Cyfluthrin was applied as a foliar spray (35 – 38 g ai/ha; 1000 – 1087 L/ha) three times to apple trees (spraying interval 14

days) with harvest three days after the last application. Washing of apples was completed using domestic practices. The preparation of dried apples, apple juice, dried pomace and apple sauce was designed to simulate industrial practice but on a laboratory scale.

Apples (approximately 2.3 – 2.4 kg) were washed in standing water under slow movement (washed apples). For preparation of apple juice and dried pomace, apples (approximately 14.2 – 14.4 kg) were washed in standing water under slow movement. After washing, the apples were cut into small pieces and mashed in a cutter. The mash was pressed to raw juice and wet pomace. The wet pomace (approximately 2.2 – 2.7 kg) was dried at approximately 60 °C to a water content of < 10% (dry pomace). The raw juice was heated to approximately 90 °C for about 30 seconds and subsequently cooled down to 50 – 55 °C. Enzyme was added (Novo Pectinex 3XL (200 µL/kg juice) and Amylase AG 200 L (80 µL/kg juice)). After enzymation the juice was centrifuged, subject to ultra-filtration and pasteurised at 87 – 88 °C for around 50 seconds (juice).

Apple sauce was prepared from apples (approximately 2.5 – 3.3 kg) that were first washed in standing water under slow movement. After washing, the apples were cut into small pieces. The cut apples were heated with the addition of 250 ml water/2 kg apples to 98 – 100 °C for around 10 minutes and the apples passed through a strainer to separate apple sauce and pomace. Sugar (l00 g/kg raw apple sauce) was added and the apple sauce was filled into 1/1 preserving cans and pasteurised at 82 – 88 °C.

For preparation of dried apples, washed apples were peeled, the apple cores removed and the fruit cut into apple slices. The apple slices were dipped in a solution of potassium metabisulfite and then in a solution of citric acid to prevent enzymatic reactions. The apple slices were then rinsed off with water and dried at about 65 °C to a water content of approximately 17.6%.

All samples were stored deep frozen until required for analysis according to Method 00255/E003 (fruit washed) and Method 00255/E005 (fruit-dried, juice, pomace-dried and sauce). Recovery values ranged from 97 to 121% over all sample materials with relative standard deviations from 2.0 to 8.9%. The LOQ was 0.01 mg/kg for fruit-washed, juice and apple-sauce and 0.05 mg/kg for fruit-dried and pomace dried.

Table 86. Results of processing on apples

Country	FL	N	g ai/ha	L/ha	Growth stage at last application	Sample	PHI (days)	Cyfluthrin (mg/kg)	PF	Reference
F-82100 St. Aignan France, 1995, Fuji	EC	3	35 – 38	1000 – 1087	mature	fruit	3	0.13		Heinemann, O.; Seym, M. 19.11.1996 M-053919-01-1
						juice	3	< 0.01	< 0.08	
						pomace, dried	3	2.1	16.2	
						sauce	3	0.03	0.23	
						Fruit washed	3	0.07	0.54	
						Fruit dried	3	< 0.05	< 0.38	

Wiedmann and Jablonski (1990 100203). Ten applications of cyfluthrin (EC) were made at an exaggerated rate of 280 g ai/ha/application. This represents three times the normal application rate.

Applications were made at the 'fruit-maturing' stage. Harvest was seven days after the final treatment. Whole fruit were processed into wet pomace, dry pomace, and juice.

Apples that were not previously frozen were chopped using a Hobart food chopper. Frozen apples were thawed and cut vertically into quarters using a sharp knife. One quarter from each apple (aliquot for whole fruit analysis) was placed into a plastic bag on an enamel pan such that the pieces are distributed on a flat surface and frozen. The remaining three quarters from each apple were chopped or quartered and the chopped fruit added to an apple press and the juice collected. A sample was retained from the combined wet pomace from the press and the later was dried at 77 °C for 6.5 h or until dry. The pomace was stirred periodically to promote uniform drying. Although the volatility

of cyfluthrin is not high, the significant reduction of residue was attributed to the atypical drying process.

Apple and apple processed fractions were held in frozen storage for a maximum period of 205 days prior to analysis. Recoveries for samples fortified at 0.01 to 2.5 mg/kg ranged from 85 to 115%.

Table 87. Results of processing on apples

Country	FL	N	g ai/ha	L/ha	Growth stage at last application	Sample	PHI (days)	Cyfluthrin (mg/kg)	PF	Reference
Howe, Indiana, USA, 1989 Rome	EC	10	281	257	mature	fruit pomace, dried pomace, wet juice	7 7 7 7	0.83 0.09 2.3 0.05	 0.11 2.7 0.06	Wiedmann, J. L.; Jablonski, J. E. 24.09.1990 M-053945-01-1

In a separate study, Harbin (2002 110032) evaluated the fate of residues in apples and apple processed commodities. A single foliar spray application of cyfluthrin (WP) was made to apple trees at a rate of 250 g ai/ha. Mature apples were collected at a six day pre-harvest interval (PHI) and stored refrigerated. A sub sample of the unwashed apples (RAC) was removed for analysis prior to the washing step of the procedure, and the remainder of the apples were processed to generate washed fruit, wet pomace, juice, concentrated juice, applesauce, and dried fruit. Processing was performed using methods designed to simulate commercial practices.

Bulk apple samples were stored at 3 °C until processing was initiated seven days after harvest. Sub-samples of the unwashed apples (RAC) were taken and immediately placed in frozen storage (-5 °C). The remaining apples were washed and an aliquot of the washed apples pulverized in a hammer mill, and the macerate transferred to an apple press. The mash was pressed to remove the juice and pressed mash pulverized to produce the wet pomace. The resultant juice was heated, de-pectinised, cooled, filtered, and concentrated to produce the juice concentrate. Another aliquot of the washed apples was peeled, cored, chopped, steamed and screened to produce a coarse sauce. The moisture content and sweetness of the sauce were adjusted, and the sauce heated (> 155°C) to produce the finished apple sauce. A third aliquot of the washed apples was peeled, cored, trimmed, sliced, dipped in a sulfite solution, and dried using hot air to produce dried fruit.

Cyfluthrin residues were quantified by GC-MS. Recoveries of cyfluthrin from unwashed apples ranged from 83 to 95% when fortified at 0.01 and 0.05 mg/kg. Recoveries of cyfluthrin from washed fruit ranged from 106% to 114% when fortified at 0.01 and 0.05 mg/kg. Recoveries of cyfluthrin from wet pomace ranged from 87 to 117% when fortified at 0.01 and 0.30 mg/kg. Recoveries of cyfluthrin from juice ranged from 114 to 121% when fortified at 0.01 mg/kg. Recoveries of cyfluthrin from concentrated juice ranged from 77 to 108% when fortified at 0.01 and 0.02 mg/kg. The LOQ for cyfluthrin residue in all of the apple matrices was 0.01 mg/kg.

The unwashed apple (RAC) samples were held in frozen storage for a maximum of 444 days prior to extraction. The apple processed commodities of washed fruit, wet pomace, juice, concentrated juice, applesauce, and dried fruit were held in frozen storage for a maximum of 364 days prior to extraction.

Table 88. Results of processing on apples

Country	FL	N	g ai/ha	L/ha	Growth stage at last appl'n	Sample	PHI (days)	Cyfluthrin (mg/kg)	PF	Reference
North Rose, New York, USA, 1999,	WP	1	250	748	85	fruit whole fruit, washed pomace, wet juice Juice, concentrated	6 6 6 6 6	0.05 0.04 0.21 < 0.01 < 0.01	 0.8 4.2 < 0.2 < 0.2	Harbin, A. M. 11.03.2002 M-058414-02-1

Country	FL	N	g ai/ha	L/ha	Growth stage at last appl'n	Sample	PHI (days)	Cyfluthrin (mg/kg)	PF	Reference
Idared						sauce fruit, dried	6 6	< 0.01 0.01	< 0.2 0.2	

Leslie (1988 98399), reported details of processing of tomatoes. A single residue study was conducted in Texas, USA in which six foliar spray applications of an EC formulation of cyfluthrin were made to tomato plants at the rate of 50 g ai/ha/application. Intervals between applications ranged from four to seven days with spray volumes of 374 L/ha applied using ground equipment. Whole tomatoes were processed into juice, purée and wet and dry pulp (i.e. pomace). Tomato ketchup and paste were produced from the processed purée.

Tomatoes were processed according to simulated commercial practice. Tomatoes were cooked in a steam jacket kettle, and then put through a grinder to separate out the skins and seeds. Some of the pulp and juice were returned to the steam jacket kettle and cooked to a Brix rating of 12 to produce purée. A 200 g sample of purée was evaporated in a beaker on a hot plate (medium heat) to ketchup consistency. Half of the ketchup was saved for analysis and the remainder was heated further to paste consistency. The moisture content of the ketchup and paste was determined by heating sub-samples to constant weight and determining the weight loss on heating. Samples were held in frozen storage for a maximum of 214 days prior to analysis.

Residues in the processed products were: 0.02 mg/kg, juice; 0.05 mg/kg, ketchup; 0.04 mg/kg, purée; 0.11 mg/kg, paste; 0.39 mg/kg, wet pulp and 1.3 mg/kg, dry pulp. The whole fruit contained a residue of 0.06 mg/kg prior to processing.

Based upon the residue values in the unprocessed tomato fruit and resulting processed component values, the concentration factors for the tomato processed products would be: 1.8, paste; 6.5, wet pulp; and 22, dry pulp.

Table 89. Results of processing trials conducted with cyfluthrin on tomatoes

Country	FL	No	g ai/ha	L/ha	Growth stage at last application	Sample	PHI (days)	Cyfluthrin (mg/kg)	PF
Adams Garden, Texas, USA, 1988, Flora Dade	EC	6	50	374	fruit 5.08 – 7.62 cm	fruit	0	0.06	-
						juice	0	0.02	0.3
						ketchup	0	0.05	0.8
						purée	0	0.04	0.7
						paste	0	0.11	1.8
						pulp, wet	0	0.39	6.5
						pulp, dry	0	1.3	22

Recoveries for tomatoes and processed commodities fortified at 0.05 mg/kg were 70 – 112%.

A study was conducted to evaluate the quantity of cyfluthrin residue in cotton and cotton processed commodities following the application of cyfluthrin (Anon. 1983 84368). Ten foliar spray applications of cyfluthrin (EC) were made to cotton plants (5×500 and 5×100 g ai/ha). The application occurred when most of the cotton bolls were open and samples of cotton bolls were taken immediately after the final application. Cyfluthrin residues were quantified by Method 085823 (Mobay Chemical Corporation).

Lots of approximately 4.5 to 23 kg of cottonseed were hulled and the de-hulled seed separated from the hulls by screening. The percentage of de-hulled seed recovered was usually just under 50 percent of the weight of the whole seed. The de-hulled seeds were flaked to about 0.038 to 0.051 mm thick and the flakes placed into batch solvent extractor. Solvent (Skellysolve F or B) was run into the bottom of the extractor after being heated to just under its boiling point until it just covers the charge of de-hulled seeds. The charge was allowed to stand in contact with the solvent for 30 minutes.

Solvent at a temperature of 38 °C was then run into the top of the extractor so as to keep the level above the flakes while solvent oil mixture (miscella) was drawn off. The extracted flakes were at < 66 °C and contained a residual oil content of < 1%. Oil was recovered from the miscella by evaporating the solvent (38 to 60 °C) under reduced pressure. The oil recovered contained 6 to 8% total moisture and volatile matter. A portion of the oil was refined according to the official laboratory methods of the American Oil Chemists' Society (AOCS. Official Method Ca -9a-52.)

Table 90. Results of processing of cotton seed

Country	FL	N	eg ai/ha	L/ha	Growth stage at last applic'n	Portion analysed	PHI (days)	Cyfluthrin (mg/kg)	PF	Reference
Adams Gardens, Texas, USA, 1982, McNair 220	EC	10	5×100 5×500	19	majority of buds open	seed hull fuzzy seed meale oil, native oil, refined oil, refined, deodorized soap stock	0 0 0 0 0 0 0 0	0.13, 0.01 [u] 0.25, 0.01 [u] 0.04 0.01 0.25, 0.04 [u] 0.15, 0.04 [u] 0.18, 0.03 [u] < 0.01	 e1.92 0.31 0.08 1.92 1.15 1.38 < 0.08	Anon. 30.08.1983 M-049103-01-1

u - Sample from control (untreated) plot.

Burger and Lenz (1992 103825). Four applications of cyfluthrin were made at a rate of 245 g ai/ha per application. Samples were collected 45 days following the last application. Field treated soya bean was processed into hulls, meal, crude oil, refined oil, and soap stock. The processing procedure simulated typical commercial practices as closely as possible. Cyfluthrin residues in soya bean seed and soya bean processed commodities were determined by GC-ECD.

The moisture content of the seed was reduced to 7 – 10% by drying at 61 – 71 °C in a forced air oven and the seed cleaned by aspiration and screening. The hulls were cracked in a mill and aspirated to separate the hulls from the kernels. Kernels were heated to 66 – 74 °C prior to flaking with a flaking roll, set to a 0.02 to 0.03 mm roll gap setting. The crude oil in the flaked kernels is solvent extracted with hexane (49 – 61 °C) in a steam-jacketed, stainless steel, batch extractor. At the end of the extraction the flakes were dried using a flow of warm air forced through the flakes. The hexane is evaporated from the miscella (crude oil and hexane). During this procedure the crude oil reaches a temperature of 75 – 85 °C. Refined oil is obtained using AOCS Method Ca9a52. The percentage free fatty acid is determined and an appropriate amount of NaOH added to the crude oil which is mixed at initially at 20 – 24 °C and then at 60 – 65 °C prior to refrigeration for at least 12 hours. At the end of this period, the refined oil is decanted and filtered. The fraction settling to the bottom of the refrigerated container is the soap stock. Soya bean processed commodities were held in frozen storage for a maximum possible period of 316 days (0.9 years). Recoveries for commodities fortified at 0.01 – 0.2 mg/kg ranged between 70 and 107%.

Table 91. Results of processing of soya bean seed

Country	Application					Residues		
	FL	No	g ai/ha	L/ha	Growth stage at last applic'n	Sample	PHI (days)	Cyfluthrin (mg/kg)
Stilwell, Kansas, USA, 1990, Williams 82	EC	4	245	180	pods filling	pod, empty	45	< 0.05
						meal	45	< 0.05
						oil, crude	45	< 0.05
						seed, dry	45	0.09
						oil, refined	45	< 0.05
						soap stock	45	< 0.05

A field study was conducted to determine if cyfluthrin residues would concentrate in sunflower processed products following field treatment with cyfluthrin (EC) (Burger and Lenz 1992 103835). Three applications of cyfluthrin were made at a rate of 245 g ai/ha per application. Samples were collected 30 days following the last application. The processing procedure simulated typical commercial practices as closely as possible. Cyfluthrin residues in sunflower seed and sunflower seed processed products were determined by GC-ECD.

The sunflower samples were cleaned by aspiration and screening. The hull surrounding the kernel was mechanically cracked and aspirated away from the kernel. The kernels were heat conditioned and pressed in an expeller for the purpose of liberating a majority of the crude oil. The residual crude oil remaining in the solid material (press cake) exiting the expeller was later extracted with the solvent, hexane. The solvent from the solvent extracted press cake (meal, if ground to a finer particle size) was removed. The crude oil recovered from the expeller and solvent extraction was combined and refined.

Residue data in this study were obtained using the analytical procedure described in Mobay report No. 85823 with minor modifications. Recoveries for commodities fortified at 0.008 to 0.4 mg/kg ranged from 70 – 113%. Samples were held in frozen storage for a maximum period of 433 days.

Table 92. Results of processing of sunflower seed

Country	FL	No	g ai/ha	L/ha	GS	Portion analysed	DALT (days)	Cyfluthrin (mg/kg)	PF
Stilwell, Kansas, USA, 1990, Cargill	EC	3	245	180	Maturity	seed	30	0.16	
						hull	30	0.18	1.1
						meal	30	< 0.08	< 0.5
						oil, crude	30	0.36	2.3
						oil, refined	30	0.17	1.1

GS = growth stage at last application

The fate of beta-cyfluthrin/cyfluthrin residues has been examined in potato, cabbage, tomato, citrus fruit, apples and oil seed crops processing studies. In processing of tomatoes into pulp and paste showed a slight increase of cyfluthrin residues in the processed commodities compared to the RAC. Whilst there was a decrease in residues found in the corresponding juice, ketchup and purée. Citrus and apples also both showed a decrease in residues found in the juice, but a slight increase into the pomace and/or oil and molasses. There was a concentration into the oil of cottonseed and sunflower. Processing studies on potatoes (Harbin 2000 109672), cabbages, soya bean and rape seed did not show any indication regarding the fate of beta-cyfluthrin/cyfluthrin residues during processing as residues in the RAC or processed products were all below the LOQ.

Table 93. Summary of processing factors for cyfluthrin residues

Raw agricultural commodity (RAC)	Processed commodity	Calculated processing factors	Mean, median or best estimate
Orange	Pulp dry	5.3	5.3
Orange	Peel	1.2	1.2
Orange	Oil	5.3	5.3
Orange	Molasses	2.9	2.9
Orange	Juice	< 0.05	< 0.05
Apple	Juice	< 0.08, 0.06, < 0.2	0.06
Apple	Pomace, dry	0.11, 16	0.11, 16
Apple	Sauce	< 0.2, 0.23	0.23
Apple	Fruit, washed	0.54, 0.8	0.67
Apple	Dried	0.2, < 0.38	0.2
Apple	Pomace, wet	2.7, 4.2	3.4
Apple	Juice, concentrated	< 0.2	< 0.2
Tomato	Juice	0.3	0.3
Tomato	Ketchup	0.8	0.8
Tomato	Purée	0.7	0.7
Tomato	Paste	1.8	1.8

Raw agricultural commodity (RAC)	Processed commodity	Calculated processing factors	Mean, median or best estimate
Tomato	Pulp, wet	6.5	6.5
Tomato	Pulp, dry	22	22
Cotton	Hulls	1.9	1.9
Cotton	Fuzzy seed	0.3	0.3
Cotton	Meal	0.08	0.08
Cotton	Oil, native	1.9	e1.9
Cotton	Oil, refined	1.2	1.2
Cotton	Oil, refined and de-odorised	1.4	1.4
Cotton	Soap stock	< 0.08	< 0.08
Sunflower	Hull	1.1	1.1
Sunflower	Meal	< 0.5	< 0.5
Sunflower	Oil, crude	2.3	2.3
Sunflower	Oil, refined	1.1	1.1

Residues in Animal Commodities

Shaw and Ayers (1983 MR-86045) dosed Holstein dairy cows (average daily weights for dose groups 394 – 473 kg, average 12 – 16 kg milk/day) with cyfluthrin at levels corresponding to the equivalent of 4.5, 13 and 40 ppm in the diet based on actual feed consumption and doses (target levels were 5, 15 and 50 ppm). Cyfluthrin was administered *via* capsule orally once per day after the morning milking. Although 29 morning doses were administered to each animal, only 28 full test days constituted the study (28-day feeding study), since the animals were sacrificed shortly after dosing on the morning of day 29.

Aliquots of evening milk and the following morning's milk were mixed together to form a sample representative of a single day. The milk samples were retained frozen for up to 43 days before extraction and analysis. Samples of muscle (composite of flank, loin and round), fat (composite of renal, omental and subcutaneous), liver and kidney were collected at sacrifice and stored retained frozen for up to 35 days prior to analysis.

Cyfluthrin was removed from the sample matrix, by organic solvent extraction, acetone/chloroform (2:1) for all tissues except fat for which hexane was used. The organosoluble extract was partitioned with various solvents to remove lipids and polar and non-polar interferences. The final purification step was column chromatography of the sample on either a silica gel column or a Florisil Sep-Pak. Determination of cyfluthrin residues was by GC-ECD. Recoveries for milk fortified at 0.02 mg/kg were 90 to 125% and for tissues fortified at 0.05 mg/kg 67 to 100%.

Cyfluthrin residues in milk from animals in the 40 ppm dose group ranged from 0.08 to 0.26 mg/kg over the intervals tested. At 28 days, the 13 ppm cows produced milk containing cyfluthrin residues ranging from 0.10 to 0.17 mg/kg and the 4.5 ppm cows showed residues of 0.01 to 0.02 mg/kg in their milk. Residue levels peaked at day 14 and declined thereafter through the end of the study. Partitioning of residues between whole milk and cream was not studied.

Residue levels of cyfluthrin in tissues of animals dosed at the highest rate (40 ppm) averaged < 0.01 mg/kg for liver, 0.01 mg/kg for kidney, 0.03 mg/kg for muscle and 2.64 mg/kg for fat. At the intermediate dose rate (13 ppm), average residue levels were < 0.01 mg/kg for liver, kidney and muscle and 0.70 mg/kg for fat. Residue levels of cyfluthrin for the low dose (4.5 ppm) were < 0.01 mg/kg for liver, kidney, and muscle and 0.25 mg/kg for fat.

Table 94. Cyfluthrin residues in milk from cows dosed with cyfluthrin daily for 29 days

Dose Level (mg/kg)	Residues of Cyfluthrin (mg/kg)			
	Day 7	Day 14	Day 21	Day 28
4.5	NA NA. NA	NA NA NA	NA NA NA	0.02 0.02 0.01
Average				0.02
13	NA	NA	NA	0.03

Dose Level (mg/kg)	Residues of Cyfluthrin (mg/kg)			
	Day 7	Day 14	Day 21	Day 28
	NA	NA	NA	0.03
	NA	NA	NA	0.08
Average				0.05
40	0.16	0.25	0.21	0.17
	0.19	0.26	0.21	0.16
	0.08	0.16	0.12	0.10
Average	0.14	0.22	0.18	0.14

Table 95. Cyfluthrin residues in tissues from cows dosed with cyfluthrin daily for 29 days

Dose Level (mg/kg)	Residues of Cyfluthrin (mg/kg)			
	Fat	Muscle	Liver	Kidney
4.5	0.30	< 0.01	NA	NA
	0.24	< 0.01	NA	NA
	0.21	< 0.01	NA	NA
Average	0.25	< 0.01		
13	0.66	< 0.01	< 0.01	< 0.01
	0.71	< 0.01	< 0.01	< 0.01
	0.73	0.02	< 0.01	< 0.01
Average	0.70	< 0.01	< 0.01	< 0.01
40	2.4	0.03	< 0.01	0.01
	2.5	0.03	< 0.01	< 0.01
	3.0	0.03	< 0.01	0.02
Average	2.6	0.03	< 0.01	0.01

Re-analysis of the 40 ppm liver and kidney samples using stronger mechanical homogenization/extraction method (Tekmar Tissuemizer instead of Omni-mixer) gave residues of cyfluthrin in liver of 0.14, 0.13 and 0.13 mg/kg and in kidney 0.18, 0.16 and 0.16 mg/kg (Murphy 1985, 88970).

Kidney and liver samples were subsequently analysed for COOH-cyfluthrin as well as combined residues of FPBald, FPBacid and FPBalc (all oxidized in the method to FPBacid). Residues of COOH-cyfluthrin were < 0.01 mg/kg in all samples while residues of combined residues of FPBald, FPBacid and FPBalc (expressed in cyfluthrin equivalents) were 0.02, 0.02 and 0.03 mg/kg in liver of the 40 ppm dose group, < 0.01, < 0.01 and 0.01 mg/kg in kidney of the 13 ppm group and < 0.01, 0.02 and 0.05 mg/kg in kidney of the 40 ppm dose group.

The lactating cow feeding study does not agree with the lactating cow metabolism where after five days of dosing at the equivalent of 12 – 17 ppm, residues identified in kidney were 0.1 mg/kg for cyfluthrin and 0.08 mg/kg for combined residues of FPBald, FPBacid and FPBalc (expressed in cyfluthrin equivalents). Residues identified in liver from the lactating cow metabolism study were 0.53 mg/kg for cyfluthrin and 0.09 mg/kg for combined residues of FPBald, FPBacid and FPBalc (expressed in cyfluthrin equivalents). The metabolism study is in broad agreement with the results observed for the related pyrethroids, permethrin and cypermethrin.

In a separate study Holstein Friesian dairy cows (*Bos Taurus*; body weights of 300 to 510 kg, milk yield 5.3 – 19.5 kg/d) were administered target doses equivalent of 15, 50, and 150 ppm cyfluthrin in the dry diet *via* capsule orally once per day after the morning milking (Lemke 1994 106628). The animals received water, hay, and salt *ad libitum* throughout the study. Mixed grain was also given to the cattle during milking.

The dose rates were calculated assuming a feed consumption for a dairy cow of 3% of body weight, however intakes for animals of similar bodyweight and milk production are reported to be 3.75% of body weight and when corrected for actual body weights and content in the capsule doses the estimated equivalent levels in feed are 11, 36 and 112 ppm. Analysis of the capsules showed the actual doses were 95, 92, and 102% of dose levels.

For each cow, the evening milk aliquot was mixed with the next morning's milk aliquot to create a days milk sample. At sacrifice, composite fat (omental, renal, and subcutaneous), composite muscle (round, flank, and loin), liver, and kidney tissues were collected and reserved for residue analysis. All milk samples were analysed between 10 and 64 days after collection Tissue samples were analysed within 36, 21, 19, and 21 days of collection for fat, muscle, liver, and kidney. Cyfluthrin was removed from the sample matrix, by organic solvent extraction, acetone/chloroform (2:1) for all tissues except fat or hexane for the fat. The organosoluble extract was partitioned with various solvents to remove lipids and polar and non-polar interferences. The final purification step was column chromatography of the sample on either a silica gel column or a Florisil Sep-Pak. The purified sample was subjected to GC-ECD for determination of cyfluthrin.

Cyfluthrin residue in milk from the 11 ppm treatment group ranged from 0.05 to 0.08 mg/kg, from the 36 ppm treatment group 0.12 to 0.24 mg/kg and for the 112 ppm group 0.45 to 0.70 mg/kg. Residue levels in the milk peaked within 14 to 21 days and were declining by the end of the study. The milk residue showed a linear relationship to the treatment rates used in the study.

Cyfluthrin residue in tissues from the 11 ppm nominal treatment group were < 0.01 mg/kg in all tissues except fat, which showed a value of 1.2 mg/kg. The 36 ppm treatment group showed average residue values of 2.7, 0.04, < 0.01, and 0.03 mg/kg in fat, muscle, liver, and kidney, respectively. The 112 ppm treatment group showed average residue values of 6.8, 0.07, 0.02, and 0.05 mg/kg in fat, muscle, liver, and kidney, respectively.

Table 96. Cyfluthrin residues in milk from cows dosed daily for 28 days

	Residues of Cyfluthrin (mg/kg)			
Dose Level (mg/kg)	Day 7	Day 14	Day 21	Day 28
11	0.07	0.07	0.04	0.06
	0.08	0.1	0.07	0.06
	0.07	0.06	0.05	0.06
Avg.	0.07	0.08	0.05	0.06
36	0.21	0.24	0.22	0.13
	0.26	0.27	0.2	0.16
	0.2	0.2	0.16	0.08
Avg.	0.22	0.24	0.19	0.12
112	0.49	0.56	0.5	0.44
	0.68	0.89	0.96	0.49
	0.5	0.41	0.65	0.43
Avg.	0.56	0.62	0.7	0.45

Control cow, day 14 milk had residues of 0.02 mg/kg.

Table 97. Cyfluthrin residues in tissues from cows dosed daily for 28 days

	Residues of Cyfluthrin (mg/kg)			
Dose Level (ppm)	Fat	Muscle	Liver	Kidney
11	1.2	0.01	< 0.01	0.01
	1.4	< 0.01	< 0.01	< 0.01
	0.98	< 0.01	< 0.01	< 0.01
avg.	1.2	< 0.01	< 0.01	< 0.01
36	3.3	0.07	< 0.01	0.07
	2.2	0.02	< 0.01	0.02
	2.6	0.03	< 0.01	< 0.01
avg.	2.7	0.04	< 0.01	0.03
112	6.5	0.05	0.01	0.05
	4.0	0.04	0.03	0.02
	9.9	0.11	< 0.01	0.07
avg.	6.8	0.07	0.02	0.05

Control cows had residues in fat of 0.08 and 0.09 mg/kg.

The average cyfluthrin residue in milk was ≤ 0.08 mg/kg at the 11 ppm nominal feeding level, < 0.24 mg/kg at the 36 ppm nominal feeding level, and ≤ 0.70 mg/kg at the 112 ppm nominal feeding level. The average cyfluthrin residue in tissues at the 11, 36, and 112 ppm nominal feed levels were

1.2, 2.7 and 6.8 mg/kg for fat, < 0.01, 0.04 and 0.07 mg/kg for muscle, < 0.01, < 0.01 and 0.02 mg/kg for liver and < 0.01, 0.03, and 0.05 mg/kg, respectively, in kidney.

In a separate report, Minor and Gronberg (1985 90288) reported combined residues of FPBald, FPBalc and FPBacid in liver and kidney of lactating cows from an additional feeding study where cows were dosed at the equivalent of 150 ppm in the diet. No details of the in-life phase of the trial were available except that the study duration was 28 days (dosing) with the dose equivalent to 150 ppm fed daily after milking in the feed. The analysis was carried out using Mobay method 86217 where FPBald, FPBalc were converted to FPBacid and measured as Me-FPBacid (gross residues were expressed as cyfluthrin equivalents). Samples were stored for approximately 190 days prior to analysis.

Table 98. Metabolite residues in liver and kidney of cows dosed daily for 28 days

Dose Level	Liver		Kidney	
(ppm)	Metabolites oxidised to FPBacid (mg/kg)	Metabolites oxidised to FPBacid (mg/kg cyfluthrin equivalents)	Metabolites oxidised to FPBacid (mg/kg)	Metabolites oxidised to FPBacid (mg/kg cyfluthrin equivalents)
150	0.07 0.1 0.18	0.14 0.19 0.35	0.1 0.1 0.22	0.2 0.19 0.41
Average	0.12	0.23	0.14	0.27

Similar levels of residues of the combined residues of FPBald, FPBalc and FPBacid were found in both the liver and the kidney of cattle dosed at 150 ppm for 28 days. When expressed as cyfluthrin equivalents the residues are 0.23 mg/kg in the liver and 0.27 mg/kg in the kidney. The combined residues of FPBald, FPBalc and FPBacid in liver and kidney are consistent with the lactating cow metabolism study.

Poultry

In a feeding study laying hens (White Leghorn) were fed a diet (Purina Layena poultry chow) containing 2, 5.6 or 20 ppm cyfluthrin for 28 days (Chopade and Gentile 1983 MR86046). At slaughter samples of liver, muscle (composite of breast, leg and thigh), heart, gizzard (minus lining and contents), fat, kidney, and skin were collected for analysis. Samples were stored frozen for intervals of up to 6 weeks prior to analysis.

For the tissues cyfluthrin was removed from the sample matrix, by organic solvent extraction, acetone/chloroform (2:1) for all tissues except fat and skin for which hexane was used. The organosoluble extract was partitioned with various solvents to remove lipids and polar and non-polar interferences. The final purification step was column chromatography of the sample on either a silica gel column or a Florisil Sep-Pak. The purified sample was subjected to GC-ECD for determination of cyfluthrin.

The average values obtained for cyfluthrin levels in fortified feed were 2, 5.6 and 20 ppm for the 2, 5.6, and 20 ppm batches. The analyses indicated that cyfluthrin was thoroughly mixed with the feed. Egg production appeared to decline in all groups during the study although the average weight of an individual egg remained the same when compared to the control group. The decline in egg production may have been influenced by the relatively high temperatures of 29 to 33 °C inside the experiment room for the last three weeks of the study when the outside temperatures were near 38 °C.

Individual body weights were recorded at the beginning and the end of the 28 day feeding period. The results of the body weight analysis indicated that laying hens in all the groups including the control group had lost 3 to 11% of the pre-treatment weight. As all groups lost body weight during the study, the relatively high temperature inside the poultry room during the last three weeks of the study may have been influential.

The cyfluthrin residue in the 5.6 and 20 ppm treatment egg samples were < 0.01 mg/kg. As the residue was < 0.01 mg/kg in the highest (20 ppm) treatment group, egg samples from the 2 ppm treatment group were not analysed.

The residue level of cyfluthrin averaged 0.05 mg/kg for fat and < 0.01 mg/kg for other tissues from the chickens fed at the 20 ppm level. For the 5.6 ppm treatment group, the cyfluthrin residue averaged < 0.01 mg/kg for all the tissues including fat.

Table 99. Cyfluthrin residues in tissues from chickens fed treated feed for 28 days

Dose Level (ppm)	Residues of Cyfluthrin (mg/kg)				
	Gizzards [a]	Skin	Muscle	Fat	Liver
5.6	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01
20	< 0.01 < 0.01	0.01 0.01	< 0.01 < 0.01	0.05 0.05	< 0.01 < 0.01

a - Minus lining and contents, and external fat

Samples of liver, gizzard, muscle and skin were analysed for the cyfluthrin metabolites COOH-cyfluthrin, FPBalc, FPBald and FPBacid. No residues of COOH-cyfluthrin were detected. FPBalc, FPBald and FPBacid were converted to FPBacid prior to analysis. No residues were detected in gizzard, skin, muscle or fat at any of the feed levels. Levels of combined residues of FPBacid and metabolites oxidised to FPBacid were 0.02 mg/kg in liver of the 5.6 and 20 ppm feed groups.

APPRAISAL – RESIDUE AND ANALYTICAL ASPECTS

Cyfluthrin was identified as a priority compound under the Periodic Re-evaluation Programme at the 37th Session of the CCPR. The Meeting received information on cyfluthrin metabolism and environmental fate, methods of residue analysis, freezer storage stability, national registered use patterns, supervised residue trials, farm animal feeding studies, fate of residues in processing and national MRLs. The Meeting also received information on beta-cyfluthrin methods of residue analysis, freezer storage stability, national registered use patterns and supervised residue trials. The metabolism and environmental fate, transfer from feeds to farm animals and fate of residues in processing provided for cyfluthrin are used to support both pesticides.

Cyfluthrin was evaluated by the 48th JECFA for residues in animal commodities arising from direct animal treatment. In the case of animal commodities, the maximum residue limit recommendations of the 48th JECFA for cattle are fat 0.2 mg/kg, muscle, liver and kidney 0.02 mg/kg and milk 0.04 mg/kg. The residue definition (marker residue) chosen by JECFA was cyfluthrin.

The 2006 JMPR established common ADIs and ARfDs for beta-cyfluthrin and cyfluthrin of 0-0.04 mg/kg bw per day and 0.04 mg/kg bw respectively.

Cyfluthrin is a mixture of 8 stereoisomeric esters derived from esterification of the dichloro analogue of chrysanthemic acid, (2,2-dimethyl-3-(2,2-dichlorovinyl)cyclopropane carboxylic acid, DCVA) with α -cyano-3-phenoxy-4-fluorobenzyl alcohol. Beta-cyfluthrin is an enriched isomeric form of the 2 biologically active diastereoisomeric pairs of isomers.

Conclusions reached in discussing cyfluthrin equally apply to beta-cyfluthrin. In the presence of water and other protic solvents, the isomer composition of beta-cyfluthrin changes through epimerisation such that with sufficient time the isomer ratio becomes the same as cyfluthrin.

The following abbreviations are used for the metabolites discussed below:

DCVA	2,2-dimethyl-3-(2,2-dichlorovinyl)cyclopropane carboxylic acid
FPBald	4-fluoro-3-phenoxybenzaldehyde
FPBacid	4-fluoro-3-phenoxybenzoic acid
FPBalc	3-phenoxy-4-fluorobenzyl alcohol
OH-FPBacid	3-(4'-hydroxyphenoxy)-4-fluorobenzoic acid

Me-FPBacid methyl 4-fluoro-3-phenoxybenzoate
FPBamide 4-fluoro-3-phenoxybenzamide
FPB 1-fluoro-2-phenoxybenzene
COOH-cyfluthrin α-[[[3-(2,2-dichloroethenyl)-2,2-dimethylcyclopropyl]carbonyl]oxy]-4-fluoro-3-phenoxybenzeneacetic acid

Animal metabolism

Two radiolabelled cyfluthrin preparations separately [^{14}C] labelled at the phenyl-UL-^{14}C- and fluorophenyl-UL positions, were used in the metabolism and environmental studies. The metabolism of laboratory animals was qualitatively the same as for farm animals. The proposed major route of cyfluthrin metabolism in livestock is via hydrolysis of the ester linkage. Hydrolysis gives initially DCVA and the unstable cyanohydrin which rapidly breaks down to the aldehyde (FPBald) and is oxidized to the corresponding acid/or hydroxylated acids (FPBacid, OH-FPBacid). A very minor route was observed in which a small amount of FPBald was converted to its corresponding alcohol, FPBalc.

Lactating cows were orally dosed with [phenyl-UL-^{14}C]-cyfluthrin at 0.5 mg/kg bw for 5 consecutive days. Cyfluthrin was the major identifiable product in milk (98% TRR, 0.039 – 0.079 mg/kg). The radiocarbon content of tissues, reported in cyfluthrin equivalents, was highest in liver (0.62 mg/kg), kidney (0.19 mg/kg), and fat (0.12 – 0.23 mg/kg) with low levels (< 0.05 mg/kg) present in other tissues. Cyfluthrin was the main component of the [^{14}C] in liver and kidney (56 – 86%) and in muscle and fat (93 – 100%). Hydrolysis products, formed from hydrolysis of the ester and oxidation, were only detected in liver (14% FPBald), kidney (43% FPBalc) and heart (29%, FPBalc).

Laying hens were orally dosed with [phenyl-UL-^{14}C]-cyfluthrin for 3 consecutive days at 5 mg/kg bw/hen/day. Radioactive residues in eggs collected in the 24 hour period prior to slaughter were 0.05 mg/kg cyfluthrin equivalents. Radioactive residues in tissues of birds slaughtered at 2 h after the last dose were highest in kidney (4.7 mg/kg cyfluthrin equivalents) and liver (3.0 mg/kg) with low levels observed in other tissues. Residues of [^{14}C] in fat were 0.1 – 0.2 mg/kg while those in muscle were 0.2 – 0.3 mg/kg, both expressed in cyfluthrin equivalents. The major radiolabelled fraction identified in eggs (56%), fat (75%) and muscle (21 – 39%) was cyfluthrin. Significant metabolites were FPBacid (12% liver, 11% kidney, 15 – 21% muscle) and OH-FPBacid (10% liver, 12% kidney, 11 – 20% muscle).

Plant metabolism

The Meeting received information on the fate of [phenyl-UL-^{14}C]cyfluthrin after foliar application to on apple, cotton, potato, soya bean and wheat and of [cyclopropyl-1-^{14}C]cyfluthrin on wheat. Studies were also available on the fate of [fluorophenyl-UL-^{14}C]cyfluthrin on tomatoes and stored wheat grain.

The metabolism of [^{14}C]cyfluthrin was studied in apples. The majority of the [^{14}C] was associated with the fruit surface (rinses and peel) with 4% or less associated with pulp. Cyfluthrin was the major component of the radioactivity detected at 0 to 28 days after application accounting for 91 – 84% of the [^{14}C] in rinse solutions and peel. Several minor components, principally FPBald and FPBacid, were present at ≤ 2% of the [^{14}C].

Unchanged cyfluthrin accounted for > 90% of the TRR present tomato fruit and leaves from 1 to 35 days after application to both fruit and leaves.

Potato plants were treated in a greenhouse study with [phenyl-UL-^{14}C]cyfluthrin as a foliar treatment. There was limited translocation of [^{14}C] to potato tubers. Residues in leaves sampled at 0 – 98 days after application mostly comprised unchanged cyfluthrin (70 – 95%) together with a number of free and conjugated metabolites (FPBald, FPBacid, OH-FPBacid, FPB) each present at < 5%TRR.

Cyfluthrin was also the major component of [^{14}C] residues following application of [phenyl-UL-^{14}C]cyfluthrin to soya bean plants raised in a greenhouse (43 – 92% TRR at 4 – 88 days after

application). Individual metabolites identified in whole plants, leaves and stalks were present in both free and conjugated forms and were all individually < 10% TRR (FPBald, OH-FPBacid, FPBacid, FPBalc, Me-FPBacid, FPBamide, FPB). There was limited translocation of [^{14}C] to pods. Incubation of [phenyl-UL-^{14}C]cyfluthrin with soya bean tissue cultures produced a similar range of metabolites.

Cotton plants were treated with [phenyl-UL-^{14}C]cyfluthrin as a foliar treatment and exposed to natural sunlight or grown in a greenhouse. Cyfluthrin was the major component of the [^{14}C] accounting for 61 – 99% of the TRR at 0 – 63 days after application. All metabolites identified were present at levels ≤ 10% TRR in free and conjugated forms (FPBald, FPBalc, FPBacid, Me-FPBacid, OH-FPBacid). Little translocation was observed when individual leaves or bolls were treated. Degradation of cyfluthrin was greater for plants exposed to field conditions than those grown in the glasshouse.

The metabolism of [phenyl-UL-^{14}C]cyfluthrin was also studied in wheat. In forage, straw and heads at up to 21 days after application, 51 – 69% of [^{14}C] residues were identified as cyfluthrin. Minor metabolites were present at ≤ 5% TRR and occurred in both free and conjugated forms (COOH-cyfluthrin, FPBacid, OH-FPBacid). In a study where [cyclopropyl-1-^{14}C]cyfluthrin and [phenyl-UL-^{14}C]cyfluthrin were applied to wheat plants as seven applications with harvest one day after the last application, cyfluthrin was the major component of [^{14}C] in both heads and straw (77 – 86%). Metabolites were individually < 10% of TRR as would be expected with application of the last spray so close to harvest. Metabolites identified were DCVA, COOH-cyfluthrin, FPBald, FPBacid, FPBalc and OH-FPBacid.

In a study of the degradation of cyfluthrin on stored wheat grain treated with [fluorophenyl-UL-^{14}C]cyfluthrin at 0.3 – 0.8 mg/kg, the majority of the radioactivity was located on the grain surface and released into rinse solutions. Unchanged cyfluthrin was the major component identified. After 9 months of storage, 79% of the TRR was cyfluthrin with a further 1.9% identified as FPBald and 0.8% as FPBalc.

Metabolism studies in apples, tomato, cotton, potatoes, soya beans and wheat demonstrated that cyfluthrin was slowly degraded and that the degradation pattern was similar in all crops. The major identified products of cyfluthrin metabolism in plants are analogous to those in mammals. The proposed degradation pathway consists of epimerisation, hydrolysis, ester cleavage, reduction, oxidation and hydroxylation. Cyfluthrin is not systemic, with only limited translocation in plants.

Environmental fate in soil

The half-life for degradation of cyfluthrin in soil is estimated to be < 6 months. Degradation occurred via ester hydrolysis followed by oxidation and mineralisation to [^{14}C]O_2.

Photodegradation on soil surfaces is fast with half-lives for degradation of cyfluthrin that are < 20 days. During irradiation with artificial or natural sunlight cyfluthrin in soils underwent ester hydrolysis with FPBald, FPBacid and DCVA identified as degradates.

Hydrolysis in water is pH dependent. Cyfluthrin is considered stable at pH 4 and 7 but is rapidly hydrolysed at pH 9 with a half-life of < 2 days. Two degradation products were identified, FPBald and traces of DCVA, presumably formed from cyfluthrin on hydrolysis of the ester. Abiotic hydrolysis is unlikely to contribute significantly to the degradation of cyfluthrin residues in aquatic systems unless the pH is high.

In confined and field rotational crop studies, no significant residues of cyfluthrin (< 0.01 mg/kg) were found in any crop material. It is concluded that succeeding or rotational crops are unlikely to contain significant residues of cyfluthrin.

Analytical methods

Several different analytical methods have been reported for the analysis of cyfluthrin (and isomers) in plant material and animal commodities. The basic approach involves extraction by homogenisation with an organic solvent mixture incorporating varying proportions of polar and non-polar solvents depending upon the nature of the matrix being extracted and its water content. In general, a primary

liquid – liquid partition follows extraction to transfer cyfluthrin residues to less polar solvents prior to column clean-up. Residues are finally determined by gas chromatography with electron capture or mass spectra detectors. In a small number of the methods the four pairs of diastereoisomeric enantiomers that make up cyfluthrin were resolved.

The methods for cyfluthrin and beta-cyfluthrin have been extensively validated with numerous recoveries on a wide range of substrates with LOQs typically in the range 0.01 to 0.05 mg/kg.

Stability of pesticide residues in stored analytical samples

Freezer storage stability was tested for a range of representative substrates. Residues of cyfluthrin were generally stable in crops and their processed products.

Cyfluthrin was stable in homogenized samples fortified at 1 mg/kg and stored frozen for at least 1118 days for apple, 1145 days for cantaloupe, 1130 days for corn, 1145 days for corn oil, 1125 days for corn starch, 1145 days for cucumber, 739 days for oranges, 1145 days for orange juice, 739 days for orange pulp, 1145 days for peanut shells, 1126 days for potatoes, 1130 days for potato chips, 1126 days for potato granules, 95 days for potato peel (wet), 1146 days for potato peel (dry), 102 days for rice, 207 days for rice hulls, 1155 for sugar cane stalks, 1125 days for molasses, 1151 days for tomatoes, 1130 days for wheat and for wheat bran, 1118 days for wheat flour and 1126 days for wheat dust. Incurred cyfluthrin residues were stable in bovine muscle, fat, milk and kidney tissues for at least 6 months and liver for at least 1 month. Fortified liver samples were stable on freezer storage for at least 1 year.

Residue definition

The residue following use of cyfluthrin on crops is predominantly cyfluthrin. Methods are available that can measure cyfluthrin however they do not generally resolve the individual diastereoisomers.

The ratio of cyfluthrin to major metabolites differed in the lactating cow metabolism and feeding studies. In the feeding study, cyfluthrin is the major component of the residue in edible animal commodities, tissues, milk and eggs. Major metabolites derived from hydrolysis of the ester are DCVA and FPBald, FPBacid and FPBalc. None of these metabolites are unique to cyfluthrin. DCVA is a metabolite common to permethrin, cypermethrin and cyfluthrin while metabolites derived from FPBald are common to cyfluthrin and flumethrin. Separate methods are required for measuring the metabolites. The metabolites were not identified in the evaluation of the toxicological data for cyfluthrin and cypermethrin by the 2006 JMPR as being of toxicological concern.

No metabolism studies were available that specifically used beta-cyfluthrin however the data for cyfluthrin can be used to support beta-cyfluthrin. The residue following its use on crops is predominantly cyfluthrin. Methods are available that can measure both cyfluthrin and beta-cyfluthrin however they do not generally resolve the individual diastereoisomers. Epimerisation of beta-cyfluthrin leads to a change in isomer composition.

Based on the actual residue measured, the Meeting recommended that the residue definition for plant and animal commodities for compliance with MRLs and for estimation of dietary intake should be cyfluthrin. The log K_{ow} of cyfluthrin (pH 6) and the animal metabolism and feeding studies suggest that cyfluthrin should be described as fat-soluble. In the lactating cow metabolism study cyfluthrin residues were approximately 10 times greater in fat than muscle.

Definition of the residue (for compliance with MRL and for estimation of dietary intake) for plant and animal commodities: cyfluthrin (sum of isomers).

The residue is fat-soluble.

Results of supervised residue trials on crops

Supervised trials were available for the use of cyfluthrin on numerous crops: apples, pears, Brassica vegetables (broccoli, Brussels sprouts, cabbage, cauliflower and Chinese cabbage), cotton, oranges,

grapefruit, lemons, peppers, potatoes, rape, soya beans, sunflower, sweet corn, mangoes and tomatoes. Specific supervised trials based on unvalidated analytical data (from Craven Laboratories) could not be considered further for sweet corn and soy beans.

Supervised trials were also available for the use of beta-cyfluthrin on several crops: mango, cabbage, cotton, soya beans and rape.

Trial data or relevant GAP was not submitted for maize for which there is a current recommendation for a maximum residue level. The Meeting agreed to withdraw its previous maximum residue level recommendation of 0.05 mg/kg for maize.

Citrus (cyfluthrin)

Cyfluthrin is registered in the USA for use on citrus fruits at 28 – 112 g ai/ha, PHI 0 days with a maximum seasonal application of 112 g ai/ha and no more than 112 g ai/ha to be applied in a seven day period. Trials conducted in the USA that approximated GAP were often conducted such that more than one plot was treated per trial location. Often treatments involved high and low spray volumes and in some cases different formulations (EC and WP). The Meeting decided that for the purposes of estimation of maximum residue levels that only one result per trial location be used. Seven trials from the USA on grapefruit were selected as complying with US GAP. Residues in whole fruit (n=7) were 0.02, 0.02, 0.03, 0.04, 0.04, 0.07 and 0.11 mg/kg. Residues in oranges from US trials conducted according to GAP (n=7) were 0.03, 0.05, 0.05, 0.05, 0.06, 0.06 and 0.2 mg/kg. Residues in lemons from US trials conducted according to GAP (n=5) were 0.08, 0.08, 0.10, 0.10 and 0.11 mg/kg.

The Meeting decided to combine the trials in the various citrus fruit for the purposes of estimating a maximum residue level and STMR. Residues in rank order are: 0.02, 0.02, 0.03, 0.03, 0.04, 0.04, 0.05, 0.05, 0.05, 0.06, 0.06, 0.07, 0.08, 0.08, 0.10, 0.10, 0.11, 0.11 and 0.2 mg/kg.

The Meeting estimated a maximum residue level, an STMR value and an HR value for cyfluthrin in citrus whole fruit of 0.3, 0.06 and 0.2 mg/kg respectively.

Apples and pears (cyfluthrin)

Data were available from supervised trials on apples in the USA (GAP: 25 – 49 g ai/ha, PHI 7 days with a maximum seasonal application of 49 g ai/ha and no more than 49 g ai/ha to be applied in a fourteen day period). Residues of cyfluthrin from twelve trials in USA at 49 g ai/ha with a PHI of 7 days were 0.01, 0.01, 0.02, 0.02, 0.02, 0.02, 0.02, 0.03, 0.03, 0.03, 0.04 and 0.06 mg/kg.

Data were available from supervised trials on pears in the USA (GAP: 25 – 49 g ai/ha, PHI 7 days with a maximum seasonal application of 49 g ai/ha and no more than 49 g ai/ha to be applied in a fourteen day period). Residues of cyfluthrin from six trials in USA at 49 g ai/ha with a PHI of 7 days were 0.02, 0.02, 0.02, 0.02, 0.04 and 0.05 mg/kg.

The Meeting noted that the use patterns for apple and pears in the USA were the same and that the residues populations for each crop could be used to support the other. Therefore the Meeting decided to combine the data for apples and pears to increase the database for the purposes of estimating a maximum residue level, STMR and HR but to make separate recommendations as a general pome fruit use pattern does not exist in the USA.

Residues in rank order (n=18), median underlined, were: 0.01, 0.01, 0.02, 0.02, 0.02, 0.02, 0.02, 0.02, 0.02, 0.02, 0.02, 0.02, 0.03, 0.03, 0.03, 0.04, 0.04, 0.05 and 0.06 mg/kg.

The Meeting estimated maximum residue levels, STMR values and HR values for cyfluthrin in apples and pears of 0.1, 0.02 and 0.06 mg/kg respectively. The Meeting agreed to withdraw its previous recommendation of 0.5 mg/kg for apples.

Mangoes - cyfluthrin

Results from three supervised trials on mangoes conducted in the Philippines were made available to the Meeting. One trial was conducted using cyfluthrin and two with beta-cyfluthrin.

One cyfluthrin trial matched GAP for the Philippines (2.5 g ai/hL, PHI 14 days) with residues of 0.02 mg/kg in whole fruit. The Meeting considered a single trial insufficient to estimate a maximum residue level for cyfluthrin in mangoes.

Mangoes (beta-cyfluthrin)

Results from two supervised trials on mangoes conducted in the Philippines were made available to the Meeting. One beta-cyfluthrin residue trial matched GAP of the Philippines (10 g ai/hL, PHI 28 days) with residues in fruit of < 0.01 mg/kg. The Meeting considered one trial insufficient to estimate a maximum residue level.

Brassica vegetables (cyfluthrin)

Cyfluthrin is registered in the USA for use on Brassica vegetables at 15 – 56 g ai/ha, PHI of 0 days and a maximum application per season of 224 g ai/ha and a maximum of 56 g ai/ha in a 7 day period.

Trials were available from USA of Brussels sprouts approximating GAP with residues of 0.39 and 0.44 mg/kg. The Meeting considered two trials are not sufficient to recommend a maximum residue level.

Thirteen trials approximating GAP were available for broccoli: 0.04, 0.05, 0.19, 0.19, 0.19, 0.19, 0.20, 0.26, 0.28, 0.29, 0.30, 0.46 and 1.5 mg/kg. The Meeting estimated a maximum residue level of 2 mg/kg, STMP of 0.20 mg/kg and an HR of 1.5 mg/kg for residues of cyfluthrin in broccoli.

Six trials on cauliflower that matched GAP of the USA were: < 0.01, 0.11, 0.17, 0.31, 0.32 and 0.91 mg/kg. The Meeting estimated a maximum residue level of 2 mg/kg, STMR of 0.24 mg/kg and HR of 0.91 mg/kg for cauliflower.

In eighteen trials on cabbage from the USA that matched GAP residues were: 0.01, 0.03, 0.03, 0.06, 0.07, 0.10, 0.10, 0.18, 0.24, 0.25, 0.33, 0.42, 0.58, 0.62, 1.0, 1.2, 1.3 and 2.1 mg/kg for cabbage. The Meeting estimated a maximum residue level, an STMR value and an HR value for cyfluthrin in cabbages of 4, 0.25 and 2.1 mg/kg respectively.

Cabbage (beta-cyfluthrin)

Results from four supervised trials on cabbage conducted in Germany (no GAP) were made available to the Meeting. The Meeting decided to evaluate the German trials against the GAP of Sweden (10 g ai/ha, PHI 7 days). Four trials matched the GAP of Sweden with beta-cyfluthrin residues of < 0.01, < 0.01, 0.06 and 0.08 mg/kg.

Tomatoes (cyfluthrin)

Trials on tomatoes were reported from the USA (GAP: 28 – 49 g ai/ha, PHI of 0 days and a maximum application per season of 295 g ai/ha and a maximum of 49 g ai/ha in a 7 day period). All trials were for field grown tomatoes with no data for tomatoes grown under protective cover.

Cyfluthrin residues in eleven trials from the USA matching GAP in rank order were (median underlined): < 0.01, 0.01, 0.02, 0.06, 0.07, 0.07, 0.07, 0.08, 0.08, 0.09 and 0.10 mg/kg.

The Meeting estimated a maximum residue level, an STMR value and an HR value for cyfluthrin in tomatoes of 0.2, 0.07 and 0.10 mg/kg respectively. The recommendation replaces the previous recommendation of 0.5 mg/kg for tomatoes.

Peppers (cyfluthrin)

Trials on peppers were reported from the USA (GAP: 28 – 49 g ai/ha, PHI of 7 days and a maximum application per season of 295 g ai/ha and a maximum of 49 g ai/ha in a 7 day period). All trials were for field grown peppers (including chilli) with no data for peppers grown under protective cover.

The Meeting agreed to combine the three trials on chilli peppers (0.06, 0.08, 0.08 mg/kg) with the six trials on sweet peppers (0.01, 0.01, 0.05, 0.06, 0.12 and 0.12 mg/kg) matching GAP in the

USA. Residues matching GAP in rank order were (median underlined): 0.01, 0.01, 0.05, 0.06, 0.06, 0.08, 0.08, 0.12 and 0.12 mg/kg.

The Meeting estimated a maximum residue level, an STMR value and an HR value for cyfluthrin in peppers of 0.2, 0.06 and 0.12 mg/kg respectively. The recommendation for peppers replaces the previous recommendation of 0.2 mg/kg for peppers sweet.

Egg plant (cyfluthrin)

The Meeting noted that the registered use of cyfluthrin in the USA also includes egg plant (GAP: 28 – 49 g ai/ha, PHI of 7 days and a maximum application per season of 295 g ai/ha and a maximum of 49 g ai/ha in a 7 day period). The meeting considered the results from the trials conducted on peppers and tomatoes that comply with GAP for egg plants could be extrapolated to egg plants for the purposes of estimating maximum residue, STMR and HR levels. Residues on tomatoes that matched GAP for egg plants were < 0.01, 0.01, 0.02, 0.02, 0.03, 0.03, 0.04, 0.04, 0.04, 0.05, 0.05, 0.05, 0.05, 0.05, 0.06, 0.08 and 0.09 mg/kg. Residues on peppers that matched GAP for egg plants were 0.01, 0.01, 0.05, 0.06, 0.06, 0.08, 0.08, 0.12 and 0.12 mg/kg. The Meeting estimated a maximum residue level, an STMR value and an HR value for cyfluthrin in egg plant of 0.2, 0.05 and 0.12 mg/kg respectively.

Sweet corn (cyfluthrin)

Trials on sweet corn were reported from the USA (GAP: 15 – 49 g ai/ha, PHI of 0 days and a maximum application per season of 493 g ai/ha and a maximum of 49 g ai/ha in a 2 day period).

Cyfluthrin residues in three trials from the USA matching GAP in rank order were (median underlined): < 0.01 (2) and 0.01 mg/kg.

The Meeting considered three trials insufficient to estimate a maximum residue level for cyfluthrin in sweet corn.

Potatoes (cyfluthrin)

Trials on potatoes were reported from Canada (no GAP) and the USA (GAP: 15 – 49 g ai/ha, PHI of 0 days and a maximum application per season of 295 g ai/ha and a maximum of 49 g ai/ha in a 7 day period).

Cyfluthrin residues in seventeen trials from the USA matching GAP in rank order were (median underlined): < 0.01 (17) mg/kg. Residues were not detected in residue trials and metabolism results on plants including potatoes confirm that cyfluthrin is not translocated by plants. The Meeting considered detectable residues in potato tubers to be unlikely.

The Meeting estimated a maximum residue level, an STMR value and an HR value for cyfluthrin in potatoes of 0.01*, 0 and 0 mg/kg respectively.

Soya beans (cyfluthrin)

Trials on soya beans were reported from the USA (GAP: 15 – 49 g ai/ha, PHI of 45 days and a maximum application per season of 196 g ai/ha and a maximum of 49 g ai/ha in a 7 day period).

Cyfluthrin residues in five trials from the USA matching GAP in rank order were (median underlined): < 0.01 (5) mg/kg. In addition, residues ranging from < 0.01 to 0.02 mg/kg were reported in unvalidated trials.

The Meeting considered five trials insufficient to estimate a maximum residue level for cyfluthrin in soya beans (dry).

Soya beans (beta-cyfluthrin)

Four trials on soya beans employing beta-cyfluthrin were reported from Brazil (12.5 g ai/ha, PHI 21 days) that complied with GAP for Brazil. Residues were < 0.01 and < 0.05 (3) mg/kg. The Meeting

considered four trials on soya beans insufficient to estimate a maximum residue level for residues arising from the use of beta-cyfluthrin in soya beans.

Cotton seed (cyfluthrin)

Trials on cotton were reported from the USA (GAP: 15 – 49 g ai/ha, PHI of 0 days and a maximum application per season of 560 g ai/ha and a maximum of 56 g ai/ha in a 3 day period).

Cyfluthrin residues in seven trials from the USA matching GAP in rank order were (median underlined): < 0.01, 0.02, 0.03, < 0.1, < 0.1, 0.1 and 0.52 mg/kg.

The Meeting estimated a maximum residue level and an STMR value for cyfluthrin in cotton seed of 0.7 and 0.1 mg/kg respectively. The recommendation for cotton seed replaces the previous recommendation of 0.05 mg/kg.

Cotton seed (beta-cyfluthrin)

Beta-cyfluthrin trials on cotton were reported from the USA (GAP: 7 – 28 g ai/ha, PHI of 0 days and a maximum application per season of 280 g ai/ha and a maximum of 28 g ai/ha in a 3 day period). Beta-cyfluthrin residues in three trials from the USA matching GAP in rank order were: < 0.1, < 0.1 and 0.38 mg/kg.

Rape seed (cyfluthrin)

Cyfluthrin trials on rape were reported from Germany (no GAP). The Meeting decided to assess the German trials against the GAP of Belgium (15 g ai/ha, application according to growth stage, maximum 2 applications per crop, one spray from seed to 3 leaf BBCH 10 – 13, one at bud development BBCH 50 – 59 and one at pod development BBCH 70-75). Seven trials matched GAP of Belgium with residues of < 0.05 (6) and 0.05 mg/kg.

The Meeting estimated a maximum residue level and an STMR value for cyfluthrin in rape seed of 0.07, 0.05 and < 0.05 mg/kg respectively. The recommendation for rape seed replaces the previous recommendation of 0.05 mg/kg.

Rape seed (beta-cyfluthrin)

Trials conducted on rape using beta-cyfluthrin trials were reported from the Germany (GAP: 5.2 – 7.7 g ai/ha, 0.2-0.3 g ai/hL, PHI of 56 days). Beta-cyfluthrin residues in nine trials from the Germany matching GAP in rank order were (median underlined): < 0.01, < 0.01, 0.01, < 0.02 (4), < 0.05, and < 0.05 mg/kg.

Sunflower seed (cyfluthrin)

Trials on sunflower were reported from Canada (no GAP) and the USA (GAP: 15 – 49 g ai/ha, PHI of 30 days and a maximum application per season of 147 g ai/ha and a maximum of 49 g ai/ha in a 7 day period).

Cyfluthrin residues in five trials from Canada and the USA matching GAP of the USA in rank order were (median underlined): < 0.01 (3) and 0.01 (2) mg/kg.

The Meeting considered five trials insufficient to estimate a maximum residue level for cyfluthrin in sunflower seed.

Animal feedstuffs

Sweet corn forage (cyfluthrin)

Field trials on sweet corn were made available to the Meeting from the USA (GAP: 15 – 49 g ai/ha, PHI of 0 days and a maximum application per season of 493 g ai/ha and a maximum of 49 g ai/ha in a 2 day period).

Residues on sweet corn forage were 3.7, 3.7 and 7.7 mg/kg (fresh weight basis).

Residues on sweet corn cannery waste were 0.20, 0.43 and 0.90 mg/kg (fresh weight basis).

The Meeting considered three trials insufficient to estimate median and high residues for sweet corn livestock feeds.

Cotton gin-trash (cyfluthrin)

Cyfluthrin field trials on cotton were made available to the Meeting from the USA (GAP: 15 – 49 g ai/ha, PHI of 0 days and a maximum application per season of 560 g ai/ha and a maximum of 56 g ai/ha in a 3 day period; Do not graze treated fields).

Cyfluthrin residues on cotton gin-trash were 2.4, 2.8 and 9.2 mg/kg (fresh weight basis). The Meeting considered three trials insufficient to estimate median residues for cotton gin-trash as a livestock feed.

Cotton gin-trash (beta-cyfluthrin)

Beta-cyfluthrin field trials on cotton were made available to the Meeting from the USA (GAP: 7 – 28 g ai/ha, PHI of 0 days and a maximum application per season of 280 g ai/ha and a maximum of 28 g ai/ha in a 3 day period; Do not graze treated fields).

Beta-cyfluthrin residues on cotton gin-trash were 2.3, 2.6, 2.9 mg/kg (fresh weight basis). The Meeting considered three trials insufficient to estimate median residues for cotton gin-trash as a livestock feed.

Rape forage and straw (cyfluthrin)

Field trials on rape seed were made available to the Meeting from the Germany (GAP: 15 g ai/ha, application according to growth stage, maximum 2 applications per crop, one spray from seed to 3 leaf BBCH 10 – 13, one at bud development BBCH 50 – 59 and one at pod development BBCH 70 – 75).

Residues on rape straw were < 0.02, < 0.02, < 0.02, < 0.02, < 0.02 and 0.06 mg/kg. The Meeting estimated an STMR and a high residue value for cyfluthrin in rape straw of < 0.02 and 0.06 mg/kg, respectively, both on an as received basis.

As the registered use pattern for rape in Germany does not restrict grazing, the Meeting assumed that according to GAP in Germany rape could be grazed at the earliest time after application. Residues on rape forage were 0.13, 0.15, 0.18, 0.20, 0.21, 0.27, 0.32 and 0.34 mg/kg (fresh weight basis). The Meeting estimated an STMR and a high residue value for cyfluthrin in forage of 0.205 and 0.34 mg/kg, respectively, both on a fresh weight basis.

Rape fodder (beta-cyfluthrin)

For beta-cyfluthrin trials on rape were reported from Germany (GAP: 5.2 – 7.7 g ai/ha, 0.2 – 0.3 g ai/hL, PHI of 56 days). Beta-cyfluthrin residues from rape straw in seven trials from Germany matching GAP in rank order were (median underlined): < 0.05, < 0.05, < 0.05, 0.02, 0.06, 0.07, 0.08 mg/kg (fresh weight basis). As the directions for use in Germany do not provide specific guidance for livestock feeding it is assumed forage rape can be grazed without restriction anytime after application. Residues in rape forage at 0 days after application were: < 0.05, 0.08, 0.16, 0.17, 0.19, 0.24, 0.26, 0.27 and 0.33 mg/kg. The Meeting estimated an STMR and a high residue value for cyfluthrin in rape forage of 0.19 and 0.33 mg/kg, respectively, both on a fresh weight basis.

Soya bean forage and vines (cyfluthrin)

Field trials on soya beans were made available to the Meeting from the USA (GAP: 15 – 49 g ai/ha, PHI of 45 days and a maximum application per season of 196 g ai/ha and a maximum of 49 g ai/ha in a 7 day period; dry vines and green forage may be fed 45 and 15 days, respectively after last application).

Residues on soya bean forage were 0.10, 0.26, 0.33, 0.34, 0.38, 0.45, 0.96 and 3.3 mg/kg (fresh weight basis). The Meeting estimated an STMR and a high residue value for cyfluthrin in soya bean forage of 0.36 and 3.3 mg/kg, respectively, both on a fresh weight basis.

Residues on soya bean dry vines were 0.01, 0.09, 0.21, 0.31 and 2.66 mg/kg (fresh weight basis). The Meeting considered five trials insufficient to estimate median and high residues for soya vines that may be used as livestock feed.

Sunflower fodder (cyfluthrin)

Trials on sunflowers were reported from Canada (no GAP) and the USA (GAP: 15 – 49 g ai/ha, PHI of 30 days and a maximum application per season of 147 g ai/ha and a maximum of 49 g ai/ha in a 7 day period; pre-grazing or foraging interval, 30 days).

Cyfluthrin residues in sunflower fodder in five trials from Canada and the USA matching GAP of the USA were 0.04, 0.13, 0.30, 0.33 and 0.63 mg/kg (fresh weight basis). The Meeting estimated an STMR and a high residue value for cyfluthrin in sunflower fodder of 0.30 and 0.63 mg/kg, respectively, both on a fresh weight basis.

Fate of residues during processing

The fate of cyfluthrin residues has been examined in potato, cabbage, tomato, citrus fruit, apples and oil seed crops processing studies. Processing of tomatoes into pulp and paste showed a slight increase of cyfluthrin residues in the processed commodities compared to the RAC. Whilst there was a decrease in residues found in the corresponding juice, ketchup and purée. Citrus and apples also both showed a decrease in residues found in the juice, but a slight increase in pomace and/or oil and molasses. There was a concentration into the oil of cottonseed and sunflower. Processing studies on potatoes, cabbages, soya bean and rape seed did not show any indication regarding the fate of beta-cyfluthrin/cyfluthrin residues during processing as residues in the RAC or processed products were all below the LOQ. Estimated processing factors, HRs and STMRs are summarized below.

Summary of processing factors for cyfluthrin residues.

Raw agricultural commodity (RAC)	Processed commodity	Calculated processing factors	PF (Mean, median or best estimate)	RAC-STMR	RAC-STMR×PF
Orange	Pulp dry	5.3	5.3	0.06	0.318
Apple	Pomace, dry	0.11, 16	16	0.02	0.32
Cotton	Hulls	1.9	1.9	0.1	0.19
Cotton	Meal	0.08	0.08		0.008
Cotton	Oil, crude	1.9	1.9		0.19
Cotton	Oil, refined	1.2	1.2		0.12

The Meeting decided to make maximum residue level recommendations for citrus pulp (dry) and cotton seed hulls. Based on an estimated high residue value of 1.06 mg/kg (5.3 × 0.2 mg/kg) for citrus pulp (dry), the meeting recommended a maximum residue level of 2 mg/kg for citrus pulp (dry). The Meeting also recommended a maximum residue level of 1 mg/kg for cotton seed oil, crude based on an estimated high residue of 0.988 mg/kg (1.9 × 0.22 mg/kg).

The Meeting also decided to use the default generic processing factor of 7 to estimate a maximum residue level for chilli pepper (dry) of 1 mg/kg based on an HR-P of 0.84 mg/kg (7 × 0.12) and STMR-P of 0.42 mg/kg (7 × 0.06).

Residues in animal commodities

Farm animal feeding studies

The Meeting received information on the residue levels arising in animal tissues and milk when dairy cows were dosed with cyfluthrin for 28 days at the equivalent of 4.5, 13 and 40 ppm in the diet. Average residues in milk of the 40 ppm dose group were 0.22 mg/kg at day 14 and 0.14 mg/kg at day 28. Cyfluthrin residues in the fat were higher than in other tissues. Transfer factors (average residue level in tissue ÷ residue level in feed) for each tissue and milk for the three dosing levels (3 animals

per dose group) were: fat, 0.056, 0.054, 0.066; muscle, < 0.002, < 0.001, 0.00075; kidney, 0.0042; liver, 0.0032; milk 28 days, 0.0037, 0.0036, 0.0036.

In an additional dosing study conducted at levels equivalent to 11, 36 and 112 ppm in the diet average residues in milk at day 28 were 0.45 mg/kg for the 112 ppm dose group. As for the previous study, residues were highest in fat with only low levels of cyfluthrin detected in other tissues. Transfer factors for each tissue and milk for the three dosing levels (3 animals per dose group) were: fat, 0.11, 0.074, 0.061; muscle, < 0.0009, 0.001, 0.00063; kidney, < 0.0009, < 0.0008, 0.00045; liver, < 0.0036, < 0.00028, 0.00018; milk 28 days, 0.0055, 0.0033, 0.0041.

The Meeting also received information on the residue levels arising in tissues and eggs when laying hens were dosed with cyfluthrin for 28 days at the equivalent of 6 and 20 ppm in the diet. Residues in eggs were below the LOQ for both feed levels. At the 2 ppm feeding level the residues in tissues were below the LOQ of the analytical methods. For the 20 ppm feed level, residues in fat were substantially higher than residues in other tissues 0.05 mg/kg compared to < 0.01-0.01 mg/kg. Transfer factors based on residues for fat were 0.0025 for the 20 ppm feed levels. Transfer factors (mean residue) for muscle and liver were < 0.0005 and < 0.0005 respectively for the 20 ppm feeding level while that for skin was 0.0005.

Farm animal direct treatment

No studies were received on the residues of cyfluthrin arising from direct animal treatment. The Meeting noted that JECFA has evaluated cyfluthrin residues arising from direct animal treatment at its 48th Meeting in 1997 and recommended maximum residue limits for cattle of 20 µg/kg for muscle, liver and kidney, 40 µg/kg for milk and 200 µg/kg for fat. The marker residue that applied to the residue limits was cyfluthrin.

Farm animal dietary burden

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

Estimated maximum and mean dietary burdens of farm animals

Dietary burden calculations for beef cattle, dairy cattle, broilers and laying poultry are provided in Annex 6 of the 2007 JMPR Report. The calculations were made according to the animal diets from US-Canada, EU and Australia in the OECD Table (Annex 6 of the 2006 JMPR Report).

	Animal dietary burden, cyfluthrin, ppm of dry matter diet					
	US-Canada		EU		Australia	
	max	mean	max	mean	max	mean
Beef cattle	1.87	0.31	3.00	0.49	5.89 [a]	0.68 [c]
Dairy cattle	1.84	0.26	3.00 [b]	0.49 [d]	2.47	0.36
Poultry - broiler	0.009	0.009	0.0152	0.015	0.003	0.003
Poultry - layer	0.009	0.009	1.3 [e]	0.16 [f]	0.003	0.003

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

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

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

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

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

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

The cyfluthrin dietary burdens for animal commodity MRL and STMR estimation (residue levels in animal feeds expressed on dry weight) are: beef cattle 5.89 and 0.68 ppm, dairy cattle 3.06 and 0.50 ppm and poultry 1.3 and 0.16 ppm.

Animal commodity maximum residue levels

The maximum dietary burden for beef and dairy cattle is 5.89 and 3.06 ppm respectively, so the levels of residues in tissues can be obtained by interpolation between the high residues obtained in tissues and at the 4.5 and 13 ppm feeding levels for milk, muscle and fat and from the 40 ppm feed level for kidney and liver as these are the only kidney and liver samples subjected to strong extraction required to release the majority of cyfluthrin residues. Maximum residues expected in tissues are: fat 0.37 mg/kg, muscle < 0.01 mg/kg, liver 0.021 mg/kg, kidney 0.027 mg/kg and the mean residue for milk 0.0136 mg/kg. No data was available on the partitioning of residues in milk between aqueous and fat phases of milk.

The Meeting estimated maximum residue levels for meat (from mammals other than marine mammals) 1 mg/kg (fat); kidney of cattle, goats, pigs and sheep 0.05 mg/kg; liver of cattle, goats, pigs and sheep 0.05 mg/kg and milks 0.04 mg/kg. The recommendation of 0.04 mg/kg milk replaces the previous recommendation of ML 0812 Cattle milk 0.01 F mg/kg, which also incorporated direct animal treatment. The Meeting noted the recommendation for cattle milk arising from exposure to cyfluthrin through the cattle diet is the same as proposed by JECFA for direct animal treatment.

The STMR dietary burdens for beef and dairy cattle are 0.68 and 0.50 ppm respectively. Transfer factors from the average residues from the 4.5 ppm feeding level were used to estimate STMR values as for cyfluthrin. The estimated STMRs are: meat (from mammals other than marine mammals) < 0.01 mg/kg, fat (from mammals other than marine mammals) 0.0378 mg/kg, kidney of cattle, goats, pigs and sheep < 0.01 mg/kg, liver of cattle, goats, pigs and sheep < 0.01 mg/kg and milks 0.0027 mg/kg.

The highest individual tissue residue from the relevant feeding group was used in conjunction with the highest residue dietary burden to calculate the likely highest animal commodity residue level. As only a single animal is available per feeding group, the tissue residues from the animals in the relevant feeding groups were used in conjunction with the STMR dietary burden to estimate the animal commodity STMR values. For milk, the mean milk residue at the plateau level from the relevant feeding group was used to estimate both the maximum residue level and the STMR.

Dietary burden (mg/kg)[a] Feeding level [ppm][b]		Cyfluthrin residues, mg/kg [c]								
		Milk	Fat		Muscle		Liver		Kidney	
		Mean	High	mean	High	mean	high	mean	High	mean
MRL beef	(5.89) [4.5] high		*(0.37)* 0.30		*(< 0.01)* < 0.01		*(0.021)* 0.14[d]		*(0.027)* 0.18[d]	
MRL dairy	(3.06) [4.5] av	*(0.0136)* 0.02								
STMR beef	(0.68) [4.5] av		*(0.0378)* 0.25		*(< 0.01)* < 0.01		*(< 0.01)* < 0.01		*(< 0.01)* < 0.01	
STMR dairy	(0.50) [4.5] av	*(0.0022)* 0.02								

a - Values in parentheses are the estimated dietary burdens

b - Values in square brackets are the actual feeding levels in the transfer study

c - Residue values in parentheses in italics are interpolated from the dietary burden, feeding levels in the transfer study and the residues found in the transfer study. High is the highest individual animal tissue residue in the relevant feeding group. Mean is mean animal tissue (or milk) residue in the relevant feeding group.

d - Residue values for kidney and liver were obtained from the dosing level equivalent to 40 ppm in the feed as only these samples were subject to reanalysis using a stronger extraction process

The maximum dietary burden for poultry is 1.3 ppm. No residues above the LOQ of the analytical method used were observed in the feeding study for laying hens at the lowest dose level equivalent to 2 ppm in the diet. Maximum residues expected are: muscle, fat, liver, kidney and eggs are all < 0.01 mg/kg.

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

As no residues are observed at the maximum feeding level for poultry, the STMRs for poultry meat, edible offal and eggs are the same as the maximum residue levels.

RECOMMENDATIONS

On the basis of the data from supervised trials the Meeting concluded that the residue levels listed below are suitable for establishing maximum residue limits and for IEDI assessment.

Definition of the residue for compliance with MRL and for estimation of dietary intake: *cyfluthrin (sum of isomers)*

The residue is fat-soluble

Recommendations for maximum residue levels arising from the use of cyfluthrin or beta-cyfluthrin

CCN	Commodity	MRL mg/kg		STMR or STMR-P mg/kg	HR or HR-P mg/kg
		New	Previous		
FP 0226	Apple	0.1	0.5	0.02	0.06
VB 0400	Broccoli	2		0.20	1.5
VB 0041	Cabbages, Head	4		0.245	2.1
VB 0404	Cauliflower	2		0.24	0.91
HS 0444	Peppers Chili (dry)	1		0.42	0.84
FC 0001	Citrus fruit	0.3		0.06	0.2
AB 0001	Citrus pulp (dry)	2		0.318	
SO 0691	Cotton seed	0.7	0.05	0.1	
OC 0691	Cotton seed oil, crude	1		0.19	
VO 0440	Egg plant	0.2		0.05	0.12
PE 0112	Eggs	0.01 *		0	0
MO 0099	Liver of cattle, goats, pigs and sheep	0.05		< 0.01	0.021
MO 0098	Kidney of cattle, goats, pigs and sheep	0.05		< 0.01	0.027
GC 0645	Maize	W	0.05		
ML 0106	Milks	0.04		0.0022	
MM 0095	Meat (from mammals other than marine mammals)	1 fat		0.0378 fat < 0.01 muscle	0.37 fat < 0.01 muscle
ML 0812	Cattle milk	W	0.01 F		
FP 0230	Pear	0.1		0.02	0.06
VO 0051	Peppers	0.2		0.06	0.12
VO 0445	Peppers sweet	W	0.2		
VR 0589	Potato	0.01 *		0	0
PM 0110	Poultry meat	0.01 * fat		0	0
PO 0111	Poultry, edible offal of	0.01 *		0	0
SO 0495	Rape seed	0.07	0.05	0.05	
VO 0448	Tomato	0.2	0.5	0.07	0.10

* the MRL is estimated at or about the LOQ

DIETARY RISK ASSESSMENT

Long-term intake

The evaluation of cyfluthrin has resulted in recommendations for MRLs and STMRs for raw and processed commodities. Consumption data were available for 22 food commodities and were used in the dietary intake calculation. The results are shown in Annex 3 of the 2007 Report of the JMPR.

The International Estimated Daily Intakes for the 13 GEMS/Food regional diets, based on estimated STMRs were in the range 0 – 2% of the maximum ADI of 0.04 mg/kg bw (Annex 3 of the 2007 Report of the JMPR). The Meeting concluded that the long-term intake of residues of cyfluthrin from uses that have been considered by the JMPR is unlikely to present a public health concern.

Short-term intake

The international estimated short-term intake (IESTI) for cyfluthrin was calculated for the food commodities (and their processing fractions) for which maximum residue levels and HRs were estimated and for which consumption data were available. The results are shown in Annex 4 of the 2007 Report of the JMPR.

For the general population the IESTI varied from 0 – 120% of the ARfD (0.04 mg/kg bw) while for children the IESTI varied from 0 – 240% of the ARfD. The IESTI (as a percentage of the ARfD) for broccoli for children was 120% and 70% for the general population, 240% for head cabbage for children and 100% for the general population.

The Meeting concluded that the short-term intake of residues of cyfluthrin resulting from uses that have been considered by the JMPR, except the uses on broccoli and head cabbage, is unlikely to present a public health concern.

The Meeting noted that no residue data relating to alternative GAP were submitted for broccoli and head cabbage. The information provided to the JMPR precludes an estimate that the dietary intake would be below the ARfD for consumption for broccoli and head cabbage by children.

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