FLUDIOXONIL (211)

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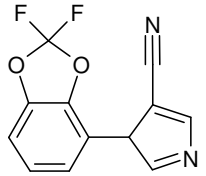
EXPLANATION

At the 33rd Session of the CCPR (ALINORM 01/24A), fludioxinil was identified as a priority for evaluation as a new pesticide by the 2004 JMPR. The manufacturer submitted information on physical and chemical properties, metabolism (plant and animal), environmental fate, analytical methods, freezer storage stability, good agricultural practices (GAP), supervised field trials, processing, and livestock feeding. Information on GAP was reported by the government of Australia, and on analytical methods by the government of the USA.

IDENTITY

ISO common name: fludioxonil
Chemical name:
IUPAC: 4-(2,2-difluoro-benzo[1,3]dioxol-4-yl)pyrrole-3-carbonitrile
CA: 4-(2,2-difluoro-1,3-benzodioxol-4-yl)-1*H*-pyrrole-3-carbonitrile
CAS no.: 131341-86-1
Syngenta code no.: CGA 173506

Structural formula:

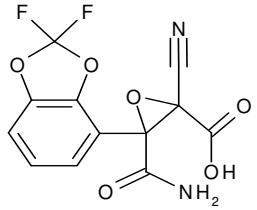
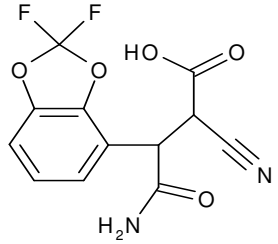
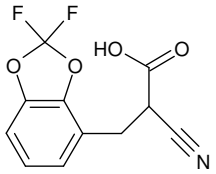
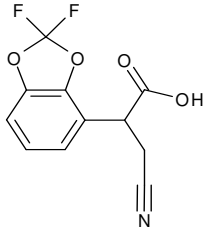


Molecular formula: $C_{12}H_6F_2N_2O_2$
Molecular mass: 248.2

PHYSICAL AND CHEMICAL PROPERTIES

Property	Results	Method Test material[1]	Reference
Melting point	199.8°C	EEC A.1 99.8% (PAI/1)	Rodler, 1992 Report EA169432
Boiling point	Thermal decomposition starts at about 306°C	EEC A.2 99.8% (PAI/1)	Das, 2000 Report 80806
Temperature of decomposition or sublimation	Thermal decomposition starts at about 306°C	EEC A.2 99.8% (PAI/1)	Das, 2000 Report 80806
	No thermal effect found between room temperature and 150°C	OECD 113 96.8%(TGAI)	Schürch, 1992 Report EA175120
Relative density	1.54-10^3 kg/m^3 at 20°C corresponding to a relative density of 1.54	OECD 109 99.8% (PAI/1)	Füldner, 1992 Report PP-92/11P.DES
Vapour pressure	Vapour pressure curve in the liquid state: ^{10}log P [Pa] = 16.8495-6936.15 · 1/T [K] from fit of measurements between 60.3°C and 186.5°C Vapour pressure at 25°C : 3.9 · 10^{-7} Pa (extrapolated)	EEC A.4 99.8% (PAI/1)	Nickler, 1992 Report PP92/11P.VPC
Volatility	Henry's law constant at 25°C 5.4 · 10^{-5} Pa · m³/mol	calculation	Burkhard, 1994 Archive CGA173506/0489

Property	Results	Method Test material1	Reference
Physical state and colour	Pure active substance : faintly yellow fine powder	visual 99.9% (PAI/2)	Das, 1998 Report EZA62940
	Technical grade active substance . light olive green powder	96.8%(TGAI)	Rodler, 1992 Report EA175120
Odour	Pure active substance : odourless	organoleptic 99.9% (PAI/2)	Das, 1998 Report EZA62940
	Technical grade active substance : odourless	96.8%(TGAI)	Rodler, 1992 Report EA175120
Spectra active substance	IR KBr pellet 3289, 2223, 1652, 1530, 1236 cm^{-1} ^{1}H-NMR (300 MHz, DMSO) chemical shift (ppm): 7.2-7.4 (3 protons), 7.5-7.6 (1 proton), 7.8 (1 proton), 12.2 (1 proton) ^{13}C-NMR (75 MHz, DMSO) chemical shift (ppm): 143, 139 (C1, C2), 135, 131, 127 (C12), 129, 120 (C8, C11), 125, 122, 108 (C4, C5, C6), 117 (C3, C10, C7), 90 (C9) mass spectra (quad EI): 248 (M+), 182, 154, 127 UV (methanol) Absorption Characteristics : Molar extinction coefficients (ε, l/mol.cm) were determined to be: solution wavelength (nm) ε (l/mol.cm) neutral 266 12384 acidic 265 12327 basic 271 11790 12384 (neutral solution), No absorption maximum between 340 nm and 750 nm was observed.	99.9% (PAI/2) UV/VIS OECD 101	Stulz, 1998 Report EZA63115 Stulz, 1998 Report EZA64462
Solubility in water including effect of pH	The solubility in pure water at 25°C was determined to be: 1.8 mg/l Fludioxonil has no dissociation within the range pH 2 to pH 12. pH has no effect on the water solubility of the compound in the pH range 4 to 10	OECD 105 99.8% (PAI/1)	Rodler, 1992 Report EA169432
Solubility in organic solvents	The solubility in different organic solvents at 25°C was determined to be : acetone 190 g/l dichloromethane 7.3 g/l ethyl acetate 86 g/l hexane 10 mg/l methanol 42 g/l octanol 20 g/l toluene 2.7 g/l	CIPAC MT 157.3 96.8% (TGAI)	Kettner, 2000 Archive CGA173506/5141
Partition coefficient n-octanol/water	The octanol/water partition coefficient (P_{ow}) and its logarithm to base 10 (log P_{ow}) was determined to be: P_{ow} = 13100 ± 472 at 25°C log P_{ow} = 4.12 ± 0.016	OECD 107 corresponding to EEC A.8 99.8% (PAI/1)	Rodler, 1992 Report EA169432
Hydrolysis rate	No degradation observed at pH 5, 7 and 9 respectively, during 32 days at 25°C	EPA 161-1 corresponding to EEC C.7 98% (PAI/3)	Kirkpatrick, 1991 Report HRC/CBG 487/891775

Property	Results	Method Test material1	Reference
Photochemical degradation	The experimental photolytic half-lives of fludioxonil at 25°C in sterile aqueous solutions buffered to pH 7 using xenon arc light were found to be : 8.7 summer sunlight days at 40°N ([phenyl-U-^{14}C]) 9.9 summer sunlight days at 30°N ([pyrrole-4-^{14}C]) Dark control experiments revealed negligible degradation of fludioxonil. Approximately 95.9% to 98.3% of the applied radioactivity was recovered. Up to 20% volatiles (CO_2, which indicates breakdown of the phenyl ring); three photoproducts >10% were found: CGA 339833  3-carbamoyl-2-cyano-3-(2,2-difluoro-benzo[1,3]dioxol-4-yl)-oxirane-2-carboxylic acid *(IUPAC)* CGA 344623  2-cyano-3-(2,2-difluoro-benzo[1,3]dioxol-4-yl)-succinamic acid *(IUPAC)* The third photoproduct >10% could be assigned either of the two isomeric structures:  2-cyano-3-(2,2-difluoro-benzo[1,3]dioxol-4-yl)-propionic acid *(IUPAC)*  3-cyano-2-(2,2-difluoro-benzo[1,3]dioxol-4-yl)-propionic acid *(IUPAC)*	EPA 161-2 is in accordance with European Community Commission Directive 95/36/EC of July 14, 1995 amending Council Directive 91/414/EEC 98% (PAI/3) 98% (PAI/4)	Kirkpatrick, 1994[a] Report CBG569A Kirkpatrick, 1994, Report CBG 49064 Kirkpatrick, 1994, Report HRC/CBG 516/901362 Kirkpatrick, 1996 Report CBG 720

Property	Results	Method Test material1	Reference
Quantum yield	The quantum yield of direct photolysis was found to be Φ = 0.026 Photolytic half-life in shallow waters was estimated for midspring time and mid summer time at the geographical latitudes of 40°N and 50°N. Under clear sky conditions half-lives between 160 days (at 40°N and midsummer time) and 1237 days (at 50°N and midspring time) are expected for fludioxonil.	ECETOC Guideline of the German Federal Environment Agency: "Phototransform-ation of Chemicals in Water; Part A : Direct Phototransfor-mation", Berlin, FRG, January 1990" 99.9% (PAI/2)	Abildt, 1994 Report 93UA02
Dissociation constant	The estimated dissociation constants of fludioxonil in water were found to be: pK_{a1} = <0 (basic) pK_{a2} = ~14.1 (acidic)	by estimation 99.8% (PAI/1)	Jäkel, 1992 Report FF92/11P.DCW
Stability in the air, photo-chemical degradation, identity of breakdown product(s)	Based on the calculation according to Atkinson the estimated half-life of fludioxonil in the atmosphere by hydroxyl radical oxidation is approximately 2-4 hours ($1.5 \cdot 10^6$ OH-radicals/cm³ and a 12 hour day)	calculation according to Atkinson, R., Environ. Toxicol. Chem., 7, 435 (1988)	Stamm, 1999 Report 95A99001SM
Flammability	Fludioxonil is not considered highly flammable	EEC A.10 96.8% (TGAI)	Schürch, 1992 Report EA 175120
Auto-flammability	No self-ignition	EEC A.16 96.8% (TGAI)	Schürch, 1992 Report PF 92/21T.AFS
Flash point	Not required, fludioxonil is a solid with a melting point >40°C		None
Explosive properties	Fludioxonil is not considered an explosive.	EEC A.14 96.8% (TGAI)	Schürch, 1992 Report PP 94/66 C.FLS
Surface tension	Surface tension of aqueous suspensions at 20°C by the Wilhelmy plate method was determined to be : σ = 47.7-48.5 mN/m (filtrates of 10 g/l suspension)	OECD 115 ≅ EEC A.5 96.8% (TGAI)	Ryser, 1992 Report PP 92/23T.SUR
Oxidizing properties	Fludioxonil is not considered an oxidizing substance	EEC A.17 96.8% (TGAI)	Schürch, 1992 Report PP92/23T.OXP

[1]PAI/1 . pure active substance, CGA 173506 pure; AMS 273/103, purity 99.8%; Details of purification method see Stulz 1994

PAI/2 . pure active substance, CGA 173506 pure;AMS 273/104, purity 99.9%

PAI/3 . [pyrrole-4-^{14}C] CGA 173506; radiochemical purity 98%;Batch K-736.3A

PAI/4 . [phenyl-U-^{14}C] CGA 173506;radiochemical purity 98%; Batch CFQ 7117

TGAI . technical grade active substance; Batch P.206006, purity 96.8%

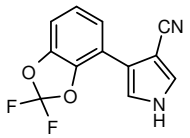
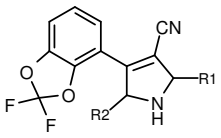
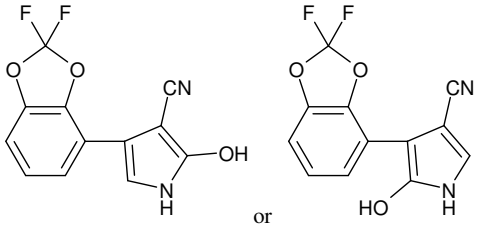
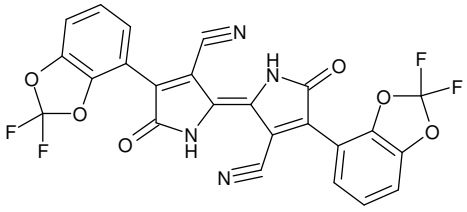
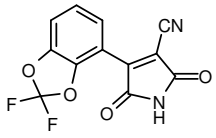
Formulations

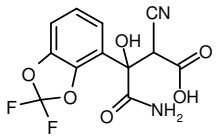
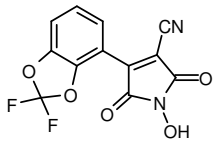
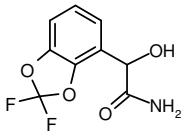
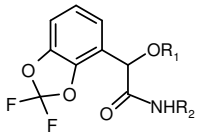
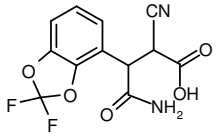
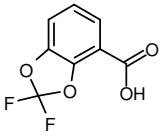
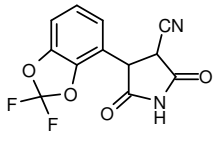
The formulations include DS (powder for dry seed treatment), ES (emulsion for seed treatment), FS (flowable concentrate for seed treatment), SE (suspo-emulsion), WG (water dispersible granules), and WP (wettable powder).

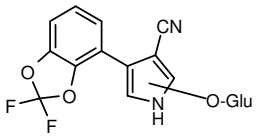
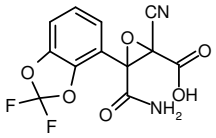
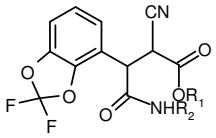
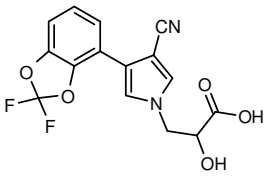
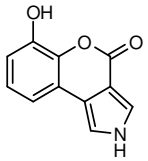
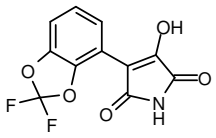
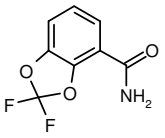
METABOLISM AND ENVIRONMENTAL FATE

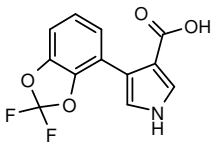
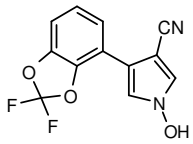
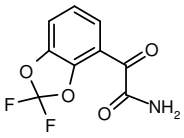
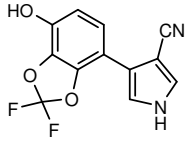
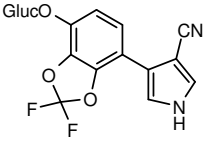
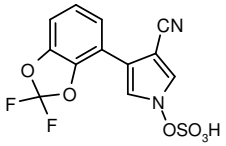
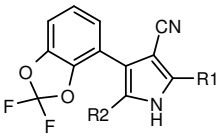
The following table links manufacturer code number, metabolite code, compound name,, and compound structure and applies to the various metabolism and rotational crop studies.

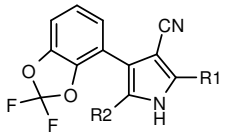
Table 1. Fludioxonil and metabolites: compound code, name, and structure

Code Number	Description / Chemical name	Plant/animal and metabolite code	Structure
Fludioxonil CGA 173506	Parent compound 4-(2,2-difluorobenzo[1,3]dioxol-4-yl)-1H-pyrrole-3-carbonitrile	Grape (II_4), tomato (III_4), peach (P19), green onion (A: peak 11), lettuce (I_{15}), potato (I_{12}/II_4), wheat (II_4), goat (C), hen (E1)	
SYN 518579	4-(2,2-difluorobenzo[1,3]dioxol-4-yl)-5-hydroxy-2-oxo-2,5-dihydro-1H-pyrrole-3-carbonitrile or its tautomeric form 4-(2,2-difluorobenzo[1,3]dioxol-4-yl)-2-hydroxy-5-oxo-2,5-dihydro-1H-pyrrole-3-carbonitrile	Grape (II_2), tomato (III_2), peach (P15 and P16), green onion (P15), wheat (II_2),	 R1 = -OH, R2 = :O or R1 = :O, R2 = -OH
SYN 518577 or SYN 518578	4-(2,2-difluorobenzo[1,3]dioxol-4-yl)-2-hydroxy-1H-pyrrole-3-carbonitrile or 4-(2,2-difluorobenzo[1,3]dioxol-4-yl)-5-hydroxy-1H-pyrrole-3-carbonitrile	Goat, Hen Rat	
SYN 518582	4,4'-bis-(2,2-difluorobenzo[1,3]dioxol-4-yl)-5,5'-dioxo-1,5,1',5'-tetrahydro-[2,2']bipyrrolylidene-3,3'-dicarbonitrile	Rat	
CGA 265378	4-(2,2-difluorobenzo[1,3]dioxol-4-yl)-2,5-dioxo-2,5-dihydro-1H-pyrrole-3-carbonitrile	Grape (II_{3b}), green onion (B: peak 10), lettuce (I_{14}), wheat (II_{3b}), rotational wheat, radish and mustard, hen (E2)	

Code Number	Description / Chemical name	Plant/animal and metabolite code	Structure
SYN 518581	2-cyano-3-(2,2-difluoro-benzo[1,3]dioxol-4-yl)-3-hydroxysuccinamic acid	Grape (I_{3b}), tomato (I_{3b})	
SYN 518580	4-(2,2-difluorobenzo[1,3]dioxol-4-yl)-1-hydroxy-2,5-dioxo-2,5-dihydro-1H-pyrrole-3-carbonitrile	Grape (II_{3a}), tomato (III_3), wheat (II_3)	
CGA 308103	2-(2,2-difluorobenzo[1,3]dioxol-4-yl)-2-hydroxyacetamide	Grape (I_{11}), peach (P14), green onion (E peak 4), lettuce (I_{11}), wheat (I_{13}), rotational wheat radish and mustard,	
	Glucose conjugate of CGA 308103	Grape (I_6), tomato (I_6), lettuce (I_5)	 R_1 = Glu, R_2 = -H or R_1 = -H, R_2 = Glu
CGA 344623	2-cyano-3-(2,2-difluorobenzo[1,3]dioxol-4-yl)succinamic acid	Tomato (I_4), peach (P5), green onion (H: peaks 2/3), lettuce (I_4), wheat (I_4), hen (P2/P3)	
CGA 192155	2,2-difluorobenzo[1,3]dioxole-4-carboxylic acid	Potato (I_{10}), tomato (I_5), peach (P17), green onion (F: peak 1), lettuce (I_{10}), wheat (I_{10}), soybean, rotational wheat, radish and mustard, hen (P1)	
CGA 308565	4-(2,2-difluorobenzo[1,3]dioxol-4-yl)-2,5-dioxo-pyrrolidine-3-carbonitrile	Peach (P18), rotational wheat, radish and mustard,	

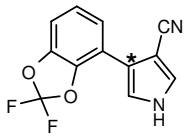
Code Number	Description / Chemical name	Plant/animal and metabolite code	Structure
	Glucose conjugate of oxidised CGA 173506 (pyrrole ring)	Peach (P10a, P10b P8b)	
	Sugar conjugate of oxidised CGA 173506 (pyrrole ring)	Peach (P8)	
CGA 339833	3-carbamoyl-2-cyano-3-(2,2-difluorobenzo[1,3]dioxol-4-yl)-oxirane-2-carboxylic acid	Peach (P6), green onion (I: peak 5), lettuce (I_{3b}), wheat (I_3), rotational wheat, radish and mustard,	
	N- or O-glucose conjugate of CGA 344623	Lettuce (I_2)	 R_1 = Glu, R_2 = H or R_1 = H, R_2 = Glu
	N-lactic acid conjugate of fludioxonil	Lettuce(I_{4b}+I_{4c})	
CGA-227731	6-hydroxy-2H-chromeno[3,4-c]pyrrol-4-one	Soybean, rotational spring wheat	
CGA-260766	3-(2,2-difluorobenzo[1,3]dioxol-4-yl)-4-hydroxy-pyrrole-2,5-dione	Soybean, rotational spring wheat	
CGA-340351	2,2-difluorobenzo[1,3]dioxole-4-carboxylic acid amide	Soybean, rotational spring wheat	

Code Number	Description / Chemical name	Plant/animal and metabolite code	Structure
CGA-257777	4-(2,2-difluorobenzo[1,3]dioxol-4-yl)-1H-pyrrole-3-carboxylic acid	Rotational spring wheat	
CGA-335892	4-(2,2-difluorobenzo[1,3]dioxol-4-yl)-1-hydroxy-1H-pyrrole-3-carbonitrile	Hen (G)	
CGA 344624	2-(2,2-difluorobenzo[1,3]dioxol-4-yl)-2-oxo-acetamide	Hen (P7)	
SYN 518576	4-(2,2-difluoro-7-hydroxybenzo[1,3]dioxol-4-yl)-1H-pyrrole-3-carbonitrile	Hen (F)	
	Glucuronide conjugate of 4-(2',2'-difluoro-7'-hydroxy-1',3'-benzodioxol-4'-yl)-1H-pyrrole-3-carbonitrile	Goat (B2)	
	Sulfate conjugate of CGA-335892, 4-(2,2-difluorobenzo[1,3]dioxol-4-yl)-1-hydroxy-1H-pyrrole-3-carbonitrile	Hen (I1)	
	Glucuronide conjugate of SYN 518577, 4-(2,2-difluorobenzo[1,3]dioxol-4-yl)-2-hydroxy-1H-pyrrole-3-carbonitrile or SYN 518578, 4-(2,2-difluoro-benzo[1,3]dioxol-4-yl)-5-hydroxy-1H-pyrrole-3-carbonitrile	Goat (B1, A) Hen (D)	 R1 = -OGluc, R2 = -H (B1) or R1 = -H, R2 = -OGluc (A)

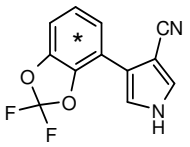
Code Number	Description / Chemical name	Plant/animal and metabolite code	Structure
	Sulfate conjugate of SYN 518577,4-(2,2-difluorobenzo[1,3]dioxol-4-yl)-2-hydroxy-1H-pyrrole-3-carbonitrile or SYN 518578, 4-(2,2-difluorobenzo[1,3]dioxol-4-yl)-5-hydroxy-1H-pyrrole-3-carbonitrile	Goat (G), Hen (I2)	 R1 = -OSO_3H, R2 = -H or R1 = -H, R2 = -OSO_3H

In studies of metabolism, fludioxonil was labelled with ^{14}C at one of two sites.

[Pyrrole-4-^{14}C]fludioxonil:



[Phenyl-U-^{14}C]fludioxonil:



*=Position of carbon-14 label

bel

Animal metabolism

The Meeting received information on the fate of fludioxonil administered orally to lactating goats and laying hens.

Goats. ^{14}C-pyrrole-labelled fludioxonil was administered orally in gelatin capsules at 150 mg/day to two lactating goats for four consecutive days (Pfeffer, 1992a, Report F-00088), corresponding to a dietary intake of 94 and 111 ppm in the feed. The radioactive pesticide was stable in the capsule from the time of preparation through the administration period (TLC analyses).

Urine, faeces and milk were collected daily from day -2 through day 4 of dosing. Most of the radioactivity was excreted in the faeces (50% to 60%) and in the urine (15% to 23%). The total recovery (including the gastrointestinal contents) was 94% to 98%. Both goats were killed about six hours after the last dose, and samples of liver, kidneys, tenderloin muscle, thigh muscle, omental fat, perirenal fat, heart, bile, blood, and gastrointestinal contents were stored at approximately -20°C until analysis.

Liquid scintillation counting (LSC) was used to determine the total radioactive residues (TRR) in the samples. The results are shown in Table 2.

Table 2. [^{14}C]fludioxonil residues found in goat tissues and milk (Peffer, 1992a, Report F-00088).

Sample	mg/kg[1], animal no. 80	mg/kg[1], animal no. 78
blood	0.47	0.49

Sample	mg/kg[1], animal no. 80	mg/kg[1], animal no. 78
plasma	0.64	0.68
liver	6.18	5.37
kidney	2.92	2.89
heart	0.22	0.16
tenderloin	0.09	0.05
leg muscle	0.07	0.06
omental fat	0.26	0.11
perirenal fat	0.28	0.10
milk[2]		
day 1	1.1	1.2
day 2	1.1	1.8
day 3	1.2	2.0
day 4	1.6	2.9

[1] fludioxonil equivalents. Limits of detection for tissues ranged from 0.002 mg/kg(blood) to 0.01 mg/kg (liver, kidney, fat), typical aliquot 30 to 200 mg
[2] Day -2 and day -1 milk samples had dpm values below the limit of detection, 0.004 mg/kg, typical aliquot 100 mg.

The highest ^{14}C residues were in the liver and kidneys. Residues in the fat were 2–4 times those in muscle. Resides in the milk rose steadily during the four days, with maximum levels of 1.6 and 2.9 mg/kg on day 4.

The radioactivity in tissue and milk samples was extracted by two techniques. The first for liver, kidney, and tenderloin muscle, involved extraction of tissue aliquots with multiple volumes of acetonitrile, followed by multiple volumes of water. The multiple extracts were combined and the radioactivity determined in each by LSC. Weighed aliquots of the remaining solid pellets were combusted to determine the amount of unextracted radioactivity.

The second method, which involved mixing 0.5 g of homogenized tissue or milk with octadecyl (C-18) column packing (2 g) in a mortar and pestle, was used for milk, omental fat, and tenderloin (animal no. 80 only). The mixture was packed into a column and radioactive residues eluted with hexane, ethyl acetate/acetonitrile, methanol, and distilled water. The radioactivity in each fraction and in the exhausted C-18 packing material was determined by LSC. The results are summarized in Tables 3 and 4.

Table 3. Distribution of radioacativity in goat tissue extracts (Peffer, 1992a, Report F-00088). Values uncorrected for recoveries.

Sample (^{14}C as fludioxonil)	% of TRR			
	Extract		Post extraction solid (PES)	Extract + PES
	Acetonitrile	Water	Pellet	Total
liver #80 (6.2 mg/kg)	38	18	44	100
liver #78 (5.4 mg/kg)	32	14	44	90
kidney #80 (2.9 mg/kg)	47	7.6	39	94
kidney #78 (2.9 mg/kg)	52	6.2	29	87

Sample (^{14}C as fludioxonil)	% of TRR			
	Extract		Post extraction solid (PES)	Extract + PES
	Acetonitrile	Water	Pellet	Total
tenderloin muscle #80 (0.09 mg/kg)	76	5.7	29	111
tenderloin muscle #78 (0.05 mg/kg)	76	9.1	42	127

Table 4. Distribution of radioacativity in goat milk, fat, and muscle (Peffer, 1992a, Report F-00088). Values uncorrected for recoveries.

Sample (^{14}C as fludioxonil)	Hexane	Ethyl acetate/ acetonitrile	Methanol	Water	Remaining on Column	Total
milk, day 3						
#80 (1.2 mg/kg)	0.16	88.	19	1.4	7.0	116
#78 (2.0 mg/kg)	0.53	69	22	0.3	2.7	95
omental fat						
#80 (0.26 mg/kg)	13	86	1.3	0.1	2.5	103
#78 (0.11 mg/kg)	6.3	88	4.4	0.9	9.2	109
tenderloin muscle #80 (0.09 mg/kg)	2.6	70	7.0	0.7	11	91

Most of the radioactivity in the milk, omental fat and muscle (76 to 109%) was extractable with organic solvent, and small amounts with water (0.1 to 9.1%), whereas in liver and kidney moderate amounts (38 to 52%) were extracted with organic solvents and smaller amounts with water (6.2 to 18%), leaving 29 to 44% unextracted.

Before analysis the extracts were also treated by TLC and/or reverse-phase HPLC, and the organic phases were further treated with beta glucuronidase or sulfatase enzymes to hydrolyse any glucuronide or sulfate conjugates.

The unextracted residues in liver, kidney, and tenderloin tissue pellets (from acetonitrile and water extractions) were treated with protease, then at various pH levels with 2,4-dinitrofluorobenzene. Protease treatment liberated 75% to 91% of the bound activity from liver, kidney, and tenderloin. Treatment with dinitrofluorobenzene, DNFB, which reacts with the terminal amino groups of amino acids, derivatized only 24 to 41% of the protease-liberated activity. This suggests that the radioactive residues remaining in the liver and kidney are associated with biological molecules, in part proteins. Other treatments used to release bound or conjugated residues in the liver included treatment with Raney nickel, derivatization with *m*-toluoyl chloride followed by extraction and TLC, and lyophilization followed by extraction and HPLC. These additional treatments liberated very little activity.

To accomplish exhaustive extraction of the hitherto unextracted residues the liver and kidney pellets were also subjected to 24 h hot methanol Soxhlet extraction, followed by acid/base hydrolysis (6N HCL; 15%KOH; $\leq$95^{o}C), which resulted in the extraction of only slightly more radioactivity than the original acetonitrile/water extractions.

Qualitative TLC and HPLC profiles of the organic-phase extracts of kidney, milk, liver, and fat from the two goats were similar (typically containing one or two major components), indicating metabolism had proceeded similarly, although profiles in the ACN (acetonitrile) extracts of tenderloin differed because animal 78 had much lower ^{14}C residue levels. The HPLC profile of the ACN extract of animal 80 indicated the presence of one major (76% of activity) and one minor (7.6%) component while the HPLC profile of animal 78 indicated the same two components in differing quantities (34.5% and 31.9%). A qualitative comparison of the enzyme-treated with the untreated organic-phase extracts indicated the presence of conjugated residues from the shifts in retention times.

Attempts were made to identify the activity in the aqueous (polar) extracts of liver (initial extracts as well as acid/base hydrolysis products). Additional treatment included protease (kidney and tenderloin were treated with protease also) and analysis by 2 dimensional TLC. However no additional metabolites were isolated.

Metabolites were isolated from HPLC fractions or TLC bands from the tissue analyses where adequate levels of activity were present, analysed by GC/MS or HPLC/MS, and the spectra obtained compared to known standards. Metabolites identified in rat urine (see below) were co-chromatographed with goat metabolites by TLC and HPLC to help assign structures to the minor metabolites which had undergone biological transformations. Rat-bile metabolites were also co-chromatographed with the rat urine metabolites to determine the site of hydroxylation for glucuronide conjugates that had similar mass spectra. The main components were

component A: 4-(2',2'-difluoro-1',3'-benzdioxol-4'-yl)-1*H*-pyrrole-3-carbonitrile 5-*O*-glucuronide (present in rat urine)

component B-1: 4-(2',2'-difluoro-1',3'-benzodioxol-4'-yl)-1*H*-pyrrole-3-carbonitrile 2-*O*-glucuronide (also present in rat urine)

component C: fludioxonil

component G: 4-(2',2'-difluoro-1',3'-benzdioxol-4'-yl)-1*H*-pyrrole-3-carbonitrile, 2- or 5-*O*-sulfate (sulfate conjugate of SYN 518577 or SYN 518578)

Component B-2 was tentatively identified when isolated from the kidney as 4-(2',2'-difluoro-1',3'-benzdioxol-4'-yl)-1*H*-pyrrole-3-carbonitrile, 7'-*O*-glucuronide on the basis of similar chromatography to a biosynthetic standard from rat bile. HPLC retention times changed after isolation by TLC or HPLC of two labile metabolites in the liver, L-1 and L-2. Storage of a liver extract sample in HPLC mobile phase (formate buffer, pH 6.5) resulted in disappearance of these peaks. Attempts to stabilize them were unsuccessful so they could not be identified. They are presumed to be reactive intermediates.

The total radioactivity identified as components A, B-1, B-2, C, and/or G ranged from 14% in liver to 83% in fat (Table 5).

Table 5. Identified metabolites in the tissues and milk of goats dosed with 150 mg/day of [pyrrole-4-^{14}C]fludioxonil for four consecutive days (Peffer, 1992a, Report F-00088).

Sample	Mg/kg as fludioxonil and (% of TRR)								
	B-1	B-2	Total B	A	C	G	L-1	L-2	Total % of identified TRR
Kidney	0.67 (23)	0.23 (7.9)	0.90 (31)	0.44 (15)	0.05 (1.7)	0.02 (0.7)			48
Liver					0.86 (14)		0.6 (9.8)	0.33 (5.3)	29
Muscle (tenderloin)									
Animal 78			0.003 (5.6)	0.001 (2.3)	0.01 (24)	0.01 (22)			53
Animal 80					0.04 (43)	0.007 (7.2)			50
Omental fat					0.21 (83)				83
Milk, day 3	1.32 (65)	Trace	1.32 (65)			0.28 (14)			78

The metabolism of ^{14}C pyrrole-labelled CGA 173506 in ruminants appears to involve hydroxylation of the pyrrole ring at the 2- or 5- position, followed by the formation of either glucuronide or sulfate conjugates. The free hydroxylated-pyrrole compounds, themselves intermediates, were not observed, presumably owing to inherent instability.

To determine the stability of metabolites a sample of liver was extracted four times with acetonitrile (ACN) and the extract concentrated and analysed in two different TLC solvent systems, and a day-3 urine sample was filtered and analysed directly using the 2 TLC systems. Analysis was initially within one month of receipt of the samples at the laboratory (January 1990), and again after all tissue extracts had been profiled (January 1991). The profiles produced were sufficiently similar to suggest that the metabolites were stable throughout the study.

Laying hens. A study (Peffer, 1992, Report F-00089) on the metabolism of fludioxonil in White Leghorn hens was reported to the Meeting, with amendments subsequently issued (Peffer, 1994; 1996; Archive CGA173406/0237).

After a seven-day acclimatizing period, five laying hens were dosed with gelatine capsules (10 mg of ^{14}C-fludioxonil (^{14}C-pyrrole-labelled) per day) for eight consecutive days, with a sixth control hen. Based on feed consumption, the dose was equivalent to an average of 89 ppm (76 ppm to 106 ppm) in the diet (target dose: 100 ppm). Eggs and excreta were collected daily from day -2 to day 8 and the hens were killed 6 hours after the last dose. Blood samples were taken immediately before the hens were killed, and thereafter samples of lean meat (breast and thigh muscle), skin plus attached fat, peritoneal fat, and liver, gizzard, kidneys, and heart were taken.

The TRR in the tissues and excreta were determined by combustion and liquid scintillation counting (LSC) after homogenization in the presence of solid CO_2, in egg yolks and whites and plasma by LSC, and in whole blood by LSC after solubilization in a mixture of Soluene and ethanol and treatment with H_2O_2 and HCl.

Samples were extracted by several methods, similar to those used in the ruminant study. Liver and muscle samples were extracted four times with acetonitrile and then four times with water. In a second method used to extract larger amounts of radioactivity for characterisation, liver samples were extracted with acetonitrile followed by overnight Soxhlet extraction with methanol. The resultant

tissue pellet was divided into two portions and treated with either 6N HCl or 15% KOH aqueous solutions. The acid and base hydrolysates were extracted with ethyl acetate. Kidney samples were extracted four times with acetonitrile and then three times with water. Egg yolk samples were extracted by solid-phase dispersion: homogenised specimens were mixed with C-18 silica and packed into a glass syringe barrel and the mixture was then extracted by elution with hexane, ethyl acetate/acetonitrile (1:3), methanol and water. Egg white was extracted four times with acetonitrile. Skin samples with attached fat were extracted with hexane followed by five extractions with acetonitrile and two extractions with water.

Characterisation of extracted radioactivity was accomplished using TLC and HPLC. In some instances the extracts were treated with enzymes (β-glucuronidase or sulfatase) to characterise conjugated metabolites. The unextracted residue from the liver, kidney and muscle samples was further characterised by treatment with protease and extraction at different pHs with ethyl acetate, with and without reaction with 2,4-dinitrofluorobenzene.

Identifications were by co-chromatography (TLC and HPLC), NMR and MS.

A mean of 102% (88% to 112%) of the total administered radiolabelled dose was eliminated in the excreta.

The results are shown in Tables 6 and 7.

Table 6. Radioactive residues (as fludioxonil equivalents) found in tissues and blood of hens dosed with 10 mg/hen/day of [pyrrole-4-^{14}C]fludioxonil for eight consecutive days (Peffer, 1992, Report F-00089).

Sample	Residue (mg/kg)		Limit of detection (mg/kg)
	Mean (n = 5)	Std. dev.	
Liver	8.9	7.3	0.011
Peritoneal fat	0.17	0.06	0.013
Lean meat			
Breast muscle	0.11	0.02	0.005
Thigh muscle	0.12	0.03	0.006
Skin plus attached fat	0.25	0.08	0.008
Other			
Plasma	2.4	0.82	0.006
Whole blood	1.8	0.68	0.003
Gizzard	11.	4.0	0.009
Kidneys	5.3	2.3	0.011
Heart	1.09	0.78	0.014

Table7. Radioactive residues (as fludioxonil equivalents) in the egg yolks and whites of hens dosed with 10 mg/hen/day of [pyrrole-4-^{14}C]fludioxonil for eight consecutive days (Peffer, 1992, Report F-00089).

Day	Residue (mg/kg)			
	Egg yolks		Egg whites	
	Mean (n = 5)	Std. deviation	Mean (n = 5)	Std. deviation
-2	<0.005	-	<0.002	-
-1	<0.005	-	<0.002	-
1	<0.005	-	0.018	0.016
2	0.41	0.52	0.035	0.018
3	0.55	0.33	0.041	0.021
4	1.53	0.73	0.045	0.019
5	1.8 (n=3)	0.99	0.038	0.020
6	1.70	0.65	0.046	0.017
7	1.85	0.58	0.054	0.027
8	2.2 (n=2)		0.043	

Acetonitrile and methanol extracted 61% (65% corrected for recovery) of the TRR in liver. Acid and base solubilized the unextracted rsidues, yielding 9.6 and 16% of the TRR respectively as organosoluble material. ACN and water extracted 64%-69% of the TRR in lean meat and 46% in kidney. Acetonitrile extracted 74% of the TRR in egg whites, and solid-phase dispersion 88% of that in the yolks. The results for tissues, eggs, and excreta are shown in Table 8.

Table 8. Extraction of radioactive residues in the tissues, eggs and excreta of hens dosed with 10 mg/hen/day of [pyrrole-4-^{14}C]fludioxonil for eight consecutive days (Peffer, 1992, Report F-00089).

Sample and (mg/kg TRR as fludioxonil)	% of TRR										
	Hex-ane	Aceto-nitrile	EtOAc/ ACN	MeOH	Water	MeOH Soxhlet	Unex-tracted pellet	Base hydrolysis[1] (15% KOH, 95ºC, overnight)		Acid hydrolysis (6N HCl, room temperature, 3 h)	
								organic	aqueous	organic	aqueous
Liver (8.9)		36				24.7	33	16	16	9.6	29
Kidney (5.3)		33			12		54				
Breast muscle (0.11)		62			2.3		36				
Thigh muscle (0.12)		62			6.3		31				
Skin with attached fat (0.15)	4.3	42			9.2		45				
Excreta				24	7.9						
Egg white (0.043)		74					26				
Egg yolk [2] (2.2)	2.6		68	15	2.4		12				

[1] Hydrolysate characterised as polar, acidic compounds by HPLC and TLC.
[2] Yolk mixed with C-18 material, transferred to syringe barrel and eluted sequentially with hexane, ethyl acetate/acetonitrile (1:3), methanol and water.

The radioactivity remaining in the tissue pellets after extraction of liver, kidney and breast muscle samples, 33%, 54%, and 36% of the TRR respectively, was solubilized with protease and characterised by treatment with DNFB and extraction with ethyl acetate at pH 2 and at pH 9. Approximately 54% and 63% of the previously unextracted radioactivity in liver and kidney samples and 67% of that in muscle was solubilized by protease, and of this, treatment with DNFB at pH 2 released 50%, 61% and 56% respectively extractable with ethyl acetate at pH 2 compared with 25%, 33% and 34% at pH 2 from the underivativised samples (Table 9). These results suggest that a portion of the radioactivity remaining in the pellet after solvent extraction represents covalent binding of reactive metabolite(s) to endogenous material, some of which is protein. HPLC showed the radioactivity in protease-treated liver to be highly polar and unresolved. Further identification was not pursued.

Table 9. Protease treatment of acetonitrile and water-extracted liver, kidney and muscle from hens dosed with 10 mg/hen/day of [pyrrole-4-^{14}C]fludioxonil for eight consecutive days (Peffer, 1992, Report F-00089).

Sample (extracted tissue pellets)	Protease treatment (radioactivitiy in extracted pellet)		Treated with DNFB (% of supernatant extracted into ethyl acetate, normalized for total recovery)		Untreated (% of supernatant extracted into ethyl acetate, normalized for total recovery)	
	% supernatant	% pellet	pH 9	pH 2	pH 9	pH 2
Liver	54	57	10	50	6.6	25
Kidney	63	43	41	61	23	33
Tenderloin muscle	67	-	14	56	24	34

Metabolites, isolated primarily from excreta, were identified by mass spectrometry (with GC or HPLC) and/or NMR. Additional information was obtained by glucuronidase or sulfatase incubations. The metabolites in the excreta were co-chromatographed with tissue and egg extracts, and in addition metabolites in rat urine identified by mass spectrometry were co-chromatographed with the poultry tissue and egg metabolites using TLC and HPLC. Biosynthetic standards were also co-chromatographed with the rat-urine metabolites to determine the site of hydroxylation in glucuronide conjugates that had similar mass spectra.. The following compounds were identified:

metabolite P1	CGA-192155: 2,2-difluorobenzo[1,3]dioxole-4-carboxylic acid
metabolite P2/P3	Form 1 SYN 518577: 4-(2,2-difluorobenzo[1,3]dioxol-4-yl)-2-hydroxy-1H-pyrrole-3-carbonitrile
	Form 2: CGA 344623: 1 2-cyano-3-(2,2-difluoro-benzo[1,3]dioxol-4-yl)succinamic acid
metabolite P7	CGA 344624: 2-(2,2-difluorobenzo[1,3]dioxol-4-yl)-2-oxoacetamide
E1	parent fludioxonil
metabolite E2	CGA-265378: 4-(2,2-difluorobenzo[1,3]dioxol-4-yl)-2,5-dioxo-2,5-dihydro-1H-pyrrole-3-carbonitrile
metabolite F	SYN 518576: 4-(2,2-difluoro-7-hydroxybenzo[1,3]dioxol-4-yl)-1H-pyrrole-3-carbonitrile
metabolite G	CGA-335892: 4-(2,2-difluoro-benzo[1,3]dioxol-4-yl)-1-hydroxy-1H-pyrrole-3-carbonitrile
metabolite I-1	sulfate conjugate of CGA-335892
metabolite I-2	Sulfate conjugate of SYN 518577,4-(2,2-difluorobenzo[1,3]dioxol-4-yl)-2-hydroxy-1H-pyrrole-3-carbonitrile or SYN 518578, 4-(2,2-difluorobenzo[1,3]dioxol-4-yl)-5-hydroxy-1H-pyrrole-3-carbonitrile

The analyses suggested the presence of additional polar metabolites P4, P5, P6, P7, P8, P9, P10, A, B, C, and D, although definitive identification was not possible, mainly because insufficient activity was present. Structures for P7, P10 and D were tentatively assigned.

Characterisation of the extracted residues by several HPLC systems yielded approximately twenty peaks, .the distribution and quantification of which are shown in Table 10.

Table 10. Identification and characterisation of radioactive residues in the tissues and eggs of hens dosed with 10 mg/hen/day (ca. 89 mg/kg diet) of [pyrrole-4-^{14}C]fludioxonil for eight consecutive days.

Compound or fraction		Egg white	Egg yolk	Egg total	Liver	Kidney	Breast muscle	Thigh muscle	Skin + attached fat
P1:CGA 192155	mg/kg	0.003	0.012	0.006	0.206	n.a.[1]	n.a.	n.a.	n.a.
	% TRR	4.6	0.7	0.9	2.3	n.a.	n.a.	n.a.	n.a.
P2/P3: CGA 344623	mg/kg	0.015	0.168	0.067	0.531	n.a.	n.a.	n.a.	n.a.
	% TRR	28	9.1	10.	5.9	n.a.	n.a.		n.a.
P4	mg/kg	0.001	0.002	0.001	0.074	n.a.	n.a.	n.a.	n.a.
	% TRR	1.1	0.1	0.2	0.8				
P5	mg/kg	0.002	0.015	0.006	0.091	n.a.	n.a.	n.a.	n.a.
	% TRR	3.4	0.8	0.9	1.0				
P6	mg/kg	0.001		0.001	0.110	n.a.	n.a.	n.a.	n.a.
	% TRR	2.1		0.1	1.2				
P7:CGA 344624	mg/kg	0.004	0.023	0.010	0.304	n.a.	n.a.	n.a.	n.a.
	% TRR	6.6	1.2	1.5	3.4				
P8	mg/kg				0.233	n.a.	n.a.	n.a.	n.a.
	% TRR				2.6				
P9	mg/kg	<0.001		<0.001	0.149	n.a.	n.a.	n.a.	n.a.
	% TRR	0.7		<0.1	1.7				
P10	mg/kg				0.154	n.a.	n.a.	n.a.	n.a.
	% TRR				1.7				

Compound or fraction		Egg white	Egg yolk	Egg total	Liver	Kidney	Breast muscle	Thigh muscle	Skin + attached fat
A	mg/kg	n.a.	n.a.	n.a.	n.a.	0.376			
	% TRR					7.1			
B	mg/kg	n.a.	n.a.	n.a.	n.a.	0.063	0.005		
	% TRR					1.2	4.3		
C	mg/kg	n.a.	n.a.	n.a.	n.a.	0.151			
	% TRR					2.9			
D: glucuronide conjugate of SYN 518577/518578	mg/kg				0.166	0.247			
	% TRR				1.9	4.7			
E1: fludioxonil	mg/kg		0.041			0.135	0.032	0.010	0.025
	% TRR		2.2			2.6	30	7.9	9.8
E2:CGA 265378	mg/kg				0.113				
	% TRR				1.3				
F:SYN 518576	mg/kg				0.235	0.146			
	% TRR				22.6	2.8			
G: CGA 335892	mg/kg	<0.001	0.025	0.008	0.073	0.121			
	% TRR	0.4	1.3	1.3	0.8	2.3			
I1: sulfate conjugate of CGA-335892	mg/kg	0.001	0.780	0.264	0.046	0.070	0.012	0.036	0.036
	% TRR	1.4	42	40	0.5	1.3	11	30	14
I2: sulfate conjugate of SYN 518577/518578	mg/kg	<0.001	0.258	0.087			0.004	0.008	0.012
	% TRR	0.4	14.0	13.3			3.9	6.9	4.9
% of TRR in analysed extracts		61	83.	n.a.	52	33.	62	62	33
% of TRR identified		42	71	69	24	14	44	45	29

[1] n.a.: not applicable

Fludioxonil was present in muscle (7.9-28% of the TRR) and skin plus attached fat (9.8%). It accounted for 1.2% of the TRR in the liver, 2.6% in kidney and 2.2% in egg yolk (equivalent to 2.1% of the egg TRR).

Unextracted radioactivity was 33% (35% corrected for recovery) of the TRR in liver, 5% of the TRR in kidney, 45% in skin with attached fat, and 31-36% of the TRR in muscle. Further treatment of liver, kidney and muscle with protease and with acid or base in the case of liver released a significant proportion (<50%). Lesser amounts of unextracted ^{14}C were found in egg whites (26%) and yolks (12%).

Rats. In four studies on the fate of fludioxonil (Thanei, 1992, Report 12/92; Bissig, 1990, Report 32/90; Thanei, 1995, Report 13/93, and Muller and Thanei, 1995, Report 4/95) six metabolites were identified in the urine, faeces and bile (see Table 11).

Table 11. Approximate amounts of fludioxonil metabolites in the rat (Thanei, 1992, Report 12/92; Bissig, 1990, Report 32/90; Muller and Thanei, 1995, Report 4/95; and Thanei, 1995, Report 13/93).

Designation	Identification	Fraction[1]	% of applied dose	
			male	female
SYN 518577	4-(2,2-difluorobenzo[1,3]dioxol-4-yl)-2-hydroxy-1H-pyrrole-3-carbonitrile			
MET 1G (glucuronyl-conjugate)	2-β-D-glucuronyl-4--(2,2-difluoro-benzo[1,3] dioxol-4-yl)-1H-pyrrole-3-carbonitrile	U11, G6	0.5-0.8	0.5-56
MET 2G (sulfate conjugate)	4-(2,2-difluorobenzo[1,3]dioxol-4-yl)-1H-pyrrole-3-carbonitrile-2-hydrogen sulfate	U10, G5	0.5-0.8	0.7-1.1
total			*1.2-1.6*	*1.5-56.7*

Designation	Identification	Fraction[1]	% of applied dose	
			male	female
SYN518578	4-(2,2-difluoro-benzo[1,3]dioxol-4-yl)-5-hydroxy-1H-pyrrole-3-carbonitrile			
MET 3G (glucuronyl-conjugate)	4-(2,2-difluoro-benzo[1,3]dioxol-4-yl)-5-β-D-glucuronyl-1H-pyrrole-3-carbonitrile	U9, G4	0.6-0.9[3]	1.0[3]-2.8
MET 1U (sulfate conjugate)	4-(2,2-difluoro-benzo[1,3]dioxol-4-yl)-1H-pyrrole-3-carbonitrile-5-hydrogen sulfate	U13	1.8-2.2	1.1-1.9
total			*2.4-2.8*	*2.5-3.9*
SYN 518576	4-(2,2-difluoro-7-hydroxy-benzo[1,3]dioxol-4-yl)-1H-pyrrole-3-carbonitrile			
MET 4G (glucuronyl-conjugate)	4-(2,2-difluoro-7--β-D-glucuronyl-benzo[1,3] dioxol-4-yl)-1H-pyrrole-3-carbonitrile	U6, G2	1.1-4.8	2.2 0.9-1.5
SYN 518582 [= MET 1G(G4)]	4,4'-bis-(2,2-difluoro-benzo[1,3]dioxol-4-yl)-5,5'-dioxo-1,5,1',5'-tetrahydro-[2,2']bipyrrolylidene-3,3'-dicarbonitrile	U19	0.2-0.4 (3.2)	0.1-0.7 (3.5)

[1] U: Urine, G: Bile

The postulated metabolic pathways of fludioxonil in goats, hens and rats are shown in Figure 1.

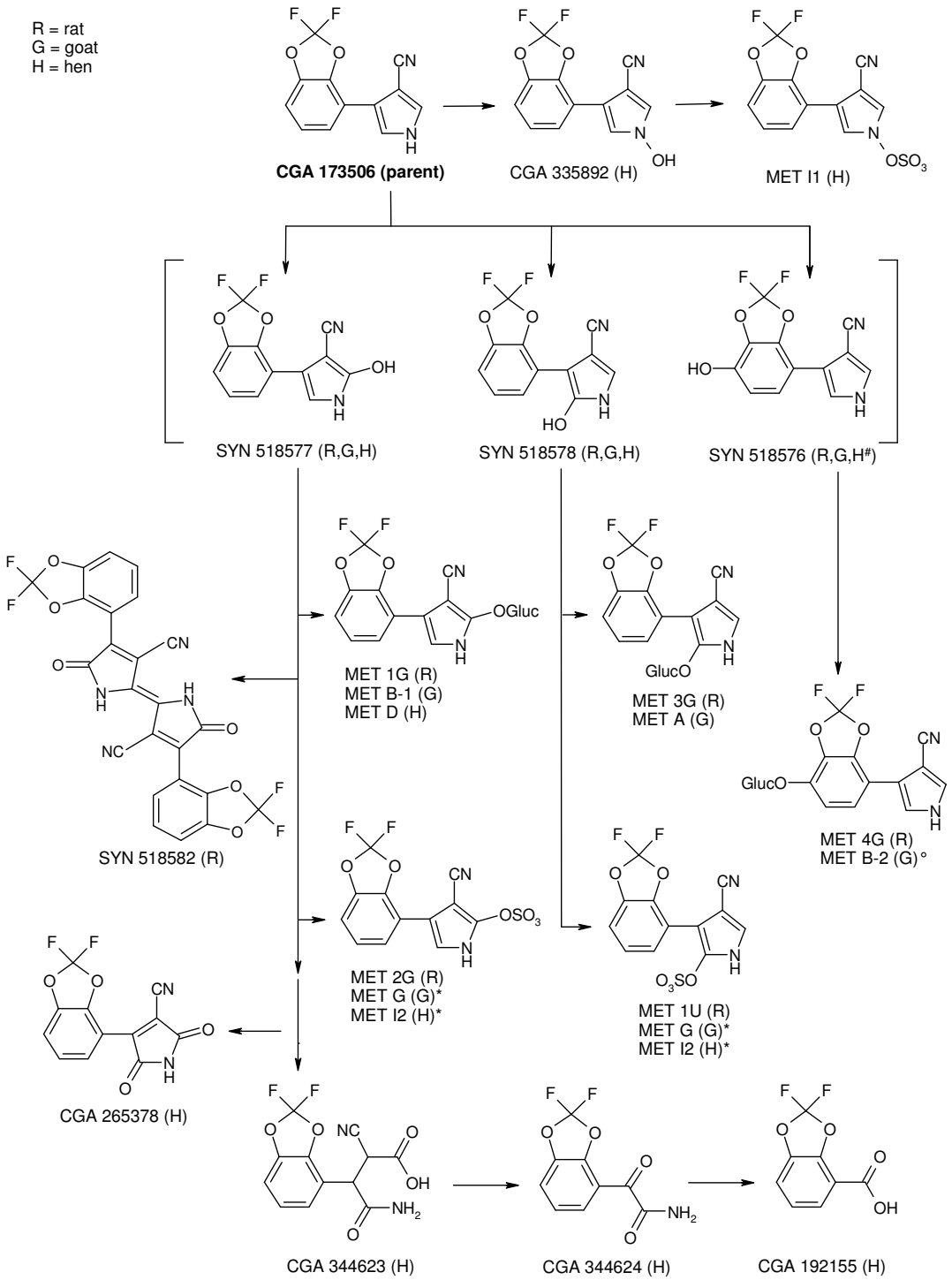


Figure 1: Postulated metabolic pathways of fludioxonil in rats, hens and goats.

Plant metabolism

Studies on the metabolism of fludioxonil when applied as a seed treatment and as foliar treatment were reported to the Meeting but no information was provided on post-harvest degradation. Pyrrole and phenyl-labelled compounds were used with radiochemical purity >97–>99%.

Foliar application

Grapes (Nicollier, 1991, Report 3/91; 1993, Report 89GN14PR2). In a field study in Sisseln, Switzerland three grapevines (Riesling/Sylvaner) were sprayed three times at 3-week intervals with [pyrrole-4-^{14}C]fludioxonil formulated as WP 50 at an approximate rate of 500 g ai/ha (50 g ai/100 l), twice the typical maximum field use rate of 250 g ai/ha. The application to two of the vines was directed at the fruit, and to the third included the leaves.

Grapes and leaves were sampled 0-day (Interval 1) and 1 month (Interval 2) after the first application, and 0-day (Interval 3) and 14 days (Interval 4) and at maturity (Interval 5, 35 days) after the third.

The grapes and leaves were washed with methanol/water (1:1), and the grapes pressed. At harvest, a portion of the *unwashed* grapes was frozen, crushed and pressed. Unwashed grapes were fermented to produce wine. Grapes, presscake, seeds and leaves were homogenised with liquid nitrogen and the TRR was measured by combustion and liquid scintillation counting (LSC) of sub-samples. Methanol/water (80:10) followed by hot methanol (Soxhlet) were used to extract samples, and extracts and washes analysed by 1- or 2-dimensional TLC. Some samples were cleaned up first using silica gel column chromatography. Unextracted radioactivity was determined by combustion and LSC of the extracted residue. Enzyme cleavage was used to determine sugar-conjugated metabolites.

Ancillary cell culture and grape leaf incubation experiments were conducted to aid the identification of radiolabelled residues. Liquid chromatography employing silica gel or XAD-4 was used in the clean-up stages. Metabolites were identified by TLC and HPLC, enzyme cleavage, and acetylation and methylation, and finally either by co-chromatography or mass spectrometry and NMR.

At harvest (Interval 5) fruit and leaves contained 56% and 43% of the radioactivity respectively immediately after the final application when 57% remained on the surface of the fruit and 32%, 2.6% and 8.2% was in the presscake, seeds, and juice respectively. The surface radioactivity on the grapes decreased from 87% shortly after the third application to 57% of the TRR at harvest. Total residues *after washing* and pressing the harvested grapes were 6.0 mg/kg as fludioxonil in the presscake (fludioxonil 2.0 mg/kg), 1.7 mg/kg in the seeds (fludioxonil 0.63 mg/kg), and 0.30 mg/kg in the juice (fludioxonil 0.14 mg/kg). Results are shown in Table 12.

Table 12. Distribution of radioactivity and residual fludioxonil (CGA 173506) after three applications of [pyrrole-4-^{14}C]fludioxonil (Nicollier, 1991, Report 3/91; 1993, Report 89GN14PR2).

Interval	Sample	Total residues (mg/kg) [1]	(%) [2]	Balance (%) [3]	Parent (mg/kg)	Surface (%) [4]	Extract (%) [4]	Soxhlet (%) [4]	Unext. (%) [4]	Total (%) [4]
0.5 h after 1st application Interval 1 (07/13/1989)	Leaves Grapes	4.9 21.	100 100		4.1 19.	87.9 98.5	11.8 1.4	0.3 0.1	0.0 0.0	100.0 100.0
26 days after 1st application, 1 day before 2nd Interval 2	Leaves Grapes	3.0 2.1	79 52		1.9 1.7	84.0 85.4	9.4 11.5	2.6 0.4	4.4 2.2	100 100

Interval	Sample	Total residues (mg/kg) [1]	(%) [2]	Balance (%) [3]	Parent (mg/kg)	Surface (%) [4]	Extract (%) [4]	Soxhlet (%) [4]	Unext. (%) [4]	Total (%) [4]
0.5 h after 3rd application	Leaves	12	100		10	92.8	4.8	1.5	1.8	101
	Grapes									
	wash	-		87.0		87.0				
Interval 3	cake	2.1		9.0	1.0		83.1	3.5	12.0	99
(08/30/1989)	juice	0.28		4.0	0.12		100.0			100
	Total Grapes	5.0	100	100.0	4.5	87.0	11.5	0.3	1.1	100
	Leaves	13	120		11.	91	7.5	0.8	0.9	100
14 days after	Grapes									
3rd application	wash			74		74				
	cake	2.5		19	0.59		61.	5.1	42	110
Interval 4	juice	0.37		7.5	0.17		100			100
	Total Grapes	3.4	64	100.	2.4	74	19.	1.0	8.0	102
	Leaves	5.2	42.		3.6	52.	42.	1.8	3.8	100.
35 days after 3rd	Grapes									
application	wash	-		57.		57.				
	cake	6.0		32.	2.02		67.	4.4	23.	94.
Interval 5	seeds	1.7		2.6	0.63		74.	4.0	16.	94.
(10/04/1989)	juice	0.30		8.2	0.14		100.			100.
	Total Grapes	2.8	58	100	2.0	57	31.	1.5	7.6	98

n.a.: not analysed.
[1] Fludioxonil equivalents.
[2] Leaf and grape radioactivity at interval 2 as % of that recovered at interval 1, and at intervals 4 and 5 as % of that recovered at interval 3
[3] 100%: total ^{14}C found in the grapes
[4] % relative to combustion value except for interval 1 where it was determined by sum of extracted + unextracted radioactivity

Unextracted total grape radioactivity increased from 1.1% shortly after the third application to 7.6% of the TRR at harvest, and from 1.8% to 3.8% of the TRR in the leaves at harvest.

Unwashed harvested grapes used for juice contained total radioactive residues of 2.5 mg/kg, including 1.7 mg/kg fludioxonil. Pressing these grapes demonstrated that 77% of the radioactivity remained in the presscake, 23% in the juice, and combined residues were 10 mg/kg (fludioxonil-6.8 mg/kg) and 0.77 mg/kg (fludioxonil 0.59mg/kg) respectively. Wine and sediment residues were 0.43 mg/kg (fludioxonil 0.34 mg/kg) and 53 mg/kg (fludioxonil 39. mg/kg) respectively. Results are shown in Tables 13 and 14.

Table 13: Distribution of radioactivity in unwashed grapes at harvest after three applications of [pyrrole-4-^{14}C]fludioxonil, PHI 35 days (Nicollier, 1991, Report 3/91; 1993, Report 89GN14PR2).

Sample	Total residues (mg/kg) [1]	Balance (%) [2]	Parent (mg/kg)
Grapes			
Cake	10	77	6.8
Juice	0.77	23	0.59
Total Grapes	2.5	100	1.7

[1] Fludioxonil equivalents.
[2] % of total radioactivity found in grapes

Table 14. Distribution of radioactivity in wine from grapes at harvest after three applications of [pyrrole-4-^{14}C]fludioxonil, PHI 35 days (Nicollier, 1991, Report 3/91; 1993, Report 89GN14PR2).

Sample	Total Residues (mg/kg)[1]	Parent (mg/kg)[1]	Balance (%)[2]	Extracted radioactivity (%)[3]	Soxhlet (%)[3]	Unextracted (%)[3]	Total (%)[3]
Juice	0.77						
Wine	0.43	0.34	60	100	-	-	100
Sediment	53	39	40	91	1.2	6.1	98
Juice total	0.72	0.55	100				

[1] Fludioxonil equivalents
[2] % relative to total radioactivity found in juice.
[3] % of radioactivity in individual processed fractions (wine and sediment)

90-100% of the radioactive residues in the grapes could be extracted with organic solvents (Table 14). Methanol/water extracted 61% to 83% of the TRR in the presscake and 74% of the TRR in the harvested seeds, and Soxhlet extraction an additional 3.5% to 5.1% from the former and 4% from the latter.

The partitioning of extracted residues is shown in Table 15.

Table 15. Partitioning of extracted radioactivity in vines and processed grapes after three applications of [pyrrole-4-^{14}C]fludioxonil (Nicollier, 1991, Report 3/91; 1993, Report 89GN14PR2).

Sample	Extracted radioactivity (%)		
	Organic phase	Water phase	Total
Leaves	81	13	94
Juice	58	51	110
Cake	78	16	95
Seeds	78	18	96
Wine	63	34	97
Sediment	94	3.2	97

TLC analysis of the extracted radioactive residues from the juice, seeds and presscake yielded numerous components (Table 16). Fludioxonil constituted 70% of the radioactivity in the whole fruit with 16 further components, none of which exceeded >2.4% of the TRR. The metabolites SYN 518579 (II_2), SYN 518581 (I_{3b}), SYN 518580: (II_{3a}), CGA 344623: (I_4), CGA 308103: (I_{11}) and the glucose conjugate of CGA 308103: (I_6) were identified. Fewer metabolites were found in the leaves than in the fruit, with fludioxonil representing 69% of the TRR (Table 16).

TLC quantification showed that fludioxonil represented most of the radioactivity in wine and sediment (79% and 74% respectively**,** Table 17)**.** The metabolites were the same as those in the juice. None were >3.1% of the radioactivity in the processed fraction.

Table 16. Quantification of metabolites in grapes at harvest and separated fractions after three applications of [pyrrole-4-^{14}C]fludioxonil (Nicollier, 1991, Report 3/91; Nicollier, 1993, Report 89GN14PR2).

Sample	Metabolite fractions (%) [1,2,3]																		Par-ent II_4	Un-re-solved	Sub-total	Soxh-let (%) [2]	Unext. (%) [2]	Total (%) [2]
	I_1	I_2	I_3 * I_{3b}	I_4	I_5	I_6	I_7	I_8	I_9	I_{10}	I_{11}	I_{12}	I_{13}	I_{14}	I_{15}	II_1	II_2	II_3						
Grapes																								
Washings a)			0.3									0.5					1.7		96	1.8	100.	-	-	100.
Washings b)			0.2									0.3					1.0		55	1.1	58.	-	-	58.
Juice a)	9.7	2.6	8.6	2.0	1.8	2.9	2.3	1.1	0.9	1.0	1.4	2.0	1.0	1.7		0.8	3.2	1.5	45.	19.	109.	-	-	109.
Juice b)	0.8	0.2	0.7	0.2	0.1	0.2	0.2	0.1	0.1	0.1	0.1	0.2	0.1	0.1		0.1	0.3	0.1	3.7	1.6	9.0	-	-	9.0
Cake a)	4.0		1.6	0.4		0.2	0.8	-	0.2	0.2	0.2	0.2	-	0.3		3.1	1.0	2.0	34.	15.	64.	4.4	23.	91.
Cake b)	1.3		0.5	0.1		0.1	0.3	-	0.1	0.1	0.1	0.1	-	0.1		1.0	0.3	0.4	11.	4.9	20.	1.4	7.2	29.
Seeds a)	4.0	0.5	2.6	0.5		0.3	1.7	0.8	0.9	0.5	-	-	0.5	0.6	0.5	-	3.7	1.6	36.	17.	72.	4.0	16.	92.
Seeds b)	0.1	-	0.1	-	-		0.1	-	-	-	-	-	-	-	-	-	0.1	0.1	0.9	0.4	1.8	0.1	0.4	2.3
Whole grape	2.4		1.5	0.2	0.2	0.3	0.6	0.1	0.2	0.2	0.2	0.6	0.1	0.2		1.1	1.7	0.6	70.	8.0	88.	1.5	7.6	98.
Leaves																								
Washings	0.2		0.2	-															50.	1.5	52.	-		520
Leaves	1.6	0.8	2.4	-		0.9	0.1	0.2	-	-	-	-	-	0.3	-	-	1.8	5.6	19.	6.6	39.	1.8	3.8	45.
Total	1.8	0.8	2.6	-		0.9	0.1	0.2	-	-	-	-	-	0.3	-	-	1.8	5.6	69.	8.1	91.	1.8	3.8	97.

n.d.: not detected or <LOQ (<0.003 mg/kg)
a) % of radioactivity in individual fruit parts
b) % of radioactivity in whole grape
[1] Quantification by 2-dimensional TLC in two solvent systems.
[2] % of sum of surface and penetrated radioactivity
[3] Metabolite fractions numbered according to position in systems SS I and SS II
* In identification phase, zone I_3 was renamed I_{3b} to differentiate it from an I_3 zone in wheat
Metabolite I_{3b}: SYN 518581
Metabolite I_4: CGA 344623
Metabolite I_6:glucose conjugate of CGA 308103
Metabolite I_{11}: CGA 308103.
Metabolite II_2: SYN 518579
Metabolite II_3 contains trace amounts of II_{3b} identified as CGA 265378.
Metabolite II_{3a}: SYN 518580

Table 17. Quantification of metabolite fractions in wine and grape sediment from grapes at harvest after three applications of [pyrrole-4-^{14}C]fludioxonil (Nicollier, 1991, Report 3/91; 1993, Report 89GN14PR2).

Sample	Metabolite Fractions (%) [1,2,3]																		Par-ent II_4	Un-re-solved	Sub-total	Soxh-let (%) [2]	Unext. (%) [2]	Total (%) [2]
	I_1	I_2	I_3 * I_{3b}	I_4	I_5	I_6	I_7	I_8	I_9	I_{10}	I_{11}	I_{12}	I_{13}	I_{14}	I_{15}	II_1	II_2	II_3						
Wine	1.7		0.6	3.1	0.6	1.3		0.3			2.1					1.9	2.1		79	4.4	97.	-	-	97.
Sediment	0.3	0.4	0.6	0.2						0.1	0.5					2.1	1.5	2.3	74.	6.6	88	1.2	6.1	96.

[1] Quantification by 2-dimensional TLC using systems SS I and SS II
[2] % of total radioactivity found in individual parts
[3] Metabolite fractions according to position in two solvent systems.
* In identification phase, zone I_3 was renamed I_{3b} to differentiate it from an I_3 zone identified in wheat
Metabolite I_{3b}: SYN 518581
Metabolite I_4: CGA 344623
Metabolite I_6:glucose conjugate of CGA 308103
Metabolite I_{11}: CGA 308103.
Metabolite II_2: SYN 518579
Metabolite II_3 contains trace amounts of II_{3b}, CGA 265378.
Metabolite II_{3a}: SYN 518580

Tomatoes. In a trial on greenhouse-grown plants (Krauss, 1992, Report 1/92, addenda 1, 04/07/1995 and 2, 12/11/1966) eight 2- to 3-week old Capello plants were sprayed three times at two-week intervals with ^{14}C-fludioxonil formulated as WP 50 at a rate of 50g ai/ 100 l/application (1500 l/ha), equivalent to 750 g ai/ha/application. This is a grossly exaggerated rate for field uses.

Samples of tomatoes and leaves were collected at the first (Interval 1) and last (Interval 2) application. At maturity (Interval 3, harvest, 40 days after the last application) all tomatoes and leaves were sampled.

Tomatoes were washed with methanol/water (1:1) to determine surface residues, frozen in liquid nitrogen and homogenised for analysis. The sum of the radioactivity found in the washings and in the fruit (by combustion) was considered as the total ^{14}C-residue. Homogenised plant material was extracted with methanol/water (8:2) by blending and shaking, followed by Soxhlet extraction with methanol. The remainder was combusted to determine the unextracted residue. The combined methanol/water extracts were concentrated and partitioned with dichloromethane. The resultant organic and aqueous phases were analysed by two-dimensional thin-layer chromatography (2-D TLC). Liquid chromatography using Amberlite XAD-4 resin, HPLC, and enzyme cleavage were employed for clean-up and isolation. The metabolites were also compared by 2-D TLC with those obtained in the grape study. The grape metabolites were identified by spectroscopic means.

The distribution of TRR in the tomatoes is summarised in Table 18.

Penetration of radioactivity from the treated fruit surface to the interior of the fruit increased over time. Surface radioactivity represented 84%, 72% and 41% of the fruit TRR after the first application, after the last application, and at harvest (40-day PHI) respectively. The residues in the tomatoes decreased from 0.36 mg/kg shortly after the final application to 0.28 mg/kg at harvest, and in the leaves from 11 mg/kg to 7.0 mg/kg.

Extraction of radioactivity from fruit at all three sampling intervals was almost quantitative (93% to 102% of the TRR).

Table 18. Total radioactivity and residual ^{14}C-fludioxonil in greenhouse-grown tomato plants after three foliar applications of [pyrrole-4-^{14}C]fludioxonil at approximately 50g ai/100 l/application (1500 l/ha/application) (Krauss, 1992, Report 1/92, and addenda 1, 04/07/1995 and 2, 12/11/1966).

Interval	Sample	Total residue (mg/kg) [1]	Fludioxonil (mg/kg)	Surface (%)	Non-surface			Total (%)
					Cold ext. (%)	Soxhlet (%)	Unextracted. (%)	
0 days after application 1 Interval 1	Tomatoes	0.20	0.19	84.	16.	0.2	0.1	100.
	Leaves	5.8	5.4		97.	0.6	0.2	98
28 days after application 1 0 days after application 3 Interval 2	Tomatoes	0.36	0.31	72.	29.	0.6	0.9	102
	Leaves	11.	9.5		95.	0.2	0.6	96
68 days after application 1 40 days after application 3 Interval 3	Tomatoes	0.28	0.20	41.	50.	2.0	5.8	98
	Leaves	7.0	4.8		92.	1.2	4.2	98.

LOQ for combustion: 0.001 mg/kg, LOQ for TLC analysis of plant material: 0.001 mg/kg
[1] fludioxonil equivalents: Tomatoes: sum of surface rinse and extracts. Leaves: direct combustion (no rinse).

Most of the radioactivity from the harvested tomatoes was organosoluble with 88% of the radioactivity of the surface wash and 79% of the fruit extract partitioning into the organic phase.

Water-soluble radioactivity was 3.7% and 12% respectively. Leaf extracts showed 90% organosoluble and 8.5% water-soluble radioactivity.

The metabolite pattern in tomatoes was complex. Fludioxonil represented 73% of the radioactivity in the harvested fruit. TLC analysis produced 11 metabolite zones in addition to fludioxonil (Table 19). In total the metabolite fractions represented 6.6% of the total radioactivity, with no single fraction above 1.6% of the TRR. Metabolites SYN 518579 (III_2), SYN 518581 (I_{3b}), SYN 518580: (III_3), CGA 344623: (I_4) and the glucose conjugate of CGA 308103: (I_6) were identified by 2-D TLC comparison with the metabolites found in grapes together with metabolite CGA 192155 (I_5), identified by co-chromatography with its authentic reference standard. The identified metabolites represented a total of 3.6% of the TRR in tomato fruit. Trace amounts of sugar conjugates (approximately 0.2% of the TRR) could be detected in water-soluble material of the tomato extracts.

A similar metabolic pattern was found in the leaves. Fludioxonil represented 69% of the harvested leaf radioactivity. In total the metabolite fractions constituted 7.9% of the TRR in the leaves with 4.6% identified as SYN 518579 (III_2), SYN 518581 (I_{3b}), SYN 518580 (III_3), CGA 344623 (I_4), the glucose conjugate of CGA 308103 (I_6) and CGA 192155 (I_5).

At harvest (Interval 3) unextracted radioactivity in the fruit and leaves was 5.8% and 4.2% of the TRR respectively (Table 19).

Table 19. Quantification (TLC) of metabolite fractions in tomatoes at maturity after three foliar treatments with [pyrrole-4-^{14}C]fludioxonil (Krauss, 1992, Report 1/92 plus addenda 1, 04.07.1995 and 2, 12.11.1966).

Sample	Radioactivity in metabolite fractions (% of TRR)													Soxhlet (%)	Un-extracted (%)	Total (%)
	I_1	I_2	I_{3a}	I_{3b}	I_4	CGA 192155 I_5	I_6	I_{11}	III_1	III_2	III_3	Fludioxonil III_4	Un-resolved			
Tomatoes																
Surface (0.11 mg/kg)	1.0	0.2	--	1.0	0.2	0.2	-	-	-	1.0	0.7	85	3.2	-	-	93
Fruit (after wash) (0.16 mg/kg)	2.1	0.7	-	1.4	0.5	-	<LOQ [2]	0.2	1.4	2.1	-	67 [3]	10.	2.6 [4]	10.	98
Total fruit [1]	1.6	0.5	-	1.2	0.4	0.1	<LOQ [2]	0.1	0.8	1.6	0.3	73 [3]	7.3	1.5 [4]	5.8	94
Leaves (7.0 mg/kg)	0.6	0.1	0.2	0.7	0.2	0.1	0.1	-	2.4	1.8	1.7	69. [3]	12.	1.0 [4]	4.2	94

[1] % of total residues in surface and fruit.
[2] LOQ: 0.001 mg/kg.
[3] Includes parent content of Soxhlet and all other fractions.
[4] Parent content subtracted from Soxhlet fraction.

Metabolite I_{3b}: SYN 518581
Metabolite I_4: CGA 344623
Metabolite I_5: CGA 192155
Metabolite I_6: the glucose conjugate of CGA 308103
Metabolite III_2: SYN 518579
Metabolite III_3: SYN 518580

Peaches. US studies were conducted during the 1996 and 1997 growing seasons at the Northeast Research Station, Hudson, NY and Western Research Station, Sanger, CA. The test substance, uniformly labelled on the phenyl ring, was formulated as 50% WP and diluted in water/acetone (9:1) for application.

For each of the three tests an isolated branch on a mature peach tree grown outdoors that would yield ~2 kg of fruit and could be covered by a predetermined volume of formulated test substance to achieve complete coverage with minimal run-off was selected. In the NY tests three foliar applications were made at 280 or 2800 g ai/ha/application beginning at petal fall and repeated at intervals of 30 and 33 days for a total of 840 or 8400 g ai/ha/season. Samples of leaves were collected 0 and 28 days and mature fruit harvested 28 days after treatment. In the CA test [^{14}C]fludioxonil was applied at petal fall at 2100g ai/ha and again 35 days later at 6300 g ai/ha (total 8400g ai/ha/season, tenfold rate. Samples of leaves were collected 0 and 114 days after treatment and of immature and mature fruit 30 and 114 days after the second application.

Samples stored frozen at the test site were later shipped frozen, combusted for TRR determination within 14-65 days of harvest, and radioassayed by LSC. The LOQs for the radioassays were 0.005-0.006 mg/kg for the 1x NY test and 10x CA test, and 0.022 mg/kg for the 10x NY test. ^{14}C-Residues were <LOQ in all fruit and leaf control samples. ^{14}C residues in the fruit and leaves harvested 28 days after the last application at the two rates were 3.5 and 46. mg/kg in leaves, and 0.083 and 0.98 mg/kg in fruit, reflecting approximately the 10-fold difference in application rates. ^{14}C residues in fruit from the 10x CA test (0.26 mg/kg; 114-day PHI) were four times lower than those detected in the fruit from the 10x NY test (0.98 mg/kg; 28-day PHI); residues in the immature fruit from the 10x CA test 30 days after treatment were 0.83 mg/kg (Table 20).

Table 20. TRR in or on fruit and leaves of peaches harvested after 2-3 foliar applications of [phenyl^{14}C]fludioxonil, 0.84 or 8.4 kg ai/ha, 1 or 10x foliar rate (Peffer, Report 156-96, 1999).

Location	Sample	PHI (days)	Total radioactive residues (mg/kg)[1]	
			1x	10x
NY	Leaves	0	13	140
		28	3.5	46
	Mature fruit	28	0.083	0.98
CA	Leaves	0	Not determined	360
		114		38
	Immature fruit	30		0.83
	Mature fruit	114		0.26

[1] Mean of triplicate analyses as fludioxonil equivalents.

Radioactive residues in homogenized fruit and leaves were extracted 3-4 times with ACN/water/acetic acid (80:20:1), and the extracts combined, concentrated, and analysed by TLC and HPLC. Cold extraction released 88-101% of the TRR from fruit and >94% from mature leaves. A small amount remained in the post-extraction solids of 1x-treated fruit (9.5% of the TRR, 0.008 mg/kg), which was not further analysed.

The unextractable residues in 10x treated fruit (7-10% of the TRR, 0.026-0.069 mg/kg) were twice subjected to sequential microwave-assisted extraction, twice with 2-propanol/water (8:2) and once with 3.0 N HCl, each for a total of 37 minutes at sequential temperatures of 100°, 120° and 150°C. The released radioactivity in the alcohol and acid fractions was partitioned with methyl *tert*-butyl ether (MtBE), and the organosoluble ^{14}C-residues (1-7% of the TRR) were analysed by TLC and/or HPLC; the aqueous fractions (0.8-1.5% of the TRR) were not further analysed.

Extracts from 1x treated leaves were purified before analysis on a C_{18}-SPE column to remove plant pigments and other non-polar interferences. The column was eluted successively with ACN/water (80:20) and solvents such as ACN, MeOH, and $CHCl_3$. The ACN eluates and MeOH/$CHCl_3$ fractions were each combined, concentrated, and analysed by TLC and HPLC; components of the combined ACN fraction (71% of the TRR, 2.5 mg/kg) were resolved by TLC and HPLC. Radioactivity in the combined MeOH/$CHCl_3$ fraction (12.3% of the TRR, 0.43 mg/kg) was not resolved by TLC owing to extensive sample interference, and HPLC analysis produced peaks eluting after those from the standards and mature fruit extracts. The study author suggested that the late-eluting radioactivity may have been associated with sample components or perhaps incorporated in leaf pigments. As these peaks were not observed in fruit samples or in 10x treated leaves, they were not examined further. Unextractable radioactivity accounted for 9.5-13% of the TRR (0.45-4.4 mg/kg) in leaves and was not further analysed.

Extractable radioactive residues were analysed 2-D TLC on silica gel plates developed with ethyl acetate/1-propanol/water (62/24/12) and chloroform/methanol/formic acid/water (75:20:4:2) for definitive sample analyses and seven other solvent systems were also used for selected analyses. Radioactive residues in mature fruit extracts were detected using an AMBIS Radioanalytic Imageing System or Fuji BAS 1000 Phosphor Imager; visualized ^{14}C-residues were scraped from the plates and quantified by LSC. For TLC of leaf extracts radioactivity was quantified by direct integration using the AMBIS system. Identification was by comparison with fludioxonil, CGA-339833, CGA-344623, CGA-308103, CGA-192155, CGA-340351, CGA-265378, CGA-260766, CGA-257777, CGA-227731, CGA-308565, CGA-335892, CGA-336293, and CGA-339836 standards visualized with UV light. A C_{18} HPLC column equipped with an in-line radioactivity monitor and UV detection (271 nm) and using a mobile phase gradient of ACN to 0.05 M ammonium formate with 5% ACN was also used with additional HPLC systems for chromatography of selected isolated metabolites.

After an additional bulk extractions of 10x-treated fruit for further characterisation, selected aliquots of the XAD-4 organic eluants from aqueous extracts were incubated overnight in 0.1M NaOAc buffer (pH 4.6) at 37°C with either cellulase to cleave sugar-conjugated metabolites, or glucosidase to cleave specifically glucose-conjugated metabolites.

The following compounds were isolated from 10x treated fruit and leaves and identities determined using MS and/or NMR analysis: the parent compound, CGA-339833, the 2-keto-5-hydroxy and 2-hydroxy-5-keto analogues of fludioxonil, glucose conjugates of oxidized fludioxonil (metabolites P10a, P10b, P8b), the hexose conjugate of oxidized fludioxonil (metabolite P8), and CGA-339833.

The distribution and identification of ^{14}C-compounds in the fruit and leaves treated with [^{14}C]fludioxonil are shown in Tables 21 and 22.

Table 21. ^{14}C-residues in peaches harvested after 2-3 foliar applications of [phenyl-U-^{14}C]fludioxonil (Peffer, Report 156-96, 1999).

Compound or fraction	Fruit 1x (28-day PHI) (TRR 0.083 mg/kg)		Fruit 10x (28-day PHI) (TRR 0.98 mg/kg)		Fruit 10x (114-day PHI) (TRR 0.27 mg/kg)	
	% of TRR [1]	mg/kg [2]	% of TRR	mg/kg	% of TRR	mg/kg
Fludioxonil [3]	22	0.018	62	0.60	36	0.091
CGA-344623	3.7	0.003	0.8	0.008	2.8	0.007
CGA-339833	5.6	0.005	2.3	0.022	4.1	0.010
P8 (oxidized fludioxonil sugar conjugates)	1.3	0.001	0.6	0.006	0.7	0.002
P8b (oxidixed fludioxonil glucose conjugate)	0.4	<0.001	ND	--	ND	--

Compound or fraction	Fruit 1x (28-day PHI) (TRR 0.083 mg/kg)		Fruit 10x (28-day PHI) (TRR 0.98 mg/kg)		Fruit 10x (114-day PHI) (TRR 0.27 mg/kg)	
	% of TRR [1]	mg/kg [2]	% of TRR	mg/kg	% of TRR	mg/kg
P10a, P10b (oxidized fludioxonil glucose conjugates)	11	0.009	3.7	0.036	7.1	0.018
CGA-308103	3.7	0.003	1.4	0.014	1.4	0.004
2-keto-5-hydroxy-fludioxonil SYN518579			0.8	0.007	0.3	0.001
2-hydroxy-5-keto-fludioxonil SYN518579	1.6	0.001	1.4	0.014	0.7	0.002
CGA-192155	1.7	0.001	1.5	0.015	1.0	0.002
Total identified	**51**	**0.042**	**74**	**0.72**	**54**	**0.14**
CGA-308565[4]	2.0	0.002	2.1	0.021	0.9	0.002
Unknown TLC Regions [5]	21.5	0.018	9.5	0.093	18.9	0.048
Total characterised/identified	**74**	**0.062**	**86**	**0.84**	**74**	**0.19**
Unresolved [6]	21	0.018	14	0.14	15	0.038
Post-extraction Solids (PES)	9.5	0.008	7.1	0.069	10	0.026

[1] Uncorrected for recovery.
[2] [^{14}C]fludioxonil equivalents.
[3] CGA-265378 co-eluted with parent and was characterised by HPLC as possible minor metabolite in fruit accounting for 0.9-1.7% of the TRR.
[4] Tentative identification.
[5] 6-9 regions each consisting of ≤8.3 % of the TRR, except for the origin of 114-day PHI fruit (10.2% of the TRR; 0.026 mg/kg) which was shown, after enzyme treatment and A25 chromatography, to be a multicomponent mixture (each ≤0.013 mg/kg) including entrapped parent and neutral and acid metabolites.
[6] TLC quadrants 1-4 scraped into eight vials (two each quadrant) after scraping of major spots.

Table 22. ^{14}C-residues in peach leaves harvested 28 days after three foliar applications of [phenyl-U-^{14}C]fludioxonil (Peffer, Report 156-96, 1999).

Compound or fraction	Leaves (1x rate) (TRR 3.5 mg/kg)		Leaves (10x rate) (TRR 46. mg/kg)	
	% of TRR [1]	mg/kg [2]	% of TRR	mg/kg
Fludioxonil	3.6	0.13	67	30.
CGA-344623	4.0	0.14	0.9	0.41
CGA-339833	5.5	0.19	2.5	1.1
P8 (oxidized fludioxonil-sugar conjugate)	1.1	0.040	0.3	0.16
P8b (oxidized fludioxonil-glucose conjugate)	1.8	0.062	0.6	0.26
P10a, P10b (oxidized fludioxonil-glucose conjugates)	4.0	0.14	1.8	0.82
CGA-308103	2.6	0.092	1.4	0.62
2-keto-5-hydroxy-fludioxonil	1.4	0.050	1.2	0.57
2-hydroxy-5-keto-fludioxonil	5.4	0.19	3.2	1.4
CGA-192155	1.7	0.060	1.6	0.72
Total identified	**31**	**0.90**	**80**	**37.**
CGA-308565[3]	4.5	0.16	3.9	1.8
CGA-265378	4.8	0.17	5.7	2.6
CGA-339833 [4]	4.0	0.14	3.0	1.4

Compound or fraction	Leaves (1x rate) (TRR 3.5 mg/kg)		Leaves (10x rate) (TRR 46. mg/kg)	
	% of TRR [1]	mg/kg [2]	% of TRR	mg/kg
CGA-344623 [4]	1.3	0.045	0.6	0.26
Unknown TLC Regions [5]	27	0.95	13	6.0
Organosoluble	12	0.43	NA	--
Total characterised/identified	**85**	**2.8**	**106**	**49**
Unresolved [6]	2.6	0.092	12.	5.6
Post-extraction Solids (PES)	13	0.45	9.5	4.4

[1] Not corrected for recovery.
[2] [^{14}C]fludioxonil equivalents.
[3] Tentatively identified by HPLC.
[4] ^{14}C-activity co-migrating with leaf components characterised as CGA-344623 or CGA-339833.
[5] 7 or 8 regions each accounting for4.3% of the TRR, except for the origin of 1x leaves which accounted for 10.9% of the TRR (0.384 mg/kg).
[6] TLC quadrants 1-4 scraped into eight vials (two each quadrant) after scraping of major spots.

Samples of fruit were extracted and profiled within 4 months of harvest, and final characterisation of residues was within 14-32 months. HPLC profiles from the original extracts and those at the end of the study period were similar, indicating that fludioxonil residues are stable in peaches.

Onions. The metabolism of [phenyl-U-^{14}C]fludioxonil in green onions was studied at a California field location in the USA (Kennedy, Report 153-97, 1999). A 50 WP formulation was applied as a foliar spray twice with a 14-day interval, at intended rates equivalent to seasonal use rates of 1116 g ai/ha and 5580 g ai/ha. Actual treatment rates were 557 g ai/ha and 683 g ai/ha (total 1240 g ai/ha) and 2793g ai/ha and 3376 g ai/ha (total 6169 g ai/ha)

Green onions (whole plants) were sampled immediately after each application, 7 days after the last application (early harvest), 14 days after the last application (mature harvest), and 28 days after the last application (delayed harvest).

Homogenized samples were combusted and the TRR determined by LSC. Uncombusted samples were extracted with acetonitrile/water (80:20) or acetonitrile/water/acetic acid (80:20:1) using a Polytron homogeniser, concentrated and profiled by TLC and HPLC. The extracts from preparative extractions were chromatographed on a C-18 column, which was washed with acetonitrile and eluted with chloroform. The fractions from the loading and washing steps were combined. Selected solutions were evaporated to leave an aqueous fraction, which was partitioned with heptane and then methyl *tert*-butyl ether (MTBE) to separate the organosoluble radioactivity. The water-soluble radioactivity from the partition was subjected to C-18 solid-phase extraction (SPE). Additional clean-up was by A-25 anion exchange chromatography. Organic and aqueous fractions were analysed by TLC and HPLC. Selected extracts were hydrolysed with β-glucosidase. Microwave-assisted extractions of selected unextracted residues (post-extraction solids, PES) were with 2-propanol followed by acid and base hydrolysis. MS and NMR were used to identify metabolites.

The distribution of the TRR in the onion plants is shown in Table 23. Control samples did not contain any detectable residues.

The residues in the whole plants decreased from 2.4 mg/kg shortly after the second application to 0.98 mg/kg 28 days later at the lower treatment rate, and from 13. mg/kg to 4.7 mg/kg at the higher treatment rate. Extractable radioactivity for the lower rate decreased from 95% after the first application to 62% after 28 days, and for the higher treatment from 96% to 79%. Unextracted radioactivity increased from 1.2% and 4.4% after the two lower treatments to 25% in the 14-day PHI samples, with similar results at the higher rate.

Table 23. Distribution and extraction characteristics of radioactivity in extracts of field-grown green onions after foliar treatment with [phenyl-U-^{14}C]fludioxonil (Kennedy, Report 153-97, 1999).

Treatment /Location	Sample	TRR (mg/kg)	Extracted [1]		Unextracted		Recovery[2]
			% of TRR	mg/kg	% of TRR	mg/kg	% of TRR
Low rate/California							
	Whole plant post 1st application	1.5	95	1.4	1.2	0.018	96
	Whole plant post 2nd application	2.4	95	2.2	4.4	0.10	99
	Whole plant – 7-day PHI	1.8	76[3]	1.4[3]	21[3]	0.38[3]	98
	Whole plant-14-day PHI	1.6	73	1.2	25	0.39	98
	Whole plant-28-day PHI	0.98	62.	0.60	22.	0.21	84.
High rate/California							
	Whole plant post 1st application	14	96	13	0.8	0.11	96
	Whole plant post 2nd application	13	98	13	4.3	0.58	102
	Whole plant-7-day PHI	10	79	7.9	18	1.8	97
	Whole plant-14-day PHI	10	78	7.8	28	2.8	106
	Whole plant-28-day PHI	4.7	79	3.7	28	1.3	107

[1] Toal extracted before C-18 acetonitrile flash chromatography.
[2] Extracted % of TRR + unextracted (PES) % of the TRR.
[3] Average: extraction carried out in two parts

The distribution of extracted residues between organic and aqueous phases is shown in Table 24. For the 1X treatment 44% of the radioactivity in the 7-day PHI samples was organosoluble, and for the 5X treatment the proportion decreased from 67% in 7-day PHI samples to 48% in 28-day PHI samples. The corresponding water-soluble radioactivity increased from 17% to 26% over the same period.

Table 24. Characterisation of extracted radioactivity from field-grown green onions treated with foliar applications of [phenyl-U-^{14}C]fludioxonil (Kennedy, Report 153-97, 1999).

Sample	TRR	Heptane soluble	MTBE soluble	Total organosoluble [1]		C-18 ACN eluant	C-18 MeOH eluant	C-18 aqueous L+W[2]	Total water-soluble [3]	
	mg/kg	% of TRR	% of TRR	% of TRR	mg/kg	% of TRR	% of TRR	% of TRR	% of TRR	mg/kg
1X, 7-day PHI	1.8	15	29	44	0.79	12	6.5	7.7	26	0.47
5X, 7-day PHI	10	40	27	67	6.7	8.9	3.8	4.6	17	1.7
5X, 14-day PHI	10	24	27	51	5.1	5.0	3.0	13.	21	2.08
5X, 28-day PHI	4.7	25	24	49	2.3	4.3	3.9	17	26	1.2

[1] Heptane soluble fraction + MTBE soluble fraction
[2] Load to wash fractions
[3] C-18 ACN eluant + C-18 MeOH eluant + C-18 aq L+W fractions.

Quantification of the metabolites is shown in Table 25. Fludioxonil was the main residue with 38% to 54% of the TRR in 7-day PHI samples, 36% to 49% of the TRR in 14-day PHI samples and 12% to 31% of the TRR in 28-day PHI samples. Metabolites CGA 265378, CGA 308103, SYN 518579, CGA 192155, CGA 344623, and CGA 339833 were also found, with none above 7% of the TRR. The residue percentages characterised ranged from 64% to 82% at all intervals and both treatment rates, and percentage identified from 29% to 67%.

Microwave-assisted extraction of the 7-day PHI samples, followed by acid hydrolysis for the 14- and 28-day PHI samples, released an additional 11% to 18% of the TRR, most of which was organosoluble. Small amounts (<2.0%) of fludioxonil, CGA-265378, CGA-308103, and CGA-192155 were found after the microwave extraction. The residues remaining (PES) varied from <0.1% to as much as 7.6% of the TRR.

Table 25. Quantification of metabolites in green onions treated with foliar applications of [phenyl-U-^{14}C]fludioxonil (Kennedy, Report 153-97, 1999).

Source	Components	1X rate		5X rate	
		% of TRR	mg/kg	% of TRR	mg/kg
7-day PHI					
	Total mg/kg	100	1.8	100	10.
Aqueous acetonitrile extracts		76		79	
	% of TRR profiled	77		79	
A [1]	Fludioxonil	38	0.69	54	5.4
B [1]	CGA-265378	1.2	0.022	4.1	0.41
E	CGA-308103	4.4	0.079	2.4	0.24
F	CGA-192155	2.6	0.047	1.2	0.12
P15	SYN 518579	1.2	0.022	0.9	0.090
O16	MS/partial structure	1.5	0.027	4.0	0.40
H	CGA-344623	2.6	0.047	0.9	0.090
I	CGA-339833	0.8	0.014	1.4	0.14
Unknowns	Multiple zones	17.	0.30	2.9	0.29
Unresolved	Low level spots			1.3	0.13
O	Origin	7.7	0.14	5.0	0.50
Total		77	1.4	79	7.8
Initial unextracted[2]		23	0.41	18	1.8
Microwave-extracts		16	0.29	11	1.1
Microwave-aqueous[3]	(not analysed)	2.9	0.052	3.1	0.31
Microwave-MTBE [4]		11	0.20	8.2	0.82
	% of TRR profiled [4]	8.6	0.16	8.2	0.82
A	Fludioxonil	0.3	0.005	0.3	0.030
B	CGA-265378	0.5	0.009	0.5	0.050
E	CGA-308103	0.3	0.005	0.2	0.020
F	CGA-192155	0.3	0.005	0.2	0.020
P15	SYN 518579	0.5	0.009	0.2	0.020
O16	MS/partial structure	1.0	0.018	0.8	0.080
Unknowns	Multiple zones	2.0	0.036	2.2	0.22
Unresolved		3.7	0.067	3.6	0.36
O	Origin	0.1	0.002	0.1	0.010
Total		12	0.21	11	1.1
Final unextracted[2]	Post-microwave	7.6	0.14	4.8	0.48
Overall total		96	1.7	95	9.4
% Characterised [6]		82	1.5	82	8.2
% Identified [7]		53	0.95	67	6.7
14-day PHI					
	Total mg/kg	100	1.6	100	10.

Source	Components	1X rate		5X rate	
		% of TRR	mg/kg	% of TRR	mg/kg
Aqueous acetonitrile extracts		73		78	
	% of TRR profiled	67		78	
A [1]	Fludioxonil	36	0.56	49	4.9
E	CGA-308103	4.5	0.071	2.0	0.20
F	CGA-192155	1.7	0.027	2.2	0.22
P15	SYN 518579	0.9	0.014	0.5	0.050
O16	MS/partial structure			1.7	0.17
H	CGA-344623	1.3	0.020	1.3	0.13
I	CGA-339833	2.7	0.042	1.4	0.14
Unknowns	Multiple zones	5.6	0.088	4.2	0.42
Unresolved				7.9	0.79
O	Origin	15	0.24	7.3	0.73
Total		67	1.1	78	7.8
Initial unextracted[2]		25	0.39	28	2.8
Microwave-extracts	Microwave	15	0.24	11	1.1
Acid/base-extracts [3]	Acid/base hydrolysis			4.5	0.45
Microwave-aqueous	(not analysed)	4.1	0.064	2.7	0.27
Microwave-MTBE[4]		10	0.16	9.0	0.90
	% of TRR profiled [4]	10	0.16	8.3	0.83
A	Fludioxonil	1.1	0.017	0.4	0.04
B	CGA-265378			0.5	0.05
E	CGA-308103	0.4	0.006	0.3	0.03
F	CGA-192155	0.2	0.003	0.3	0.03
P15	SYN 518579	0.9	0.014	0.3	0.03
O16	MS/partial structure	1.4	0.022	0.9	0.09
Unknowns	Multiple zones	3.2	0.050	2.0	0.20
Unresolved		3.0	0.047	3.5	0.35
O	Origin	0.3	0.005	0.2	0.02
Total		15	0.229	15	1.6
Final unextracted[2]	Post microwave	7.5	0.12		
Final unextracted [5]	Post acid/base hydrolysis			<0.1	<0.010
Overall Total		90	1.4	93	9.4
% Characterised [6]		75	1.2	75	7.5
% Identified [7]		47	0.74	58	5.8
28-day PHI					
	Total mg/kg	100	0.98	100	4.7
Raw extracts		62		79	
	% of TRR profiled	56		75	
A [1]	Fludioxonil	12	0.112	31	1.4
B [1]	CGA-265378	2.6	0.025	6.8	0.32
E	CGA-308103	5.5	0.054	3.0	0.14
F	CGA-192155	2.5	0.024	2.2	0.10
P15	SYN 518579	1.4	0.014	1.6	0.075
O16	MS/partial structure			4.0	0.19
H	CGA-344623	1.7	0.017	2.0	0.093
I	CGA-339833	2.8	0.027	2.2	0.10
Unknowns	Multiple zones			7.0	0.33
Unresolved				5.4	0.25
O	Origin	28	0.275	9.4	0.44
Total		56	0.549	75	3.5
Initial unextracted[2]		22	0.213	28	1.3
Microwave-Extracts	Microwave	13	0.123	18	0.86
Acid -Extracts [3]	Acid hydrolysis			1.8	0.084
Microwave-Aqueous[4]	(not analysed)	2.5	0.024	3.8	0.18
Microwave-MTBE[4]		9.5	0.093	15	0.71
	% of TRR profiled [4]	7.9	0.077	15	0.69
A	Fludioxonil CGA-173506	0.5	0.005	0.9	0.042

Source	Components	1X rate		5X rate	
		% of TRR	mg/kg	% of TRR	mg/kg
B	CGA-265378	0.8	0.008	1.1	0.051
E	CGA-308103	0.4	0.004	0.5	0.023
F	CGA-192155	0.4	0.004	0.6	0.028
P15	SYN 518579	0.5	0.005	0.7	0.033
O16	MS/partial structure	1.3	0.013	1.8	0.084
Unknowns	Multiple zones	3.9	0.038	4.0	0.19
Unresolved		0.2	0.002	4.6	0.22
O	Origin			0.4	0.019
Total		10	0.102	20	0.94
Final unextracted[2]	Post microwave	6.7	0.065		
Final unextracted [5]	Post acid/base hydrolysis			6.2	0.29
Overall total		73	0.716	101	4.7
% Characterised [6]		64	0.625	79	3.7
% Identified [7]		29	0.280	53	2.4

[1] CGA 265378 co-eluted with fludioxonil in zone A/B. Quantitative values based on ratio of metabolites by HPLC.
[2] Initial unextracted (post-extraction solids): residues remaining after aqueous acetonitrile extraction, further extracted using microwave techniques or acid/base hydrolysis. Final unextracted (post-extraction solids): residues remaining after microwave extraction.
[3] Extract from acid hydrolysis not fractionated or further analysed.
[4.] Metabolites in MTBE-soluble fraction of microwave extracts identified by co-chromatography (TLC) with available standards and/or characterised by comparison of chromatographic behaviour (TLC and HPLC) with known metabolites. Water-soluble fraction of extracts not analysed.
[5] For the 5X sample unextracted residue combusted after acid hydrolysis.
[6] Sum of values for all quantified chromatographic peaks.
[7] Sum of values for regions A, B, E, F, P15, H, and I.

Lettuce. Iceberg Floreal lettuce plants were treated three times at 10-day intervals with a WP 50 formulation of [pyrrole-4-^{14}C]fludioxonil in St Aubin, Switzerland (Stingelin, Report 98JS29, 2000, amendments 01/10/2000 and 10/02/2003) at a rate corresponding to 200 g ai/ha/application and sampled 1 h, and 6 and 13 days after the last application.

In an auxiliary experiment a threefold treatment rate (600 g ai/ha/application) was used for characterisation purposes. Plants and soil were sampled as above.

Plant samples cut just above the soil surface and free of soil were chopped, homogenized with liquid nitrogen and combusted and the TRR determined by LSC. An aliquot of the homogenised plant material was extracted with methanol/water (80:20) five times or until any additional extraction yielded <5% of the first extract using a mechanical shaker and centrifugation. For metabolite isolation a single extraction with methanol and then was used. The unextracted residues (PES) were determined by combustion. The methanol/water extracts were analysed by TLC. The crude extracts were also concentrated to the aqueous phase and extracted with dichloromethane. The organosoluble material was analysed by TLC and the water-soluble remainder cleaned up by C-18 and Serdolit® PAD I solid-phase extraction. Samples were further cleaned up and analysed using HPLC. Selected aqueous phases were hydrolysed using cellulase and β-glucosidase. Metabolites were identified by mass spectrometry and NMR.

The TRR in lettuce heads were 5.3 mg/kg (0-day PHI), 1.3 mg/kg (6-day PHI), and 0.64 mg/kg (13-day PHI) for the 1X rate, and for the 3X rate correspondingly higher. Fludioxonil residues from the 1X treatment decreased from 4.0 mg/kg to 0.29 mg/kg and those from the 3X rate from 11. mg/kg to 0.58 mg/kg during the 13 days after treatment. Most of the radioactivity in the heads was extracted, decreasing from 109% to 94% of the TRR in the 1X samples and from 104% to 91% in the 3X samples over the 13-day period, while the unextracted increased from 1.9% to 9.0% of the TRR in the 1X lettuce and from 1.7% to 6.4% in the 3X lettuce over the same period (Table 26).

Table 26. Distribution of radioactivity in heads of field-grown lettuce treated with foliar applications of [pyrrole-4-^{14}C]fludioxonil in Switzerland (Stingelin, Report 98JS29, 2000).

Treatment/Days after 3rd treatment	TRR, mg/kg [1]	Parent, mg/kg	% of TRR [2]		
			Extracted	Unextracted	Total
1X rate					
0	5.3	4.0	109	1.9	110
6	1.3	0.76	97	5.7	103
13	0.64	0.29	94	9.0	103
3X rate					
0	19	11	104	1.7	105
6	5.8	2.7	104	3.6	108
13	2.0	0.58	91	6.4	97

n.a. :not analysed
LOQ: lettuce heads. 0.002 mg/kg
[1] Fludioxonil equivalents
[2] TRR determined by combustion

The quantification of the parent and metabolite fractions in the lettuce extracts after the 1X and 3X treatments is summarised in Tables 27 and 28.

54% to 74% of the TRR from the 1X rate was the parent compound and the results for the 3X samples were similar, 62% to 87%. Twenty metabolite fractions were detected and no metabolite or zone constituted more than 4% of the TRR at either rate.

The metabolites identified by TLC co-chromatography with reference compounds or by MS and NMR were CGA 308103, CGA 339833, CGA 344623, CGA 192155, CGA 265378, the lactic acid conjugate of fludioxonil, the N-or O-glucose conjugate of CGA 308103, and the glucose conjugate of CGA 344623.

Table 27. Quantification of metabolite fractions in heads of lettuce treated with foliar applications of [pyrrole-4-^{14}C]fludioxonil (1X rate) (Stingelin, Report 98JS29, 2000).

pHI (days)[1]	0		6		13	
TRR (mg/kg)	5.3		1.3		0.64	
Extract	E1		E1		E1 + E2	
Metabolite fractions	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg
I_1 (start)	2.3	0.12	2.3	0.030	3.5	0.022
I_2 CGA 344623 glucose conjugate	1.9	0.10	1.1	0.015	2.4	0.015
I_{2a}	<LOQ	<LOQ	0.8	0.011	1.5	0.010
I_3[2]	1.5	0.082	1.7	0.022	1.8	0.011
I_4 CGA 344623	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
I_{4a}	<LOQ	<LOQ	0.3	0.004	0.4	0.002
I_{4b} fluxioxonil lactic acid conjugate	<LOQ	<LOQ	<LOQ	<LOQ	0.4	0.002
I_{4c} fludioxonil lactic acid conjugate	0.2	0.012	1.0	0.013	1.7	0.011
I_{4d}	0.2	0.012	0.4	0.005	0.6	0.004
I_{4e}	0.4	0.022	0.8	0.011	1.5	0.010
I_5 CGA 308103 N- or O- glucose conjugate	<LOQ	<LOQ	0.5	0.007	0.9	0.006
I_6	<LOQ	<LOQ	0.7	0.009	1.2	0.007
I_{10} CGA 192155	2.2	0.116	0.5	0.006	0.6	0.004
I_{10a}	<LOQ	<LOQ	0.3	0.004	0.3	0.002
I_{11} CGA 308103	<LOQ	<LOQ	0.2	0.003	0.2	0.001
I_{12}	0.5	0.025	1.1	0.014	0.8	0.005

pHI (days)[1]	0		6		13	
TRR (mg/kg)	5.3		1.3		0.64	
Extract	E1		E1		E1 + E2	
Metabolite fractions	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg
I_{12a}	<LOQ	<LOQ	0.7	0.009	0.4	0.003
I_{12b}	<LOQ	<LOQ	1.2	0.015	0.8	0.005
I_{12c}	<LOQ	<LOQ	0.3	0.004	0.3	0.002
I_{12d}	<LOQ	<LOQ	0.3	0.004	0.2	0.001
I_{13}	1.3	0.067	1.8	0.023	1.6	0.010
I_{14} CGA 265378	1.1	0.057	2.2	0.029	1.8	0.011
I_{15} fludioxonil (CGA 173506)	74	3.9[3]	68	0.90[3]	54	0.34[3]
I_{16}	0.9	0.049	1.6	0.021	1.2	0.007
I_{17}	<LOQ	<LOQ	0.4	0.005	0.5	0.003
Unresolved radioactivity	23.	1.2	8.8	0.12	16.	0.103
Sub total	109.0	5.815	97.4	1.275	94.	0.60
Extract E2	n.a.		n.a.		1.1	
Unextracted	1.9		5.7		9.0	
Total	110		103		104	
Identification	81		76		64	

LOQ: c. 0.2-1.2% of the TRR depending on the size of the radioactive zone. 0 days - c. 0.047 mg/kg, 6 days - c. 0.007 mg/kg, 13 days - c. 0.003 mg/kg.
n.a.: not analysed
[1] after 3rd application
[2] I_3: two fractions I_{3a} and I_{3b} : 1st is (CGA 339833)
[3] fludioxonil determined by TLC system 1 generally gave higher values than those by TLC system II

Table 28. Quantification of metabolite fractions in heads of lettuce treated with foliar applications of [pyrrole-4-^{14}C]fludioxonil (3X rate) (Stingelin, Report 98JS29, 2000).

PHI (days)[1]	0		6		13	
TRR (mg/kg)	19.		5.8		2.0	
Extract	E1		E1		E1 + E2	
Metabolite fractions	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg
I_1 (start)	0.9	0.17	1.7	0.098	2.6	0.052
I_2 CGA 344623 glucose conjugate	0.9	0.174	1.8	0.104	2.2	0.044
I_{2a}	<LOQ	<LOQ	0.5	0.029	0.7	0.014
I_3[2]	1.3	0.252	1.5	0.086	1.9	0.038
I_4 CGA 344623	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
I_{4a}	<LOQ	<LOQ	0.4	0.023	0.3	0.006
I_{4b} fudioxonil lactic acid conjugate	0.2	0.039	<LOQ	<LOQ	0.2	0.004
I_{4c} fludioxonil lactic acid conjugate	0.2	0.039	0.4	0.023	1.1	0.022
I_{4d}	<LOQ	<LOQ	0.2	0.012	0.4	0.008
I_{4e}	0.1	0.019	0.6	0.035	1.2	0.024
I_5 CGA 308103 N- or O- glucose conjugate	<LOQ	<LOQ	0.5	0.029	0.6	0.012
I_6	<LOQ	<LOQ	<LOQ	<LOQ	0.7	0.014
I_{10} CGA 192155	0.2	0.039	0.5	0.029	0.5	0.010
I_{10a}	<LOQ	<LOQ	<LOQ	<LOQ	0.2	0.004
I_{11} CGA 308103	<LOQ	<LOQ	0.3	0.017	0.2	0.004
I_{12}	0.4	0.078	0.3	0.017	0.5	0.010
I_{12a}	<LOQ	<LOQ	<LOQ	<LOQ	0.3	0.006
I_{12b}	<LOQ	<LOQ	<LOQ	<LOQ	0.5	0.010
I_{12c}	<LOQ	<LOQ	<LOQ	<LOQ	0.2	0.004
I_{12d}	<LOQ	<LOQ	<LOQ	<LOQ	0.4	0.008
I_{13}	1.2	0.233	1.4	0.081	1.1	0.022
I_{14} CGA 265378	1.2	0.233	1.1	0.063	1.7	0.034
I_{15} fludioxonil (CGA 173506)	87	17[3]	80	4.6[3]	62	1.2[3]
I_{16}	1.2	0.23	1.1	0.063	0.9	0.018
I_{17}	<LOQ	<LOQ	<LOQ	<LOQ	0.4	0.008
Unresolved radioactivity	8.5	1.6	11.	0.63	9.9	0.20
Sub total	104.	20.	103.	6.0	91.	1.8

PHI (days)[1]	0		6		13	
TRR (mg/kg)	19.		5.8		2.0	
Extract	E1		E1		E1 + E2	
Metabolite fractions	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg
Extract E2	n.a.		n.a.		1.1	
Unextracted	1.7		3.6		6.4	
Total	105		107		98	
Identification	91		86		70	

LOQ: c. 0.1-1.7% of TRR depending on size of radioactive zone. 0 days - c. 0.095 mg/kg, 6 days - c. 0.030 mg/kg, 13 days - c. 0.007 mg/kg.
n.a.: not anaysed
[1] after 3rd application
[2] I_3: two fractions, I_{3a} and I_{3b} . 1st is CGA 339833
[3] Fludioxonil determined by TLC system 1 generally gave higher values than those by TLC system II .

Seed treatment

Potatoes. In a field trial in Klus, Switzerland, Bintje seed potatoes were treated with [pyrrole-4-^{14}C]fludioxonil, formulated as FS 100, at a rate corresponding to 2.5 g ai/100 kg seed (Kruass, Report 13/93, 1993). After being dried, the potatoes were wrapped and stored until planting the next day. Plants were sampled at 0, 40 days 71 and at harvest 95 days after treatment (Intervals 1, 2, 3, and 4). Tubers were present on the plants after 71 days, and at harvest were separated into peel and flesh.

Samples were frozen with liquid nitrogen and homogenized. The TRR were determined by combustion and LSC of representative sub-samples. Homogenized plant material was extracted with methanol/water (8:2) by blending and shaking, followed by Soxhlet extraction with methanol. Unextracted residues were determined by combustion after the Soxhlet extraction. The extracts were analysed by 2-D TLC. In some cases, the methanol/water was concentrated and extracted with dichloromethane to produce an organosoluble and water-soluble fraction, which were also analysed by 2-D TLC. Selected samples were subjected to liquid chromatography clean-up using XAD-4 resin before TLC. Some water-phase material was subjected to enzyme cleavage to release sugar conjugates.

The distribution of radioactivity in the plants and in various samples at intervals is shown in Table 29. Total residues in the tubers shortly after treatment were 8.6 mg/kg with a fludioxonil content of 8.4 mg/kg. Translocation of radioactivity from treated tubers to leaves or into new growth was limited. In the new tubers, the TRR was only 0.006 mg/kg at 71 days after treatment and at harvest. The radioactivity at harvest was 48/52% peel/flesh: approximately half was in the peel (equivalent to a residue of 0.031 mg/kg) and half in the flesh (equivalent to 0.004 mg/kg). In leaves, the TRR was 0.022 mg/kg (mg/kg), 0.019 mg/kg and 0.024 mg/kg, 40 days and 71 days after treatment and at harvest respectively.

The characterisation, identification, and quantification of the extracted radioactive residues are shown in Table 30.

Only the peel at harvest had sufficient radioactivity for chromatographic characterisation. Five zones, I_1, I_{1a}, I_2, I_{4a}, and fludioxonil (II_4), were defined and quantified by TLC. The major component was fludioxonil (21% of the TRR in the whole new tuber at harvest with the highest remaining component being 1.6% of the TRR.

Four zones were defined and quantified by TLC in the leaves at harvest. These were I_1, I_2, CGA 192155 (I_{10}) and fludioxonil (II_4). CGA 192155 and fludioxonil constituted 1.9% and 0.8% of the TRR respectively in leaves at harvest.

Unextracted radioactivity represented 35% of the TRR in new peels at harvest and was too low in the flesh to characterise, and in leaves increased over time from 21% 40 days after treatment to 46% at harvest.

Table 29: Distribution and penetration of radioactivity and residual fludioxonil in potato substrates after treatment of the seed potatoes with [pyrrole-4-^{14}C]fludioxonil at 2.5 g ai/100 kg (Kruass, Report 13/93, 1993).

Days after treatment	Sample	Total residues (mg/kg)[1]	(%)[2]	Parent (mg/kg)	Extracted radioactivity cold ext. (%)[3]	Soxhlet ext. (%)[3]	Unextracted (%)[3]	Total (%)[3]
0 days Interval 1	Treated tubers	8.6	100	8.4	99	n.a.	1.3	100
40 days Interval 2	Leaves	0.022	100	<0.001	77.	5.3	21.	103
	Treated peels	3.4	96	3.2	101	1.8	5.5	109
	Treated flesh	0.024	4.3	0.009	105.	1.0	5.3	111
	Whole treated tuber	0.49	100	0.44				
71 days Interval 3	Leaves	0.019	100	<0.001	75	3.2	28.	106
	New tubers	0.006	100	n.a.	n.a.	n.a.	n.a.	n.a.
95 days Interval 4	Leaves	0.024	100	<0.001	49	2.2	46	97
	New tuber peels	0.031	48	0.014	67	6.7	35	109
	New tuber flesh	0.004	52	n.a.	n.a.	n.a.	n.a.	n.a.
	Whole new tuber	0.006	100	0.003				

n.a.: not analysed
LOQ: combustion: 0.001 mg/kg, LOQ: TLC analyses of plant material: 0.001 mg/kg
[1] fludioxonil equivalents
[2] % of radioactivity in sample.
[3] % of radioactivity in sample.

Table 30. Quantification of metabolite fractions in potatoes at maturity after seed treatment with [pyrrole-4-^{14}C]fludioxonil at 2.5 g ai/100 kg (Kruass, Report 13/93, 1993).

Sample	Metabolite Fractions (% of TRR)							Soxhlet (% of TRR)	Unextracted (% of TRR)	Total (%) [1]
	I_1 [2]	I_{1a} [3]	I_2 [3]	I_{4a}	CGA 192155 I_{10}	Fludioxonil II_4 [4]	Un-resolved			
New tuber peels	3.4	2.5	1.4	1.2	-	44 [5]	6.3	5.6 [6]	35	100
New tuber flesh	n.a.									
Whole new tuber	1.6	1.2	~0.7	~0.6	-	21[5]	n.a.			
Leaves	18	-	1.3	-	1.9	0.8	27	2.2	46	97

[1] Sum of fractions: % by combustion of total residues.
[2] Radioactivity remaining at the origin in TLC analysis in analytical system I.
[3] Diffuse spot in TLC analysis and/or partial overlap
[4] $I_{12} = II_4$ = fludioxonil.
[5] Includes parent content of Soxhlet and all other fractions.

Rice. Plant uptake, distribution, and metabolism of [pyrrole-4-^{14}C]fludioxonil were studied in a greenhouse trial (Fleishmann, Report ABR-90099, 1991, amendment 10/12/1993). Rice seeds (variety Labonnet) were soaked in a 267 mg/kg solution of ^{14}C-fludioxonil formulated as a 5% powder, a treatment rate equivalent to 6.5 g ai/100 kg seed, and plants grown from the seed.

Whole plants were sampled at 25% maturity (38 days) and 50% maturity (76 days), and stalk, hulls, and grain at maturity (152 days).

The seed coating solution was analysed by TLC to establish that degradation was insignificant. Treated seeds were combusted to determine the initial seed dressing. Whole plants and mature fractions were homogenized before combustion. Samples were combusted and the TRR determined by LSC.

The distribution of the TRR in the plants is shown in Table 31. Treated seeds contained an initial coating equivalent to 65. mg/kg of fludioxonil.

Translocation of radioactivity from treated seeds to aerial plant parts and/or uptake of radioactivity by roots were minimal. The maximum total radioactive residue of 0.004 mg/kg was in whole plants sampled at 25% maturity and the TRR 38 and 76 days after treatment was 0.004 mg/kg and <0.002 mg/kg respectively. At harvest the mature stalks, hulls, and grain contained total residues of <0.002 mg/kg, 0.002 mg/kg and <0.002 mg/kg respectively.

Both extracted and unextracted radioactivity was too low in all the plant samples to pursue characterisation and/or identification.

Table 31. Residues found in greenhouse-grown rice plants after seed treatment with [pyrrole-4-^{14}C]fludioxonil (Fleishmann, Report ABR-90099, 1991).

Sampling	Interval (days)	TRR in ^{14}C-fludioxonil equivalents (mg/kg)				
		Treated seed	Whole plant	Stalk	Hulls	Grain
Treated seeds	0	65	-	-	-	-
25% maturity	38	-	0.004	-	-	-
50% maturity	76	-	<0.002	-	-	-
100% maturity	152	-	-	<0.002	0.002	<0.002

Wheat. The metabolism of [pyrrole-4-^{14}C]fludioxonil was studied in greenhouse- and field-grown spring wheat plants by Gentile (Report 15/91, 1991, addendum 11/28/1996). In the greenhouse seeds were individually syringed with a mixture of blank formulation (A-8207) and [pyrrole-4-^{14}C]fludioxonil, corresponding to approximately 13 g ai/ha and covered with soil in beakers. Plants were sampled 11, 18, 25, 32, 39, 46, and 53 days after application and separated into leaves and roots.

For the field experiment seeds were treated at a rate of 7.4 g ai/100 kg seed with ^{14}C-fludioxonil as an FS 037 formulation containing A-8207, corresponding to approximately 15 g ai/ha. Plants were sampled 48 (stalks), 83 (ears and stalks) and 106 days (straw, husks and grain) after treatment, corresponding to ear emergence (Interval 1), milky stage (Interval 2) and maturity (Interval 3).

Plant fractions were homogenized with liquid nitrogen, extracted with acetonitrile/water (8:2) and analysed by TLC. The extracts were cleaned up by XAD-4 resin and analysed by HPLC. Enzyme cleavage was used to determine sugar conjugates. Mass spectrometry was attempted on selected fractions. Reference metabolites isolated from grapes treated with ^{14}C-fludioxonil were also used to aid in the identification process.

To obtain higher residues for characterisation in an auxiliary experiment, one-month old spring wheat plants were injected with a DMSO solution of radiolabelled fludioxonil (Gentile, Report 27/92, 1993) and harvested 40 and 69 days later.

In a second short-term stem injection experiment using an acetone solution of ^{14}C-fludioxonil for the injections immature plants were after harvested three weeks and the work-up of green plant samples was as described above.

The distribution of the TRR in the greenhouse-grown wheat plants is shown in Table 32. Translocation of radioactivity from treated seeds to aerial plant parts and/or uptake of radioactivity by roots was low ranging from 0.9% and 22.6% of the applied radioactivity 11 days after treatment to 3.1% and 13.0% after 53 days in the leaves and roots respectively.

Table 32. Distribution of fludioxonil in wheat after treatment of the seeds with [pyrrole-4-^{14}C]fludioxonil (greenhouse experiment) (Gentile, Report 15/91, 1991).

Interval	Sample	Total residues		Parent (mg/kg)[1]	Extracted radioactivity (%) [3]	Unextracted (%)[3]	Total (%)[3]
		mg/kg [1] ± SD (%)	(%) [2]				
11 days	Leaves	0.32 ± 55.	0.9	0.005	96	3.6	100
	Roots	8.6 ± 30	23	2.8	86	14	100
	Plant total		24				
	Soil total	0.015 ± 3.8	78	0.013	97	3.3	100
	Total		102				
18 days	Leaves	0.36 ± 10.	3.9	0.015	86	14	100
	Roots	5.5 ± 42.	20	1.4	79	21	100
	Plant total		24				
	Soil total	0.015 ± 3.9	75	0.012	94	6.2	100
	Total		99				
25 days	Leaves	0.16 ± 28.	3.1	<0.001	86	14	100
	Roots	2.9 ± 11.	14	0.81	68	32	100
	Plant total		17				
	Soil total	0.015 ± 3.8	78	0.012	91	8.6	100
	Total		95				
32 days	Leaves	0.14 ± 12.	3.8	0.004	76	24	100
	Roots	3.3± 7.5	18	0.89	58	42	100
	Plant total		22				
	Soil total	0.015 ± 0.0	77	0.012	89	11	100
	Total		99				
39 days	Leaves	0.081 ± 15.	3.1	<0.001	88	12	100
	Roots	2.2 ± 8.2	13	0.46	48	52	100
	Plant total		16				
	Soil total	0.016 ± 3.7	80	0.012	89	11	100
	Total		96				
46 days	Leaves	0.067 ± 3.9	3.3	<0.001	87	13	100
	Roots	1.3 ± 20.	10	0.15	38	62	100
	Plant total		13.5				
	Soil total	0.015 ± 10	75	0.010	85	15	100

Interval	Sample	Total residues		Parent (mg/kg)[1]	Extracted radioactivity (%) [3]	Unextracted (%)[3]	Total (%)[3]
		mg/kg [1] ± SD (%)	(%) [2]				
	Total		89				
53 days	Leaves	0.056 ± 30.	3.1	<0.001	78	22	100
	Roots	1.9 ± 22.	13	0.20	32	68	100
	Plant total		16				
	Soil total	0.016 ± 6.2	83	0.010	83	17	100
	Total		99				

[1] Fludioxonil equivalents.
[2] % of radioactivity applied.
[3] % of total radioactivity found in sample, determined by sum of extracted and unextracted radioactivity.
n.a.: not analysed

Results from the field experiment are summarized in Table 33. Translocation of radioactivity from treated seeds to aerial plant parts (stalks, husks, and grain) was low. Radioactive residues at harvest were 0.015 mg/kg, 0.005 mg/kg and 0.003 mg/kg in stalks, husks, and grain respectively.

Table 33: Distribution of radioactivity and residual fludioxonil in spring wheat after seed treatment with [pyrrole-4-^{14}C]fludioxonil (long-term experiment) (Gentile, Report 15/91, 1991).

Interval	Sample	Total residues		Parent (mg/kg)[1]	Extracted radioactivity (%)		Unextracted (%)[3]	Total (%)[3]
		mg/kg [1]	(%) [2]		Cold [3]	Soxhlet [3]		
48 days	Stalks	0.005	100	n.a.	80	n.a.	36	120
Ear emergence	Soil							
Interval 1	0-5 cm	0.035	91	0.017	70	7.4	29	106
	5-10	0.002	4.5	n.a.	n.a.	n.a.	n.a.	
	10-20	<0.001	3.3	n.a.	n.a.	n.a.	n.a.	
	20-30	<0.001	1.3	n.a.	n.a.	n.a.	n.a.	
	Soil 0-30 cm	0.007	100					
83 days	Stalks	0.004	100	n.a.	n.a.	n.a.	n.a.	
Milk stage	Ears	0.002	100	n.a.	n.a.	n.a.	n.a.	
Interval 2	Soil							
	0-5 cm	0.013	91	0.004	53	6.2	53	112
	5-10	<0.001	5.2	n.a.	n.a.	n.a.	n.a.	
	10-20	<0.001	3.2	n.a.	n.a.	n.a.	n.a.	
	20-30 cm	<0.001	0.9	n.a.	n.a.	n.a.	n.a.	
	Soil 0-30 cm	0.003	100					
106 days	Stalks	0.015	100	n.a.	41	14.	63	120
Maturity	Husks	0.005	100	n.a.	n.a.	n.a.	n.a.	
Interval 3	Grain	0.003	100	n.a.	n.a.	n.a.	n.a.	
	Soil							
	0-5 cm	0.048	90	0.017	53	6.1	43	102
	05-10	0.003	5.5	n.a.	n.a.	n.a.	n.a.	

Interval	Sample	Total residues		Parent (mg/kg)[1]	Extracted radioactivity (%)		Unextracted (%)[3]	Total (%)[3]
		mg/kg [1]	(%) [2]		Cold [3]	Soxhlet [3]		
	10-20	<0.001	2.7	n.a.	n.a.	n.a.	n.a.	
	20-30	<0.001	2.2	n.a.	n.a.	n.a.	n.a.	
	Soil 0-30 cm	0.008	100					

[1] fludioxonil equivalents.
[2] % of radioactivity in sample; % of radioactivity in soil layers.
[3] % of radioactivity in sample, determined by combustion.
n.a. not analysed

In the injection experiment 78% of the radioactivity remained at the injection point. At harvest, mature stalks, husks and grain contained 20%, 0.9% and 0.2% respectively of the applied radioactivity (Table 35).

Table 34. Distribution of radioactivity in greenhouse-grown spring wheat after stem injection with [pyrrole-4-^{14}C]fludioxonil (Gentile, Report 27/92, 1993).

Interval	Sample	Total Residues		Parent (mg/kg)	Extracted radioactivity (%)		Unextracted (%)[2]	Total (%)[3]
		mg/kg [1]	(%) [2]		Cold [2]	Soxhlet [2]		
Harvest (69 days after injection)	Grain	0.46	0.2	0.19	79	0.7	20	102
	Husks	8.8	0.9	4.2	89	0.7	10	101
	Stalks	75.	20.	41.	84	1.5	15	99
	Injection point	-	78					
	Plant total	-	100					

[1] Fludioxonil equivalents.
[2] % of total extracted + unextracted activity.
[3] % of total radioactivity determined by combustion.

The quantification of the metabolite fractions from the stem injection experiment is shown in Table 35. Fifteen zones were quantified. Fludioxonil (II_4) represented the largest fraction at 36%, 49% and 49% of radioactivity in the grain, husks and stalks, and unresolved activity 8.7%, 13% and 13% of the TRR respectively. Cellulase treatment released only 1% of sugar conjugates.

Metabolites SYN 518579 (II_2), SYN 578580 (II_3), CGA 308103 (I_{13}), CGA 192155 (I_{10}), CGA 339833 (I_3) and CGA 344623 (I_4) were identified in the grain, husks and straw, but none above 2.6% of the TRR. The short-term stem injection experiment yielded an organosoluble fraction in green parts that co-chromatographed on 2-D TLC with CGA 265378 (II_{3b}).

Table 35. Quantification of metabolite fractions in mature spring wheat after stem injection with [pyrrole-4-^{14}C]fludioxonil (Gentile, Report 27/92, 1993).

Sample	Metabolite fractions (% of TRR) [1]																Soxhlet (%) [1]	Un-extracted (%) [1]	Total (%) [1]
	I_1	I_2	I_3	I_4 + I_5	I_6	I_7	I_8	I_9	I_{10}	I_{11}	I_{12}	I_{13} II_1	I_{14} II_2	II_3	II_4 *	Un-resolved			
Grain (0.37 mg/kg)	8.2	14	0.7	2.6	n.a.	0.3	n.a.	n.a.	1.0	n.a.	1.0	1.1	0.8	0.3	36	8.7	0.7	20	95

Sample	Metabolite fractions (% of TRR) [1]																Soxhlet (%) [1]	Un-extracted (%) [1]	Total (%) [1]
	I_1	I_2	I_3	I_4 + I_5	I_6	I_7	I_8	I_9	I_{10}	I_{11}	I_{12}	I_{13} II_1	I_{14} II_2	II_3	II_4 *	Un-resolved			
Husks (7.8 mg/kg)	7.7	6.2	2.1	1.3	0.9	0.2	n.a.	0.5	1.7	0.2	0.2	ca 2.5	ca 1.5	0.8	49	12.7	0.7	10.0	98
Straw (63 mg/kg)	3.1	2.6	2.5	1.1	0.3	0.3	0.1	0.2	1.2	0.1	0.6	1.7	1.6	0.6	49.	13.	1.5	14.7	95

* II_4 : parent fludioxonil
[1] % of total residues found in sample by combustion. Quantified by 2-D TLC two TLC systems.

Metabolite I_3: CGA 339833
Metabolite I_4: CGA 344623
Metabolite I_{10}: CGA 192155.
Metabolite I_{13}: CGA 308103.
Metabolite II_2: SYN 518579
Metabolite II_3: SYN 518580

Cotton. Plant-uptake, distribution and metabolism of [pyrrole-4-^{14}C]fludioxonil were studied in greenhouse-grown cotton plants (Close, 1998). Cotton seeds (Delta & Pineland 5415) were treated with an FS formulation at 2.5 g or 5.0 g fludioxonil per 100 kg seeds and grown in pots in a sandy loam soil.

Samples at maturity (186 days after planting) consisting of cotton seed, lint, and gin trash (leaves and stalks, forage, allowed to dry) were combusted and the TRR determined by LSC.

Samples with sufficient radioactivity were extracted and partitioned. The residues in the cotton seed were extracted with hexane and then, together with those in seed and gin trash, with acetonitrile/water (80:20). After evaporation of the acetonitrile, the aqueous fraction was partitioned with methylene chloride. Radioactivity was measured by combustion and LSC. This characterised radioactive residues as organosoluble, water-soluble, and unextracted.

The distribution of theTRR in the cotton plants is shown in Table 36. Translocation of radioactivity from treated seeds to aerial plant parts and/or uptake of radioactivity by roots were very low for both treatment rates. For the 2.5 g ai/100 kg seed treatment, mature cotton seed, lint, and gin trash contained radioactive residues equivalent to 0.003 mg/kg, for the 5 g ai/100 kg mature seed (undelinted) and lint 0.012 mg/kg, and for gin trash 0.011 mg/kg.

At 2.5 g ai/100 kg seed residues were too low to be characterised. Extracted radioactive residues in seed and gin trash from the 5 g ai/100 kg seed treatment were <0.005 mg/kg. In cotton seed, extracted residues constituted approximately 33% of the TRR, with organosoluble radioactivity representing 24% (0.003 mg/kg), and in gin trash approximately 19% of the TRR, with organosoluble 4.2% (<0.001 mg/kg). In cotton seed unextracted radioactivity constituted approximately 74% of the TRR, and in gin trash approximately 83%.

Table 36. Distribution of radioactivity in greenhouse-grown cotton plants after treatment of the seed with [pyrrole-4-^{14}C]fludioxonil at 2.5 g/100 kg and 5.0 g/100 kg seed (Close, 1998).

Treatment (g ai/100 kg seed)	Sample	Residue (mg/kg)	Fraction	% of TRR	Conc. (mg/kg)
2.5	Mature cotton seed	0.003			
	Lint	0.003			
	Cotton forage (gin trash)	0.003			

Treatment (g ai/100 kg seed)	Sample	Residue (mg/kg)	Fraction	% of TRR	Conc. (mg/kg)
5.0	Mature cotton seed	0.012		100	0.012
			Hexane	24	0.003
			Acetonitrile/water (80:20) [2]	88	0.011
			Dichloromethane	0.0	<0.001
			Aqueous	8.7	0.001
			Unextracted	74	0.009
			Total recovery [1]	106	0.013
	Lint	0.012			
	Cotton forage (gin trash)	0.011		100	0.011
			Acetonitrile/water (80:20) [2]	54	0.006
			Dichloromethane	4.2	<0.001
			Aqueous	15	0.002
			Unextracted	83	0.009
			Total recovery [3]	103	0.011

[1] calculated by adding hexane fraction, aqueous fraction, dichloromethane fraction, and unextracted residues.
[2] procedural problems led to over-estimation of results.
[3] calculated by adding aqueous fraction, dichloromethane fraction, and unextracted residues.

Soya beans. In a study by Close of the plant-uptake, distribution and metabolism of [pyrrole-4-^{14}C]fludioxonil in greenhouse-grown plants (Report ABR-97033, 1998b) seeds (Novartis Seeds 3474) were treated with a 4FS formulation at 5.0 g ai/100 kg seed.

Plants were sampled at the sixth node stage (above ground portion, soya bean forage, 28 days after planting), at mid full-bloom (above ground portion, soya bean hay, 38 days after planting) and at maturity (stalks, dry beans and dry hulls 133 days after planting).

Homogenized samples were combusted and the TRR determined by LSC, then extracted with acetonitrile/water (80:20), and the extract evaporated to leave an aqueous fraction which was partitioned with dichloromethane. Both the aqueous and organic layers were assayed by LSC. The water-soluble radioactivity was passed through a C-18 SPE cartridge. Non-retained fractions were collected and retained residues eluted with acetonitrile and methanol. All the cartridge fractions were analysed by LSC. The acetonitrile and methanol fractions were combined and analysed by HPLC and TLC. Selected samples of water-soluble fractions were incubated with β-glucosidase, and of unextracted residues treated with cellulase.

The uptake and distribution of the radioactivity into the soya bean plants is summarized in Table 37. Early forage taken 28 days after planting contained the highest radioactive residues at 0.096 mg/kg, the hay 38 days after planting contained a TRR of 0.041 mg/kg, and at harvest (133 days) stalks, beans, and hulls contained residues of 0.005 mg/kg, 0.015 mg/kg, and 0.012 mg/kg respectively.

Table 37. Distribution of radioactivity in greenhouse-grown soya bean plants after treatment of the seed with [pyrrole-4-^{14}C]fludioxonil at 5.0 g/100 kg seed (Close, Report ABR-97033, 1998b).

Sample	Residue (mg/kg)	Fraction	% of TRR	mg/kg
Soya bean forage 6th node	0.096		100	0.096
28 days		Acetonitrile/water (80:20)	1201	0.12
		Organosoluble	9.5	0.009
		Water-soluble	73	0.070
		Non-retained	3.5	0.003
		Acetonitrile	41	0.040
		Methanol	15	0.015
		Unextracted	22	0.021
		Recovery 2	104	0.10
Soya bean forage 6th node	0.096		100	0.096
38 days		Acetonitrile/water (80:20)	85	0.082
		Organosoluble	7.7	0.007
		Water-soluble	66	0.063
		β-glucosidase, dichloromethane	3.2	0.003
		β-glucosidase, aqueous	67	0.065
		Unextracted	19	0.018
		Recovery 2	92	0.088
Soya bean hay 50% flowering	0.041		100	0.041
		Acetonitrile/water (80:20)	2801	0.12
		Organosoluble	2.1	0.001
		Water-soluble	54	0.022
		Non-retained	6.1	0.002
		Acetonitrile	40.	0.016
		Methanol	2.9	0.001
		Unextracted	32	0.013
		Recovery 2	88	0.036
Soya bean stalks	0.005			
Dry beans, harvest	0.015		100	0.015
133 days		Acetonitrile/water (80:20)	2.0	0.000
		Organosoluble	4.4	0.001
		Water-soluble	10	0.002
		Non-retained	8.7	0.001
		Acetonitrile	4.2	0.001
		Methanol	1.1	0.000
		Unextracted	93	0.014
		Cellulase extracted		
		Organosoluble	0.0	0.000
		Water-soluble	16	0.002
		Recovery[2]	107	0.017
Dry hulls	0.012			

[1] high owing to poor replication/counting.
[2] calculated by adding aqueous and organic fractions, and unextracted residues.

The organosoluble residue from forage, hay, and dry beans was analysed using HPLC. The major component in forage matched the retention time of CGA 227731 (1.9% of the TRR and 0.002 mg/kg). Components matching the retention times of CGA 260766, CGA 340351, and CGA 192155 were also present each at levels of <1.4% of the TRR and ≤ 0.001 mg/kg. CGA 227731 was tentatively identified as the major component in soya bean hay (1.5% of the TRR and <0.001 mg/kg) with a small amount of CGA 260766 (0.5% of the TRR and <0.001 mg/kg). One peak was present in dry beans (1.5% of the TRR and <0.001 mg/kg) that did not match any standard. Residues were not confirmed using a second chromatographic system owing to the low level of radioactivity in these fractions (<10% of the TRR, <0.01 mg/kg).

The water-soluble radioactive fraction from forage, hay, and dry beans was passed through C-18 cartridges. The retained fractions after elution with organic solvent contained 57% (0.055 mg/kg) of the forage TRR, 43% (0.017 mg/kg) of the hay and 5% (c. 0.001 mg/kg) of the dry bean, and the corresponding unretained fractions were <10% of the TRR or 0.003 mg/kg. HPLC indicated the presence of CGA 260766, CGA 340351, CGA 227731, CGA 308103, CGA 192155 and CGA 257777 in some of the C18-fractions but confirmatory chromatography was inconclusive. HPLC fractionation showed that no component could be >0.01 mg/kg. Treatment of the forage water-soluble residue with β-glucosidase released only 3.2% of the TRR. The radioactivity was too low for further analysis.

Unextracted radioactivity represented 19%-22%, 32%, and 93% of the TRR in forage, hay, and dry beans respectively, and the residue from dry beans was treated with cellulase which released 18% of the TRR. None of the released radioactivity was organosoluble and only 16% was water-soluble.

Metabolic pathways consistent with the foliar and seed treatment metabolism studies are shown in Figure 2.

Figure 2: Proposed metabolic pathways of fludioxonil in or on plants (foliar, seed treatment).

Environmental fate in soil

Numerous studies were reported on degradation, dissipation under field conditions, adsorption/desorption, mobility, resdiues in succeeding crops, and fate in water. However on the basis of decisions made by the 2003 JMPR (Report 2003, General Items) and confirmed by the 2004 CCPR only aerobic soil degradation and dissipation (seed treatment) and rotational crops are of concern for fludioxonil. Some information on the photolysis of fludioxonil on soil was also considered.

Aerobic degradation

Laboratory studies on fludioxonil under dark conditions in a range of soils showed slow breakdown. Mineralisation to CO_2 was the main breakdown route (4-45%), together with formation of bound residues (8–27%). Mineralisation of [phenyl-U-^{14}C]fludioxonil (Minet, Report 4/93, 1994; Reishmann, Report 7/95, 1994) occurred more rapidly than that of the [pyrrole-4-^{14}C]-labelled compound at 20°C (Kirkpatrick, Report HRC/CBG485/90818, 1991; Abildt, Report 1/91, 1991; Ellgehausen, Report 1/92, 1992; Ellgehausen, Report 91EH08, 1992; Minet, Report 92MU01-1, 1994; Minet, Report 15/93, 1994*).* Extractable radioactivity was <10% of that applied; individual degradation products could not be isolated or characterised owing to the low amounts formed.

The extent of mineralisation and formation of bound residues under aerobic conditions depended on the initial concentration of fludioxonil, label position, soil moisture content and temperature, and initial microbial activity of the soil. In laboratory studies carried out under sterile aerobic conditions degradation of both phenyl and pyrrole-labelled fludioxonil was negligible (Kirkpatrick, Report HRC/CBG485/90818, 1991; Minet, Report 4/93, 1994; Adam, Report 97DA01, 1998).

In contrast degradation of fludioxonil on soil exposed to light was rapid. [Pyrrole-4-^{14}C]fludioxonil at 5 kg ai/ha equivalent on 1 mm layers of sandy loam soil was exposed to a xenon arc light source for up to 44 days (natural sunlight at 30°N equivalent). There was extensive degradation of the ai over this period and a biphasic decline curve (Kirkpatrick, Report CBG569A, CBG 569B, 1994).

On the basis of the best fit to a first-order model, fludioxonil in the irradiated compartment (i.e. at or near the soil surface) initially represented about 40% of the applied radioactivity (AR) and degraded with a half life of <1 day. Fludioxonil in the unirradiated compartment (partially or completely shielded from irradiation) initially represented about 50-60% of the AR with a half-life of 50 days.

In a similar study with [phenyl-U-^{14}C]fludioxonil, photoproducts extracted from the soil were co-chromatographed with those extracted after irradiation of the [pyrrole-4-^{14}C]fludioxonil-treated soil (Kirkpatrick, Report CBG 49064, 1994; Report HRC/CBG516/901362, 1994). Many of the degradates contained both radiolabels, the most significant ones formed from breakdown of the pyrrole ring. Three metabolites were identified: CGA 265378 with a maximum of about 1% of the AR, CGA 339833 9% and CGA 192155 10%. No other photoproduct represented >2% of the AR.

Cumulative evolution of volatiles (mainly CO_2) over the equivalent to about 44 days of natural sunlight accounted for 8% and 9% of the AR for the phenyl and pyrrole-labelled fludioxonil respectively

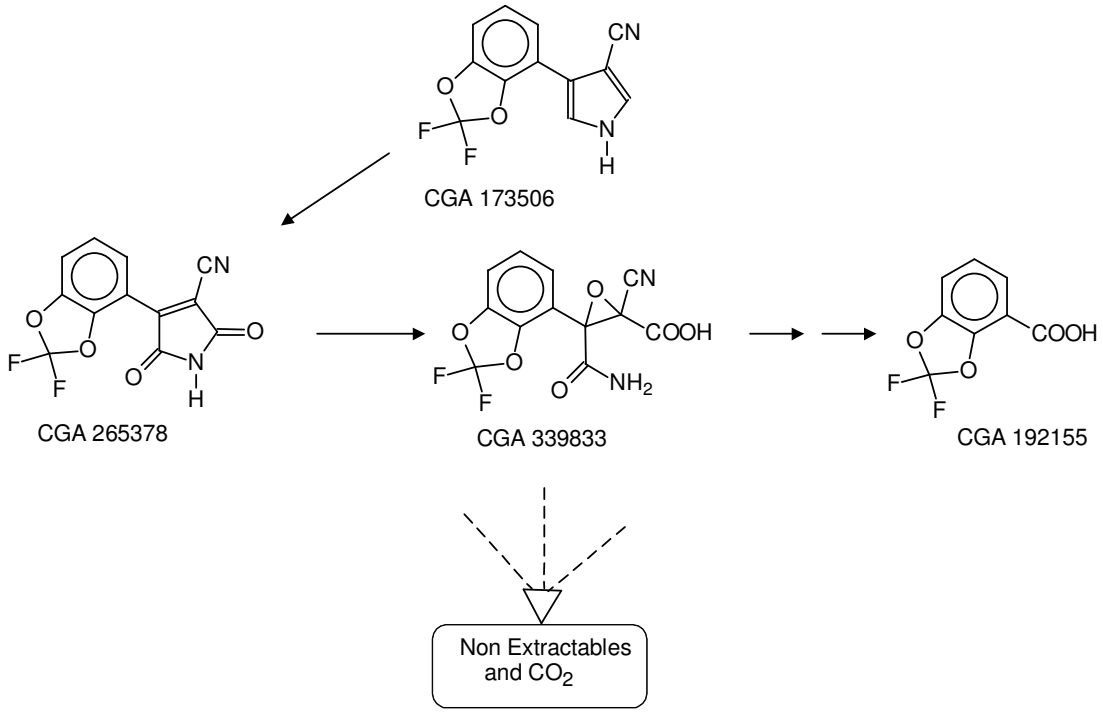
The importance of photodegradation of fludioxonil on the soil surface in field dissipation was demonstrated using [pyrrole-4-^{14}C]fludioxonil (Gentile, Report 89BG02PR3, 1993). A sandy loam plot was sprayed with the labelled compound as a WP 50 formulation at 550 g ai/ha, and divided into two areas, one of which was covered with 2 cm soil immediately after application (covered area) and the other was left uncovered. Over a period of 62 days both areas were protected with a tarpaulin at night and during rain but otherwise were unprotected. Over the initial 62 days the fludioxonil concentration in the 0-10 cm layer decreased from 0.43 mg/kg to 0.40 mg/kg in the covered and from 0.43 mg/kg to 0.34 mg/kg in the exposed area. Unextractable radioactivity increased to about 12% and 30% of the total soil residue in the covered and exposed areas respectively, indicating that the

formation of bound residues is the result of degradation and not of irreversible adsorption of the ai. The two main products in soil extracts from the light-exposed area were identified as CGA 192155 and CGA 339833 and amounted to 13% and 8% of the total residue in the light-exposed soil but only about 2% each in the covered area.

The biphasic decline curves of fludioxonil in the exposed and covered areas were analysed by a two-compartment model. In the former fludioxonil dissipated rapidly with half-lives of <0.1 days in the upper exposed layer and 60 days in the lower (40% and 60% of the applied radioactivity respectively), and in the covered soil 0.4 days in the upper and 255 days in the lower layer (19% and 81% of the AR respectively). Combined rates of both layers gave half-lives of 16 and 187 days for the light-exposed and covered plots respectively.

The proposed pathways of the degradation of fludioxonil in light-exposed soil are shown in Figure 3.

Figure 3: Proposed pathways for the degradation of fludioxonil in light-exposed soil.



Wheat. In two field studies in England *(*Walser, Report 211/98, 1999; Report 212/98, 1999*)* fludioxonil-treated winter wheat seeds were drilled into a clay and a sandy loam soil over a period of 3 years at 12.5 g ai/ha. No residues ≥0.02 mg/kg (LOQ) were found in the soil. In a third field study at two sites in Canada *(*Purdy, Report CER04110/97, 1998*)* maize seed was treated at 2.2 g ai/100 kg seed (corresponding to 0.59 g ai/ha). Dissipation in the soil was monitored using analytical techniques with an LOD of 0.001 mg/kg for fludioxonil, CGA 265378 and CGA 192155. Only fludioxonil was detected and only in the 0-10 cm soil layer. Residues were variable for the first 5-12 days and then steadily decreased from 0.011-0.013 mg/kg to about 0.003 mg/kg between days 79 and 121. By inclusion of a calculated initial (uniform) concentration of 0.028 and 0.024 mg/kg at the two sites, half-lives of 26 and 54 days were calculated assuming first order kinetics.

Rotational crops. Four studies were reported to the Meeting.

In an outdoor confined accumulation study (Gentile, Report 89BG03PR1, 1992) [pyrrole-4-^{14}C]fludioxonil was sprayed onto bare ground in a WP50 formulation in Switzerland at a rate of 750 g ai/ha. The uptake, distribution, and degradation in the following rotational crops were investigated:

Transplanted 90 days after application:	Lettuce (Soraya)
Sown 140 days after application:	Winter wheat (Zenta)
Sown 320 days after application:	Sugar beets (KWS)
Sown 345 days after application:	Maize (Blizzard)

Winter wheat, sugar beets, and corn were sown directly onto the plot and lettuce seedlings were transplanted after the specified ageing periods. Plant samples were collected at selected intervals and stored frozen. Soil cores (0-5 cm, 5-10 cm, 10-20 cm, and 20-30 cm layers) were taken at each planting and plant sampling.

Fresh and dry plant samples were homogenized and the TRR determined by combustion and LSC. The results are shown in Tables 38-41. Characterisation or identification of residues was not attempted owing to the very low residues observed in all four crops.

Table 38. Distribution of radioactivity and residual fludioxonil in rotation lettuce after a bare ground application of [pyrrole-4-^{14}C]fludioxonil at 750 g ai/ha (Gentile, Report 89BG03PR1, 1992).

Days after treatment/ growth stages	Sample	Total residues		Parent	Extracted radioactivity		Unextracted	Total
					Cold	Soxhlet		
		mg/kg [1]	% of TRR [2]	mg/kg	% of TRR [3]	% of TRR [3]	% of TRR [3]	% of TRR [3]
93 days	Soil							
	0-5 cm	0.43	84	0.12	49	3.6	48	101
	5-10 cm	0.052	9.2	0.007	46	3.1	52	100
	10-20 cm	0.010	4.7	n.a.	52	2.6	44	99
	20-30 cm	0.003	2.1	n.a.	n.a.	n.a.	n.a.	
	Soil 0-30 cm	0.069	100					
120 days/ 50% maturity	Lettuce heads	0.006	100	n.a.	n.a.	n.a.	n.a.	
	Soil							
	0-5 cm	0.154	70	0.006	38	4.8	58	101
	5-10 cm	0.036	16	0.003	32	3.5	62	97
	10-20 cm	0.011	12	<0.001	37	2.4	59	98
	20-30 cm	0.002	1.7	n.a.	n.a.	n.a.	n.a.	
	Soil 0-30 cm	0.033	100					
152 days/ maturity	Lettuce heads	0.006	100	n.a.	n.a.	n.a.		
	Soil							
	0-5 cm	0.234	59	0.051	38	3.8	58	100
	5-10 cm	0.080	26	0.015	36	2.9	59	98
	10-20 cm	0.013	11	0.001	51.	1.9	46.	99
	20-30 cm	0.005	3.9	n.a.	n.a.	n.a.	n.a.	
	Soil 0-30 cm	0.044	100					

n.a. : not analysed
LOD for combustion: 0.001 mg/kg; for the TLC of plant and soil material: 0.001 mg/kg
[1] fludioxonil equivalents
[2] % of summed radioactivity in soil layers
[3] % of radioactivity in sample determined by combustion

Table 39. Distribution of radioactivity and residual fludioxonil in rotational winter wheat after a bare ground application of [pyrrole-4-14C]fludioxonil at 750 g ai/ha (Gentile, Report 89BG03PR1, 1992).

Days after treatment/ growth stages	Sample	Total residues		Parent	Extracted radioactivity		Unextracted	Total
		mg/kg [1]	% of TRR [2]	mg/kg	Cold	Soxhlet	% of TRR [3]	% of TRR [3]
					% of TRR [3]	% of TRR [3]		
152 days before sowing of winter wheat	Soil							
	0-5 cm	0.23	59.	0.051	38	3.8	58	100
	5-10 cm	0.08	25.	0.015	36	2.9	59	98
	10-20 cm	0.013	11.	0.001	51	1.9	46	99
	20-30 cm	0.005	3.9	n.a.	n.a.	n.a.	n.a.	
	Soil 0-30 cm	0.044	100					
196 days/ fall cutting	Whole tops	0.005	100	n.a.	n.a.	n.a.	n.a.	
	Soil							
	0-5 cm	0.275	49	0.071	34	7.3	61	102
	5-10 cm	0.116	21	0.023	30	6.5	66	103
	10-20 cm	0.045	22	n.a.	n.a.	n.a.	n.a.	
	20-30 cm	0.011	7.8	n.a.	n.a.	n.a.	n.a.	
	Soil 0-30 cm	0.065	100					
321 days/ 25% maturity	Whole tops	0.002	100	n.a.	n.a.	n.a.	n.a.	
	Soil							
	0-5 cm	0.19	58	0.038	31	4.2	61	96
	5-10 cm	0.055	17.1	0.008	26	4.1	68	98
	10-20 cm	0.030	16	n.a.	n.a.	n.a.	n.a.	
	20-30 cm	0.009	8.7	n.a.	n.a.	n.a.	n.a.	
	Soil 0-30 cm	0.047	100					
377 days/ 50% maturity	Whole tops	0.002	100	n.a.	n.a.	n.a.	n.a.	
	Soil							
	0-5 cm	0.13	55	0.022	26	4.4	67	97
	5-10 cm	0.060	20	0.007	27	4.2	76	107
	10-20 cm	0.037	21	n.a.	n.a.	n.a.	n.a.	
	20-30 cm	0.004	3.8	n.a.	n.a.	n.a.	n.a.	
	Soil 0-30 cm	0.044	100.0					
429 days/ maturity	Stems	0.008	100	n.a.	n.a.	n.a.	n.a.	
	Husks	0.004	100	n.a.	n.a.	n.a.	n.a.	
	Grains	0.002	100	n.a.	n.a.	n.a.	n.a.	
	Soil							
	0-5 cm	0.290	70	0.066	33	3.2	53	89
	5-10 cm	0.077	18	0.012	27	11.	58	97
	10-20 cm	0.021	9.2	n.a.	n.a.	n.a.	n.a.	
	20-30 cm	0.005	2.9	n.a.	n.a.	n.a.	n.a.	
	Soil 0-30 cm		100					

n.a. - not analysed
LOD: for combustion: 0.001 mg/kg, for TLC of plant and soil material: 0.001 mg/kg
[1] fludioxonil equivalents
[2] % of summed radioactivity in soil layers
[3] % of radioactivity in sample determined by combustion

Table 40. Distribution of radioactivity and residual fludioxonil in rotational sugar beets after a bare ground application of [pyrrole-4-^{14}C]fludioxonil at 750 g ai/ha (Gentile, Report 89BG03PR1, 1992).

Days after treatment/ growth stages	Sample	Total residues		Parent	Extracted radioactivity		Unextracted	Total
		mg/kg [1]	% of TRR [2]	mg/kg	Cold	Soxhlet	% of TRR [3]	% of TRR [3]
					% of TRR [3]	% of TRR [3]		
321 days/ before sowing of sugar beets	Soil							
	0-5 cm	0.37	49	0.10	38	9.8	51	98
	5-10 cm	0.33	30	0.10	52	13.	36.	101
	10-20 cm	0.096	20	n.a.	n.a.	n.a.	n.a.	
	20-30 cm	0.006	1.9	n.a.	n.a.	n.a.	n.a.	
	Soil 0-30 cm	0.14	100					
377 days/ 25% maturity	Tops	0.002	100	n.a.	n.a.	n.a.	n.a.	
	Roots	0.002	100	n.a.	n.a.	n.a.	n.a.	
	Soil							
	0-5 cm	0.190	71	0.036	30	9.7	57.	96

Days after treatment/ growth stages	Sample	Total residues		Parent	Extracted radioactivity		Unextracted	Total
					Cold	Soxhlet		
		mg/kg [1]	% of TRR [2]	mg/kg	% of TRR [3]	% of TRR [3]	% of TRR [3]	% of TRR [3]
	5-10 cm	0.058	16	0.007	24	15.	65.	104
	10-20 cm	0.018	11	n.a.	n.a.	n.a.	n.a.	
	20-30 cm	0.003	2.2	n.a.	n.a.	n.a.	n.a.	
	Soil 0-30 cm	0.051	100					
419 days/ 50% maturity	Tops	<0.001	100	n.a.	n.a.	n.a.	n.a.	
	Roots	0.002	100	n.a.	n.a.	n.a.	n.a.	
	Soil							
	0-5 cm	0.201	48	0.038	27.9	6.2	62	96
	5-10 cm	0.122	27	0.022	27.8	17	62	107
	10-20 cm	0.030	19	n.a.	n.a.	n.a.	n.a.	
	20-30 cm	0.009	6.3	n.a.	n.a.	n.a.	n.a.	
	Soil 0-30 cm	0.056	100					
519 days/ maturity	Tops	<0.001	100	n.a.	n.a.	n.a.	n.a.	
	Roots	0.001	100	n.a.	n.a.	n.a.	n.a.	
	Soil							
	0-05 cm	0.147	44	0.019	25	6.4	66	97
	5-10 cm	0.135	29	0.019	24	5.4	68	97
	10-20 cm	0.048	24	n.a.	n.a.	n.a.	n.a.	
	20-30 cm	0.004	3.0	n.a.	n.a.	n.a.	n.a.	
	Soil 0-30 cm	0.058	100					

n.a. : not analysed. LOD for combustion: 0.001 mg/kg; for TLC of plant and soil material: 0.001 mg/kg
[1] fludioxonil equivalents
[2] % of summed radioactivity in soil layers
[3] % of radioactivity in sample determined by combustion

Table 41. Distribution of radioactivity and residual fludioxonil in rotational maize after a bare ground application of [pyrrole-4-^{14}C]fludioxonil at 750 g ai/ha (Gentile, Report 89BG03PR1, 1992).

Days after treatment/ growth stages	Sample	Total residues		Parent	Extracted radioactivity		Unextracted	Total
					Cold	Soxhlet		
		mg/kg [1]	% of TRR [2]	mg/kg	% of TRR [3]	% of TRR [3]	% of TRR [3]	% of TRR [3]
	Soil							
352 days before sowing of corn	0-5 cm	0.18	66	0.034	31	4.4	61	96
	5-10 cm	0.053	14	0.008	26	4.7	63	94
	10-20 cm	0.028	18	n.a.	n.a.	n.a.	n.a.	
	20-30 cm	0.003	2.2	n.a.	n.a.	n.a.	n.a.	
	Soil 0-30 cm	0.047	100					
377 days 25% maturity	Whole tops	0.003	100	n.a.	n.a.	n.a.	n.a.	
	Soil							
	0-5 cm	0.330	56	0.070	34	8.4	57	99
	5-10 cm	0.18	23	0.035	31	5.4	63	100
	10-20 cm	0.051	16	n.a.	n.a.	n.a.	n.a.	
	20-30 cm	0.011	5.2	n.a.	n.a.	n.a.	n.a.	
	Soil 0-30 cm	0.093	100					
419 days 50% maturity	Whole tops	<0.001	100	n.a.	n.a.	n.a.	n.a.	
	Soil							
	0-5 cm	0.19	50	0.030	27	5.8	66	99
	5-10 cm	0.10	24	0.011	22	2.9	69	93
	10-20 cm	0.033	19	n.a.	n.a.	n.a.	n.a.	
	20-30 cm	0.011	8.3	n.a.	n.a.	n.a.	n.a.	
	Soil 0-30 cm	0.055	100					
519 days maturity	Stalks	0.005	100	n.a.	n.a.	n.a.	n.a.	
	Cobs	<0.001	100	n.a.	n.a.	n.a.	n.a.	
	Grains	<0.001	100	n.a.	n.a.	n.a.	n.a.	
	Soil							
	0-5 cm	0.15	44	0.019	25	6.4	66	97
	5-10 cm	0.14	29	0.019	24	5.4	68	97
	10-20 cm	0.048	24	n.a.	n.a.	n.a.	n.a.	
	20-30 cm	0.004	3.0	n.a.	n.a.	n.a.	n.a.	
	Soil 0-30 cm	0.058	100					

n.a. - not analysed
LOD for combustion: 0.001 mg/kg
[1] fludioxonil equivalents
[2] % of summed radioactivity in soil layers
[3] % of radioactivity in sample determined by combustion

[Pyrrole-4-^{14}C]fludioxonil in methanol was sprayed onto bare ground in California, USA at a rate of 50 g ai/acre (124 g ai/ha). Uptake, distribution, and degradation were investigated in the following rotational crops (Thalacker, Report CHW 6117-329, 1996):

Sown 33 days and 90 days after application:	Spring wheat (Aldura), mustard (Florida Broadleaf), and turnips (Purple Top White Globe)

Spring wheat, mustard, and turnips were sown directly into the plot after the specified ageing periods. (The trial initially set up was discontinued owing to high background values of fludioxonil found in pre-treatment soil cores). Plant samples were collected at selected intervals as were soil cores but the latter will not be discussed further.

Plant samples were homogenized with solid CO_2 to keep them frozen. Subsamples were combusted to determine the TRR, and residues in each rotational crop sample were extracted with methanol/water (80:20) fractionated by evaporation of the methanol and partitioning of the resulting aqueous solution with dichloromethane. Additional fractionation of the water-soluble and the unextracted radioactivity was by enzymatic, acidic or basic procedures. Selected extract fractions were profiled using HPLC and/or 2D-TLC. Unextracted radioactivity was determined by combustion and LSC. The results are shown in Table 42.

Table 42. Distribution of radioactivity and residual fludioxonil in rotation crops after a bare ground application of [pyrrole-4-^{14}C]fludioxonil at 124 g ai/ha (Thalacker, Report CHW 6117-329, 1996).

Sample	Description	Harvest interval (days)	TRR mg/kg	Extracted						Unextracted		Recovery[1]	
				MeOH:H_2O (80:20)		Aqueous		Organic					
				% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg
33 day plant back													
0-15 cm Soil	Pre-application	0	<0.001										
0-15 cm Soil	Post application	0	0.029										
0-15 cm Soil	Day of planting	33	0.041										
Root crop	Mature turnip tops	129	0.007	63	0.004	63	0.004	18	0.001	30	0.002	110	0.007
Root crop	Mature turnip roots	129	0.002										
Leafy vegetable	Mature mustard greens	129	0.006	95	0.006	70	0.004	32	0.002	29	0.002	130	0.008
Small Grain	25% mature wheat forage	109	0.058	80	0.047	57	0.033	26	0.015	32	0.019	115	0.067
				65	0.038	59	0.034	7.9	0.005	32	0.018	98	0.057
	Mature wheat straw	175	0.12	59	0.071	56	0.067	6.5	0.008	35	0.042	98	0.12
				63	0.075	56	0.067	7.0	0.008	34	0.041	98	0.12
	Mature wheat grain	175	0.006	39	0.002	33	0.002	14	0.001	49	0.003	97	0.006

[1] Sum of aqueous and organic fraction, and unextracted residues.

HPLC indicated that fludioxonil constituted 2.4% of the TRR (0.001 mg/kg) in the organosoluble fraction of 25% maturity wheat forage. Other radioactive peaks corresponded to CGA 265378, CGA 192155, and CGA 308103, but all were <1.5% of the TRR (<0.001 mg/kg). The residues were not verified by a second chromatographic system. At full maturity in harvest wheat straw fludioxonil had decreased to 0.3% of the TRR (<0.001 mg/kg). CGA 308103 (1.1% of the TRR, 0.001 mg/kg) and CGA 192155 (0.9% of the TRR, 0.001 mg/kg) were detected by co-chromatography with non-radiolabelled standards. The residues were not verified using a second chromatographic system.

HPLC analysis of the water-soluble fraction of 25% maturity wheat forage suggested the presence of CGA 227731 (10.7% of the TRR, 0.006 mg/kg), CGA 260766 (4.2% of the TRR, 0.002 mg/kg), and CGA 192155 (2.3% of the TRR, 0.001 mg/kg). Two unidentified polar peaks were 18.3% (0.011 mg/kg), and 12.7% (0.007 mg/kg) of the TRR. Co-chromatography using 2D-TLC confirmed the presence of CGA 227731.

HPLC of the water-soluble fraction from wheat straw harvested at full maturity suggested the presence of CGA 227731 (11.1% of the TRR, 0.013 mg/kg), CGA 260766 (4.8%, 0.006 mg/kg), CGA 340351 (1.2%, 0.001 mg/kg), and CGA 192155 (1.8%, 0.002 mg/kg). Two unidentified polar peaks were 13.1% (0.016 mg/kg), and 11.7% (0.014 mg/kg) of the TRR. Co-chromatography using 2D-TLC did not confirm HPLC identifications.

Enzyme treatment of the aqueous fractions from immature forage and mature straw with β-glucosidase released approximately 3% of the TRR (0.002-0.003 mg/kg). The HPLC profiles were similar to those of the untreated fractions.

Treatment of the wheat forage and straw aqueous fractions with three concentrations of HCl or NaOH released a maximum of 7% of the TRR into the organosoluble fraction. HPLC of the organosoluble fractions indicated the presence of CGA 340351, CGA 308103, CGA 260766, CGA 227731, CGA 265378 and CGA 192155 but these metabolites were not confirmed by a second system. None of these components was above 3.4% of the TRR (0.004 mg/kg). HPLC of the water-soluble radioactivity indicated the presence of CGA 308103, CGA 227731, CGA 192155 and CGA 340351. No component tentatively identified was above 5.1% of the TRR (0.003 mg/kg) except CGA 227731, which appeared to be highest in full maturity wheat straw at 15.9% of the TRR (0.019 mg/kg) after treatment with 1.0 N NaOH. This was not confirmed by a second chromatography system.

Treatment of the unextracted residues from wheat forage with 0.1N NaOH under reflux released 24.5% of the TRR (0.014 mg/kg) and of the full maturity straw 13.8% (0.017 mg/kg). HPLC of the forage extract produced five radioactive peaks that appeared to co-elute with CGA-308103, CGA 227731, CGA 260766, CGA 192155, and CGA 257777. None of these components was above 2.3% of the TRR (0.001 mg/kg) and none were confirmed by a second chromatography method. No defined peak areas were detected when wheat straw was subjected to the same treatment.

Treatment of the unextracted residues with cellulase and base reduced the unextracted radioactivity to 0.35% of the TRR (<0.001 mg/kg) and 2.04% (0.002 mg/kg), and with cellulase and acid to 11.4% of the TRR (0.007 mg/kg) and 22.1% (0.027 mg/kg) in the immature wheat forage and mature wheat straw respectively. The HPLC profiles of the compounds released by cellulase was similar to the aqueous profile. The components released by acid treatment were more polar than the standards by HPLC.

[Pyrrole-4-^{14}C]fludioxonil in methanol was sprayed onto bare ground in California, USA at a rate of 62 g ai/ha. Uptake, distribution, and degradation of ^{14}C-fludioxonil were investigated in the following rotational crops (Close, Report ABR-97005, 1997):

Sown 32 and 90 days after application:	Spring wheat (Yacaro rojo), mustard (Florida Broadleaf), and radishes (Cherry Belle)

Spring wheat, mustard, and radishes were sown directly into the plot after the specified ageing periods.

Plant samples were extracted, fractionated and analysed as above

The results are summarized in Table 43.

Table 43. Distribution of radioactivity and residual fludioxonil in rotation crops after a bare ground application of [pyrrole-4-14C]fludioxonil at 25 g ai/acre (Close, Report ABR-97005, 1997):

Sample	Description	PHI (days)	TRR (mg/kg)	Extracted						Unextracted		Recovery[1]	
				MeOH:H2O (80:20)		Aqueous		Organic					
				% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg
32 day plant back													
0-15 cm Soil	Pre-application	0	<0.002										
0-15 cm Soil	Post application	0	0.021										
0-15 cm Soil	Day of planting	32	0.018										
0-15 cm Soil	Final harvest	206	0.010										
Root crop	Mature radish tops	117	0.002										
Root crop	Mature radish roots	117	0.002										
Leafy vegetable	Mature mustard greens	117	<0.002										
Small grain	25% mature wheat forage	117	0.011	58	0.0064	44	0.0048	15	0.0017	40	0.0044	99	0.0109
	50% mature wheat forage	135	0.008										
	Mature wheat straw	204	0.056	54	0.030	47	0.026	3.6	0.002	49	0.027	99	0.055
				60	0.034	52	0.029	2.5	0.001	47	0.026	102	0.056
	Mature wheat grain	204	0.015	34	0.005	29	0.004	8.9	0.001	69	0.010	107	0.015

[1] Sum of aqueous and organic fraction, and unextracted residues.

The radioactivity in the mature mustard and radishes was too low to characterise, as it was below or at the limit of quantification (0.002 mg/kg).

The organic fraction of wheat straw was analysed by HPLC and suggested one major component, CGA 308103 (1.1% of the TRR, <0.001 mg/kg). The residue was not confirmed owing to the low level of radioactivity in the fraction (4.6% of the TRR, 0.003 mg/kg). The organosoluble radioactivity in wheat grain (8.9% of the TRR, 0.001 mg/kg) was too low to analyse by HPLC.

The water-soluble fraction of the straw was also analysed by HPLC (C-18 retained and unretained fractions) and eluted in the vicinity of CGA 227731 (22% of the TRR, 0.012 mg/kg), CGA 260766 (6.5%, 0.004 mg/kg), CGA 192155 (1.4%, <0.001 mg/kg) and CGA 265378 (1.1%, <0.001 mg/kg). The HPLC assignments were not confirmed owing to the low level of radioactivity in the fraction (32% of the TRR, 0.018 mg/kg, C-18 retained fraction and 12% of the TRR, 0.007 mg/kg, C-18 unretained fraction).

The water-soluble fraction of wheat grain was analysed by HPLC (C-18 retained and non-retained fractions). Most of the radioactivity eluted near CGA 308103 (12.2% of the TRR, 0.001 mg/kg), some eluting near CGA 340351 (2.0%, <0.001 mg/kg). The HPLC assignments were not confirmed.

Treatment of the wheat straw aqueous fraction with enzyme released 2.1% of the TRR (0.001 mg/kg).

Treatment of the unextracted residue of wheat straw and grain with enzyme released 16% of the TRR (0.009 mg/kg) and 21% of the TRR (0.003 mg/kg) respectively. In straw 1.0% of the TRR (0.001 mg/kg) was organosoluble and 7.6% (0.004 mg/kg) was water-soluble, and in grain none was organosoluble and 11% (0.002 mg/kg) was water-soluble. The extracted residue was not analysed chromatographically.

Tentative identifications of CGA 265378, CGA 192155, CGA 308103, CGA 227731 and CGA-260766 in the wheat straw extracts and CGA 308103 and CGA 340351 in the grain were made using one chromatographic system. All were <0.01 mg/kg except CGA 227731 found at 0.012 mg/kg in the straw.

[Phenyl-U-^{14}C]fludioxonil in methanol was sprayed onto bare ground in California, USA at an exaggerated rate of 452 g ai/acre (1117 g ai/ha). Uptake, distribution, and degradation of ^{14}C-fludioxonil were investigated in the following rotational crops (Thalacker, Report 117-97, 1999):

Planted 30, 90 and 210 days after application:	Spring wheat (Yacaro rojo), Mustard (Florida Broadleaf), and Radishes (Cherry Belle)

Spring wheat, mustard, and radishes were sown directly into the plot after the specified ageing periods and plant and soil samples collected at intervals.

Plant samples were homogenized using a food grinder or a Wiley Mill with liquid nitrogen or solid CO_2 to keep the samples frozen. Sub-samples were combusted to determine the TRR. The radioactive residues in the crop samples were extracted with acetonitrile (ACN)/water (80:20) and initially cleaned up using C-18 chromatography. The acetonitrile was evaporated and the remaining aqueous solution was partitioned with methyl *tert*-butyl ether. Further fractionation of the water-soluble components was with cellulase, and of the unextracted radioactive residues by enzymatic, acidic and basic procedures. Selected organic and water-soluble fractions were profiled using HPLC and/or two-dimensional 2D-TLC. Unextracted radioactivity was determined by combustion and LSC.

The results are summarized in Tables 44 and 45.

Table 44. Distribution of radioactivity in rotation crops and soil after a bare ground application of [phenyl-U-14C]fludioxonil at 1120 g ai/ha (Thalacker, Report 117-97, 1999):

Plant-back interval, days	Sample	TRR mg/kg	ACN/H_2O extract		Organic (MtBE) fraction		Aqueous fraction		Unextracted		Recovery, % of TRR
			% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg	
30	Mature Mustard Leaves	0.033	56	0.018	11.	0.004	34	0.011	52	0.017	108
	Mature Radish Leaves	0.11	62	0.066	18	0.019	41	0.043	42	0.045	104
	Mature Radish Tuber	0.14	49	0.066	22	0.029	21	0.029	54	0.073	103
	25% Mature Wheat Forage	0.080	65	0.052	14.	0.011	48	0.039	26	0.021	91.
	50% Mature Wheat Forage	0.067	70.	0.047	22.	0.015	51	0.034	29	0.020	100
	Mature Wheat Straw	0.36	44	0.16	16.	0.059	24	0.087	53	0.190	97.
	Mature Wheat Grain	0.058	16	0.009	8.2	0.005	13	0.008	81	0.047	97.
	Soil post application	0.43	101	0.44	NA	NA	NA	NA	1.8	0.008	103.
	Soil at planting	0.32	70.	0.23	NA	NA	NA	NA	45	0.089	94
	Soil at 50% mature wheat harvest	0.20	53	0.11	NA	NA	NA	NA	45	0.089	98
90	Mature Mustard Leaves	0.044	79	0.035	40	0.017	48	0.021	13	0.006	92
	Mature Radish Leaves	0.021	75	0.016	19.	0.004	31	0.006	37	0.008	112.
	Mature Radish Tuber	0.019	94	0.016	53	0.010	35	0.007	12	0.002	106
	25% Mature Wheat Forage	0.15	76	0.012	30	0.045	49	0.075	18	0.027	94
	50% Mature Wheat Forage	0.095	78	0.074	19	0.018	59	0.056	22	0.021	100
	Mature Wheat Straw	0.14	46	0.063	16.	0.022	27	0.036	59	0.080	105
	Mature Wheat Grain	0.021	10	0.002	4.3	0.001	8.3	0.002	87	0.018	98
	Soil post application	1.03	99	1.02	NA	NA	NA	NA	1.6	0.016	101
	Soil at planting	0.31	64	0.20	NA	NA	NA	NA	38	0.12	101
	Soil at 50% mature wheat harvest	0.15	26	0.039	NA	NA	NA	NA	68	0.105	93
210	Mature Mustard Leaves	0.050	84	0.042	40.	0.020	40	0.020	32	0.016	116
	Mature Radish Leaves	0.022	67	0.015	21.	0.005	41	0.009	40	0.009	106
	Mature Radish Tuber	0.019	63	0.012	23	0.004	29	0.005	40	0.008	103
	25% Mature Wheat Forage	0.11	73	0.080	38	0.042	52	0.057	19	0.021	92
	50% Mature Wheat Forage	0.089	66	0.058	30	0.027	35	0.031	31	0.027	96
	Mature Wheat Straw	0.11	40	0.043	17	0.018	22	0.023	57	0.061	97
	Mature Wheat Grain	0.019	13	0.003	3.9	0.001	7.0	0.001	85	0.016	98
	Soil at planting	0.28	45	0.13	NA	NA	NA	NA	48	0.14	93
	Soil at 50% mature wheat harvest	0.22	18	0.039	NA	NA	NA	NA	71	0.16	88

Table 45. Identification of metabolites in rotation crops after a bare ground application of [phenyl-U-14C]fludioxonil at 1120 g ai/ha (Thalacker, Report 117-97, 1999).

Sample	TRR mg/kg	% of TRR Organic	Fludioxonil		CGA-192155		CGA-308103		CGA-265378		CGA-308565		CGA-339833		CGA-344623		Total characterised	
			% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg
30-DAT																		
Mature Mustard Leaves	0.033	11	Not profiled															
Mature Radish Leaves	0.11	18	2.0	0.002	8.0	0.008	1.6	0.002	1.2	0.001	1.6	0.002	0.7	<0.001	1.6	0.002	16.7	0.018
Mature Radish Tuber	0.14	22	11.8	0.016	4.8	0.007	0.3	<0.001	1.6	0.002	0.7	<0.001	0.4	<0.001	0.7	0.001	20.	0.027
25% Mature Wheat Forage	0.080	14	0.5	<0.001	7.2	0.006	1.1	<0.001	0.9	<0.001	0.9	<0.001	1.1	<0.01	1.1	<0.001	13.	
50% Mature Wheat Forage	0.067	22	3.5	0.002	7.5	0.005	2.1	0.001	1.4	<0.001	1.0	<0.001	1.7	0.001	1.3	<0.001	18.	0.012
Mature Wheat Straw	0.36	16	0.2	<0.001	4.4	0.015	0.2	0.001	0.1	0.001	ND	NA	0.1	<0.001	0.3	0.001	5.3	0.019
Mature Wheat Grain	0.058	8.2																
90-DAT																		
Mature Mustard Leaves	0.044	40	0.8	<0.001	28	0.012	1.1	<0.001	ND	NA	ND	NA	0.9	<0.001	ND	NA	30	0.013
Mature Radish Leaves	0.021	19.	Not profiled															
Mature Radish Tuber	0.019	53	4.3	<0.001	38	0.007	ND	NA	ND	NA	ND	NA	ND	NA	ND	NA	43	0.008
25% Mature Wheat Forage	0.15	30	0.7	0.001	19	0.029	0.7	0.001	0.9	0.001	1.7	0.003	3.1	0.005	2.1	0.003	28	0.044
50% Mature Wheat Forage	0.095	19	0.5	<0.001	13	0.012	0.5	<0.001	0.5	<0.001	0.8	<0.001	1.4	0.001	1.6	0.002	18	0.017
Mature Wheat Straw	0.14	16	0.2	<0.001	7.0	0.010	0.6	<0.001	0.3	<0.001	0.3	<0.001	ND	NA	1.7	0.002	10	0.014
Mature Wheat Grain	0.021	4.3																
210-DAT																		
Mature Mustard Leaves	0.050	40	2.1	0.001	10.4	0.005	1.5	<0.001	ND	NA	ND	NA	ND	NA	ND	NA	14	0.007
Mature Radish Leaves	0.022	21	Not profiled															
Mature Radish Tuber	0.019	23	Not profiled															
25% Mature Wheat Forage	0.11	38	1.9	0.002	18.2	0.020	3.9	0.004	1.6	0.002	2.2	0.002	3.7	0.004	3.0	0.003	34	0.038
50% Mature Wheat Forage	0.089	30	1.3	0.001	1.0	0.009	1.8	0.002	ND	NA	ND	NA	1.6	0.001	1.1	<0.001	16	0.014
Mature Wheat Straw	0.11	17	0.2	<0.001	7.8	0.008	0.5	<0.001	0.2	<0.001	0.3	<0.001	4.6	0.005	0.9	<0.001	14	0.016
Mature Wheat Grain	0.019	3.9	Not profiled															

HPLC and 2 D-TLC analyses of the organosoluble fractions of various crop tissues from the 30-day plant back identified residues of fludioxonil, CGA 192155, CGA 308103 and CGA 344623. CGA 265378, CGA 308565 and CGA 339833 were indicated by HPLC. The same compounds were indicated in the organosoluble fractions of crop tissues from the 90 and 210-day plant back. Only fludioxonil and CGA 192155 were detected at or above 0.01 mg/kg. Fludioxonil constituted 0.2% to 11.8% of the TRR (<0.001 to 0.016 mg/kg), the main metabolite CGA 192155 4.8% to 38.3% of the TRR (0.005 to 0.029 mg/kg), and the minor metabolites CGA 308103, CGA 265378, CGA 308565, and CGA 344623 each <4% of the TRR (<0.005 mg/kg). CGA339833 reached 4.6% in mature wheat straw.

HPLC analysis of the water-soluble fractions from mature straw indicated the presence of fludioxonil and CGA 192155 below 0.02 mg/kg but these assignments were not confirmed by TLC. CGA 308103, CGA 265378, CGA 308565, CGA 344623 and CGA 339833 were also indicated by HPLC in some fractions but again unconfirmed.

Treatment of aqueous samples from 25% mature (90 and 210-day plant back) and 50% mature (90-day plant back) forage and mature wheat straw (30-day plant back) with cellulase released 3.1% to 10.0% of the TRR (0.009 to 0.014 mg/kg) as organosoluble residues. CGA 192155 was the major component at a maximum of 0.01 mg/kg. No other components exceeded 0.002 mg/kg.

Enzyme treatment of the unextracted residues from 30-day plant back radish tuber (containing 54.4% of the TRR) and mature wheat straw (containing 53.4% of the TRR) released 3.41% of the TRR (0.005 mg/kg) and 4.58% (0.016 mg/kg) respectively. HPLC analyses of the released material showed a void volume peak and diffuse base-level radioactive residues. Further sequential treatment of these unextracted fractions with 0.1M HCl, 1.0 M HCl, 0.1M NaOH and 1.0M NaOH released totals of 24.8% of the TRR (0.033 mg/kg) and 37.1% (0.132 mg/kg) of radioactive residues. After the base treatment, 8.5% (0.011 mg/kg) and 7.1% (0.025 mg/kg) remained unextracted. After each treatment more of the released radioactivity was water-soluble than organosoluble. Chromatographic profiling of the organosoluble residues indicated the presence of fludioxonil, CGA 192155, CGA 308103, CGA 265378, CGA 308565, CGA 339833 and CGA 344623. None of these were above 2.1% of the TRR (0.007 mg/kg). Most of the water-soluble residues were more polar than available reference standards by HPLC..

The highest radioactive residues were seen in cereal straw (0.36 mg/kg) and radish tuber (0.14 mg/kg) at the 30-day planting interval. At the 90 and 210-day planting intervals total radioactive residues were at or below 0.05 mg/kg in all food crops and <0.2 mg/kg. in animal feed items. Fludioxonil was present between 0.2% and 12% of the TRR (<0.001 to 0.016 mg/kg). CGA 192155, the major identified metabolite, was present between 4.4% and 38% of the TRR (0.005 to 0.046 mg/kg), and was no higher than 0.012 mg/kg in food crops. The minor metabolites CGA 308103, CGA 265378, CGA 308565, CGA 339833 and CGA 344623 were <4% of the TRR (0.005 mg/kg).

In further rotational crop trials fludioxonil (50WP formulation at a rate of 282 g ai/ha/application, total 1126 g ai/ha) was applied four times at 7-day intervals to bare soil in California, Florida, and New York (Joseph, Report 174-97, 1999). Cyprodinil was also applied as a 75 WG formulation at a rate of 561 g ai/ha. The California site was the location of previous confined rotational studies using ^{14}C-fludioxonil (see above).

Wheat, turnips, and leaf lettuce were sown or planted 30, 90, 150 and 210 days after the last application. Plant samples collected at selected intervals and kept frozen before analysis were homogenised and extracted with acetonitrile/water (90:10). An aliquot was evaporated to an aqueous solution, methanol and water added, and the sample loaded onto a phenyl solid-phase extraction cartridge. Fludioxonil was eluted with acetone/water and the eluate evaporated to the aqueous solution and partitioned with methyl *tert*-butyl ether. After solvent exchange to hexane, the residues were further cleaned up on a silica extraction cartridge. Residues were determined by normal phase HPLC with UV detection at 268 nm. These procedures are described in Analytical Method AG-597B (see below). The limit of quantification was 0.01 mg/kg for all samples.

Samples of wheat forage, hay, straw and grain, turnip tops and roots, and leaf lettuce from the 30-day planting were analysed for fludioxonil (and cyprodinil). No detectable residues of fludioxonil (<0.01 mg/kg, duplicate samples) were found in any of the samples at any location so the samples from the longer plant-back intervals were not analysed.

Results with pyrrole- and phenyl-labelled fludioxonil were similar.They showed that fludioxonil was not taken up into rotational crops. The metabolism of fludioxonil in rotational crops was the same as that seen in target crop studies and is characterised by oxidation and cleavage of the pyrrole ring. No metabolites indicating cleavage of the bond between the phenyl and the pyrrole rings were observed, showing that either ^{14}C label is suitable for metabolic studies.

RESIDUE ANALYSIS

Analytical methods

Analytical methods for the determination of residues of fludioxonil in crops and animal products (meat, fat, liver, kidney, milk and eggs) were reported.

Two HPLC methods, one with column switching (REM 133.04, AG-631A) and one without (AG- 597), and a multiresidue method (DFG S19) are described for determination of fludioxonil residues in plant materials. They determine fludioxonil only. Method REM 133.04 (Mair, Report REM-133-04, 1993) was developed from REM 133.01 (Hohl, Report REM-133-01, 1989*)* and REM 133.03 (Maffezzoni, Report RES 133.03, 1996). Homogenized plant samples are extracted with methanol. An aliquot of the filtered extract is diluted with water and passed through a phenyl solid-phase extraction cartridge. The analyte is eluted from the cartridge with acetone. The eluate is diluted with water, saturated with sodium chloride and partitioned against hexane/diethyl ether (8:2). The organic phase is evaporated to dryness and reconstituted with hexane/isopropanol (9:1). Determination is by normal-phase HPLC using a two-column switching system with UV detection at 268 nm. Typically, the column is switched from a C-18 column to a phenyl column immediately before elution of fludioxonil.

Wine is diluted with water and treated in the same way as the methanol extract of plant material.

Residues of fludioxonil can be confirmed by GC-MS using an ion trap and diagnostic ions m/z 248, 127, 154 and 182.

Validation was carried out as part of the method development and showed that the overall average recovery for the method from 30 samples was 91% (range 71-111%), Table 46. The lowest practical level of determination is stated to be 0.02 mg/kg for plant material and soil, and 0.005 mg/kg for wine. The demonstrated limit of quantification was 0.04 mg/kg, except 0.01 mg/kg for wine.

Table 46: Recoveries by analytical method REM 133-04 for determination of fludioxonil in crops (Mair, Report REM-133.04, 1993).

Sample	Fortification level	Recovery [%]		SD (rel)	No.
	[mg/kg]	Mean	Range	[%]	
Grapes	0.04	88	83, 93	-	2
	0.4	88	88, 88	-	2
Wine	0.01	90	90	-	1
	0.1	89	89	-	1
Tomatoes	0.04	91	81-97	7.1	4
	0.4	89	87-91	1.7	4
Strawberries	0.04	104	104	-	1
	0.4	87	87	-	1
Maize (on cob)	0.04	90	90	-	1
	0.4	87	87	-	1
Egg plant	0.04	87	79, 95	-	2

Sample	Fortification level [mg/kg]	Recovery [%]		SD (rel) [%]	No.
		Mean	Range		
	0.4	80	79, 82	-	2

The range of crops and limit of quantification was extended by Tribolet (Report 210/01, 2001). Four crop samples and a wine sample were each fortified at 0.02 mg/kg and 0.2 mg/kg. Control samples were also analysed for each crop, and in each case the residues were quoted as <0.02 mg/kg. The data are shown in Table 47. The overall mean recoveries for each of the samples range from 83-89% with relative standard deviations in the range 7.4-14.7%. The LOQ was 0.02 mg/kg for the crops and 0.005 mg/kg for wine.

Table 47. Recoveries by analytical method REM 133-04 for determination of fludioxonil in crops (Tribolet, Report 210/01, 2001).

Sample	Fortification level [mg/kg]	Recovery [%]		SD (rel) [%]	No.
		Mean	Range		
Strawberries	0.02	85	73-97	12	5
	0.2	81	72-87	7.7	5
Apples	0.02	95	84-112	12.	5
	0.2	83	76-88	6.4	5
Wheat grain	0.02	92	81-106	11	5
	0.2	81	70-89	10.	5
Grapes	0.02	88	71-119	21.	5
	0.2	84	79-89	4.7	5
Wine	0.005	84	76-95	9.8	5
	0.05	84	77-88	5.2	5

Radiovalidation of the method was provided by Mair (Report 10/96, 1996) using samples of tomato from the metabolism study. Three sub-samples of tomato previously treated with radiolabelled fludioxonil and harvested at maturity were analysed to determine the amount of fludioxonil. The total radioactive residue (TRR) in the tomato sample was 0.106 mg/kg fludioxonil equivalents and 73% was identified as fludioxonil. In the sub-samples taken, the expected residue of fludioxonil was therefore 0.077 mg/kg. The extraction method solubilized 89% (0.094 mg/kg) of the total radioactivity and the residue of fludioxonil in the samples was determined as 0.051 mg/kg, or 48% of the TRR. The method showed a good extraction of the residues and an overall recovery of fludioxonil of 66% (0.051/0.077 mg/kg).

The second HPLC method (AG-597A, AG-597B) for the determination of fludioxonil in plant commodities (Campbell, Report CGA173506/0773, 1996; Williams, Report ABR-97080, 1998) is typically used in the USA for crop field trials. Crop samples are homogenized with acetonitrile/water (9:1) and filtered. An aliquot is evaporated to remove acetonitrile, diluted with saturated salt solution and partitioned with methyl *tert*-butyl ether (MTBE). For fodder only, the aliquot is partitioned with hexane before evaporation of acetonitrile, diluted with saturated salt solution and partitioned with hexane/MTBE (4:1). Toluene is added to the organic phase, the MTBE evaporated and hexane added to the sample. Grape, wine, grain and tuber samples are cleaned up on a silica SPE cartridge and the analyte eluted with dichloromethane/toluene (1:1). Forage, hay and fodder samples are cleaned up on a silica cartridge and the analyte eluted with dichloromethane/toluene (1:3).

Eluates from the silica cartridges are further cleaned up on a phenyl SPE cartridge, the analyte being eluted with acetone. The eluate is evaporated to dryness and reconstituted in mobile phase. Determination is by normal phase HPLC (amino column) with UV detection at 268 nm. No column switching is involved.

Maize oil samples are dissolved in hexane and partitioned with acetonitrile. The acetonitrile extract is evaporated to dryness and reconstituted in toluene. Clean up is on a Florisil SPE cartridge eluted with 2% acetone in toluene. The eluate is evaporated to dryness and reconstituted in mobile phase. Determination is by reverse-phase HPLC (C-18 column) with UV detection at 268 nm.

Validation results are shown in Table 48. The LOQ of the method is commodity-specific and in the 0.01–0.05 mg/kg range

The extraction efficiency of the method was determined using the rice grain and stalk samples from greenhouse-grown rice given a foliar treatment with ^{14}C fludioxonil. Mean extractabilities of the TRR were 53% (CV 5.7%, n = 3) for 1x treated grain (0.28 kg ai/ha, PHI 52 days), 90% (CV = 0.6%, N=3) for 1x treated stalks, 119% (CV 7.8%, n=3) for 5x treated grain (1.4 kg ai/ha, PHI 50 days) and 86% (CV 3.7%, n=3) for 5x treated rice stalks. HPLC analysis of residues was good from the 1x treated stalks and 5x treated stalks and grain (range 61-112%), but poor from the 1x treated grain (range 29-39%). No comparison could be made with the percentage of the TRR determined as fludioxonil in the metabolism study as the study was not reported to the Meeting.

Table 48. Recoveries by analytical method AG-597B for determination of fludioxonil in crops (Campbell, Report CGA173506/0773, 1996).

Sample	Fortification level [mg/kg]	Recovery [%]		No.
		Mean	Range	
Maize grain	0.01	109	105-114	3
	0.05	87	87	1
	0.1	95	95	1
Maize forage	0.01	83	79, 86	2
	0.05	86	86	1
	0.1	86	86	1
	0.2	83	83	1
Maize forage	0.02	81	72-90	1
	0.1	100	100	1
	0.5	72	72	1
Maize fodder	0.01	72	70, 73	2
	0.05	89	89	1
	0.10	82	82	1
	0.20	87	87	1
Sorghum grain	0.05	66	66	1
	0.10	102	102	1
Sorghum hay	0.01	96	95-96	2
	0.05	90	90	1
	0.10	76	76	1
	0.20	71	70, 72	2
Sorghum fodder	0.01	83	75, 90	2
	0.05	70	70	1
	0.10	86	86	1
	0.20	81	81	1
Potato tuber	0.01	85	75-101	3
	0.05	92	92	1
	0.10	79	79	1
Rice grain	0.01	83	72, 94	2
	0.05	113	109-117	2
Rice stalks	0.05	79	79	1
	0.10	94	94	1
	2.0	85	85	1
	8.0	94	94	1
White grapes	0.02	91	88, 93	2
	0.2	134	134	1
	0.5	73	73	1
Red grapes	0.02	90	86, 93	2

Sample	Fortification level [mg/kg]	Recovery [%]		No.
		Mean	Range	
	0.1	96	96	1
	1.0	93	93	1
White wine	0.02	96	92, 100	2
	0.05	73	73	1
	0.5	96	96	1
Red wine	0.02	114	114	1
	0.2	76	76	1
	1.0	79	79	1

In a further validation of the method (Williams, Report ABR-97080, 1998) the range of crops was extended and minor modifications made to the method for particular samples. Recoveries are shown in Table 49.

LC/MS conditions were developed for confirmatory detection and quantification of residues in lima beans and as a primary determination system for alfalfa hay, clover hay and black pepper. LC/MS was used in single ion monitoring mode on ion 247.

Table 49. Recoveries by analytical method AG-597B for determination of fludioxonil in crops (Williams, Report ABR-97080, 1998).

Sample	Fortification level [mg/kg]	Recovery [%]		SD (rel) [%]	No.
		Mean	Range		
Radish tops	0.01	97	92, 101	-	2
	0.02	95	93, 97	-	2
	0.05	98	95, 100	-	2
Radish tops	0.01	98	85-120	12	10
	0.02	104	94-112	4.9	10
Dried peas	0.01	101	99, 102	-	2
	0.02	85	64, 105	-	2
	0.05	101	97, 104	-	2
Turnip tops	0.01	110	109, 111	-	2
	0.02	104	103, 104	-	2
	0.05	100	98, 101	-	2
Succulent peas with pod	0.01	102	72-120	15	10
	0.02	101	77-120	13	10
Wheat grain	0.01	90	88, 91	-	2
	0.02	79	61-89	20	3
	0.05	79	71-94	16	3
Head lettuce[1]	0.01	107	104, 110	-	2
	0.02	87	84, 90	-	2
	0.05	112	105, 118	-	2
Celery [1]	0.01	85	82, 87	-	2
	0.02	94	93, 95	-	2
	0.05	97	96, 97	-	2
Spinach[1]	0.01	81	71, 91	-	2
	0.02	88	86, 89	-	2
	0.05	94	90, 98	-	2
Cantaloupe fruit[1]	0.01	88	81, 94	-	2
	0.02	76	60, 92	-	2
	0.05	89	85, 93	-	2
Summer squash[1]	0.01	108	104, 112	-	2
	0.02	105	104, 105	-	2
	0.05	104	101, 106	-	2
Cucumber[1]	0.01	93	84, 101	-	2
	0.02	102	101, 103	-	2
	0.05	97	96, 97	-	2

Sample	Fortification level [mg/kg]	Recovery [%]		SD (rel) [%]	No.
		Mean	Range		
Carrot root[1]	0.02	96	88-103	6.3	5
	0.05	108	102-112	4.2	4
Sugar beet top[1]	0.01	98	95, 100	-	2
	0.02	93	91, 95	-	2
	0.05	95	93-, 6	-	2
Sugar beet root[1]	0.02	109	108, 110	-	2
	0.05	108	107, 108	-	2
Bulb onion dry[1]	0.02	111	110, 112	-	2
Green onion[1]	0.01	106	101-113	5.1	4
	0.02	95	95	-	1
	0.05	97	97	-	1
Bean with pod[1]	0.01	110	109, 110	-	2
	0.02	98	96, 100	-	2
	0.05	96	95, 96	-	2
Bean without pod[1]	0.01	99	98, 100	-	2
	0.02	101	100, 101	-	2
	0.05	100	100	-	1
Pea without pod[1]	0.01	94	92, 96	-	2
	0.02	87	85, 89	-	2
	0.05	88	85, 91	-	2
Sorghum asp. grain fraction[1]	0.01	109	108, 109	-	2
	0.02	110	105, 114	-	2
	0.05	104	101, 107	-	2
Tomato fruit[1]	0.01	112	104, 120	-	2
	0.02	105	105	-	2
	0.05	105	103, 107	-	2
Pepper chilli[1]	0.01	101	99, 103	-	2
	0.02	94	93, 95	-	2
	0.05	92	91, 93	-	2
Pepper bell[1]	0.01	113	111, 115	-	2
	0.02	117	115, 118	-	2
	0.05	115	113, 116	-	2
Fresh basil[1]	0.01	102	99, 104	-	2
	0.02	101	99-103	-	2
	0.05	101	100, 101	-	2
Turnip root[1]	0.01	113	109, 116	-	2
	0.02	108	105, 111	-	2
	0.05	117	114, 119	-	2
Broccoli[1]	0.01	114	113, 115	-	2
	0.02	108	105, 111	-	2
	0.05	106	102, 110	-	2
Mustard greens[1]	0.01	107	106, 107	-	2
	0.02	104	101, 106	-	2
	0.05	99	94, 104	-	2
Chives (fresh)[1]	0.01	105	104, 106	-	2
	0.02	103	102, 103	-	2
	0.05	100	99, 101	-	2
Rape seed[1]	0.01	97	90, 103	-	2
	0.02	98	96, 99	-	2
	0.05	93	92, 93	-	2
Lettuce, leaf[1]	0.01	104	92-117	8.1	12
	0.02	99	90-110	7.9	6
	0.05	102	92-109	7.2	5
Cucumber[1]	0.01	98	88-109	8.8	11
	0.02	100	81-112	11	6
	0.05	93	75-105	14	6
Radish roots[1]	0.01	96	74-110	14	10
	0.02	108	95-123	11	8
	0.05	110	104, 116	-	2

Sample	Fortification level [mg/kg]	Recovery [%]		SD (rel) [%]	No.
		Mean	Range		
Rice straw[2]	0.01	100	99, 100	-	2
	0.02	103	96, 109	-	2
	0.05	101	99, 102	-	2
Sweet corn forage[2]	0.01	107	105, 108	-	2
	0.02	96	95, 97	-	2
	0.05	101	100, 101	-	2
Peanut hay[2]	0.02	116	115, 116	-	2
	0.05	120	117, 122	-	2
Wheat forage[2]	0.01	99	84-115	16	3
	0.02	96	75- 111	20	3
	0.05	104	99-107	4.2	3
Wheat hay[2]	0.01	96	81, 110	-	2
	0.02	101	85-108	14	3
	0.05	99	93-104	5.7	3
Soya forage[2]	0.01	108	103, 113	-	2
	0.02	91	89, 93	-	2
	0.05	95	92, 97	-	2
Clover forage[2]	0.01	101	93, 109	-	2
	0.02	99	93, 104	-	2
	0.05	91	83, 100	-	2
Alfalfa forage[2]	0.01	83	83	-	2
	0.02	84	84	-	1
	0.05	80	77, 83	-	2
Celery seed[2]	0.01	102	100, 104	-	2
	0.02	95	94, 96	-	2
	0.05	96	91, 100	-	2
Cabbage[2]	0.01	72	71, 73	-	2
	0.02	92	89, 95	-	2
	0.05	88	84, 92	-	2
Peanut meal[2]	0.01	80	73, 86	-	2
	0.02	99	86, 111	-	2
	0.05	107	105, 109	-	2
Field corn flour[3]	0.01	105	102, 107	-	2
	0.02	109	107-110	-	2
	0.05	92	92	-	2
Soya beans[3]	0.01	105	104, 106	-	2
	0.02	114	113, 114	-	2
	0.05	106	105, 107	-	2
Soya asp. grain fraction[3]	0.01	73	66, 79	-	2
	0.02	106	104, 107	-	2
	0.05	91	80, 102	-	2
Soya hay[3]	0.01	101	99, 103	-	2
	0.02	109	107, 110	-	2
	0.05	96	95, 96	-	2
Sunflower seeds[3]	0.01	110	107, 112	-	2
	0.02	92	82, 102	-	2
	0.05	99	89, 109	-	2
Maize starch[3]	0.01	111	108, 114	-	2
	0.02	111	110, 111	-	2
	0.05	101	101	-	2
Maize grits[3]	0.01	108	10, -110	-	2
	0.02	112	111, 113	-	2
	0.05	100	99, 100	-	2
Maize asp. grain fraction[3]	0.01	106	104, 107	-	2
	0.02	107	107	-	2
	0.05	103	102, 104	-	2
Maize fodder[3]	0.01	90	86, 94	-	2
	0.02	91	89, 92	-	2
	0.05	92	92	-	2

Sample	Fortification level [mg/kg]	Recovery [%]		SD (rel) [%]	No.
		Mean	Range		
Rice grain[3]	0.01	104	103, 105	-	2
	0.02	107	106, 108	-	2
	0.05	97	96, 97	-	2
Lima bean, dry[3]	0.01	95	79-109	17.	4
	0.02	98	86-110	13.	4
	0.05	99	92-106	6.3	4
Basil dried[3]	0.02	105	99, 110	-	2
	0.05	95	92, 98	-	2
Chives dried[3]	0.01	80	64, 96	-	2
	0.02	122	122	-	1
	0.05	88	87, 89	-	2
Peanut nutmeg[3]	0.01	81	79, 82	-	2
	0.02	72	64, 80	-	2
	0.05	84	80, 88	-	2
Cotton undelinted seed[3]	0.05	100	91-106	5.9	8
	0.5	103	103	-	1
Rape seed oil[4]	0.01	72	71, 73	-	2
	0.02	76	76	-	1
	0.05	127	96, 158	-	2
Peanut oil, refined[4]	0.01	94	90, 96	-	2
	0.02	85	80, 89	-	2
	0.05	89	88, 89	-	2
Cotton seed oil, refined[4]	0.05	77	75, 78	-	2
Wheat straw[3]	0.01	99	89-100	6.0	3
	0.02	94	90-97	3.7	3
	0.05	83	80-85	3.0	3
Alfalfa hay[5]	0.01	103	101, 105	-	2
	0.02	97	96, 98	-	2
	0.05	114	111, 116	-	2
Clover hay[5]	0.01	81	79, 83	-	2
	0.02	73	64, 82	-	2
	0.05	116	115, 117	-	2
Black pepper[5]	0.01	106	90, 122	-	2
	0.02	108	93, 123	-	2
	0.05	90	87, 93	-	2
Lima beans, dried[5]	0.01	106	105, 107	-	2
	0.02	95	93, 96	-	2
	0.05	86	85, 87	-	2

[1] using method AG-597B as written for grapes, wine, grain and potato tubers with alternative procedure for forage crops
[2]using method AG-597B as written for forage and hay samples
[3]using method AG-597B as written for fodder samples
[4]using method AG-597B as written for oil samples
[5]using method AG-597B as written for fodder samples using LC/MS detection

An LOQ of 0.01 mg/kg was achieved in radish roots and tops, turnip roots and tops, beet tops, green onion (whole plant), head and leaf lettuce, celery, spinach, broccoli, cabbage, mustard greens, succulent beans (with and without pod), beans (dry seed), succulent peas (with and without pod), peas (dry seed) soya (asp. grain fraction, dry beans, forage and hay), tomato, peppers (bell and non-bell), cantaloupe, cucumber, summer squash, maize (starch, grits, flour, asp. grain fraction), rice grain and straw, sorghum (asp. grain fraction), wheat (grain, straw, forage and hay), maize forage and fodder, alfalfa forage and hay, clover forage and hay, basil (fresh and dried), chives (fresh and dried), celery seed and black pepper, sunflower seeds, rape (oil and seed), cotton (undelinted seed, gin trash, hulls, meal and oil) and peanuts (nutmeat, meal, hay and oil). An LOQ of 0.02 mg/kg was achieved in carrot, sugar beet roots, dry bulb onions, peanut hay and dried basil.

Control values were quoted as less than the corresponding LOQ samples. Reagent blanks and control samples were free of interfering substances at the retention time of fludioxonil.

The original method (without modifications) was validated by the U.S. EPA (Willet, Report D217129, 1995). The method was as described in the manufacturer's document, with very minor modifications to use the available equipment. The limit of detection was estimated as 0.002 mg/kg. Findings are summarized in Table 50. Recovery from forage was unacceptable with the unmodified method.

Table 50. Recoveries by analytical method AG-597A for determination of fludioxonil in crops (Willet, Report D217129, 1995).

Sample	Fortification level [mg/kg]	Recovery [%]		SD (rel) [%]	No.
		Mean	Range		
Maize grain	0.02	95	98.0, 92.5		2
	0.04	104	106, 101		2
Sorghum grain	0.05	93	93.8, 92.0		2
	0.1	96	99.2, 91.8		2
Corn forage	0.02	67	56.5-94.0	18	4
	0.04	41	31.3-55.3	12	3

Method AG-597B was also validated by the US EPA for stone fruits, strawberries, and bulb vegetables (Donovan, Report D272959, 2001). The limit of detection and LOQ for these commodities were 0.003 mg/kg and 0.008 mg/kg respectively.

A multi-residue method, based on DFG S19 (extended revision) was developed for routine monitoring of fludioxonil in samples of plant material (Pelz, Report SYN-0103V, 2001). The extractions of fludioxonil were according to extraction module E1 for orange and tomato, E2 for wheat grain and E7 for rape seed. Extracts were separated by gel permeation chromatography and analysed by capillary gas chromatography (30 m fused silica capillary DB-5, 60–180°C) using mass selective detection (MSD) with the molecular ion (m/z 248) and 2 fragment ions (m/z 154 and 127).

In method DFG S19 (extended revision) has been validated for determination of residues of fludioxonil in fortified samples of tomato (as representative of commodities with high water content), orange (as representative of fruits with high acid content), wheat (as representative of cereals and other dry crops), and rape (as representative of commodities with high fat content). Recovery experiments were conducted with fortifications of 0.02 mg/kg and 0.2 mg/kg for each sample. Control samples were analysed in duplicate and fortified samples in quintuplet for each fortification level.

The demonstrated LOQ was 0.02 mg/kg. The stated limit of detection (LOD) was 0.004 mg/kg. Analysis of unfortified homogenized control samples yielded no residues of fludioxonil above the limit of detection, indicating that no background levels of fludioxonil or interferences were present in any of the samples before the beginning of the study. The linearity of the detector response covered a working range of 0.01-4.0 µg/ml fludioxonil.

Recoveries were within the required range 70-110% (except oranges where the mean value at 0.02 mg/kg was 114%). The data are presented in Table 51.

Table 51. Recoveries by analytical method DFG S19 (extended revision) for determination of fludioxonil in crops (Pelz, Report SYN-0103V, 2001).

Sample	Fortification level [mg/kg]	Recovery [%]		SD* (rel) [%]	No.
		Mean	Range		
Tomato	0.02	104	98-108	3.8	5
	0.2	107	97-114	6.9	5
Orange	0.02	114	107-117	4.2	5
	0.2	105	97-111	5.5	5
Wheat	0.02	97	86-106	7.7	5

Sample	Fortification level [mg/kg]	Recovery [%]		SD* (rel) [%]	No.
		Mean	Range		
	0.2	101	94-108	5.1	5
Rape	0.02	106	99-109	5.3	5
	0.2	92	84-96	5.1	5

The applicability of the multi-residue to wine was confirmed (Dieterle, Report 108-93, 1993). The LOQ was 0.01 mg/kg, defined by the lower fortification level tested. (Table 52).

Table 52. Recoveries by analytical method DFG S19 (extended revision) for determination of fludioxonil in wine (Dieterle, Report 108-93, 1993).

Sample	Fortification level [mg/kg]	Recovery [%]		No.
		Mean	Range	
Wine	0.01	102	95, 108	2
	0.1	99	92, 105	2

An independent validation of the multi-residue method was carried out on fortified samples of tomato and rape seed (Stenhauer, Report SYN-0104V, 2001). The data are presented in Table 53.

Table 53. Recoveries by analytical method DFG S19 (extended revision) for determination of fludioxonil in crops (Stenhauer, Report SYN-0104V, 2001).

Sample	Fortification level [mg/kg]	Recovery [%]		SD (rel) [%]	No.
		Mean	Range		
Tomato	0.02	104	97-113	6.5	5
	0.2	95	93-97	2.0	5
Rape	0.02	107	95-115	7.9	5
	0.2	93	80-110	14	5

Fludioxonil was tested according to the FDA Multiresidue Methods using sorghum grain, field corn grain and potatoes as the test samples according to protocols C, D and E (Willett, Report D206301, 1995). The compound was inadequately recovered from sorghum grain and field corn grain. Potatoes were tested as the representative non-fatty crop using protocol D. Recoveries ranged from 49 to 121% at the 0.05 mg/kg fortification level and from 78 to 87% at 0.5 mg/kg.

An HPLC method was reported for the determination of fludioxonil residues in livestock commodities (Vienneau, Report AG-616B, 1996). The method converts residues of fludioxonil and its metabolites to CGA 192155 (2,2-difluoro-1,3-benzodioxole-4-carboxylic acid), and the total residues are quantified in terms of CGA 192155 by external calibration. An independent laboratory has validated the in-house method.

Homogenized samples of animal tissues, milk and eggs are extracted by reflux with ammonium hydroxide/acetonitrile. The extract is filtered, acidified and partitioned with toluene. The organic phase of liver, eggs and chicken fat extracts is cleaned up by solid-phase extraction cartridge (silica for liver and eggs and C-18 for chicken fat). The cleaned up extract and the original extracts for other substrates are evaporated to dryness and heated with potassium permanganate and aqueous sodium hydroxide to oxidize fludioxonil and its oxidizable benzopyrrole metabolites to CGA-192145. The oxidation is quenched with sodium metabisulphite. The oxidized extract is filtered, acidified and partitioned with dichloromethane before clean-up by silica solid-phase extraction. Determination is by column switching reversed-phase HPLC (Supelcosil LC-C1 plus Supelcosil LC-8-DB analytical) with

UV (230 nm) detection. Residues are expressed as fludioxonil. A confirmatory HPLC system using an alternative column (Suplcosil LC-C1 plus Supelcosil LC-CN) is also described. Residue values are converted to mg/kg fludioxonil using a conversion factor of 1.23 (MW of fludioxonil ÷ MW of CGA-192155).

The method was validated. The overall recovery was found to be 83% with a standard deviation of 8.6%. Extraction and accuracy were shown to be acceptable by the analysis of samples from the goat and hen metabolism studies. Fortified samples were also analysed by the confirmatory HPLC method where the overall mean recovery was 80% with a relative standard deviation of 22%.

The limit of quantification was 0.01 mg/kg fludioxonil equivalents in meat and milk and 0.05 mg/kg in eggs, liver, kidney and fat. Recovery data are shown in Table 54.

Table 54. Recoveries by analytical method AG-616B for determination of fludioxonil in animal tissues, milk and eggs (Vienneau, Report AG-616B, 1996).

Analyte	Sample	Fortification level [mg/kg]	Recovery [%]		CV [%]	No.
			Mean	Range		
Fludioxonil	Cattle milk	0.01	83.5	83, 84	-	2
and metabolites		0.05	83	83	-	1
as	Goat milk	0.01	83.7	77-94	10.8	3
CGA-192155		0.60	82	82	-	1
	Beef round muscle	0.01	78.5	78, 79	-	2
		0.10	79	79	-	1
	Beef tenderloin	0.01	75.5	74, 77	-	2
		0.05	71	71	-	1
	Goat leg muscle	0.01	73	69, 77	-	2
		0.05	79	79	-	1
	Poultry lean meat	0.01	94	90, 98	-	2
		0.05	79	79	-	1
	Beef liver	0.05	77.5	70, 85	-	2
		0.10	69	69	-	1
	Goat liver	0.05	88	88	-	2
		6.0	94	94	-	1
	Poultry liver	0.05	89	89	-	2
		0.10	91	91	-	1
	Beef kidney	0.05	89	87, 91	-	2
		0.10	86	86	-	1
	Goat kidney	0.05	78.5	69, 88	-	2
		3.0	90	90	-	1
	Beef omental fat	0.05	89.5	80, 99	-	2
		0.10	83	83	-	1
	Goat perirenal fat	0.05	95.5	87, 104	-	2
		0.06	94	94	-	1
	Poultry perirenal fat	0.05	74.5	66, 83	-	2
		0.10	89	89	-	1
	Poultry eggs	0.05	83.5	82, 85	-	2
		0.50	75.7	68-81	-	3

The validation data for the confirmatory method (alternative HPLC column) for AG-616 is presented in Table 55.

Table 55. Recoveries by confirmatory method for AG-616B (Vienneau, Report AG-616B, 1996; Perez, Report ADPEN-901-95-1023, 1996).

Analyte	Sample	Fortification level [mg/kg]	Recovery [%]		No.
			Mean	Range	
Fludioxonil	Cattle milk	0.01	92	83, 101	2
and metabolites		0.05	76	76	1
as	Goat milk	0.01	86.5	83, 90	2
CGA-192155		0.60	81	81	1
	Beef tenderloin	0.01	76.5	71, 82	2
		0.05	65	65	1
	Goat leg muscle	0.01	63.5	37, 90	2
		0.05	78	78	1
	Poultry lean meat	0.01	133	95, 171	2
		0.05	92	92	1
	Beef liver	0.05	69.5	69, 70	2
		0.10	66	66	1
	Goat liver	0.05	101	88, 114	2
		6.0	89	89	1
	Poultry liver	0.05	86	85,	2
		0.10	95	95	1
	Beef kidney	0.05	88.5	88,	2
		0.10	88	88	1
	Goat kidney	0.05	77	69,	2
		3.0	88	88	1
	Beef omental fat	0.05	58	51, 65	2
		0.10	73	73	1
	Goat perirenal fat	0.05	64	63, 65	2
		0.06	60	60	1
	Poultry perirenal fat	0.05	45	37, 53	2
		0.10	80	80	1

An independent laboratory validation was carried out on the method (Perez, Report ADPEN-901-95-1023, 1996). Results are presented in Table 56. The validation was successful at a fortification of 0.05 mg/kg for eggs and liver and at 0.01 mg/kg for milk. A second independent laboratory validation was carried out to determine the ruggedness of the method (Tang and Baldi, 1996).

Table 56. Method validation and concurrent method recoveries of Method AG-616B from fortified untreated samples of milk and animal tissues (Perez, Report ADPEN-901-95-1023, 1996; Tang and Baldi, Study 102-96, 1996).

Commodity	Fortification level, mg/kg	% Recovery[1]
Independent Laboratory Validation (Perez, Report ADPEN-901-95-1023, 1996)		
Liver	0.05, 0.10	85-107 (n=4)
Milk	0.01, 0.10	83-109 (n=4)
Eggs	0.05, 0.10	63 (n=1); 73-99 (n=3)
Concurrent Method Recoveries		
Dairy cattle, milk	0.01-0.20	58-68 (n=6), 70-101 (n=35)
Dairy cattle, round muscle	0.01, 0.20	82, 85 (n=2)
Dairy cattle, tenderloin muscle	0.01, 0.10	72, 76 (n=2)
Dairy cattle, liver	0.05, 0.50	78, 80 (n=2)

Commodity	Fortification level, mg/kg	% Recovery[1]
Dairy cattle, kidney	0.05, 0.20	88, 100 (n=2)
Dairy cattle, perirenal fat	0.05, 0.20	80, 80 (n=2)
Dairy cattle, omental fat	0.05, 0.10	85, 88 (n=2)
Independent Laboatory Valiadation (Tang and Baldi, Reort 102-96, 1996)		
Liver	0.05, 0.25	80-85 (n=4)
Milk	0.01, 0.05	70-75 (n=4)
Eggs	0.05, 0.25	75-79 (n=4)

[1] No. of samples (n) in parentheses; recoveries outside the acceptable 70-120% range are listed separately.

Samples from the goat and poultry metabolism studies were used for radioavalidation of the method (Vienneau, Report AG-616B, 1996). Extraction of the total residue ranged from 50% to 85% for goat tissues and was 69% for eggs. In the metabolism studies the figures were 32%-76% and about 70% of the TRR respectively, so extraction by the method is acceptable. The overall recovery of fludioxonil and benzopyrrole metabolites ranged from 36% to 58%. The total percentage of the TRR identified in the metabolism studies ranged from 14 to 83% for the goat metabolism and 69% in eggs. The results are summarized in Table 57.

Table 57. Extraction and recoveries of [^{14}C]fludioxonil as determined by method AG-616B (Vienneau, Report AG-616B, 1996).

Sample	TRR [1] (mg/kg)	Extraction[1,2] (mg/kg)	Mean extraction of TRR (%)	LSC Total Residue from HPLC[1] (mg/kg)	Fludioxonil by HPLC (mg/kg)[3]	Recovery[5] (%)	Total % of TRR identified from metabolism studies[6]
Goat milk, day 2	0.59	0.50	85	0.28	0.34	56	78
Goat muscle	0.045	0.030	68	0.015	0.018	40	52
Goat liver	6.6	3.3	50	1.92	2.36	36	14
Goat kidney	2.8	1.84	65	0.95	1.16	41	48
Goat fat	0.063	0.041	65	0.026	0.031[4]	49	83
Hen eggs	0.52	0.36	69	0.24	0.30	58	69

[1] Average of three samples.
[2]Residues (fludioxonil plus benzopyrrole metabolites) determined by HPLC and converted with a factor of 1.23.
[3]Conversion factor 1.23
[4] Less than the LOQ for fat.
[5]Fludioxonil equivalents by HPLC divided by the TRR in the sample.
[6]See Tables 5 and 10 above.

Stability of pesticide residues in stored analytical samples

The stability of fludioxonil residues under deep freeze storage (<-18°C) was examined in cereal grain and straw samples (Bass, Report 621/7-1012, 1995), grapes (Mair, Report 131/93, 1996), maize, sorghum and potato substrates (Eudy, Report ABR-97108, 1997), tomatoes (Tribolet, Report 222/98, 2000a), apples (Tribolet, Report 221/98, 2000b), and peas and rape seed (Tribolet, Report 210/00, 2002).

Single period assessments were made of the storage stability of fludioxonil in fortified samples of peach (Thompson & Ediger, Report A6934, 1999a), plum (Thompson & Ediger, Report 06943, 1999b), cherry (Thompson and Ediger, Report 06933, 1999c), raspberry (Starner, Report

06838, 2001), blueberry (Thompson, Report 06724, 2001), cabbage (Arsenovic, Report 07121, 2002a), broccoli (Arsenovic, Report 07122, 2002b), carrots (Hong Chen, Report 07090, 2002), and fresh chives (Hong Chen, Report 07126, 2002),.

Samples were stored in polyethylene containers in deep freeze rooms under conditions corresponding to those used for storage of residue samples (≤ -20°C). The analytical methods were REM 133 (European samples) and AG 597 (US samples).

Fludioxonil residues were shown to be stable over the periods for which they were stored (depending on the sample) under these conditions. The results are summarized in Tables 58 and 59. Samples in Table 59 were not analysed at the time of fortification (day 0) and the results are therefore of limited value.

Table 58: Storage stability of fludioxonil residues in frozen crop substrates (multi-period studies).

Crop (fortification level, mg/kg) Reference	Storage (months)	Procedural recovery (%)	Mean procedural recovery (%)	Fludioxonil remaining in stored samples					
				Individual samples (mg/kg)			Mean (mg/kg)	Mean corrected (mg/kg)[1]	% of fortification not corrected
Cereal	0	86 82 71	80	0.43	0.41	0.36	0.40	0.50	80
grain	3	94 97	96	0.35	0.42	0.43	0.40	0.42	80
(fortified,	6	101 101	101	0.50	0.47	0.48	0.48	0.48	96
0.50 mg/kg)	12	85 91	88	0.42	0.39	0.44	0.42	0.47	84
Bass, Report 621/7-1012, 1995	24	102 98	100	0.44	0.48	0.44	0.45	0.45	90
Cereal	0	106 103 97	102	0.53	0.51	0.48	0.51	0.51	102
straw	3	72 81	77	0.38	0.38	0.34	0.37	0.48	74
(fortified,	6	72 79	76	0.46	0.42	0.37	0.42	0.55	84
0.50 mg/kg)	12	74 90	82	0.50	0.53	0.52	0.52	0.63	104
Bass, Report 621/7-1012, 1995	24	80 81	81	0.41	0.42	0.48	0.44	0.54	88
Apples	0	93 90	92	0.43	0.46	0.46 0.47	0.46	0.49	92
(fortified,	1	85 88	87	0.43		0.43	0.43	0.49	86
0.50 mg/kg)	3	87 86	87	0.45		0.40	0.43	0.49	86
Tribolet,	6	87 89	88	0.42		0.43	0.43	0.48	86
Report 221/98,	12	86 88	87	0.42		0.42	0.42	0.48	84
2000b	24	89 80	85	0.41		0.41	0.41	0.48	82
Tomatoes	0	88 89	89	0.44	0.45	0.42 0.44	0.44	0.49	88
(fortified,	1	86 90	88	0.42		0.42	0.42	0.48	84
0.50 mg/kg)	3	84 89	87	0.42		0.39	0.41	0.47	82
Tribolet,	6	91 88	90	0.42		0.41	0.42	0.46	84
Report 222/98,	12	95 88	92	0.40		0.37	0.39	0.42	78
2000a	24	91 96	94	0.39		0.41	0.40	0.43	80
Grapes	0	89 88	89	6.05	5.95	5.81	5.94	6.67	89
(field-	1.2	74 74	74	4.25	4.96	4.88	4.70	6.35	52
incurred	3.5	87 81	84	5.89	5.64	5.61	5.71	6.80	86
from study	6.5	93 91	92	5.77	5.75	5.89	5.80	6.31	87
2069/93)	13.3	74 74	74	5.21	6.55	5.79	5.85	7.91	88
	26.0	88 88	88	5.36	4.70	5.25	5.10	5.80	76
	28.9	86 88	87	6.87	7.59	7.88	7.45	8.56	111
Grapes	0	89 88	89	5.81	5.66	5.54	5.67	6.37	89
(field-	1.2	74 74	74	5.20	5.19	4.22	4.87	6.58	76
incurred	3.5	87 81	84	5.98	5.64	5.92	5.85	6.96	92
from study	6.5	93 91	92	6.02	5.71	5.61	5.78	6.28	91
2070/93)	13.3	74 74	74	5.42	5.50	4.59	5.17	6.99	81
Mair, Report	26.0	88 88	88	3.65	4.46	3.49	3.87	4.39	61
131/93, 1996	28.9	86 88	87	5.66	5.98	5.49	5.71	6.56	90

Crop (fortification level, mg/kg) Reference	Storage (months)	Procedural recovery (%)		Mean procedural recovery (%)	Fludioxonil remaining in stored samples					
					Individual samples (mg/kg)			Mean (mg/kg)	Mean corrected (mg/kg)[1]	% of fortification not corrected
Peas	0	81	88	85	0.42	0.40	0.45	0.42	0.50	84
(fortified,	3	81	70	76	0.42	0.40	0.41	0.41	0.54	82
0.50 mg/kg)	6	88	85	87	0.42	0.44	0.42	0.43	0.49	86
Tribolet,	12	84	84	84	0.41	0.41	0.44	0.42	0.50	84
Report 210/00,	18	86	85	86	0.39	0.44	0.41	0.41	0.48	96
2002	24	80	90	85	0.33	0.50	0.44	0.42	0.50	84
Rape seed	0	72	78	75	0.33	0.35	0.37	0.35	0.47	70
(fortified,	3	80	81	81	0.35	0.29	0.35	0.33	0.41	66
0.50 mg/kg)	6	70	76	73	0.29	0.28	0.32	0.30	0.41	60
Tribolet,	12	83	81	82	0.36	0.35	0.38	0.36	0.44	72
Report 210/00,	18	78	80	79	0.32	0.31	0.35	0.33	0.41	66
2002	24	84	75	80	0.27	0.24	0.29	0.27	0.33	54
Maize	0	108	99	104	0.198		0.187	0.19	0.19	95
Forage	1.8	97	89	93	0.187		0.185	0.19	0.20	95
(fortified,	5.7	93	96	95	0.186		0.191	0.19	0.20	95
0.20 mg/kg)	12.3	103	99	101	0.189		0.198	0.19	0.19	95
Eudy, Report	18.1	91	106	99	0.194		0.197	0.20	0.20	100
ABR-97108,	23.8	100	97	99	0.157		0.176	0.17	0.17	85
1997										
Maize	0	76	99	88	0.058		0.097	0.08	0.09	80
grain	1.6	116	89	103	0.084		0.088	0.09	0.09	90
(fortified,	5.9	102	107	105	0.098		0.098	0.10	0.10	100
0.10 mg/kg)	12.5	100	98	99	0.096		0.091	0.09	0.09	90
	18.1	108	108	108	0.103		0.105	0.10	0.10	100
	23.9	97	99	98	0.094		0.093	0.09	0.10	90
Maize ears	0	97	99	98	0.151		0.190	0.17	0.17	85
(fortified,	1.8	97	97	97	0.185		0.179	0.18	0.19	90
0.20 mg/kg)	6.0	113	110	112	0.197		0.204	0.20	0.20	100
Eudy, Report	12.2	101	106	104	0.208		0.199	0.20	0.20	100
ABR-97108,	18.3	112	112	112	0.205		0.199	0.20	0.20	100
1997	23.9	101	104	103	0.197		0.195	0.20	0.20	100
Maize meal	0	105	106	106	0.212		0.213	0.21	0.21	105
(fortified,	2.8	117	128	123	0.235		0.247	0.24	0.24	120
0.20 mg/kg)	3.0	123	122	123	0.225		0.237	0.23	0.23	115
	6.1	92	96	94	0.177		0.165	0.17	0.18	85
Eudy, Report	12.0	101	102	102	0.190		0.191	0.19	0.19	95
ABR-97108,	18.5	110	115	113	0.210		0.207	0.21	0.21	105
1997	26.6	102	108	105	0.206		0.214	0.21	0.21	105
Sorghum	0	100	93	97	0.480		0.488	0.48	0.50	96
Hay	2.1	94	100	97	0.464		0.477	0.47	0.49	94
(fortified,	5.7	99	103	101	0.504		0.472	0.49	0.49	98
0.50 mg/kg)	12.4	100	103	102	0.487		0.497	0.49	0.49	98
Eudy, Report	17.9	100	98	99	0.460		0.482	0.47	0.48	94
ABR-97108,	24.0	98	98	97	0.446		0.463	0.45	0.47	90
1997										
Potato	0	99	97	98	0.099		0.098	0.10	0.10	100
Tubers	1.8	100	96	98	0.090		0.069	0.08	0.08	80
(fortified,	5.6	101	93	97	0.091		0.095	0.09	0.10	90
0.10 mg/kg)	12.1	93	94	94	0.090		0.100	0.10	0.10	100
Eudy, Report	18.2	100	100	100	0.109		0.107	0.11	0.11	110
ABR-97108,	24	96	92	94	0.094		0.093	0.09	0.10	90
1997										
Potato	0	108	113	111	0.228		0.214	0.22	0.22	110
Flakes	2.8	107	98	103	0.167		0.188	0.18	0.18	90
(fortified,	6.2	112	101	107	0.170		0.178	0.17	0.17	85
0.20 mg/kg)	12.2	98	102	100	0.169		0.165	0.17	0.15	85
Eudy, Report	18.6	100	104	102	0.171		na	0.17	0.17	85
ABR-97108,	26.6	104	117	111	0.161		0.167	0.16	0.16	80
1997										

[1]corrected for control and procedural recoveries <100%
na: not analysed

Table 59. Storage stability of fludioxonil residues in frozen crop substrates (single period studies).

Crop, fortification, mg/kg, Reference	Storage (months)	Procedural recoveries (%)	Mean procedural recovery (%)	Recovery from individual stored samples (%)	Mean uncorrected recovery (%)
Peach 4.0 Thompson & Ediger, Report A6934, 1999a	3.3	Not determined	na	87 94 106	96
Plum 4.0 Thompson & Ediger, Report 06943, 1999b	2	Not determined	na	93 102 104	100
Cherry 4.0 Thompson and Ediger, Report 06933, 1999c	3.5	Not determined	na	103 86 89	93
Raspberry 2.0 Starner, Report 06838, 2001	4.5	89 93 86	93	74 68 69	70
Blueberry 2.0 Thompson, Report 06724, 2001	23 days 57 days	89 87 88	88	86 91 117 108 117 103	95 108
Cabbage 1.0 Arsenovic, Report 07121, 2002a	12	81 78 72 83	79	65 64 72	67
Broccoli 1.0 Arsenovic, Report 07122, 2002b	12	97 86 110 74	92	78 69 75	74
Carrot 1.0 Hong Chen, Report 07090, 2002	12	66 82 73	74	86 85 84	85
Chives 1.0 Hong Chen, Report 07126, 2002	9.4	78 85 79 88	83	92 87 104	94

na: not applicable

The storage stability of fludioxonil in beef muscle, beef liver, milk and eggs under freezer storage was reported to the Meeting (Eudy, Report ABR-97055, 1997). Samples of beef muscle, beef liver, milk and eggs fortified with fludioxonil were stored at -20°C for up to about 19 months. The storage conditions were chosen to represent those under which residue samples from animal studies are stored before analysis. Samples were analysed by method AG-616 with minor modifications for some samples.

No significant deterioration of fludioxonil residues with time was observed in the samples. Results are summarised in Table 60.

Table 60. Storage stability of fludioxonil residues in frozen animal tissues (Eudy, Report ABR-97055, 1997).

Tissue (fortification, mg/kg)	Storage (momths)	Procedural recoveries (%)		Mean procedural recovery (%)	Residue[1] Remaining in Store Sample				
					Individual stored samples (mg/kg)		Mean (mg/kg)	Mean corrected[2] (mg/kg)	% of fortification (not corrected)
Beef muscle									
	0	78	76	77	0.40	0.42	0.41	0.53	82
(fortified,	3.8	80	80	80	0.35	0.39	0.37	0.46	74
0.5 mg/kg)	11.2	68	136	102	**	0.51	0.51	0.50	100
	19.3	76	63	70	0.30	**	0.30	0.42	60
Beef liver	0	86	86	86	0.92	0.92	0.92	1.07	192
(fortified,	3.3	78	78	78	0.68	0.68	0.68	0.87	68
1.0 mg/kg)	11.7	96	90	93	0.76	0.63	0.70	0.75	70
	19.2	121	96	109	0.89	0.88	0.88	0.81	88
Milk	0	80	82	81	0.40	0.38	0.39	0.48	78
(fortified,	3.1	79	67	73	0.31	0.38	0.35	0.48	70
0.5 mg/kg)	11.2	61	73	67	0.32	0.33	0.32	0.48	96
	18.8	76	67	72	0.35	0.37	0.36	0.50	72
Eggs	0	89	92	91	0.95	0.87	0.91	1.00	91
(fortified,	3.1	74	79	77	0.77	0.81	0.79	1.03	79
1.0 mg/kg)	12.0	76	79	78	0.73	0.74	0.73	0.94	73
	18.7	77	78	78	0.70	0.82	0.76	0.97	76

[1]method determines fludioxonil and metabolites as CGA 192155 (2,2-difluoro-1,3-benzodioxole-4-carboxylic acid).
[2]corrected for control and procedural recovery <100%

USE PATTERNS

Fludioxonil is registered globally as a fungicide and is used as a seed treatment, as a foliar treatment, and post-harvest application on a wide variety of crops. The information available to the Meeting on registered uses relevant to the supervised field trial data is summarized in Tables 61–63. It is based on the labels or translations of labels provided by the manufacturer.

Table 61. Registered foliar uses of fludioxonil.[1]

Crop	Country	Formulation, ai %	Application rate		No. per season	PHI days
			kg ai/hl	kg ai/ha		
Fruit						
Blackberry	Switzerland	WG, 25	0.025	0.3	2	14
Blackberry	USA	WG, 25		0.25	4	0
Blueberry	USA	WG, 25		0.25	4	0
Cherry (stone fruit)	Switzerland	WG, 25		0.30	2	
Currant	USA	WG, 25		0.25	4	0
Grape	Austria	WG, 25	0.025	0.25	2	35
Grape	Chile	WG, 25		0.25	2	7
Grape	France	WG, 25		0.3	2	50
Grape	Germany	WG, 25	0.015	0.24	2	35
Grape	Italy	WG, 25	0.02	0.2	2	21
Grape	Spain	WG, 25		0.25	2	21
Grape	Switzerland	WG, 25		0.3	1	Before grape close
Lychee	USA	WG, 25		0.25	4	0
Peach	France	WG, 25	0.015			14
Peach	Italy	WG, 25	0.015	0.25	2	14
Peach	Switzerland	WG, 25	0.015	(0.3)	2	
Pear	Italy	WG, 25	0.02	0.25	3	14

Crop	Country	Formulation, ai %	Application rate		No. per season	PHI days
			kg ai/hl	kg ai/ha		
Pear	Spain	WG, 25	0.025	0.25	3	7
Plum	France	WG, 25	0.012	0.12 (up to 1000 l/ha)	3	14
Plum	Italy	WG, 25	0.025	0.25	2	14
Plum	Switzerland	WG, 25		0.3	2	
Raspberry	Switzerland	WG, 25	0.025	0.3 2	2	14
Raspberry	USA	WG, 25		0.25	4	0
Strawberry	France (glasshouse and field)	WG, 25		0.25	ca 1[2]	3
Strawberry	Germany	WG, 25	(0.0125)	0.25	3	7
Strawberry	Italy (glasshouse and field)	WG, 25	0.02	0.2	3	7
Strawberry	Spain	WG, 25		0.25	3	7
Strawberry	Switzerland	WG, 25	0.025	0.3	2	14
Strawberry	USA	WG, 25		0.25	4	0
Vegetables						
Asparagus	Austria	WG, 25	(0.042)	0.25	3	NS
Broccoli	USA	WG, 25	(≤0.13)	0.25	4	7
Cabbage	USA	WG, 25	(<0.13)	0.25	4	7
Carrot	USA	WG, 25		0.25	4	7
Cucumber	Italy (glasshouse and field)	WG, 25	0.02	0.20	3	7
Cucumber	Spain (glasshouse and field)	WG, 25	0.025		3	7
Cucumber	Switzerland (glasshouse)	WG, 25	0.025			3
Egg plant (aubergine)	Italy (glasshouse and field)	WG, 25	0.02	0.2	3	7
Egg plant (aubergine)	Spain (glasshouse and field)	WG, 25	0.025		3	7
Egg plant (aubergine)	Switzerland (glasshouse and field)	WG, 25	0.025			
Herbs (chives & basil)	USA	WG, 25		0.25	4	7
Legume (dry seed; pulse)	Austria+ Spain	WG, 25		0.25	2	14
Legume (fresh seed)	France	WG, 25	(0.083)	0.25		14
Legume (bean, n.o.s.)	Switzerland	WG, 25		0.2		
Legume (pod and seed)	Spain (glasshouse and outdoor)	WG, 25	0.025		3	14
Legume (pod and seed)	France	WG, 25	(0.083)	0.25		14
Lettuce, head	France (glasshouse and outdoor)	WG, 25		0.15	4	14
Lettuce, head	Italy (glasshouse and outdoor)	WG, 25	0.018	0.18	3	14
Lettuce, head	Spain (glasshouse and outdoor)	WG, 25		0.15	3	14
Lettuce, head	Switzerland (glasshouse and outdoor)	WG, 25		0.12	2	Early season use
Melon	USA	WP, 50	0.28	Drip irrigation	3	14
Mustard greens	USA	WG, 25		0.25	4	7
Onion	Austria	WG, 25	(0.12)	0.25	3	7

Crop	Country	Formulation, ai %	Application rate		No. per season	PHI days
			kg ai/hl	kg ai/ha		
Onion	Switzerland	WG, 25		0.25	2	
Onion, green and dry bulb	USA	WG, 25		0.25	4	7
Pepper (sweet)	Austria[2]	WG, 25	(0.025)	0.25	3	7
Pepper (sweet)	Italy (glasshouse and outdoor)	WG, 25	0.02	0.2	3	7
Pepper (sweet)	Spain (glasshouse and outdoor)	WG, 25	0.025		3	7
Tomato	Switzerland	WG, 25		0.25	2	3
Tomato	Italy (glasshouse and field)	WG, 25	0.02	0.2	3	7
Tomato	Spain (glasshouse and field)	WG, 25	0.025		3	7
Tomato	Greece[2]	WG, 25	0.025	0.38	2	7
Watercress	USA	WG, 25		0.25	4	0
Summer squash (Zucchini)	Italy (glasshouse and field)	WG, 25	0.02	0.20	3	7
Treenuts						
Pistachio	USA	WG, 25		0.25	4	7

[1] Includes only the registered uses related to the supervised field trial data supplied to the Meeting, not all possible registered national uses. Values in parenthesis are calculated from the spray volume.
[2] Fludioxonil use should not exceed one of each three fungicide uses.

Table 62. Registered post-harvest uses of fludioxonil in the USA.[1]

Crop	Formulation, ai %	Application rate		No. per season	PHI days
		kg/hl	kg/ha		
Fruit					
Apricot	WP 50	0.06	-	1	0
Cherry	WP 50	0.06	-	1	0
Citrus	WP, 50	0.06[2]	-	2	0
Kiwifruit	WP, 50	0.06[3]			
Nectarine	WP, 50	0.06	-	1	0
Pome fruit	WP, 50	0.06[2]	-	2	0
Peach	WP 50	0.06	-	1	0
Plum	WP, 50	0.06	-	1	0
Yam	WP, 50	0.06	-	1	0

[1] Includes only the registered uses related to the supervised field trial data supplied to the Meeting.
[2] Dip treatment for 30 seconds. Spray treatment low volume at 0.86 kg ai/hl or high volume at 0.24 kg/hl, with 0.5 g ai/250 000 kg fruit, or 2 mg/kg.
[3] 2.5 mg/kg fruit.

Table 63. Registered seed treatment uses of fludioxonil.[1]

Crop	Country	Formulation, ai %	Appliacation rate g/100 kg seed
Vegetables			
Bean, pea	USA	FS,48	5.0
Pea	UK	WG,5	10
Potato	Australia	FS,10	2.5
Potato	USA	FS, 48	2.5
Sweet corn	USA	FS, 48	5.0
Oilseeds			
Rape	USA	FS, 48	5.0
Rape	Germany	FS, 2.5	12
Cotton seed	USA	FS, 48	5.0

Crop	Country	Formulation, ai %	Appliacation rate g/100 kg seed
Sunflower	US	FS, 40	5.0
Soya	Argentina Paraguay Brazil	XL, 2,5 XL, 2,5 XL, 2,5	5.0
Cereals			
Barley	Austria	FS, 2.5	5.0
Barley	Belgium	FS, 2.5	5.0
Barley	UK	FS, 2.5	5.0
Barley	USA	FS, 48	5.0
Maize	Argentina	FS, 2.5	3.0
Maize	Brazil	FS, 2.5	3.8
Maize	Mexico	FS, 48	5.8
Maize (field corn)	USA	FS, 48	5.0
Oats	UK	FS, 2.5	5.0
Oats	USA	FS, 48	5.0
Popcorn	USA	FS, 48	5.0
Rye	Austria	FD, 2.5	5.0
Sorghum	USA	FS, 48	5.0
Spelt	Belgium	FS, 2.5	5.0
Sweet corn	USA	FS, 48	5.0
Wheat/Triticale	Austria	FS, 2.5	5.0
Wheat/Triticale	Belgium	FS, 2.5	5.0
Wheat/Triticale	UK	FS, 2.5	5.0
Wheat/Triticale	USA	FS, 48	5.0

[1] Includes only the registered uses related to the supervised field trial data supplied to the Meeting, not all possible registered national uses.

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

The Meeting received information on fludioxonil supervised field trials on the following crops.

Commodity	Application	Country	Table no.
Citrus (orange, lemon, grapefruit)	Post-harvest	USA	64
Apple	Post-harvest	USA	65
Pear	Post-harvest	USA	66
Pear	Foliar	France, Italy, Spain	67
Peach	Post-harvest	USA	68
Peach	Foliar	France, Italy, Spain	69
Plum	Post-harvest	USA	70
Plum	Foliar	France, Germany, Italy, Switzerland	71
Cherry	Post-harvest	USA	72
Cherry	Foliar	Grance, Germany, Italy, Switzerland	73
Grapes	Foliar	Chile, France, Germany, Greece, Italy, South Africa, Spain, Switzerland	74
Strawberry	Foliar	France, Germany, Italy, Spain, Switzerland, UK, USA	75
Strawberry	Foliar indoor	France, Italy, Spain, UK	76

Commodity	Application	Country	Table no.
Raspberry	Foliar	Germany, US	77
Blueberry and Currant	Foliar	Germany, US	78
Lychee	Foliar	USA	79
Kiwifruit	Post-harvest	USA	80
Pomegranate	Post-harvest	Usa	81
Green Onion and Bulb Onion	Foliar	France, Germany, Italy, Switzerland, USA	82
Broccoli	Foliar	Canada, USA	83
Cabbage	Foliar	USA	84
Cucumber	Foliar (indoor and outdoor)	Greece, Spain, Switzerland	85
Squash (Zucchini)	Foliar (indoor and outdoor)	Italy	86
Melon (Cantaloupe)	Foliar	USA	87
Tomato	Foliar (indoor and outdoor)	Greece, Italy, Spain, Switzerland, UK	88
Bell Pepper	Foliar (indoor and outdoor)	Italy, Spain, Switzerland	89
Egg plant (Aubergine)	Foliar (indoor and outdoor)	Italy, Spain	90
Sweet Corn	Seed treatment	USA	91
Head lettuce	Foliar (indoor and outdoor)	France, Germany, Italy, Spain, Switzerland	92
Watercress	Foliar	USA	93
Mustard Greens	Foliar	USA	94
Bean pods with seeds (succulent)	Foliar (indoor and outdoor)	France, Spain, Switzerland	95
Bean pods with seeds (succulent)	Seed treatment	Denmark, Germany	96
Peas without pod	Foliar	France, Switzerland	97
Peas without pod	Seed treatment	France, UK	98
Dry pea and kidney bean	Foliar	France	99
Dry pea	Seed treatment	France, UK	100
Potato	Seed treatment	USA	101
Yam	Post-harvest	USA	102
Carrot	Foliar	USA	103
Asparagus	Foliar	Germany	104
Wheat	Seed tretment	France, Germany, Switzerland	105
Rye	Seed treatment	Denmark	106
Barley	Seed treatment	France, Germany, Switzerland	107
Maize (Field Corn)	Seed treatment	France, Germany, Greece, Hungary, Spain, South Africa, USA	108
Sorghum	Seed treatment	USA	109
Pistachio	Foliar	USA	110

Commodity	Application	Country	Table no.
Rape (Canola)	Seed treatment	France, UK	111
Cotton seed	Seed treatment	Greece, USA	112
Chives	Foliar	USA	113
Basil	Foliar	USA	114
Wheat forage and straw	Seed treatment	France, Germany, Switzerland	115
Rye forage and straw	Seed treatment	Denmark	116
Barley forage and straw	Seed treatment	France, Germany, Switzerland	117
Maize (Field Corn) and Sweet Corn forage and fodder	Seed treatment	France, Germany, Greece, Hungary, South Africa, Spain, USA	118
Sorghum forage, hay, fodder, and silage	Seed treatment	USA	119
Rape (Canola) forage and straw	Seed treatment	France, Sweden, USA	120

In general, method AG-597 was used for trials in the USA and method REM 133 for trials in Europe and elsewhere. In some of the European trials method REM 133 was modified by using only one HPLC column with a fluorescence detector (excitation 265 nm, emission 312 nm). Unless noted otherwise, concurrent recovery analyses were conducted with the field trial analyses, including fortifications at the LOQ.

Citrus fruits

Table 64: Fludioxonil residues resulting from post-harvest application to citrus in the USA.

Location Year (variety)	Form	Application rate	No	Sample	PHI days	Fludioxonil residue mg/kg	Author Date Study No
Dip treatment with storage wax[1]							
		(g ai/hL)					
California 2001 (Valencia)	50 WP	120	1	Orange, Whole fruit	0	3.39, 2.21 (2.8)	Thompson 2003 IR4-07947
California 2001 (Eureka)	50 WP	120	1	Lemon, whole fruit	0	3.28, 3.02 (3.2)	Thompson 2003 IR4-07947
California 2001 (Ruby Red)	50 WP	120	1	Grapefruit, whole fruit	0	4.16, 3.43 (3.8)	Thompson 2003 IR4-07947
California 2001 (Eureka)	50 WP	120	1	Lemon, whole fruit	0	3.29, 2.45 (2.9)	Thompson 2003 IR4-07947
Texas 2001 (Ruby Red)	50 WP	120	1	Grapefuit, whole fruit	0	6.79, 3.53 (5.2)	Thompson 2003 IR4-07947
Florida 2001 (Valencia)	50 WP	108	1	Orange, whole fruit	0	1.56, 1.28 (1.4)	Thompson 2003 IR4-07947
Packing line spray with storage wax[1]							
		(g ai/250,000 kg fruit)					

Location Year (variety)	Form	Application rate	No	Sample	PHI days	Fludioxonil residue mg/kg	Author Date Study No
California 2001 (Valencia)	50 WP	1000 (applied in 90.7 L)	1	Orange, whole fruit	0	1.09, 0.91 (1.0)	Thompson 2003 IR4-07947
California 2001 (Eureka)	50 WP	1000 (applied in 95.9 L)	1	Lemon, whole fruit	0	1.14, 1.01 (1.1)	Thompson 2003 IR4-07947
California 2001 (Ruby Red)	50 WP	1000 (applied in 93.6 L)	1	Grapefruit, whole fruit	0	1.28, 0.61 (0.94)	Thompson 2003 IR4-07947
California 2001 (Eureka)	50 WP	930 (applied in 88.1 L)	1	Lemon Lemon - processing sample Juice Oil Pulp	0	0.54, 0.53 (0.54) 0.65 <0.02 39.7 1.39	Thompson 2003 IR4-07947
Dip treatment without storage wax							
		(g ai/hL)					
California 2001 (Valencia)	50 WP	120	1	Orange, whole fruit Peel Flesh	0	2.99, 1.41 (2.2) 1.92, 0.55 3.35, 0.92	Thompson 2003 IR4-07947
California 2001 (Eureka)	50 WP	120	1	Lemon, whole fruit	0	1.13, 1.04 (1.1)	Thompson 2003 IR4-07947
California 2001 (Ruby Red)	50 WP	120	1	Grapefruit, whole fruit	0	0.98, 0.92 (0.96)	Thompson 2003 IR4-07947
California 2001 (Eureka)	50 WP	120	1	Lemon, whole fruit	0	1.39, 0.64 (1.0)	Thompson 2003 IR4-07947
Texas 2001 (Ruby Red)	50 WP	120	1	Grapefruit, whole fruit	0	1.42, 1.31 (1.4)	Thompson 2003 IR4-07947
Florida 2001 (Valencia)	50 WP	120	1	Orange, whole fruit	0	0.96, 0.85 (0.90)	Thompson 2003 IR4-07947
Packing line spray without storage wax, followed by shipping wax[2] treatment							
		(g ai/250,000 kg fruit)					
California 2001 (Valencia)	50 WP	1000 (applied in 91.6 L)	1	Orange, whole fruit	0	0.49, 0.48 (0.48)	Thompson 2003 IR4-07947
California 2001 (Eureka)	50 WP	1000 (applied in 87.6 L)	1	Lemon, whole fruit	0	0.47, 0.46 (0.46)	Thompson 2003 IR4-07947
California 2001 (Ruby Red)	50 WP	1000 (applied in 95.9 L)	1	Grapefruit, whole fruit	0	0.62, 0.40 (0.51)	Thompson 2003
Post-harvest dip with storage wax[1] followed by dip without wax							
		(g ai/hL)					
California 2001 (Valencia)	50 WP	120 + 120	1	Orange, whole fruit	0	2.96, 2.86	Thompson 2003 IR4-07947
California 2001 (Eureka)	50 WP	120 + 120	1	Lemon, whole fruit	0	3.11, 2.56	Thompson 2003 IR4-07947
California 2001 (Ruby Red)	50 WP	120 + 120	1	Grapefruit, whole fruit	0	4.57, 4.25	Thompson 2003 IR4-07947

Location Year (variety)	Form	Application rate	No	Sample	PHI days	Fludioxonil residue mg/kg	Author Date Study No
California 2001 (Eureka)	50 WP	120 + 120	1	Lemon, whole fruit	0	4.28, 2.01	Thompson 2003 IR4-07947
Texas 2001 (Ruby Red)	50 WP	120 + 120	1	Grapefruit, whole fruit	0	6.85, 5.25	Thompson 2003 IR4-07947
Florida 2001 (Valencia)	50 WP	120 + 108	1	Orange, whole fruit	0	1.98, 1.40	Thompson 2003 IR4-07947
Packing line spray with storage wax[1], followed by wash[3], followed by packing line spray without storage wax, followed by shipping wax[2] treatment							
		(g ai/1250,000 kg fruit)					
California 2001 (Valencia)	50 WP	1000 (in 93.1 L) + 1000 (in 91.9 L)	1	Orange, whole fruit	0	0.70, 0.41	Thompson 2003 IR4-07947
California 2001 (Eureka)	50 WP	1000 (in 98.8 L) + 1000 (in 96.6L)	1	Lemon, whole fruit	0	1.01, 0.65	Thompson 2003 IR4-07947
California 2001 (Ruby Red)	50 WP	1000 (in 94.9 L) + 1000 (in 94.1 L)	1	Grapefruit, whole fruit	0	0.55, 0.49	Thompson 2003 IR4-07947

[1] Decco 202 storage wax
[2] Decco 400 shipping wax
[3] Decco Fruit & Vegetable Kleen 241

Pome fruits

Table 65: Fludioxonil residues resulting from post-harvest application to apples in the USA.

Location Year (variety)	Form	Method	Fludioxonil residue, mg/kg[4]	Author, Date Study No. Syn No
California 2001 (Fuji)	WP 50	Dip treatment[1]	1.1, 0.76 (0.93)	Thompson. Ediger 2003 IR4 07568 1751-02
	WP 50	Packing line spray[2]	1.7, 1.26 (1.5)	
	WP 50	Dip treatment[3] followed by packing line spray[2]	2.4, 2.07 (2.2)	
Idaho 2001 (Red Spur Delicious)	WP 50	Dip treatment[1]	0.75, 0.59 (0.67)	Thompson. Ediger 2003 IR4 07568 1751-02
Michegan 2001 (Red Delicious)	WP 50	Dip treatment[1]	0.52, 0.35 (0.44)	Thompson. Ediger 2003 IR4 07568 1751-02
New Jersey 2001 (McIntosh)	WP 50	Dip treatment[1]	0.56, 0.50 (0.53)	Thompson. Ediger 2003 IR4 07568 1751-02

Location Year (variety)	Form	Method	Fludioxonil residue, mg/kg[4]	Author, Date Study No. Syn No
Washington 2001 (Red Delicious)	WP 50	Dip treatment[1]	1.1, 0.72 (0.91)	Thompson. Ediger 2003 IR4 07568 1751-02
	WP 50	Packing line spray[2]	0.68, 0.57 (0.62)	
	WP 50	Dip treatment[3] followed by packing line spray[3]	2.2,1.8 (2.0)	

[1]post-harvest dip: 0.06 kg ai/hl – dip solution includes carnuba packing wax, fruit dipped 2 min (± 10 s)
[2]packing line spray 0.25 kg ai in low pressure/low volume post-harvest packing line spray in approx. 30–105 l of water+ carnuba fruit wax per 100,000 kg fruit
[3]post-harvest dip 0.06 kg ai/hl (no carnuba packing wax); fruit dipped 2 min (± 10 s)
[4] results from replicate treatments are on same line

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Table 66: Fludioxonil residues resulting from post-harvest application to pears in the USA.

Location Year variety	Form	Method	Conc. kg ai/hl	Fludioxonil residue, mg/kg	Author, Date Study No.
NJ 2000 Bartlett	WP 50	drench[1]	0.06	0.76, 0.71	Starner 2001 556-00
		dip[2]	0.06	1.2, 0.79	
CA 2000 Shinko	WP 50	drench[1]	0.06	1.6, 1.3	Starner 2001 556-00
		packing line spray[3]	0.06	2.5, 1.4	
		drench[1] then packing line spray[3]	2 x 0.06	2.8, 2.7 (2.8)	
		dip[4]	0.06	2.7, 1.6	
WA 2000 Anjou	WP 50	drench[1]	0.06	1.3, 1.1	Starner 2001 556-00
		packing line spray[5]	0.06	1.6, 1.3	
		drench[1] then packing line spray[5]	2 x 0.06	1.6, 1.5 (1.6)	
		dip[2]	0.06	0.68, 0.67	
ID 2000 Anjou	WP 50	drench[1]	0.06	3.5, 2.2	Starner 2001 556-00
		dip[2]	0.06	1.4, 0.93	

[1]fludioxonil drench 0.048 kg ai/hl water
[2]fludioxonil dip 0.048 kg ai/hl + fruit wax (10 l into 40 l water)
[3]fludioxonil packing line spray 0.2 – 0.6 kg ai/hl + undiluted carnuba wax
[4]fludioxonil dip 0.06 kg ai/hl + carnuba wax
[5]fludioxonil packing-line spray 0.2 – 0.6 kg ai/hl + undiluted carnuba wax per 200,000 lb (= 90,720 kg) fruit

Table 67. Fludioxonil residues in pears from supervised trials in Italy, France and Spain after foliar application.

Country Year (variety)	Application					PHI days	Fludioxonil residue, mg/kg	Author, Date Study No.
	Form	kg ai/ha	kg ai/hl	Water, l/ha	No.			
Italy 1996 (Kaiser)	WG 62.5	0.25	0.020	1200	3	0B*	0.08	Walser 1997 2063/96
						0	0.31	
						7	0.15	

Country Year (variety)	Application Form	kg ai/ha	kg ai/hl	Water, l/ha	No.	PHI days	Fludioxonil residue, mg/kg	Author, Date Study No.
						14 21	0.07 0.04	0975
Italy 1996 (Kaiser)	WG 62.5	0.25	0.025	1000	3	0B* 0 7 14 21	0.11 0.48 <u>0.21</u> 0.20 0.14	Walser 1997 2064/96 0976
Italy 1998 (Abate Fetel)	WG 62.5	0.25	0.017	1500	3	0B* 0 7 14 21	0.10 0.21 <u>0.18</u> 0.10 0.09	Walser 1998 2072/97 1117
Italy 1997 (Kaiser)	WG 62.5	0.25	0.020	1200	3	14	0.18, 0.13	Walser 1997 2065/96 0977
Italy 1997 (Williams)	WG 62.5	0.25	0.016	1500	3	14	0.03, 0.03	Walser 1998 2071/97 1116
France, South 1997 (Williams)	WG 62.5	0.25	0.027	931-949	3	0B* 0 3 7 14 21	0.08 0.29 0.14 <u>0.14</u> 0.12 0.10	Walser 1998 2161/97 1120
Spain 1997 (Conference)	WG 62.5	0.25	0.013	1912- 1915	3	0B* 0 3 7 14 21	0.03 0.44 0.35 <u>0.28</u> 0.08 0.04	Walser 1998 2055/97 1119
Spain 1997 (Blanquilla)	WG 62.5	0.24- 0.25	0.023 – 0.025	1433 – 1630	3	14	0.19, 0.22	Walser 1998 2054/97 1118
Spain 2001 (Blanquilla)	WG 62.5	0.25- 0.26	0.021 - 0.025	1051- 1067	2	7	0.38, 0.2 <u>(0.32)</u>	Solé 2002 2090/01 5528
Spain 2001 (Blanquilla)	WG 62.5	0.26- 0.27	0.025	1108- 1215	2	7	0.47, 0.25 <u>(0.36)</u>	Solé 2002 2091/1 5529

*0B: before final application

Stone fruits

Table 68: Fludioxonil residues resulting from post-harvest application to peaches in the USA.

Location Year (variety)	Form	Method	Treatment concentration kg ai/hl	Fludioxonil residue, mg/kg[3]	Author, Date Study No. Syn No
California 1998 (Elegant Lady)	WP 50	Packing line spray[1]	0.02 0.03 0.06	0.16, 0.10 0.18, 0.05 0.55, 0.19 <u>(0.37)</u>	Ediger & Thompson 1999 833-99
S Carolina 1998	WP 50	Packing line spray[2]	0.02 0.03	0.21, 0.15 0.37, 0.17	Ediger & Thompson

Location Year (variety)	Form	Method	Treatment concentration kg ai/hl	Fludioxonil residue, mg/kg[3]	Author, Date Study No. Syn No
(Jefferson)			0.06	0.49, 0.35 (0.42)	1999 833-99
California 1998 (Goldcrest)	WP 50	Post-harvest dip[1]	0.02 0.03 0.06	1.7, 1.5 2.2, 2.1 3.6, 3.5 (3.6)	Thompson &Ediger 1999 455-98
California 2000 (Elegant Lady)	WP 50	High volume spray Low volume spray Low volume spray Low volume spray Post-harvest dip	0.06 0.06 0.045 0.03 0.06	1.8, 1.3 (1.6) 2.8, 2.7 (2.8) 1.9, 1.3 1.7, 1.2 3.8, 3.0 (3.4)	Thompson& Ediger 2000 878-00 5482

[1]DECCO LUSTR 251 fruit wax added at 25 l/100L
[2]DECCO LUSTR 282 fruit wax added at 5 l/100L
[3]Fruit was air-dried and de-stoned after treatment

Table 69. Fludioxonil residues in peaches following foliar application in supervised trials in Italy, France, and Spain.

Country Year (variety)	Application					PHI Days	Fludioxonil residue, mg/kg	Author, Date Study No.
	Form.	kg ai/ha	kg ai/hl	l/ha	No.			
Italy 2001 (Flavorcrest)	WG 62.5	0.25	0.021	1200	2	0 3 7 14	0.10 0.12 0.11, 0.05 0.08	Ryan 2002 2047/01 5479
Italy 2001 (Red Moon)	WG 62.5	0.25	0.021	1200	2	0 3 7 14	0.43 0.09 0.06, 0.03 0.04	Ryan 2002 2045/01 5480
Italy 2001 (Stark Red Gold)	WG 62.5	0.25 - 0.26	0.021	1200 - 1247	2	0 3 7 14	0.09 0.12 0.07, 0.06 0.08	Ryan 2002 2046/01 5481
Italy 2001 (Carson)	WG 62.5	0.25	0.021	1215	2	0 3 7 14	0.60 0.31 0.31, 0.23 0.23	Ryan 2002 2048/01 5483
Italy 1996 (Fayette)	WG 62.5	0.20	0.020 - 0.025	800- 1000	3	0B* 0 7 14 21	0.16 0.43 0.29 0.23 0.17	Walser 1997 2058/96 0978
Italy 1997 (Bella di Imola)	WG 62.5	0.20	0.013 - 0.020	1000 - 1500	3	0B* 0 7 14 21	<0.02 0.10 <0.02 <0.02 0.02	Walser 1998 2068/97 1147
France, South 2002 (Symphonie)	WG 62.5	0.25 - 0.27	0.025	1002 - 1074	2	0B* 0 3 7 10 14	0.17 0.23 0.28 0.19 0.16 0.11	Solé 2003 02-2131 5599
France, South 1994 (Queen Ruby)	WG 62.5	0.15	0.018	833	3	0B 0 7 14 28	0.04 0.17 0.06 0.06 0.03	Maffezzoni 1995 OF94154 0597
France, South	WG	0.15	0.016	926	3	0B	0.08	Maffezzoni

Country Year (variety)	Application					PHI Days	Fludioxonil residue, mg/kg	Author, Date Study No.
	Form.	kg ai/ha	kg ai/hl	l/ha	No.			
1994 (Symphonie)	62.5					0 5 14 28	0.10 0.11 <u>0.04</u> 0.03	1995 OF94154 0597
Spain 2002 (Androx)	WG 62.5	0.25	0.025	990 - 1005	2	0B* 0 3 7 10 14	0.24 0.53 0.63 0.48 0.50 <u>0.33</u>	Solé 2003 02-2128 5600
Spain 2002 (Sudanel)	WG 62.5	0.24 – 0.25	0.025	954 - 1003	2	0B* 0 3 7 10 14	0.28 0.53 0.56 0.37 0.26 <u>0.29</u>	Solé 2003 02-2129c 5602

*0B: before final application

Table 70. Fludioxonil residues resulting from post-harvest application to plums in California, USA (Thompson and Ediger, Report 834-99, 1999).

Year (variety)	Form	Method	Treatment concentration kg ai/hl	Fludioxonil residue, mg/kg[3]
Trial CA31 1998 (Casselman)	WP 50	Packing line spray[1]	0.02 0.03 0.06	0.12, 0.09 (0.10) 0.05, 0.05 (0.05) 0.10, 0.09 <u>(0.10)</u>
Trial CA32 1998 (Casselman)	WP 50	Packing line controlled droplet application[2]	0.02 0.03 0.06	0.14, 0.13 (0.14) 0.47, 0.42 (0.44) 1.06, 0.79 <u>(0.92)</u>

[1]DECCO LUSTR 251 fruit wax added at 25 l/100L
[2]DECCO LUSTR 251 fruit wax added at 75 l/100L
[3]Fruit was air-dried and de-stoned after treatment. Results are duplicate samples, not duplicate trials.

Table 71. Fludioxonil residues in plums after foliar application from supervised trials in Italy, France, Germany, and Switzerland.

Country Year (variety)	Application					PHI days	Fludioxonil residue, mg/kg	Author, date Study No. Syn No
	Form	kg ai/ha[1]	kg ai/hl	Water, l/ha	No.			
Italy 2000 (Angelina)	WG 62.5	0.25	0.025	1200	3	0 3 7 14 21	0.05 0.03 0.06 <u>0.04</u> 0.02	Ryan 2002 2089/01 5505
Italy 2000 (Black Star)	WG 62.5	0.25	0.025	1000	3	14	<u>0.065</u>	Salvi 2001 2103/00 5380

Country Year (variety)	Application					PHI days	Fludioxonil residue, mg/kg	Author, date Study No. Syn No
	Form	kg ai/ha[1]	kg ai/hl	Water, l/ha	No.			
Italy 2000 (Regina)	WG 62.5	0.25	0.025	996- 1000	3	0 7 14 21	0.08 0.09 0.05 0.09	Salvi 2001 2102/00 5394
France, South 1994 (707/GF801)	WG 62.5	0.15	0.030	500	3	0B* 0 7 14 28	0.02 0.05 0.05 0.03 0.02	Maffezzoni 1995 OF94156 0599
France, South 1994 (President)	WG 62.5	0.15	0.015	1000	3	0B* 0 7 14 28	0.02 0.05 0.06 <0.02 <0.02	Maffezzoni 1995 OF94156 0599
Switzerland 1997 (Fellenberg)	WG 62.5	0.22	0.015	1500	3	0B* 0 7 11 14	0.05 0.29 0.18 0.11 0.06	Walser 1998 2342/97 1162
Switzerland 1997 (Fellenberg)	WG 62.5	0.22	0.015	1500	3	0B* 0 7 11 14	0.04 0.24 0.14 0.11 0.17	Walser 1998 2343/97 1163
Switzerland 1997 (Fellenberg)	WG 62.5	0.22	0.015	1500	3	0B* 0 7 10 14	0.02 0.25 0.09 0.08 0.11	Walser 1998 2344/97 1164
Germany 1998 (Cak-Cak's Beste)	WG 62.5	0.22	0.015	1500	3	0 7 10 14	0.05 0.03 0.04 0.05	Smith 1999 gr 91898 4976
Germany 1999 (Hauszwetschge)	WG 62.5	0.19– 0.23	0.015	1265 - 1545	3	0 7 10 14	0.19 0.09 0.10 0.10	Smith 1999 gr 92998 4977
Germany 1998 (Hauszwetschge Schäfer)	WG 62.5	0.22	0.015	1500	3	0 7 10 14	0.32 0.07 0.13 0.06	Smith 2000 gr 90898 5205
Germany 2000 (Hauszwetschge)	WG 62.5	0.22	0.015	1500	3	0 14	0.17 0.07, 0.07 (0.07)	Simon 2001 gr 36800 5438

*0B: before final application
[1]Calculated from spray volume/ha and spray concentration

Table 72. Fludioxonil residues resulting from post-harvest application to cherry in the USA (Ediger and Thompson, Report 06933, 1999).

Location, Year (variety)	Form	Method	Treatment concentraton kg ai/hl	Fludioxonil residue, mg/kg[3]
California 91 1998 (Bing)	WP 50	Packing line spray[1]	0.02 0.03 0.06	0.19, 0.16 0.42, 0.15 0.78, 0.57 (0.68)
Michegan 21	WP 50	Packing line spray[2]	0.02	0.15, 0.08

Location, Year (variety)	Form	Method	Treatment concentraton kg ai/hl	Fludioxonil residue, mg/kg[3]
1998 (Hedelfingen)			0.03 0.06	0.20, 0.19 0.27, 0.1 (0.19)
Washington 1998 (Chinook)	WP 50	Packing line spray[1]	0.02 0.03 0.12	0.73, 0.73 0.50, 0.44 1.08, 0.91
Washington 1998 (Chinook)	WP 50	Packing line spray[1]	0.02 0.03 0.12	0.34, 0.28 0.53, 0.49 1.2, 1.2

[1]Fruit wax (DECCO LUSTR 251) was added at 5 l/100L
[2]No fruit wax added

Table 73. Fludioxonil residues in cherries after foliar application from supervised trials in France, Germany, Italy, and Switzerland.

Country Year (variety)	Application					PHI days	Fludioxonil residue, mg/kg	Author, Date Study No. Syn No
	Form	kg ai/ha	kg ai/hl	water, l/ha	No.			
Germany 1998 (Schneiders späte Knorpel)	WG 62.5	0.22	0.015	1500	3	0 7 10 14	0.63 0.16 0.17 0.16	Smith gr 93898 1999 4978
Germany 1998 (Van)	WG 62.5	0.22	0.015	1500	3	0 7 10 14	0.56 0.17 0.25 0.23	Smith gr 94898 1999 4979
Switzerland 1996 (Sunburst)	WG 62.5	0.15	0.015	1000	3	0B* 0 7 14 21	0.11 0.34 0.09 0.04 0.03	Walser 1997 2023/96 0965
Switzerland 1996 (Burlat)	WG 62.5	0.15	0.015	1000	3	14	0.07, 0.03	Walser 1997 2024/96 0972
Italy 1996 (Stella)	WG 62.5	0.15	0.015- 0.019	800 - 1000	3	0B* 0 7 14 21	0.18 0.43 0.12 0.02 0.02	Walser 1997 2059/96 0979
France (North) 1997 (Stanley)	WG 62.5	0.23	0.015	1512- 1555	3	7 14	0.16 0.08	Sole 2003 02-2122 5679

*0B: before final application

Berries and other small fruits

Table 74. Fludioxonil residues in grapes (berries) from supervised trials in Chile, South Africa, Spain, Greece Italy, France, Germany and Switzerland.

Country Year (variety)	Application					PHI days	Fludioxonil residue, mg/kg	Author Date Study No. Syn No
	Form	kg ai/ha	kg ai/hl	Water, l/ha	No.			
Chile 1996	WG 62.5	0.20 – 0.25	0.016 –	1225 – 1519	2	7	0.27, 0.22 (0.24)	Walser 1996

Country Year (variety)	Application					PHI days	Fludioxonil residue, mg/kg	Author Date Study No. Syn No
	Form	kg ai/ha	kg ai/hl	Water, l/ha	No.			
(Thompson Seedless)			0.017					2218/95 0840
Chile 1996 (Thompson Seedless)	WG 62.5	0.20 – 0.25	0.017 – 0.020	975 – 1497	2	7	0.21, 0.16 (0.18)	Walser 1996 2219/95 0841
Chile 1996 (Thompson Seedless)	WG 62.5	0.20 – 0.25	0.020 – 0.025	797- 1236	2	7	0.32, 0.25 (0.28)	Walser 1996 2224/95 0846
Chile 1996 (Thompson Seedless)	WG 62.5	0.20- 0.25	0.020 - 0.025	804- 1243	2	7	0.30, 0.27 (0.28)	Walser 1996 2226/95 0848
South Africa 1998 (S.A. Reisling)	WG 62.5	0.20	0.020	1000	2	0B* 0 3 7 14 21 28	0.02 0.22 0.17 0.25 0.16 0.21 0.10	Walser 1999 2408/97 1380
Spain 1996 (Mazuelo)	WG 62.5	0.25	0.030 - 0.050	496 –826	2	0B* 0 7 14 21	<0.02 0.6 0.51 0.12 0.22	Walser 1997 2007/96 0928
Spain 1996 (Macabeo)	WG 62.5	0.24 - 0.26	0.038	613 - 678	2	0B* 0 7 14 21 28	0.10 1.62 1.32 0.76 0.41 0.30	Walser 1997 2008/96 0962
Greece 2002 (Black Corinth)	WG 62.5	0.25	0.025	995	2	0 7 15	0.34 0.41, 0.22 (0.32) 0.38	Kühne 2003 02-2111 5574
Greece 2002 (Sultana)	WG 62.5	0.25	0.025	995	2	0 7 14	0.62 0.42, 0.39 (0.40) 0.35	Kühne 2003 02-2112 5575
Greece 2002 (Sultana)	WG 62.5	0.25	0.025	996	2	0 7 14	0.59 0.37, 0.36 (0.36) 0.26	Kühne 2003 02-2113 5576
Italy 1994 (Trebbiano Romagnolo)	WG 62.5	0.25	0.025	1000	2	0 7 14 21	0.59 0.55 0.63 0.43	Kissling 1995 2109/94 0658
Germany 1995 (Dornfelder)	WG 62.5	0.30	0.038	800	2	0 13 28 36 41	1.15 0.50 0.36 0.28 0.24	Ipach 1996 gr51295 0717
Germany 1995 (Scheurebe)	WG 62.5	0.30	0.038	800	2	0 13 28 34 41	0.67 0.47 0.33 0.17 0.20	Ipach 1996 gr51195 0718
Germany 1995 (Muller-Thurgau)	WG 62.5	0.30	0.038	800	2	0 13 28 34	0.95 0.53 0.34 0.31	Ipach 1996 gr51095 0719

Country Year (variety)	Application					PHI days	Fludioxonil residue, mg/kg	Author Date Study No. Syn No
	Form	kg ai/ha	kg ai/hl	Water, l/ha	No.			
						41	0.24	
Germany 1994 (Kerner)	WG 62.5	0.30	0.038	800	2	0B* 0 14 29 34 43	0.16 0.62 0.36 0.25 <u>0.17</u> 0.16	Raum 1996 gr5094 0813
Germany 1994 (Dornfelder)	WG 62.5	0.30	0.038	800	2	0B* 0 14 29 34 43	0.27 0.68 0.32 0.20 <u>0.24</u> 0.21	Raum 1996 gr5094 0813
Germany 1999 (Kerner)	WG 62.5	0.30	0.015	2000	2	0 21 35 42	0.43 0.16 <u>0.21</u> 0.12	Smith 2000 gr42899 5123
Switzerland 1994 (Pinot Noir)	WG 62.5	0.30	0.038	800	2	0 35 42 50	5.2 1.4 1.2 <u>1.6</u>	Kissling 1995 2057/94 1136
Switzerland 1994 (Chasselas)	WG 62.5	0.30	0.038	800	2	0 35 42 49	3.8 0.66 <u>0.90</u> 0.60	Kissling 1995 2058/94 1137
Switzerland 1994 (Chasselas)	WG 62.5	0.30	0.038	800	2	0 36 43 49	2.7 1.30 1.10 <u>1.40</u>	Kissling 1995 2059/94 1138
Switzerland 1995 (Chasselas)	WG 62.5	0.30	0.038	800	2	0B* 0 14 28 35 42	0.07 1.49 1.19 0.79 <u>0.99</u> 0.41	Walser 1996 2049/95 1092
Switzerland 1995 (Pinot Noir)	WG 62.5	0.30	0.038	800	2	0B* 0 14 28 35 42	0.14 3.3 1.4 1.2 <u>1.6</u> 1.0	Walser 1996 2050/95 1093
Switzerland 1993 (Chasselas)	WP50	0.50	0.063	800	1	63	0.77, 0.60	Mair 1997 2047/92-96 0466
Switzerland 1992 (Chasselas)	WP50	0.50	0.063	800	1	57	0.10, 0.08	Mair 1997 2047/92-96 0466
France, North 1989 (Sauvignon)	WP 50	0.50	0.050	100	2	45	0.26	Tournayre 1990 106/89 0020
France, North 1989 (Chardonnay)	WP 50	0.50	0.031	160	2	60	0.20	Tournayre 1990 108/89 0021
France, North 1989 (Chenin)	WP 50	0.50	0.050	100	2	45	0.30	Tournayre 1990 109/89 0022
France, North	WP	0.50	0.040	125	2	49	0.27	Tournayre

Country Year (variety)	Application					PHI days	Fludioxonil residue, mg/kg	Author Date Study No. Syn No
	Form	kg ai/ha	kg ai/hl	Water, l/ha	No.			
1989 (Reisling)	50							1990 112/89 0023
France, North 1989 (Pinot Noir)	WP 50	0.50	0.050	100	2	53	<0.02	Tournayre 1990 113/89 0024
France, North 1990 (Pinot Noir)	WP 50	0.50	0.046	110	2	62	0.23	Tournayre 1992 02/92 0158
France, North 1991 (Pinot)	WP 50	0.50	0.046	110	1	65	0.16	Tournayre 1991 0470F91 0161
France, North 1993 (Meunier 41B)	WP 50	0.50	0.025	204	1	66	0.42	Maffezzoni 1994 OF93162 0533
France, North 1993 (Gamay)	WP 50	0.50	0.033	150	1	73	0.24	Maffezzoni 1994 OF93162 0533
France, North 1993 (Sauvignon)	WP 50	0.50	0.033	150	1	73	0.05	Maffezzoni 1994 OF93162 0533
France 1993 (Semillon)	WP 50	0.50	0.033	152	1	70	0.05	Maffezzoni 1994 OF93162 0533

*0B: before final application

Table 75. Fludioxonil residues in strawberries from supervised outdoor trials in the USA and Europe.

Location, year (variety)	Application					PHI Days	Fludioxonil residue, mg/kg	Author, Date Study No. Syn No
	Form.	kg ai/ha	kg ai/hl	Water, l/ha	No.			
USA, New York 1996 (Tribute)	WP 50	0.28	0.060	468	4	0	0.71, 0.53 (0.62)	Van Geluwe 1997 ABR-97069
USA, California 1996 (Chandler)	WP 50	0.28	0.040	701	4	0	0.45, 0.41 (0.43)	Van Geluwe 1997 ABR-97069
USA, California 1996 (Camarosa)	WP 50	0.28	0.060	468	4	0	1.1, 1.0 (1.0)	Van Geluwe 1997 ABR-97069
USA, Florida 1996 (Oso Grande)	WP 50	0.28	0.035	797	4	0	1.2, 1.1 (1.2)	Van Geluwe 1997 ABR-97069
USA, Michegan 1996 (Allstar)	WP 50	0.28	0.058	474 - 489	4	0 6	0.30, 0.14 (0.22) 0.16, 0.10 (0.13)	Van Geluwe 1997 ABR-97069
USA, California 1996 (592)	WP 50	0.28	0.030	935	4	0 3	1.3, 1.3 (1.3) 1.5, 1.1	Van Geluwe 1997 ABR-97069

Location, year (variety)	Application					PHI Days	Fludioxonil residue, mg/kg	Author, Date Study No. Syn No
	Form.	kg ai/ha	kg ai/hl	Water, l/ha	No.			
							(1.3)	
USA, Oregon 1996 (Totem)	WP50	0.28	0.037	748	4	0	0.63, 0.46 (0.54)	Van Geluwe 1997 ABR-97069
USA, North Carolina 1996 (Chandler)	WP 50	0.28	0.060	467	4	0	1.3, 1.1 (1.2)	Van Geluwe 1997 ABR-97069
Germany 1995 (Korona)	WG 62.5	0.25	0.013	2000	3	0 3 5 7 10 12	0.30 0.09 0.07 0.04 0.05 0.04	Beinhauer 1996 FRI12/95/35 0720
Germany 1995 (Korona)	WG 62.5	0.25	0.013	2000	3	0 3 5 7 10 12	0.21 0.07 0.05 0.05 0.03 0.03	Beinhauer 1996 FRI12/95/44 0721
Germany 1995 (Korona)	WG 62.5	0.24- 0.25	0.024- 0.025	991- 1069	3	0 3 5 7 10 12	0.41 0.21 0.08 0.04 0.03 0.02	Leiblein 1996 95011 R 0722
Switzerland 1995 (Elvira)	WG 62.5	0.25	0.031	800	3	0 3 5 7 10 12	0.83 0.21 0.19 0.13 0.09 0.10	Walser 1996 2051/95 0716
Switzerland 1995 (Elsenta)	WG 62.5	0.25	0.031	800	3	0 3 6 8 10 13	0.23 0.23 0.12 0.13 0.14 0.09	Walser 1996 2052/95 1100
France, North 1995 (Pandora)	WG 62.5	0.25	0.050- 0.051	486-500	3	0B* 0 3 7 10 14	0.05 0.15 0.10 0.09 0.08 0.06	Maffezzoni 1996 OF95116/DE9 8 0781
France, North 2002 (Selva)	WG 62.5	0.25	0.025	1000	3	0 1 3 7 14	0.47 0.54 0.38, 0.32 0.25 0.13	Solé 2003 02-2059
UK 2002 (Florence)	WG 62.5	0.25 - 0.26	0.050	502-509	3	0 1 3 7 14	0.22 0.24 0.16, 0.15 0.11 0.06	Solé 2003 02-2054 5668
France, South 1995 (Chandler)	WG 62.5	0.25	0.050	500	3	0B* 0 3 7 10 14	0.39 0.94 0.89 0.77 0.65 0.60	Maffezzoni 1996 OF95116/AC9 7 0780
France, South 2001 (Mamie)	WG 62.5	0.23- 0.26	0.063	395-410	3	0 3	0.31 0.19, 0.18	Pointurier 2002 0110402

Location, year (variety)	Application					PHI Days	Fludioxonil residue, mg/kg	Author, Date Study No. Syn No
	Form.	kg ai/ha	kg ai/hl	Water, l/ha	No.			
								5485
France, South 2001 (Mara Style)	WG 62.5	0.25- 0.26	0.063	397-420	3	0 3	0.31 0.24, 0.21	Pointurier 2002 0110401 5486
Italy 2002 (Marmolada)	WG 62.5	0.26	0.025	1033 - 1040	3	0 1 3 7	0.27 0.31 0.27, 0.26 0.14	Solé 2003 02-2051 5636
France (South) 2002 (Ciloe)	WG 62.5	0.25 - 0.26	0.050	501 - 509	3	0 1 3 7 14	0.94 0.77 0.73, 0.68 0.61 0.29	Solé 2003 02-2057 5677
France (South) 2002 (Tetis)	WG 62.5	0.24 - 0.27	0.050	476 - 537	3	0 1 3 10 17	1.0 0.87 0.73, 0.72 0.48 0.35	Solé 2003 02-2058 5678
Spain 2002 (Camarrosa)	WG 62.5	0.24 - 0.25	0.025	955 - 996	3	0 1 3 7 14	0.65 0.70 0.61, 0.58 0.64 0.40	Solé 2003 02-2001 5643
Spain 2002 (Camarrosa)	WG 62.5	0.25	0.025	992 - 1004	3	0 1 3 7 14	0.95 0.77 1.04, 0.98 0.83 0.46	Solé 2003 02-2002 5644

Table 76. Fludioxonil residues in strawberries from supervised indoor trials in Spain, Italy, France, and the UK.

Country Year (variety)	Application					PHI Days	Fludioxonil residue, mg/kg	Author, Date Study No. Syn No
	Form.	kg ai/ha	kg ai/hl	Water, l/ha	No.			
Spain 1994 (Tulda)	WG 62.5	0.25	0.021	1150	3	0 3 7 14	2.7 2.2 1.9 1.0	Kissling 1995 2013/94 1098
Spain 1994 (OsoGrande)	WG 62.5	0.25	0.021	1150	3	0 3 7 14	1.3 1.0 0.11 0.08	Kissling 1995 2014/94 1099
Spain 2001 (Camarrosa)	WG 62.5	0.24- 0.25	0.025	944-986	3	0 3	0.57 0.48, 0.42 (0.45)	Ryan 2002 2056/01 5512
Spain 2001 (Camarrosa)	WG 62.5	0.25	0.025 - 0.026	961 - 984	3	0 3	0.58 0.45, 0.44 (0.44)	Ryan 2002 2055/01 5504
Italy 2001 (Onda)	WG 62.5	0.24- 0.25	0.025	958- 1010	3	0 3	0.17 0.26, 0.14 (0.20)	Ryan 2002 2050/01 5503
France, North	WG 62.5	0.22-	0.063	376-392	3	0	1.0	Pointurier

Country Year (variety)	Application					PHI Days	Fludioxonil residue, mg/kg	Author, Date Study No. Syn No
	Form.	kg ai/ha	kg ai/hl	Water, l/ha	No.			
2001 (Diamante)		0.245				3	0.31, 0.27 (0.29)	2002 0110301 5484
France, North 2001 (Diamante)	WG 62.5	0.23-0.26	0.063	368-411	3	0 3	0.46 0.78, 0.54 (0.66)	Pointurier 2002 0110302 5495
France, North 2000 (Gariguette)	WG 62.5	0.25	0.063	400	3	0 3 7	0.26 0.24, 0.18 (0.21) 0.21	Pointurier 2001 0011102 5336
France, North 2001 (Chandler)	WG 62.5	0.25	0.063	400	3	0 3 7	0.71 0.44, 0.41 (0.42) 0.27	Pointurier 2002 0011101 5337
Switzerland 2001 (Marmelada)	WG 62.5	0.25	0.031	800	3	3	0.41, 0.24 (0.32)	Kühne 2003 2113-01 5629
Switzerland 2001 (Marmelada)	WG 62.5	0.25	0.031	800	3	3	0.21, 0.17 (0.19)	Kühne 2003 2114-01 5630

Table 77. Fludioxonil residues in raspberries from supervised trials in the USA and Germany.

Country Year (variety)	Application					PHI days	Fludioxonil residue, mg/kg	Author, Date Report No. Syn No
	Form	kg ai/ha	kg ai/hl	Water, l/ha	No.			
USA, NC 1998 (Southland)	WG 62.5	0.24	0.053	463	4	0	4.17, 2.99 (3.6)	Starner 2001 IR-4 PR No 06838 US 1239-01
USA, NH 1998 (Heritage)	WG 62.5	0.24	0.048	489 - 506	4	0	1.05, 1.04 (1.0)	Starner 2001 IR-4 PR No 06838 US 1239-01
USA, WA 1998 (Meeker red)	WG 62.5	0.24 - 0.26	0.033 - 0.043	584 - 762	4	0	1.11, 0.81 (0.96)	Starner 2001 IR-4 PR No 06838 US 1239-01
USA, WA 199 (Meeker red)	WG 62.5	0.24 - 0.25	0.033 – 0.043	575 - 752	4	0	1.22, 0.88 (1.0)	Starner 2001 IR-4 PR No 06838 US 1239-01
USA, WA 1998 (Canby)	WG 62.5	0.24	0.040 - 0.043	576 - 613	4	0	1.04, 0.98 (1.0)	Starner 2001 IR-4 PR No 06838 US 1239-01
Germany 1999 (Autumn Bliss)	WG 62.5	0.25	0.013	2000	3	0 7 10 14	1.1 0.51 0.46 0.19	Smith 2001 gr 93899 0958
Germany	WG	0.25	0.013	1969	3	0	2.7	Smith

Country Year (variety)	Application					PHI days	Fludioxonil residue, mg/kg	Author, Date Report No. Syn No
	Form	kg ai/ha	kg ai/hl	Water, l/ha	No.			
1999 (Himboqueen)	62.5			- 2004		7 9 14	0.61 0.26 0.24	2000 gr 94999 0959
Germany 2000 (Autumn Bliss)	WG 62.5	0.25	0.013	2000	3	0 7 10 14 21	<u>0.58</u> 0.50 0.32 0.25, 0.23 0.15	Simon 2001 gr39800 5421
Germany 2000 (Rumiloba)	WG 62.5	0.24- 0.26	0.012- 0.013	1902 - 2066	3	0 7 10 13 21	<u>1.1</u> 0.98 0.50 0.30, 0.29 0.20	Simon 2001 gr40900 5422

Table 78. Residues of fludioxonil in blueberries and currants from supervised trials in the USA and Germany.

Country, year (variety)	Application					PHI days	Fludioxonil residue, mg/kg	Author, Date Report No. Syn No
	Form	kg ai/ha	kg ai/hl	Water, l/ha	No.			
BLUEBERRY								
USA, North Carolina[1] 1998 – NC06 (Harrison)	WG 62.5	0.24	0.077	319-322	4	0	0.28, 0.25 <u>(0.26)</u>	Thompson 2001 06724
USA, North Carolina[1] 1998 –NC07 (Harrison)	WG 62.5	0.24	0.077	321-326	4	0	0.16, 0.12 <u>(0.14)</u>	Thompson 2001 06724
USA, Maine 1998 – ME04 (wild blueberry)	WG 62.5	0.24	0.089	275-279	4	0	1.70, 1.16 <u>(1.4)</u>	Thompson 2001 06724
USA, New Jersey 1998 – NJ29 (Bluecrop)	WG 62.5	0.24	0.074	324-334	4	0	0.58, 0.47 <u>(0.52)</u>	Thompson 2001 06724
USA, Oregon 1998 – OR29 (Bludecrop)	WG 62.5	0.24	0.051	476-481	4	0	<0.05, <0.05 <u>(<0.05)</u>	Thompson 2001 06724
USA, Michigan 1998 – MI23 (Rubel)	WG 62.5	0.24	0.052	461-481	4	0	0.94, 0.87 <u>(0.90)</u>	Thompson 2001 06724
USA, Michigan 1998 – MI24 (Rubel)	WG 62.5	0.24	0.053	463-468	4	0	0.68, 0.68 <u>(0.68)</u>	Thompson 2001 06724
USA, Michigan 1998 –MI 25 (Rubel)	WG 62.5	0.24	0.052	461-481	4	0	0.90, 0.77 <u>(0.84)</u>	Thompson 2001 06724
Germany 1999 (Berkley)	WG 62.5	0.25	0.013	2000	3	0 7 9 14	<u>0.78</u> 0.31 0.23 0.12	Smith 2001 gr 95899 0960
Germany 1999 (Heerma)	WG 62.5	0.24 - 0.25	0.013	1926 - 2004	3	0 7 10 13	<u>0.70</u> 0.31 0.16 0.08	Smith 2000 gr 96999 0961
Germany 2000 (Blue Crop)	WG 62.5	0.25	0.013	1995	3	0 7 10 14 22	<u>0.91</u> 0.37 0.34 0.31 0.24	2001 gr 41800 5468

Country, year (variety)	Application					PHI days	Fludioxonil residue, mg/kg	Author, Date Report No. Syn No
	Form	kg ai/ha	kg ai/hl	Water, l/ha	No.			
Germany 2000 (Heerma)	WG 62.5	0.23 - 0.24	0.012	1867 - 1948	3	0 7 10 14 21	0.48 0.15 0.10 0.07 0.04	2001 gr 42900 5469
BLACK CURRANT								
Germany 1999 (Titania)	WG 62.5	0.24 - 0.25	0.013	1961 - 2029	3	0 7 10 14	0.63 0.24 0.26 0.16	Smith 2000 gr 92999 0957
Germany 2000 (Ben Alder)	WG 62.5	0.25	0.013	1991	3	0 7 10 14 21	0.57 0.60 0.46 0.55 0.56	2001 gr 30800 5464
Germany 2000 (Ojebin	WG 62.5	0.25	0.012	2020	3	0 7 10 14 21	0.83 0.62 0.59 0.43 0.30	2001 gr 37800 5465
Germany 2000 (Titania)	WG 62.5	0.25	0.013	1975 - 1992	3	0 7 10 14 21	0.79 0.55 0.63 0.53 0.33	2001 gr 38900 5466
RED CURRANT								
Germany 1999 (Rondom)	WG 62.5	0.25	0.013	2000	3	0 7 10 14	2.7 1.4 0.97 0.45	Smith 2001 gr 91899 0956

[1]Trials were approximately 30 m apart, had same application date and shared a control plot

Assorted tropoical and sub-tropical fruit – inedible peel

Table 79. Residues of fludioxonil in lychees from supervised trials in Florida, USA.

Year (variety)	Application					PHI days	Fludioxonil residue, mg/kg	Author Date Study No. Syn No
	Form	kg ai/ha	kg ai/hl	Water, l/ha	No.			
2000 (Mauritius)	WG 62.5	0.245	0.035	701.5	5	0	1.04, 0.81 (0.92)	Chen, Moore 2002 IR-4 07760 28813
2000 (Mauritius)	WG 62.5	0.245	0.011	1221 - 1196	5	0	1.03, 0.59 (0.81)	Chen, Moore 2002 IR-4 07760 28813
2000 (Mauritius)	WG 62.5	0.245		1188 - 1235	7	0	1.48, 1.36 (1.4)	Chen, Moore 2002 IR-4 07760 28813

Table 80: Fludioxonil residues resulting from post-harvest application to kiwifruit in the USA.

Location Year (variety)	Form	Method	Conc. kg ai/hl	Fludioxonil residue, mg/kg	Author Date Report No. Syn. No.
CA 01 2000 (Hayward)	50WP	Fludioxonil spray[1] Fludioxonil dip[2]	0.06	2.7, 0.6 9.5, 7.6	Ediger 2002 217-01
CA 02 2000 (Hayward)	50 WP	Fludioxonil spray[1] Fludioxonil dip[2]	0.06	13.9, 8.4 7.0, 6.9 8.0, 4.2 7.4, 7.2	Ediger 2002 217-01
OR 02 2000 (Hayward)	50 WP	Fludioxonil dip[2]	0.06	5.4, 5.1	Ediger 2002 217-01

[1]Fludioxonil spray 0.250 kg ai post-harvest packing line spray, 29.-104 L water per 100,000 kg fruit
[2] Fludioxonil dip 0.06 kg ai/100 L water, 30 s dip

Table 81. Fludioxonil residues resulting from post-harvest application to pomegranates in California, USA.

Year (Variety)	Form	Method	Conc. kg ai/hl	Fludioxonil residue, mg/kg	Author, Date Study No. Syn No
2001 (Wonderful)	50WP	Fludioxonil dip	0.06	0.80, 0.50 (0.65)	Thompson 2003 IR4-08085
2001 (Wonderful)	50 WP	Fludioxonil dip	0.06	1.13, 0.71 (0.92)	Thompson 2003 IR4-08085

Bulb vegetables

Table 82. Fludioxonil residues in green and dry bulb onions from supervised trials in the USA, Italy, France, Germany, and Switzerland.

Country Year (variety)	Application					PHI days	Sample	Fludioxonil residue, mg/kg	Author Date Study No. Syn No
	Form	kg ai/ha	kg ai/hl	water, l/ha	No.				
Green onions									
USA, MI 1996 (Ishikura Improved)	WP 50	0.28		486	4	7	Green onions	0.17, 0.12 (0.14)	Van Geluwe 1997 ABR-97050 US 51-96
USA TX 1996 (Colossal)	WP 50	0.28		90.5	4	7	Green onions	6.6, 5.2 (0.59)	Van Geluwe 1997 ABR-97050 US 51-96
USA CA 1996 (Southport White	WP 50	0.28		468	4	0 1 3 3	Green onions	7.5, 7.1 8.0, 6.8 7.3, 5.5 6.3, 5.3	Van Geluwe 1997 ABR-97050 US 51-96

Country Year (variety)	Application					PHI days	Sample	Fludioxonil residue, mg/kg	Author Date Study No. Syn No
	Form	kg ai/ha	kg ai/hl	water, l/ha	No.				
Globe)						7 14		3.0, 2.9 (3.0) 1.9, 1.4	
Bulb onions									
USA TX 1996 (Texas Early Grand 502)	WP 50	0.28		90-99	4	7 7 7 7	Whole plant Fresh bulb Fresh bulb Dry bulb	1.2, 0.68 0.19, 0.05 0.21, 0.15 0.04, 0.03 (0.04)	Van Geluwe 1997 ABR-97050 US 51-96
USA CA 1996 (Cal Red)	WP 50	0.28		468	4	0 1 1 3 3 7 7 7 14 14 14	Whole plant Fresh bulb Dry bulb Fresh bulb Dry bulb Whole plant Fresh bulb Dry bulb Fresh bulb Fresh bulb Dry bulb	3.8, 3.3 0.06, 0.04 0.03, 0.03 0.05, 0.04 <0.02, <0.02 3.4, 2.2 0.04, 0.03 0.05, <0.02 (0.04) 0.11, <0.02 <0.02, <0.02 <0.02, <0.02	Van Geluwe 1997 ABR-97050 US 51-96
USA CA 1996 (315 Whites)	WP 50	0.28		897	4	6 6 6	Whole plant Fresh bulb Dry bulb	0.29, 0.25 0.12, 0.07 <0.02, <0.02 (<0.02)	Van Geluwe 1997 ABR-97050 US 51-96
USA CO 1996 (Vaquero)	WP 50	0.28		496	4	7	Whole plant Fresh bulb Dry bulb	0.75, 0.60 0.16, 0.09 <0.02, <0.02 (<0.02)	Van Geluwe 1997 ABR-97050 US 51-96
USA OR 1996 (Candy)	WP 50	0.28		486	4	7	Whole plant Fresh bulb Dry bulb	1.3, 0.96 0.03, 0.02 <0.02, <0.02 (0.02)	Van Geluwe 1997 ABR-97050 US 51-96
USA NY 1996 (Stulgarter Riesen)	WP 50	0.28		468	4	7	Whole plant Fresh bulb Dry bulb	0.72, 0.37 0.15, 0.14 0.11, 0.06 (0.08)	Van Geluwe 1997 ABR-97050 US 51-96
France, South 1999 (Cévennes)	WG 62.5	0.2520.26	0.063	403-423	3	0 3 7 14 21	Fresh bulb	0.06 0.03 <0.02, <0.02 (<0.02) <0.02 <0.02	Tribolet 2000 2071/99 5302
France, South 2000 (Rocodoro)	WG 62.5	0.25	0.063	400	3	0 3 7 14 21	Fresh bulb	<0.02 <0.02 <0.02, <0.02 (<0.02) <0.02 <0.02	Pointurier 2001 0010901 5391
France, South 2000 (Aldobo)	WG 62.5	0.25	0.063	400	3	0 3 7 14 21	Fresh bulb	0.05 0.02 0.02, <0.02 (0.02) <0.02 <0.02	Pointurier 2001 0010902 5392
Italy 1999 (Rojo Duro)	WG 62.5	0.25	0.025	1000	3	0 3 7 14 21	Fresh bulb	0.04 0.02 <0.02, <0.02 (<0.02) <0.02 <0.02	Tribolet 2000 2072/99 5217
Italy	WG	0.25	0.025	1000	3	7	Fresh bulb	<0.02, <0.02	Tribolet

Country Year (variety)	Application					PHI days	Sample	Fludioxonil residue, mg/kg	Author Date Study No. Syn No
	Form	kg ai/ha	kg ai/hl	water, l/ha	No.				
1999 (Rojo Duro)	62.5							(<0.02)	2000 2073/99 5218
Italy 2000 (Borrentana gialla)	WG 62.5	0.25	0.025	1000	3	0 3 7 14	Fresh bulb	0.07 0.08 0.05, 0.05 (0.05) 0.02	Salvi 2000 2032/00 5409
Italy 2000 (Borrentana gialla)	WG 62.5	0.25	0.025	1000	3	0 7	Fresh bulb	0.34 0.06, 0.06 (0.06)	Salvi 2000 2033/00 5408
Germany 1999 (Stuttgarter Reisen)	WG 62.5	0.21	0.033	620-636	3	0 7 14 21 28	Fresh bulb Or whole plant (*)	0.83* 0.07* <0.02 <0.02 <0.02	Smith 2001 gr 32899 0951
Germany 1999 (Elsa)	WG 62.5	0.20	0.033	585-621	3	0 7 13 20 27	Fresh bulb Or whole plant (*)	1.07* 0.31* <0.02 <0.02 <0.02	Smith 2001 gr 33999 0952
Germany 1999 (Stuttgarter Reisen)	WG 62.5	0.20-0.22	0.033-0.033	613-650	3	15	Fresh bulb	<0.02	Smith 2001 gr 34899 0953
Germany 2000 (Hilton)	WG 62.5	0.25	0.042	600	3	0 7 14 14 20 27	Fresh bulb Or whole plant (*)	1.4* 0.36* 0.02 0.17* <0.02 <0.02	Simon 2001 gr 34200 5419
Germany 2000 (Stuttgarter Reisen)	WG 62.5	0.27-0.28	0.042	647-660	3	0 7 14 14 21 28	Fresh bulb Or whole plant (*)	1.9* 0.27* <0.02 0.15* <0.02 <0.02	Simon 2001 gr 35800 5420
Switzerland 2000 (Copra)	WG 62.5	0.25	0.033	800	3	14	Fresh bulb	<0.02, <0.02	Salvi 2001 2010/00 5410
Switzerland 2000 (Redwing)	WG 62.5	0.25	0.033	800	3	14	Fresh bulb	<0.02, <0.02	Salvi 2009/00 2001 5411

Brassica vegetables

Table 83. Fludioxonil residues in broccoli from supervised trials in the USA and Canada.

Country Year (variety)	Application					PHI days	Fludioxonil residue, mg/kg	Author Date Study No. Syn No
	Form	kg ai/ha	kg ai/hl	water, l/ha	No.			
USA, Texas 2000 (Baccus hybrid)	WG 62.5	0.245	0.054	449-456	4	6	0.10, 0.04 (0.07)	Arsenovic 2002 A-27200-63-02

Country Year (variety)	Application					PHI days	Fludioxonil residue, mg/kg	Author Date Study No. Syn No
	Form	kg ai/ha	kg ai/hl	water, l/ha	No.			
USA, California 2000 (Patriot)	WG 62.5	0.243-0.255	0.041	477-709	4	7	0.11, 0.09 (0.10)	Arsenovic 2002 A-27200-63-02
USA, California 2000 (Marathon)	WG 62.5	0.243-0.253	0.041	477-717	4	8	0.25, 0.21 (0.23)	Arsenovic 2002 A-27200-63-02
USA, California 2000 (Greenbelt)	WG 62.5	0.245	0.047	512-522	4	6	0.27, 0.25 (0.26)	Arsenovic 2002 A-27200-63-02
USA, California 2000 (Green Sprouting)	WG 62.5	0.243-0.250	0.064	371-383	4	8	0.20, 0.15 (0.18)	Arsenovic 2002 A-27200-63-02
Canada, British Colombia 2000 (Greenbelt)	WG 62.5	0.245	0.070	348-355	4	7	0.53, 0.19 (0.36)	Arsenovic 2002 A-27200-63-02
Canada, Quebec 2000 (Marathon)	WG 62.5	0.245	0.069	355	6	7	0.36, 0.31 (0.34)	Arsenovic 2002 A-27200-63-02

Table 84: Fludioxonil residues in cabbage from supervised trials in the USA.

Location Year (variety)	Application					PHI days	Fludioxonil residue, mg/kg	Author Date Study No. Syn No
	Form	kg ai/ha	kg ai/hl	water, l/ha	No.			
ME 04 2000 (Danish Ballhead)	WG 62.5	0.230-0.245	0.079	309-315	4	7	0.27[1], 0.23[2]	Arsenovic 2002 A-27200-62-02
TX 33 2000 (Cheers)	WG 62.5	0.245	0.061	392-416	4	7	0.21[1], 0.20[2]	Arsenovic 2002 A-27200-62-02
FL 48 2000 (Asgrow Blue Dynasty)	WG 62.5	0.245-0.253	0.066	374	6	8	1.20[1],0.09[2]	Arsenovic 2002 A-27200-62-02
TX 34 2000 (Cheers)	WG 62.5	0.245	0.054	449-457	4	7	0.50[1], 0.08[2]	Arsenovic 2002 A-27200-62-02
CA 15 2000 (Red Rookie)	WG 62.5	0.240-0.255	0.042	501-717	4	7	0.17[1], 0.03[2]	Arsenovic 2002 A-27200-62-02
WI 03 2000 (Blue Thunder)	WG 62.5	0.379-0.412	0.130	303-314	4	6	0.17[1], 0.17[2]	Arsenovic 2002 A-27200-62-02

[1] wrapper leaves retained
[2] wrapper leaves removed

Fruiting vegetables, cucurbits

Table 85. Fludioxonil residues in cucumbers from supervised trials in Greece, Spain and Switzerland.

Country Year (variety)	Application					PHI days	Fludioxonil residue, mg/kg	Author Date Study No. Syn No
	Form.	kg ai/ha	kg ai/hl	water, l/ha	No.			
GLASSHOUSE (INDOOR)								
Spain 1994 (Nevada)	WG 62.5	0.25	0.020	1250	3	0 3 7 14	0.34 0.27 0.14 0.04	Mair 1996 2173/93 0789
Spain 1996 (Regal)	WG 62.5	0.25	0.015 - 0.022	1125 - 1625	3	0B* 0 3 7 14	0.03 0.18 0.09 0.07 0.02	Walser 1996 2005/96 0832
Spain 1999 (Albatros)	WG 62.5	0.24	0.025	981 - 983	3	0 3 7 14	0.12 0.09 0.08, 0.07 (0.08) 0.03	Tribolet 2000 2166/99 5219
Spain 1999 (Toril)	WG 62.5	0.24- 0.24	0.026	978 - 985	3	0 3 7 14	0.12 0.09 0.07, 0.05 (0.06) 0.03	Tribolet 2000 2167/99 5220
Greece 1996 (Biri)	WG 62.5	0.25	0.016	1600	3	0B* 0 3 7 14	0.02 0.28 0.17 0.08 0.05	Walser 1997 2101/96 1034
Spain 2000 (Rayo)	WG 62.5	0.25 - 0.27	0.024	1045 - 1115	3	0 7	0.08 0.03, 0.02 (0.02)	Salvi 2001 2031/00 5399
Spain 2000 (Toril)	WG 62.5	0.22 - 0.25	0.025	867 - 999	3	0 7	0.06 0.03, 0.02 (0.02)	Salvi 2001 2030/00 5400
Spain 2001 (Bala)	WG 62.5	0.25	0.025	1012	3	0 7	0.16 0.17, 0.05 (0.11)	Ryan 2002 2057/01 5558
Switzerland 1995 (Thyria F1)	WG 62.5	0.25	0.017	1500	3	0 7 14 21	0.09 <0.02 <0.02 <0.02	Walser 1996 2053/95 0664
Switzerland 1995 (Thyria F1)	WG 62.5	0.25	0.017	1500	3	0 7 14 21	0.17 0.06 <0.02 <0.02	Walser 1996 2054/95 0715
FIELD (OUTDOOR)								
Greece 1995 (Danimas)	WG 62.5	0.25	0.025 - 0.028	880 - 1000	3	0B* 0 3 7 14	0.02 0.60 0.08 0.02 <0.02	Walser 1996 2024/95 0725
Spain 1995 (Peto 025)	WG 62.5	0.25	0.017	1500	3	0 3 7 14	0.10 0.04 <0.02 <0.02	Walser 1996 2014/95 0710

Country Year (variety)	Application					PHI days	Fludioxonil residue, mg/kg	Author Date Study No. Syn No
	Form.	kg ai/ha	kg ai/hl	water, l/ha	No.			
Spain 1995 (Bellissima)	WG 62.5	0.25	0.017	1500	3	0 3 7 14	0.20 0.11 0.03 <0.02	Walser 1996 2184/95 0762

*0B: before final application

Table 86. Fludioxonil residues in summer squash (zucchini) from supervised indoor trials in Italy.

Year (variety)	Application					PHI days	Fludioxonil residue, mg/kg	Author Date Study No. Syn No
	Form.	kg ai/ha	kg ai/hl	water, l/ha	No.			
1995 (GB 68)	WG 62.5	0.25	0.025	1000	3	0 7 14 21	1.4 0.06 <0.02 <0.02	Walser 1995 2089/95 0670
1996 (GB 68)	WG 62.5	0.25	0.025	1000	3	0B* 0 7 14	0.03 0.56 0.05 <0.02	Walser 1997 2057/96 0961

* 0B: before final application

Table 87. Fludioxonil residues in cantaloupe melon after drip irrigation treatment from supervised trials in the USA.

Location Year (variety)	Application			PHI Days	Fludioxonil residue, mg/kg	Author, Date Study No. Syn No
	Form.	kg ai/ha	No.			
Texas 2000 (Hy-Mark)	50 WP	0.28	3	14 (±2)	<0.02, <0.02 <0.02, <0.02 <0.02, <0.02	Thompson & Ediger 02/06/2003 IR-4: 07618 38-00
California 2000 (Hale's Best Jumbo)	50 WP	0.28	3	14 (±2)	0.03, <0.02 <0.02, <0.02 <0.02, <0.02	Thompson & Ediger 02/06/2003 IR-4: 07618 38-00
Texas 2001 (Explorer)	50 WP	0.28	3	14 (±2)	<0.02, <0.02	Thompson & Ediger 02/06/2003 IR-4: 07618 38-00
California 2001 (Hale's Best Jumbo)	50 WP	0.28	3	14 (±2)	0.02, <0.02	Thompson & Ediger 02/06/2003 IR-4: 07618 38-00

Fruiting vegetables, other than cucurbits

Table 88. Fludioxonil residues in tomatoes from supervised trials in Greece, Switzerland, Spain, the UK and Italy.

Country Year (variety)	Application					PHI days	Residue, mg/kg	Author Date Study No. Syn No
	Form	kg ai/ha	kg ai/hl	water, l/ha	No.			
Indoor/Covered								
Greece 1005 (Alpado)	WG 62.5	0.25	0.025	1000	3	0B* 0 3 7 14	0.14 0.32 0.24 <u>0.32</u> 0.24	Walser 1996 2023/95 0763
Greece 1996 (Optima)	WG 62.5	0.25	0.012 -0.013	2000	3	0B* 0 3 7 14	0.10 0.14 0.16 <u>0.09</u> 0.13	Walser 1997 2100/96 1033
Switzerland 1994 (Savor)	WG 62.5	0.25	0.013	2000	3	0 3 7 14	0.20 0.13 <u>0.28</u> 0.19	Kissling 1995 2060/94 1102
Switzerland 1994 (Merano)	WG 62.5	0.25	0.013	2000	3	0 3 7 14	0.14 0.13 0.08 <u>0.10</u>	Kissling 1995 2061/94 1103
Switzerland 1997 (Cannelli)	WG 62.5	0.25	0.013	2000	3	0B* 0 3 7 14	0.06 0.19 0.15 0.10 <u>0.14</u>	Walser 1998 2265/97 1161
Spain 1993 (Daniela)	WG 62.5	0.25	0.017	1500	3	0 3 7 14	0.07 0.08 <u>0.08</u> 0.03	Mair 1994 2171/93-94 1104
Spain 1994 (Santos)	WG 62.5	0.25	0.017	1500	3	0 3 7 14	0.13 0.16 0.13 <u>0.15</u>	Mair 1994 2171/93-94 1104
Spain 1994 (Daniella)	WG 62.5	0.25	0.017 - 0.023	1111 – 1481	3	0 3 7 14	0.07 0.06 <u>0.10</u> 0.05	Mair 1994 2172/93-94 1105
Spain 1994 (Daniella)	WG 62.5	0.25	0.023	1111	3	0 3 7 14	0.14 0.09 0.14 <u>0.23</u>	Mair 1994 2172/93-94 1105
UK 1997 (Durinda)	WG 62.5	0.25	0.020 - 0.023	1092 - 1249	3	0B* 0 3 7 14	0.03 0.02 0.03 0.03 <u>0.05</u>	Walser 1998 2451/97 1217
UK 1997 (Durinda)	WG 62.5	0.25	0.020 - 0.023	1102 - 1250	3	0B* 0 3 7 14	0.08 0.03 0.05 0.05 <u>0.09</u>	Walser 1998 2450/97 1218

Country Year (variety)	Application					PHI days	Residue, mg/kg	Author Date Study No. Syn No
	Form	kg ai/ha	kg ai/hl	water, l/ha	No.			
Italy 1995 (Gincala)	WG 62.5	0.25	0.015 - 0.021	1200 - 1800	3	0 7 10 14 21 28	0.17 0.16 0.15 0.14 <u>0.21</u> 0.11	Walser 1995 2092/95 0667
Italy 1995 (114 (pre-coder))	WG 62.5	0.25	0.025	1000	3	0 7 14 21	0.19 <u>0.13</u> 0.08 0.04	Walser 1995 2090/95 0668
Italy 1995 (Max)	WG 62.5	0.25	0.017- 0.025	1000 - 1500	3	0 7 14 21	0.09 <u>0.21</u> 0.07 0.05	Walser 1995 2091/95 0669
Outdoor								
Switzerland 1997 (Selhardy)	WG 62.5	0.25	0.013	2000	3	0B* 0 3 7 14	0.06 0.17 0.07 <u>0.07</u> 0.03	Walser 1998 2264/97 1160
Switzerland 1999 (Petula)	WG 62.5	0.25	0.017	1500	3	7	0.05, 0.04 (<u>0.04</u>)	Tribolet 2000 2126/99 5345

*0B: before final treatment

Table 89. Fludioxonil residues in sweet (bell) peppers from supervised trials in Italy, Spain, and Switzerland.

Country, Year (variety)	Application					PHI days	Fludioxonil residue, mg/kg	Author Date Study No. Syn No
	Form.	kg ai/ha	kg ai/hl	water, l/ha	No.			
Glasshouse (indoor)								
Spain 1996 (Estar)	WG 62.5	0.25	0.020 - 0.025	1000 - 1250	3	0B* 0 3 7 14	0.62 1.04 0.93 <u>0.56</u> 0.38	Walser 1996 2001/96 0791
Spain 1996 (Tanger)	WG 62.5	0.25	0.020 - 0.022	1125 - 1250	3	7	0.47, 0.46 <u>(0.46)</u>	Walser 1996 2002/96 0792
Spain 1996 (Saxo)	WG 62.5	0.25	0.025	1000	3	0B* 0 3 7 14	0.08 0.22 0.26 <u>0.22</u> 0.13	Walser 1996 2006/96 0833
Spain 1997 (Estar)	WG 62.5	0.25	0.035	714	3	0B* 0 3 7 14	0.03 0.30 0.35 <u>0.14</u> 0.14	Walser 1997 2057/97 1041
Spain 1997 (Italico)	WG 62.5	0.25 - 0.2	0.017 - 0.026	981 - 1555	3	0B* 0 3 7 14	0.08 0.67 0.36 <u>0.29</u> 0.16	Walser 1998 2058/97 1131

Country, Year (variety)	Application					PHI days	Fludioxonil residue, mg/kg	Author Date Study No. Syn No
	Form.	kg ai/ha	kg ai/hl	water, l/ha	No.			
Spain 1997 (Italico)	WG 62.5	0.25 - 0.26	0.018	1427 - 1450	3	0B* 0 3 7 14	0.38 1.16 0.78 0.60 0.23	Walser 1998 2059/97 1132
Switzerland 2002 (Evident)	WG 62.5	0.23 - 0.24	0.025	933 - 944	3	0 1 4 8 14	0.23 0.21 0.17 0.09, 0.06 (0.08) 0.03	Solé 2003 02-2006 5564
Switzerland 2002 (Evident)	WG 62.5	0.24	0.025	937 - 980	3	0 1 3 7 14	0.21. 0.26 0.19 0.21 0.12, 0.07 (0.10) 0.09	Solé 2003 02-2007 5565
Outdoor								
Italy 1996 (Phatos)	WG 62.5	0.25	0.025	1000	3	0B* 0 7 14	0.08 0.30 0.06 0.04	Walser 1997 2060/96 0960
Spain 1997 (Italico)	WG 62.5	0.24 - 0.26	0.016 - 0.024	1021 - 1573	3	0B* 0 3 7 14	0.03 0.18 0.19 0.13 0.09	Walser 1998 2370/97 1144

*0B: before final application

Table 90. Fludioxonil residues in egg plants (aubergines) from supervised indoor trials in Italy and Spain.

Country Year (variety)	Application					PHI days	Fludioxonil residue, mg/kg	Author Date Study No. Syn No
	Form.	kg ai/ha	kg ai/hl	water, l/ha	No.			
Italy 1997 (Violetta)	WG 62.5	0.25	0.025	1000	3	0B* 0 3 7 14	0.02 0.05 0.05 0.03 <0.02	Walser 1998 2073/97 1145
Italy 1997 (Napoletana)	WG 62.5	0.25	0.028 - 0.031	800 - 900	3	0B* 0 3 7 14	<0.02 0.15 0.13 0.06 0.03	Tribolet 1998 2074/97 1146
Spain 1999 (Cava)	WG 62.5	0.24 0.25	0.025	941 - 1000	3	0 7	0.14, 0.20 0.07, 0.08 (0.08)	Tribolet 2000 2014/99 5088
Spain 1999 (Cava F1)	WG 62.5	0.25	0.025	988 - 1000	3	0 7	0.12, 0.14 0.06, 0.07 (0.06)	Tribolet 2000 2013/99 5089

*0B: before final application

Table 91. Fludioxonil residues in sweet corn from seed treatment in the USA.

Location Year (Variety)	Form	g ai/ 100 kg seed	Sample	PHI	Fludioxonil residue mg/kg	Author Date Study No. Syn No
FL 1992 Sweet corn (Revere)	4FS	15	Ears	82	0.01[1], <0.01[1], <0.01[1] <0.01[2] (<0.01)	Selman 1993 ABR-93030 31-92
WA 1992 Sweet corn (Terminator)	4FS	25	Ears Whole corn Kernel, cleaned Cannery waste Corn, canned	98 98 98 98 98	<0.01, <0.01 <0.01 <0.01 <0.01 <0.01	Selman 1993 ABR-93030 31-92
WI 1992 Sweet corn (Terminator)	4FS	25	Ears	97	<0.01, <0.01	Selman 1993 ABR-93030 31-92

[1] replicate determinations of sample 1
[2] determination of sample 2

Leafy vegetables

Table 92. Fludioxonil residues in head lettuce from supervised glasshouse and field trials in Europe.

Country Year (variety)	Application					PHI days	Fludioxonil residue, mg/kg	Author, Date Study No. Syn No
	Form	kg ai/ha	kg ai/hl	water, l/ha	No.			
Indoor (glasshouse)								
Italy 2001 (Manita RZ, Head)	WG 62.5	0.18	0.018	1000	3	0 3 7 14	7.2 5.1 4.3, 3.2 2.5, 2.3 (2.4)	Ryan 2002 2044/01 5509
Italy 2001 (Manita, Head)	WG 62.5	0.18	0.018	1000	3	0 3 7 14	9.6 8.2 4.3, 3.7 2.6, 2.4 (2.5)	Ryan 2002 2043/01 5510
Italy 2001 (Mindoro, Head)	WG 62.5	0.18	0.018	1000 - 1024	3	0 7 14	3.6 1.8, 1.7 1.2, 0.99 (1.1)	Ryan 2002 2042/01 5516
France, South 1997 (Nalys, Head)	WG 62.5	0.15	0.038	400	4	0B* 0 7 14 21	0.84 8.4 7.4 2.7 2.5	Maffezzoni 1999 9810203 4992
France, South 1998 (Mexico)	WG 62.5	0.15	0.038	400	3	14	2.88, 2.57 (2.7)	Maffezzoni 1999 9810204 4993
France, South 1997 (Samourai, Head)	WG 62.5	0.15	0.038	399-413	3	14	3.67, 3.23 (3.4)	Maffezzoni 1999 9810205 4994

Country Year (variety)	Application					PHI days	Fludioxonil residue, mg/kg	Author, Date Study No. Syn No
	Form	kg ai/ha	kg ai/hl	water, l/ha	No.			
France, South 1998 (Cybelle, Head)	WG 62.5	0.15 – 0.16	0.038	399-413	3	14	0.73, 0.70 (0.72)	Maffezzoni 1999 9810206 4995
France, North 1997 (Angie, Head)	WG 62.5	0.15	0.038	400	3	0B* 0 7 14 21	3.9 17. 8.6 4.7 4.2	Maffezzoni 1999 9810201 4990
France, North 1997 (Sensai, Head)	WG 62.5	0.15	0.038	400	3	14	5.96, 5.94 (6.0)	Maffezzoni 1999 9810202 4991
Germany 2001 (Sensai, Head)	WG 62.5	0.15	0.025	600	3	0 7 9 15 20	3.3 1.7 1.2 1.0, 0.97 (0.98) 0.82	Simon 2002 gle 52501 5537
France, North 1999 (Angie, Head)	WG 62.5	0.15	0.038	400	3	14	3.6, 3.1 (3.4)	Tribolet 2000 2168/99 5347
Outdoor (Field)								
France, North 1996 (Newton)	WG 62.6	0.18	0.044	400	3	OB 0 7 14 21	0.30 7.25 1.68 0.17 0.04	Maffezzoni 1998 OF96103/K J85 1047
France, North 1996 (Marianna)	WG 62.5	0.18	0.044	400	3	12	0.04, 0.05 (0.04)	Maffezzoni 1998 OF96103/S J15 1048
France, North 1997 (Audran)	WG 62.5	0.18	0.045 – 0.047	370 – 385	3	0B 0 8 15 22	0.19 6.21 0.16 0.02 <0.02	Maffezzoni 1998 9713001 1074
France, North 1997 (Newton)	WG 62.5	0.18	0.044	400	3	0B* 0 7 15	0.39 3.27 0.68 0.11	Maffezzoni 1998 9713002 1075
France, North 1997 (Aurica)	WG 62.5	0.18	0.044	400	3	14	<0.02, <0.02 (<0.02)	Maffezzoni 1998 9713101 1076
France, North 1997 (Floreal)	WG 62.5	0.18	0.044	400	3	14	0.04, 0.04 (0.04)	Maffezzoni 1998 9713102 1077
Switzerland 1997 (Newton)	WG 62.5	0.20	0.040	500	3	0B* 0 7 14 21 28	0.05 7.06 0.64 0.02 <0.02 <0.02	Walser 1998 2268/97 1157

Country Year (variety)	Application					PHI days	Fludioxonil residue, mg/kg	Author, Date Study No. Syn No
	Form	kg ai/ha	kg ai/hl	water, l/ha	No.			
Switzerland 1997 (Stephanie)	WG 62.6	0.200	0.040	500	3	0B* 0 7 14 21 28	0.08 3.79 0.18 <u><0.02</u> <0.02 <0.02	Walser 1998 2267/97 1158
Switzerland 1997 (Larand)	WG 62.5	0.20	0.040	500	3	0B* 0 7 14 21 28	0.08 3.77 0.17 <u>0.07</u> <0.02 <0.02	Walser 1998 2266/97 1159
France, South 1996 (Batavia Rosia)	WG 62.5	0.18	0.044	400	3	14	0.04, 0.04 <u>(0.04)</u>	Maffezzoni 1998 OF96103/FP04 1049
France, South 1996 (Batavia Carmen)	WG 62.5	0.18	0.044	400	3	14	<0.02, <0.02 <u>(<0.02)</u>	Maffezzoni 1998 OF96103/AC14 1050
Spain 1996 (Romana, Cos)	WG 62.5	0.18	0.025	700	3	0B 0 3 7 14	0.86 3.5 2.9 2.3 <u>1.2</u>	Walser 1996 2003/96 0793
Spain 1996 (Valladolid, Cos)	WG 62.5	0.18	0.025	700	3	14	1.20, 1.15 <u>(1.2)</u>	Walser 1996 2004/96 0794
Spain 1997 (Iceberg, Cos)	WG 62.5	0.18	0.035	500	3	0B* 0 7 14 21	0.04 4.34 <0.02 <u><0.02</u> <0.02	Walser 1998 2056/97 1079
Italy 1996 (Justine, Head)	WG 62.5	0.18	0.029	600	3	14	0.33, 0.24 <u>(0.29)</u>	Walser 1996 2062/96 0974
Italy 1996 (Lido, Head)	WG 62.5	0.18	0.029	600	3	0B* 0 7 14 21	0.12 5.81 0.80 <u><0.02</u> <0.02	Walser 1997 2061/96 0991
Italy 1997 (Justine, Head)	WG 62.5	0.18	0.029	600	3	0B* 0 7 14 21	0.20 4.26 0.09 <u><0.02</u> <0.02	Walser 1998 2067/97 1113

* 0B: before final application

Table 93. Residues of fludioxonil in watercress from supervised trials in Florida, USA.

Location Year (variety)	Application					PHI days	Fludioxonil residue, mg/kg	Author, Date Study No. Syn No
	Form	kg ai/ha	kg ai/hl	Water, l/ha	No.			
1999 (B&W New Improved Variety 3)	WG 62.5	0.24	0.048 - 0.051	477 - 506	4	0	5.01, 3.99 (4.5)	Starner 2001 IR-4 06759 1269-99
1999 (B&W Standard Variety 1)	WG 62.5	0.24	0.035	702	4	0	4.79, 3.69 (4.2)	Starner 2001 IR-4 06759 1269-99

Table 94. Residues of fludioxonil in mustard greens from supervised trials in the USA.

Location Year (variety)	Application					PHI days	Fludioxonil residue, mg/kg	Author Date Study No. Syn No
	Form	kg ai/ha	kg ai/hl	Water, l/ha	No.			
NJ 2000 (Southern Giant Curled)	WG 62.5	0.24	0.064	374 - 387	4	8	7.7, 6.5 (7.1)	Arsenovic, Moore 2002 IR-4 07622 US 29147
TN 2000 (Southern Giant Curled)	WG 62.5	0.24	0.074	327 - 338	4	7	0.64, 0.45 (0.54)	Arsenovic, Moore 2002 IR-4 07622 US 29147
FL 2000 (Florida Broadleaf)	WG 62.5	0.24	0.065	370 - 386	4	7	6.9, 6.4 (6.6)	Arsenovic, Moore 2002 IR-4 07622 US 29147
NC 2000 (Southern Giant Curled)	WG 62.5	0.24	0.080	305 - 311	4	7	0.06, 0.06 (0.06)	Arsenovic, Moore 2002 IR-4 07622 US 29147
TX 2000 (India)	WG 62.5	0.24	0.054	450 - 455	4	6	0.50, 0.48 (0.49)	Arsenovic, Moore 2002 IR-4 07622 US 29147
CA 2000 (Southern Giant Curled)	WG 62.5	0.24	0.035 - 0.050	494 - 695	4	7	1.2, 0.33 (0.76)	Arsenovic, Moore 2002 IR-4 07622 US 29147
MI 2000 (Southern Giant Curled)	WG 62.5	0.39	0.105	367 - 383	4	7	1.3, 1.1 (1.2)	Arsenovic, Moore 2002 IR-4 07622 US 29147

Legume vegetables

Table 95. Fludioxonil residues in bean pods with seed from foliar applications in Europe.

Country Year (variety)	Application					PHI days	Fludioxonil residue, mg/kg	Author, Date Study No. Syn No
	Form	kg ai/ha	kg ai/hl	water, l/ha	No.			
Outdoor								
France, South 1998 French beans (Ardinal)	WG 62.5	0.23 - 0.25	0.063	375-400	2	14	0.08, 0.08 (0.06)	Maffezzoni 1999 9812803 1330
France, South 1998 French beans (Longio)	WG 62.5	0.26 - 0.27	0.063	418-426	2	14	0.04, 0.04 (0.04)	Maffezzoni 1999 9812804 1331
France, South 1998 French beans (Crugaly)	WG 62.5	0.25	0.063	394-406	2	0 3 7 14 21	0.31 0.25 0.19 0.03 0.02	Maffezzoni 1998 9812703 1335
France, South 1998 French beans (Xéra)	WG 62.5	0.26	0.063	415-419	2	0 3 7 14 21	0.44 0.21 0.08 0.02 0.02	Maffezzoni 1998 9812704 1336
France, North 1998 French beans (Booster)	WG 62.6	0.25	0.063	395-400	2	14	0.03, 0.03 (0.03)	Maffezzoni 1999 9812801 1328
France, North 1998 French beans (Longio)	WG 62.5	0.24- 0.25	0.063	377-407	2	14	0.04, 0.04 (0.04)	Maffezzoni 1999 9812802 1329
France, North 1998 French beans (Capitole)	WG 62.5	0.24- 0.25	0.063	382-405	2	0 3 7 14 21	0.43 0.19 0.08 0.03 0.02	Maffezzoni 1998 9812701 1333
France, North 1998 French beans (Longio)	WG 62.5	0.23– 0.2	0.063	367-388	2	0 3 7 14 21	0.30 0.12 0.08 0.06 0.03	Maffezzoni 1998 9812702 1334
Switzerland 1998 Common beans (Processor)	WG 62.5	0.25	0.031	800	2	14	0.03, 0.03 (0.03)	Walser 1999 2107/98 4952
Switzerland 1998 Common beans (Sonate)	WG 62.5	0.25	0.031	800	2	14	0.06, 0.05 (0.06)	Walser 1999 2109/98 4954
France, South 1996 French beans (Adagio)	WG 62.5	0.25	0.063	400	3	14	0.02, 0.02 (0.02)	Maffezzoni 1998 OF96108/LD63 1051
France, South 1996 French beans (Cupidon)	WG 62.5	0.25	0.063	400	3	14	0.03, 0.03 (0.03)	Maffezzoni 1998 OF96108/FP15 1053

Country Year (variety)	Application					PHI days	Fludioxonil residue, mg/kg	Author, Date Study No. Syn No
	Form	kg ai/ha	kg ai/hl	water, l/ha	No.			
France, North 1996 French beans (Xavo)	WG 62.5	0.25	0.063	400	3	13	<0.02, <0.02 (<0.02)	Maffezzoni 1998 OF96108/SJ29 1052
France, North 1996 Fench beans (Calypso)	WG 62.5	0.25	0.063	400	3	0B* 0 7 14 21	0.18 0.64 0.40 0.13 0.12	Maffezzoni 1998 OF96109/KJ56 1054
Spain 1996 Common beans (Buenos Aires)	WG 62.5	-0.25	0.013	1978 - 2000	3	0B* 0 7 14 21	0.07 0.64 0.17 0.09 0.03	Walser 1997 2009/96 0883
Indoor (glasshouse)								
Spain 1996 Common beans (Maite)	WG 62.5	0.25	0.017 - 0.020	1250 - 1500	3	0B* 0 7 14 21	0.11 0.64 0.13 0.03 <0.02	Walser 1996 2010/96 0834
Spain 1997 Common beans (Helda)	WG 62.5	0.25	0.025	1000	3	0B* 0 7 14 21	0.12 0.85 0.21 0.04 <0.02	Walser 1997 2002/97 1039
Spain 1997 Common beans (Maite)	WG 62.5	0.25	0.024 - 0.026	952 - 1060	3	0B* 0 7 14 21	0.05 0.92 0.28 0.06 <0.02	Walser 1997 2001/97 1040
Spain 1997 Common beans (Musica)	WG 62.5	0.24 – 0.26	0.017 - 0.022	1146 - 1437	3	0B* 0 7 14 21	<0.02 0.41 0.14 0.04, 0.04 (0.04) 0.08, 0.09	Walser 1998 157/97 1152
Spain 1997 Common beans (Musica)	WG 62.5	0.231– 0.26	0.021 - 0.025	939 - 1213	3	0B* 0 7 14 21	0.03 0.53 0.23 0.14 0.17	Walser 1998 157/97 1152
Spain 1996 Common beans (Encañe Dulce)	WG 62.5	0.22 - 0.25	0.026 - 0.036	637 – 876	3	0B* 0 7 14 21	0.11 0.43 0.44 0.09 <0.02	Walser 1998 157/97 1152
Spain 1997 Common beans (Emerite)	WG 62.5	0.25 - 0.26	0.024 - 0.025	1035 - 1064	3	0B* 0 7 14 21	0.12 0.33 0.34 0.20 0.18	Walser 1998 157/97 1152

*0B: before final application

Table 96. Fludioxonil residues in bean pods with seed and in seeds from supervised trials in Germany and Denmark (fludioxonil-treated seed).

Country Year (variety)	Application		DAS[1] (days)	Analysis			Author, Date Study No. Syn No
	Form.	g ai/ 100kg seed		Sample	BBCH Stage	Fludioxonil residue, mg/kg	
Germany 1997 French beans (Ferande)	FS 035 (A 9638A)[2]	5.0	53 77 159	Whole plant Unripe pods with seed Beans (seed)	50-52 72-73 89	<0.02 <0.02 <0.02	Smith 1999 GR 39197 1339
Germany 1997 French Beans (Ferande)	FS 035 (A 9638A)	5.0	53 75 133	Whole plant Unripe pods with seed Beans (seed)	51 73 89	<0.02 <0.02 <0.02	Smith 1999 GR 40497 1352
Denmark 1997 French beans (Bon-bon)	FS 035 (A 9638A)	4.5	49 66 113	Whole plant Unripe pods with seed Seed (dry)	50-55 71-74 99	<0.02 <0.02 <0.02	Kuhne 1999 2274/97 1367
Germany 1997 Broad beans (Scirocco)	FS 035 (A 9638A)	4.4	65 139 139	Whole plant Straw (remainder) Beans (seed)	50-52 89 89	0.06 <0.02 <0.02	Smith 1999 GR 37197 1337
Germany 1997 Broad beans (Scirocco)	FS 035 (A 9638A)	4.4	57 154 154	Whole plant Straw (remainder) Beans (seed)	51 89 89	<0.02 <0.02 <0.02	Smith 1999 GR 38497 1338
Denmark 1997 Broad beans (Cargo)	FS 035 (A 9638A)	3.3	41 113 113	Whole plant Vines (haulms) Seed (dry)	50-57 99 99	<0.02 <0.02 <0.02	Kuhne 1999 2275/97 1368

[1]days after sowing
[2]Test product designation.

Table 97. Fludioxonil residues in fresh peas from supervised outdoor trials in France and Switzerland (foliar treatment).

Fresh seed Country, year (variety)	Application				No.	BBCH stage at harvest	PHI days	Fludioxonil residue, mg/kg	Author, Date Study No. Syn No
	Form	kg ai/ha	kg ai/hl	water, l/ha					
Switzerland 1998 (Merveille Hative)	WG 62.5	0.25	0.031	800	2	79	14	Empty pod: 0.06, <0.05 Fresh seed: <0.02, <0.02	Walser 1999 2108/98 4953
Switzerland 1998 (Bördi)	WG 62.5	0.25	0.031	800	2	79	14	Empty pod: <0.05, <0.05 Fresh seed: <0.02, <0.02	Walser 1999 2115/98 4955
France, South 1998 (Cador)	WG 62.5	0.25	0.063	400	2	 79/80	0 7 14 20	0.03 <0.02 <0.02 <0.02	Maffezzoni 1998 9811501 1332
France, North 1998 (Koka)	WG 62.5	0.25	0.063	400	2	79	14	<0.02, <0.02	Maffezzoni 1998 9811601 1285
France, North 1998 (Etna)	WG 62.5	0.25	0.063	400	2	76/79	14	<0.02, <0.02	Maffezzoni 1998 9811602 1286

Fresh seed Country, year (variety)	Application				No.	BBCH stage at harvest	PHI days	Fludioxonil residue, mg/kg	Author, Date Study No. Syn No
	Form	kg ai/ha	kg ai/hl	water, l/ha					
France, North 1998 (Koka)	WG 62.5	0.25	0.063	400	2	76/77	14	<0.02, <0.02	Maffezzoni 1998 9811603 1287
France, North 1998 (Koka)	WG 62.5	0.25	0.063	400	2	76/77	14	<0.02, <0.02	Maffezzoni 1998 9811604 1288
France, South 1998 (Piano)	WG 62.5	0.25	0.063	400	2	77/79	14	<0.02, <0.02	Maffezzoni 1998 9811605 1289
France, North 2001 (Frediro)	WG 62.5	0.25	0.05	500	2	79	14	Empty pod 0.06, 0.05 Seeds <0.02, <0.02 Whole pods 0.02, 0.02	Pointurier 2002 0112201 5520
France, North 1996 (Fonado)	WG 62.5	0.25	0.060 - 0.063	400 - 417	3	74	14	0.02	Pointurier 1996 OF96105/D E18 0870
France, North 1996 (Fonado)	WG 62.5	0.25	0.060 - 0.063	400 - 417	3	74	14	<0.02	Pointurier 1996 OF96106/D E19 0871

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Table 98. Fludioxonil residues in pea without pod harvested from supervised trials in Northern France and UK (treated seed).

Fresh seeds Country Year (variety)	Application		DAS[1] (days)	Analysis			Author Date Study No. Syn No
	Form.	g ai/ 100kg seed		Sample	BBCH stage at harvest	fludioxonil residue, mg/kg	
France, North 1997 (Avola)	FS 260	11.	46 62 62	Whole plant Empty pod Seed	51 73 73	<0.02 <0.02 <0.02	Kühne 1998 2296/97 1259
France, North 1997 (Avola)	FS 260	11.	35 63 63	Whole plant Empty pod Seed	51 75/76 75/76	<0.02 <0.02 <0.02	Kühne 1998 2297/97 1260
UK 1997 (Avola)	FS 260	10.	81 81	Empty pod Seed	75-76 75-76	<0.05 <0.02	Kühne 1998 2298/97 1261
UK 1997 (Avola)	FS 260	11.	80 80	Empty pod Seed	75-76 75-76	<0.05 <0.02	Kühne 1998 2299/97 1262
UK 1998 (Avola)	WG 32.5	9.8	60 94 94	Whole plant Empty pods Seed	51 76 76	<0.05 <0.05 <0.02	Kühne 1999 2008/98 4968

Fresh seeds Country Year (variety)	Application		DAS[1] (days)	Analysis			Author Date Study No. Syn No
	Form.	g ai/ 100kg seed		Sample	BBCH stage at harvest	fludioxonil residue, mg/kg	
UK 1998 (Avola)	WG 32.5	9.1	47 71 71	Whole plant Empty pods Seed	51 76 76	<0.05 <0.05 <u><0.02</u>	Kühne 1999 2009/98 4969

[1]days after sowing

Table 99. Fludioxonil residues in dry pea seed and kidney beans from supervised outdoor trials in France (foliar treatment).

CROP Location , (variety)	Application				No.	BBCH stage at harvest	PHI days	Fludioxonil residue, mg/kg	Author Date Study No. Syn No
	Form	kg ai/ha	kg ai/hl	water, l/ha					
Pea Dry Seed									
South 1996 (Caprice)	WG 62.5	0.25	0.063	400	3	87	13	<u><0.02</u>	Pointurier 1996 OF96107/AC31 0872
South 1996 (Caprice)	WG 62.5	0.25	0.063	400	3	89	14	<u><0.02</u>	Pointurier 1996 OF96107/LD64 0873
North 2001 (Bonette)	WG 62.5	0.25	0.063	400	2	89	14	0.05, 0.04 <u>(0.04)</u> 0.52[1], 0.47[1] 0.19[2], 0.17[2]	Pointurier 2002 0112202 5519
Kidney Bean Dry									
North 2000 Kidney beans (Flavert)	WG 62.5	0.25	0.063	400	2	83	14	Pods 0.27, 0.21 Seeds 0.04, 0.04 <u>(0.04)</u>	Pointurier 2001 0011201 5396
North 2000 Kidney beans (Astoria)	WG 62.5	0.25	0.063	400	2	89	14	Pods 0.63, 0.56 Seeds 0.06, 0.04 <u>(0.05)</u>	Pointurier 2001 0011202 5397

[1]empty pods
[2]whole pods

Table 100. Fludioxonil residues in dry pea seed harvested from supervised trials in France.

Location Year (variety)	Application		DAS[1] (days)	Analysis			Author Date Study No. Syn No
	Form.	g ai/ 100kg seed		Sample	BBCH Stage at harvest	fludioxonil residue, mg/kg	
France, North 1998 (Baccara)	WG 32.5	10.	129 129	Rest of plant Dry seed	89 89	<0.05 <u><0.02</u>	Kühne 1998 2010/98 4962
France, North 1998 (Rustic)	WG 32.5	10.	113 113	Rest of plant Dry seed	89 89	<0.05 <u><0.02</u>	Kühne 1999 2011/98 4963

Location Year (variety)	Application Form.	Application g ai/ 100kg seed	DAS[1] (days)	Analysis Sample	Analysis BBCH Stage at harvest	Analysis fludioxonil residue, mg/kg	Author Date Study No. Syn No
France, North 2001 (Athos)	WG 32.5	10.	74 74 101 101	Empty pods Immature seed Rest of plant Dry seed	75 75 89 89	<0.02 <0.02 <0.02 <0.02	Pointurier 2002 0140501 5506
France, South 1998 (Baccara)	WG 32.5	10.	96 96	Rest of plant Dry seed	89 89	0.05, <0.05 <0.02, <0.02	Kühne 1999 2012/98 4964
France, South 1998 (Loto)	WG 32.5	10.	93 93	Rest of plant Dry seed	97 97	0.08, 0.05 <0.02, <0.02	Kühne 1999 2013/98 4965
France, South 1998 (Tonus)	WG 32.5	10.	105 105	Rest of plant Dry seed	89 89	<0.05, <0.05 <0.02, <0.02	Kühne 1999 2014/98 4966
France, South 1998 (Loto)	WG 32.5	10.	100 100	Rest of plant Dry seed	89 89	<0.05, <0.05 <0.02, <0.02	Kühne 1999 2015/98 4967

[1]days after sowing

Root and tuber vegetables

Table 101. Fludioxonil residues in potato after seed treatment from supervised trials in Australia, South Africa, and the USA.

Country Year (variety)	Form	g ai/100 kg seed	Sample	PHI (days)	Fludioxonil residue, mg/kg	Author Date Study No. Syn No
Australia 1997 (Russet Burbank)	FS 100	2.5	Tubers	208	0.01, <0.01 (0.01)	McKee 1998 98/7/1618 1231
Australia 1997 (Kennebec)	FS 100	2.5	Tubers	116	<0.01, <0.01 (<0.01)	McKee 1998 98/7/1618 1231
Australia 1997 (Nadine)	FS 100	2.5	Tubers	106	<0.01, <0.01 (<0.01)	McKee 1998 98/7/1618 1231
South Africa 1998 (Nicola)	FS 100	2.5	Tubers	140	<0.02, <0.02 (<0.02)	Tribolet 1999 2232/98 5084
South Africa 1998 (Bp1)	FS 100	2.5	Tubers	140	<0.02, <0.02 (<0.02)	Tribolet 1999 2233/98 5085
Australia 1997 (Russet Burbank)	FS 100	5.0	Tubers	208	0.01, <0.01	McKee 1998 98/7/1618 1231
Australia 1997 (Kennebec)	FS 100	5.0	Tubers	116	<0.01, <0.01 (<0.01)	McKee 1998 98/7/1618

Country Year (variety)	Form	g ai/100 kg seed	Sample	PHI (days)	Fludioxonil residue, mg/kg	Author Date Study No. Syn No
						1231
Australia 1997 (Nadine)	FS 100	5.0	Tubers	106	0.01,<0.01	McKee 1998 98/7/1618 1231
South Africa 1998 (Bp1)	FS 100	5.0	Tubers	140	<0.02, <0.02 <u>(<0.02)</u>	Tribolet 1999 2234/98 5086
USA, Idaho 1992 (Russett Burbank)	DP 0.5	5 15 25	Immature tubers Mature tubers Immature tubers Mature tubers Immature tubers Mature tubers	66 143 66 143 66 143	<0.01, <0.01 <u><0.01</u>, <0.01 <0.01, <0.01 0.059, <0.01 0.057, <0.01 <0.01, <0.01	Selman 1996 ABR-93027 0747
USA, Washington 1992 (Norkota)	DP 0.5	5	Immature tubers Mature tubers	90 123	0.04, <0.01 <u><0.01</u>, <0.01	Selman 1996 ABR-93027 0747
USA, Maine 1992 (Frito Lay 945)	DP 0.5	5	Immature tubers Mature tubers	83 114	<0.01, <0.01 <u><0.01</u>, <0.01	Selman 1996 ABR-93027 0747
USA, New York 1992 (Kennebec)	DP 0.5	5 15	Immature tubers Mature tubers Immature tubers Mature tubers	76 154 76 154	<0.01, <0.01 <u><0.01</u>, <0.01 <0.01, <0.01 0.014, <0.01	Selman 1996 ABR-93027 0747
USA, Wisconsin 1992 (Sunnydale Farms)	DP 0.5	5	Immature tubers Mature tubers	60 95	<0.01, <0.01 <u><0.01</u>, <0.01	Selman 1996 ABR-93027 0747
USA, Idaho 1992 (Russet Burbank)	DP 0.5	5	Immature tubers Mature tubers	66 143	<0.01, <0.01 <u><0.01</u>, <0.01	Selman 1996 ABR-93027 0747
USA, Florida 1992 (Kennebec)	DP 0.5	5	Immature tubers Mature tubers	61 76	0.013, <0.01 <u><0.01</u>, <0.01	Selman 1996 ABR-93027 0747
USA, North Dakota 1992 Red Norland	DP 0.5	5 15	Immature tubers Mature tubers Immature tubers Mature tubers	73 112 73 112	0.016, <0.01 <u><0.01</u>, <0.01 <0.01, <0.01 0.024, <0.01	Selman 1996 ABR-93027 0747
USA, Michegan 1992 Onaway	DP 0.5	5	Immature tubers Mature tubers	53 91	<0.01, <0.01 <u><0.01</u>, <0.01	Selman 1996 ABR-93027 0747
USA, California 1992 Red LA Soda	DP 0.5	5 15 25	Immature tubers Mature tubers Immature tubers Mature tubers Immature tubers Mature tubers	60 99 60 99 60 99	<0.01, <0.01 <u><0.01</u>, <0.01 0.055, 0.026 0.015, <0.01 0.48, 0.062 0.019, <0.01	Selman 1996 ABR-93027 0747
USA Minnesota 1992 (Norchip)	DP 0.5	5	Immature tubers Mature tubers	83 114	<u><0.01</u>, <0.01 <0.01; <0.01	Selman 1996 ABR-93027 0747
USA, Idaho 1992 (Russet Burbank)	DP 0.5	5 15	Immature tubers Mature tubers Immature tubers	66 143 66	<0.01, <0.01 <u><0.01</u>, <0.01 <0.01, <0.01	Selman 1996 ABR-93027

Country Year (variety)	Form	g ai/100 kg seed	Sample	PHI (days)	Fludioxonil residue, mg/kg	Author Date Study No. Syn No
		25	Mature tubers Immature tubers Mature tubers	143 66 143	0.059, <0.01 0.057, <0.01 <0.01, <0.01	0747
USA, Colorado 1992 (Frito Lay 1775)	DP 0.5	5	Immature tubers Mature tubers	60 96	<0.01, <0.01 <u><0.01</u>, <0.01	Selman 1996 ABR-93027 0747

Table 102: Fludioxonil residues resulting from post-harvest application to yams in the USA.

Year (variety)	Form	Method	Treatment concentration kg ai/hl	Fludioxonil residue, mg/kg[3]	Author Date Study No. Syn No
2000 (Guinea)	WP 50	Post-harvest dip to tuber pieces at 0.06 kg ai/l; dipping time 30 ± 3 seconds Post-harvest dip to whole tubers at 0.06 kg ai/l; dipping time 30 ± 3 seconds	0.06 0.06	4.2, 3.4 (3.8) 4.7, 4.5 (4.6)	Thompson & Ediger 2003 1654-00
2000 (Guinea)	WP 50	Post-harvest dip to tuber pieces at 0.06 kg ai/l; dipping time 30 ± 3 seconds Post-harvest dip to whole tubers at 0.06 kg ai/l; dipping time 30 ± 3 seconds	0.06 0.06	5.7, 4.2 (5.0) 4.0, 2.5 (3.2)	Thompson & Ediger 2003 1654-00

Table 103. Residues of fludioxonil in carrots from supervised trials in the USA.

Location Year (variety)	Application					PHI days	Fludioxonil residue, mg/kg	Author, Date Study No.
	Form	kg ai/ha	kg ai/hl	Water, l/ha	No.			
BC 06 2000 (Bolero)	WG 62.5	0.24	0.070	348-352	4	6	0.46, 0.37 <u>(0.42)</u>	Hong Chen 2002 07090
CA 04 2000 Minicor (baby variety)	WG 62.5	0.24	0.041	483-697 -	4	7	0.18, 0.14 <u>(0.16)</u>	Hong Chen 2002 07090
CA 86 2000 (Caropak)	WG 62.5	0.24- 0.25	0.048	496-525	4	8	0.05, 0.02 <u>(0.04)</u>	Hong Chen 2002 07090
CA 104 2000 (Nantes)	WG 62.5	0.25	0.062	400	4	7	0.31, 0.19 <u>(0.25)</u>	Hong Chen 2002 07090
CA 105 2000 (Imperator)	WG 62.5	0.24- 0.25	0.064	375-383	4	7	0.18, 0.18 <u>(0.18)</u>	Hong Chen 2002 07090
MI 01 2000 (Premium)	WG 62.5	0.38- 0.40	0.104	365-385	4	7	0.24, 0.19 (0.22)	Hong Chen 2002 07090

Location Year (variety)	Application					PHI days	Fludioxonil residue, mg/kg	Author, Date Study No.
	Form	kg ai/ha	kg ai/hl	Water, l/ha	No.			
FL 75 2000 (Navajo F1)	WG 62.5	0.12- 0.13	0.045	282-295	4	8	0.05, 0.04 (0.04)	Hong Chen 2002 07090
TX 05 2000 (Nantes coreless)	WG 62.5	0.24- 0.25	0.064	367-411	4	7	0.21, 0.19 (0.20)	Hong Chen 2002 07090
WA 43 2000 (Bolero)	WG 62.5	0.24- 0.25	0.064	373-386	4	7	0.24, 0.16 (0.20)	Hong Chen 2002 07090

Stalk and stem vegetables

Table 104: Residues of fludioxonil in asparagus from supervised trials in Germany.

Year (variety)	Application					PHI days	Fludioxonil residue, mg/kg	Author Date Study No. Syn No
	Form	kg ai/ha	kg ai/hl	Water, l/ha	No.			
2000 (Gijlim)	WG 62.5	0.245- 0.260	0.063	588 - 626	3	238 239	<0.02 <0.02	Smith 2001 gr 46999 5378
2000 (Vulkan)	WG 62.5	0.249 - 0.262	0.063	597 - 629	3	238 239	<0.02 <0.02	Smith 2001 gr 47999 5379

Cereal grains

The Meeting received numerous reports on the use of fludioxonil as a seed treatment for cereal grains.

Table 105. Fludioxonil residues in wheat grain after seed treatment from supervised trials in France, Switzerland, and Germany.

Country Year (variety)	Form	No.	g ai/ 100 kg seed	PHI (days)	Fludioxonil residue mg/kg	Author, Date Study No. Syn No
France, North 1998 (Ritmo)	FS 287.5	1	6.0	262	<0.02	Pointurier 2000 9940201 5191
France, North 1997 (Vivant)	FS 312.5	1	5.3	276	<0.02	Pointurier 1999 9840301 1927
France, North 1997 (Sidéral)	FS 312.5	1	5.4	251	<0.02	Pointurier 1999 9840302 1928
France, North 1997 (Vivant)	FS 206.25	1	5.5	276	<0.02	Pointurier 1999 9840401 1957

Country Year (variety)	Form	No.	g ai/ 100 kg seed	PHI (days)	Fludioxonil residue mg/kg	Author, Date Study No. Syn No
France, North 1997 (Sidéral)	FS 206.25	1	5.7	251	<0.02	Pointurier 1999 9840402 1956
France, North 1998 (Forence Aurore)	FS 206.25	1	5.4	156	<0.02; <0.02	Pointurier 2000 9840502 2013
France, North 1998 (Furio)	FS 312.5	1	5.3	151	<0.02; <0.02	Pointurier 2000 9840601 2016
France, North 1998 (Prinqual)	FS 312.5)	1	5.2	156	<0.02; <0.02	Pointurier 2000 9840602 2017
France, North 1989 (Maris Huntsman)	FS 25	1	5.0	267	<0.02	Tournayre 1992 04/92 0189
France, North 1993 (Soissons)	FS 300	1	5.0	240	<0.02	Argento 1995 OS94405 1116
France, North 1993 (Soissons)	FS 150	1	5.0	240	<0.02	Maffezzoni 1994 OS94402 539
France, North 1996 (Sidéral)	FS 320	1	5.0	251	<0.02	Pointurier 1998 OS97401/SJ99 1587
France, North 1996 (Tremie)	FS 320	1	5.0	270	<0.02	Pointurier 1998 OS97401/KJ99 1586
France, North 1991 (Apollo)	FS 275	1	7.5	273	<0.02	Maffezzoni 1993 OS92001 0301
France, North 1991 (Fortal)	FS 275	1	7.5	237	<0.02	Maffezzoni 1993 OS92001 0301
France, South 1991 (Festival)	FS 275	1	7.5	234	<0.02	Maffezzoni 1993 OS92001 0301
France, South 1996 (Soissons)	FS 320	1	5.0	223	<0.02 <0.04	Pointurier 1998 OS97401/AC99 1585
France, South 1993 (Sidéral)	FS 150	1	5.0	212	<0.02 <0.02	Maffezzoni 1994 OS94402 0539
France, South 1997 (Soissons)	FS 206.25	1	5.6	217	<0.02	Pointurier 1999 9840403 1955

Country Year (variety)	Form	No.	g ai/ 100 kg seed	PHI (days)	Fludioxonil residue mg/kg	Author, Date Study No. Syn No
France, South 1997 (Scipion)	FS 206.25	1	5.6	223	<0.02	Pointurier 1999 9840404 1954
France, South 1993 (Fidel)	FS 150	1	5.0	223	<0.02 <0.02	Maffezzoni 1994 OS94402 0539
France, South 1998 (Sidéral)	FS 312.5	1	6.0	242	<0.02; <0.02	Pointurier 2000 9940402 2034
France, South 1998 (Florence Aurore)	FS 206.25	1	5.4	147	<0.02; <0.02	Pointurier 2000 9840503 2014
France, South 1998 (Furio)	FS 206.25	1	5.5	145	<0.02; <0.02	Pointurier 2000 9840504 2015
France, North 1998 (Furio)	FS 206.25	1	5.3	153	<0.02, <0.02	Pointurier 2000 9840501 2012
France, South 1998 (Orqual)	FS 312.5	1	6.0	238	<0.02; <0.02	Pointurier 2000 99-40-401 2033
France, South 1998 (Florence Aurore)	FS 312.5	1	5.1	147	<0.02; <0.02	Pointurier 2000 9840603 2018
France, South 1997 (Furio)	FS 312.5	1	5.2	145	<0.02, <0.02	Pointurier 2000 984064 2019
France, South 1998 (Sidéral)	FS 287.5	1	7.4	242	<0.02; <0.02	Pointurier 2000 9940202 5192
France, South 1993 (Sidéral)	FS 300	1	5.0	212	<0.02	Argento 1995 OS94405 1116
France, South 1993 (Fidel)	FS 300	1	5.0	223	<0.02	Argento 1995 OS94405 1116
France, South 1989 (Maris Huntsman)	FS 25	1	5.0	211	<0.02	Tournayre 1992 03/92 0188
Switzerland 1989 (Albis)	FS 25	1	5.0	128	<0.02; <0.02	Altenburger 1991 2030/89 1171

Country Year (variety)	Form	No.	g ai/ 100 kg seed	PHI (days)	Fludioxonil residue mg/kg	Author, Date Study No. Syn No
Switzerland 1989 (Arina)	FS 25	1	5.0	276	<0.02; <0.02	Altenburger 1991 2028/89 0075
Germany 1991	FS 25	1	5.0	126	<0.04	Brandl 1993 gr 40191 0677
Germany 1991 (Max)	FS 25	1	5.0	142	<0.04	Brandl 1993 gr 10191 0676
Germany 1991 (Max)	FS 25	1	5.0	131	<0.04	Brandl 1993 gr 70191 0678
Germany 1992 (Star)	FS 125	1	5.0	112	<0.04	Brandl 1993 gr 6292 1145
Germany 1992 (Star)	FS 125	1	5.0	128	<0.04	Brandl 1993 gr 6292 1145
Germany 1991 (Herzog)	FS 125	1	5.0	272	<0.04	Brandl 1993 gr 6292 1145
Germany 1992 (Star)	FS 125	1	5.0	116	<0.04	Brandl 1993 gr 6292 1145
Germany 1991 (Herzog)	FS 125	1	5.0	284	<0.04	Brandl 1993 gr 6292 1145
Germany 1991 (Max)	FS 125	1	5.0	142	<0.04	Brandl 1993 gr6291 1144
Germany 1991 (Max)	FS 125	1	5.0	126	<0.04	Brandl 1993 gr6291 1144
Germany 1991 (Max)	FS 125	1	5.0	131	<0.04	Brandl 1993 gr6291 1144
Germany 1993 (Star)	FS 050	1	5.0	128	<0.02	Kühl 1994 gr6893 0546
Germany 1993 (Star)	FS 050	1	5.0	147	<0.02	Kühl 1994 gr6893 0546
Germany 1993 (Star)	FS 050	1	5.0	159	<0.02	Kühl 1994 gr6893 0546

Table 106. Fludioxonil residues in rye grain from supervised seed treatment trials in Denmark.

Year (Variety)	Form	No.	g ai/ 100 kg seed	PHI days	Fludioxonil residue mg/kg	Author Date Study No. Syn No
1991 (Petkus II)	FS 25	1	5.0	305	<0.02	Mair 1993 2020/91 0333

Table 107. Fludioxonil residues in barley grain after seed treatment from supervised trials in France, Switzerland, and Germany.

Country Year (variety)	Form.	No.	g ai/ 100 kg seed	PHI, days	Fludioxonil residue mg/kg	Author Date Study No. Syn No
France, North 1989 (Astrix)	FS 25	1	5.0	256	<0.02	Maffezzoni 1992 05/92 0190
France, North 1991 (Plaisant)	FS 275	1	7.5	265	<0.02	Maffezzoni 1993 OS92002 0300
Germany 1992 (Gaulois)	FS 60	1	5.0	287	<0.02	Kühl 1995 gr 6793 0692
Germany 1993 (Sissy)	FS 60	1	5.0	156	<0.02	Kühl 1995 gr 6793 0692
Germany 1993 (Sissy)	FS 60	1	5.0	143	<0.02	Kühl 1995 gr 6793 0692
Switzerland 1989 (Cornel)	FS 25	1	5.0	115	<0.02; <0.02	Altenburger 1990 2029/89 0074
Switzerland 1999 (Meltan)	FS 25	1	3.5	112	<0.02; <0.02	Tribolet 2000 4000/99 6895
Switzerland 1999 (Meltan)	FS 25	1	3.5	112	<0.02; <0.02	Tribolet 2000 4001/99 6896
France, North 1999 (Scarlett, Spring)	FS 425	1	2.8	133	<0.02	Pointurier 2000 9940801 5303

Country Year (variety)	Form.	No.	g ai/ 100 kg seed	PHI, days	Fludioxonil residue mg/kg	Author Date Study No. Syn No
France, North 1998 (Nevada)	FS 425	1	2.3	151	<0.02, <0.02	Pointurier 2000 9840701 5155
France, North 1998 (Prisma)	FS 425	1	2.6	156	<0.02, <0.02	Pointurier 2000 9840801 5157
France, North 1998 (Esterel)	FS 425	1	2.9	262	<0.02	Pointurier 2000 9940501 5284
France, North 1994 (Clarine)	FS 302	1	2.5	217	<0.02, <0.02	Maffezzoni 1996 OS95400/SJ99 0987
France, North 1994 (Intro)	FS 30	1	2.5	217	<0.02, <0.02	Maffezzoni 1996 OS95400/SJ98 0988
France, North 1994 (Express)	FS 302	1	2.5	237	<0.02, <0.02	Maffezzoni 1996 OS95400/KJ02 0989
France, North KJ01 1994 (Labea)	FS 302	1	2.5	237	<0.02, <0.02	Maffezzoni 1996 OS95400/KJ01 0822
France, South 1998 (Pastoral, Winter)	FS 425	1	2.9	224	<0.02, <0.02	Pointurier 2000 9940601 5211
France, South 1998 (Maeva, Winter)	FS 425	1	2.9	212	<0.02, <0.02	Pointurier 2000 9940602 5210
France, South 1998 (Plaisant, Winter)	FS 425	1	2.9	216	<0.02	Pointurier 2000 9940603 5209
France, South 1998 (Gaelic, winter)	FS 425	1	2.8	242	<0.02, <0.02	Pointurier 2000 9940604 5208
France 1999 (Scarlett, Winter)	FS 425	1	2.7	133	<0.02, <0.02	Pointurier 2000 9940801 5303
France, South 1998 (Prisma)	FS 425	1	2.6	125	<0.02, <0.02	Pointurier 2000 9840702 5156
France, South 1998 (Nevada)	FS 425	1	2.3	124	<0.02, <0.02	Pointurier 2000 9840802 5158

Country Year (variety)	Form.	No.	g ai/ 100 kg seed	PHI, days	Fludioxonil residue mg/kg	Author Date Study No. Syn No
France, South 1994 (Intro)	FS 302	1	2.5	208	<0.02, <0.02	Maffezzoni 1996 OS95400/AC98 0986
France, South 1994 (Plaisant)	FS 302	1	2.5	204	<0.02, <0.02	Maffezzoni 1996 OS95400/AC99 0985
France, South 1994 (Pastoral)	FS 302	1	2.5	209	<0.02, <0.02	Maffezzoni 1996 OS95400/LD02 0984
France, South 1994 (Plaisant)	FS 302	1	2.5	209	<0.02, <0.02	Maffezzoni 1996 OS95400/LD01 0983
Germany 1999 (Krona, Spring)	FS 43.75	1	2.6	118	<0.02	Smith 2000 gr 67299 6902
Germany 1999 (Krona, Spring)	FS 43.75	1	2.5	115	<0.02	Smith 2001 gr 68499 5189
Germany 1998 (Cabria)	FS 43.75	1	2.6	285	<0.02	Smith 2000 gr 63299 6900

Table 108. Fludioxonil residues in maize grain after seed treatment from supervised trials in France, Germany, Greece, Hungary, South Africa, Spain, and the USA.

Country Year (Variety)	Form	g ai/ 100 kg seed	Crop part	PHI	Fludioxonil residue mg/kg	Author Date Study No. Syn No
USA-MS 1992 Field corn (6138X)	4FS	25	Grain	147	<u>≤0.01,</u> <0.01	Selman 1993 ABR-93030 31-92
USA-IL 1992 Field corn (G4385)	4FS	25	Grain	166	<u>≤0.01</u>, <0.01	Selman 1993 ABR-93030 31-92
USA-IA 1992 Field corn (2073X)	4FS	15	Grain	167	<u>≤0.01,</u> <0.01	Selman 1993 ABR-93030 31-92
USA-MO 1992 Field corn (2073X)	4FS	15	Grain	179	<u>≤0.01,</u> <0.01	Selman 1993 ABR-93030 31-92
USA-PA 1992 Field corn (2073X)	4FS	15	Grain	169	<u>≤0.01</u>, <0.01	Selman 1993 ABR-93030 31-92
France, North	FS 378	5.0	cobs grain	116 160	<0.02 <u>≤0.02</u>	Maffezzoni 1995

Country Year (Variety)	Form	g ai/ 100 kg seed	Crop part	PHI	Fludioxonil residue mg/kg	Author Date Study No. Syn No
1994 (Bahia)						OS94411 3965
France, North 1994 (Antares)	FS 378	5.0	cobs grain	116 160	<0.02 <u><0.02</u>	Maffezzoni 1995 OS94411 3965
France, North 1994 (Cesar)	FS 378	5.0	cobs grain	126 162	<0.02 <u><0.02</u>	Maffezzoni 1995 OS94411 3965
France, South 1994 (Furio)	FS 378	5.0	cobs grain	105 146	<0.02 <u><0.02</u>	Maffezzoni 1995 OS94411 3965
France, South 1994 (Occitan)	FS 378	5.0	cobs grain	105 146	<0.02 <u><0.02</u>	Maffezzoni 1995 OS94411 3965
France, South 1994 (Pactol)	FS 378	5.0	cobs grain	116 164	<0.02 <u><0.02</u>	Maffezzoni 1995 OS94411 3965
Greece 1991 (Prisma)	FS 100	10.	cobs	159	<0.02, <0.02	Mair 1992 2100/91 0226
Greece 1991 (Prisma)	FS 100	5.0	cobs	159	<0.02, <0.02	Mair 1992 2099/91 0225
Germany 1992 (Garant)	FS 200	?	grain	168 183?	0.05	Heyer 1994 RCC 463306 4011
Germany 1992 (Garant)	FS 200	?	grain	170	<0.02	Heyer 1994 RCC 463307 4033
South Africa 1997 (PAN 6479)	FS 035	5.0	grain	206	<u><0.02</u>	Kühne 1998 2405/97 1182
France, North 1998 (Bahia)	FS 424.6	3.4	cobs grain	111 145	<0.02, <0.02 <0.02, <0.02	Pointurier 1999 9841301 5056
France North, 1998 (Antares)	FS 424.6	3.4	cobs grain	105 166	0.02, 0.02 <0.02 <0.02	Pointurier 1999 9841601 5057
France, North 1999 (Anjou 285)	FS 424.6	2.6	cobs grain	106 158	<0.02, <0.02 <0.02 , <0.02	Pointurier 2000 9941201 5241
France, North 1997 (Antares)	FS 035	2.5	cobs cobs grain	125 133 166	<0.02 <0.02 <0.02	Kühne 1998 2321/97 1082
France, North 1997	FS 035	2.5	cobs grain	135 157	<0.02 <0.02	Kühne 1998 2322/97

Country Year (Variety)	Form	g ai/ 100 kg seed	Crop part	PHI	Fludioxonil residue mg/kg	Author Date Study No. Syn No
(Bahia)						1083
Germany 1997 (Antares)	FS 035	2.5	cobs cobs grain	124 141 155	<0.02 <0.02 <0.02	Smith 1998 gr 44497 1088
Germany 1997 (Antares)	FS 035	2.5	cobs cobs grain	118 142 174	<0.02 <0.02 <0.02	Smith 1998 gr 43197 1089
France, South 1998 (Furio)	FS 424.6	3.4	cobs grain	92 142	<0.02, <0.02 <0.02, <0.02	Pointurier 1999 9841302 5058
France, South 1998 (Occitan)	FS 424.6	3.3	cobs grain	92 145	<0.02, <0.02 <0.02, <0.02	Pointurier 1999 9841602 5059
France, South 1999 (Occitan)	FS 424.6	2.6	cobs grain	86 132	<0.02, <0.02 <0.02, <0.02	Pointurier 2000 9941202 5243
France, South 1997 (Occitan)	FS 035	2.5	cobs cobs grain	105 132 159	<0.02 <0.02 <0.02	Kühne 1998 2323/97 1084
France, South 1997 (Furio)	FS 035)	2.5	cobs cobs grain	92 109 141	<0.02 <0.02 <0.02	Kühne 1998 2324/97 1085
Greece 1997 (Tundra)	FS 035	2.5	cobs grain	106 160	<0.02, <0.02 <0.02, <0.02	Kühne 1998 2306/97 1186
Italy 1997 (Tundra)	FS 035	2.5	cobs grain	111 138	<0.02, <0.02 <0.02, <0.02	Kühne 1998 2036/97 1087
Spain 1997 (Miguel)	FS 035	2.5	cobs grain	112 163	<0.02, <0.02 <0.02, <0.02	Kühne 1998 2025/97 1086
Spain 1997 (Miguel)	FS 035	2.5	grain	177	<0.02, <0.02	Kühne 1998 2024/97 1153
Hungary 1996	FS 045	2.5	grain	157	<0.02	Berczi 1996 CIBA AB 16961 4202
South Africa 1997 (PAN 6479)	FS 035	2.5	grain	206	<0.02	Kühne 1998 2403/97 1180
South Africa 1997 (SNK 2266)	FS 035	2.5	grains	206	<0.02	Kühne 1998 2404/97 1181

Sorghum seed treatment trials in Kansas and Texas, USA, were reported (Boyette, Report US 33-92, 1993). The analytical method was AG-597, validated by concurrent recovery experiments at

0.05 mg/kg for grain and processed fractions thereof. It was reported that there were interferences at fortification levels below 0.05 mg/kg (Tables 49 and 51 in the Analytical methods section above).

Table 109. Fludioxonil residues in sorghum grain after seed treatment from supervised trials in the USA.

Location, Year (Variety)	Form	No.	g ai/100 kg seed	PHI days	Sample	Fludioxonil residue mg/kg	Author Date Study No. Syn No
KA 1992 (Funks G1711)	4FS	1	16 26	127 127 127 127	Grain Flour Grain Flour	<0.05, <0.05 <0.05 <0.05, <0.05 <0.05	Boyette 1993 US 33-92
TX 1992	4FS	1	14 23	145 145	Grain Grain	<0.05, <0.05 <0.05, <0.05	Boyette 1993 US 33-92

Nuts and seeds

Treenuts

Table 110. Residues of fludioxonil in pistachio nuts from supervised trials in California, USA.

Location Year (variety)	Application					PHI days	Fludioxonil residue mg/kg	Author Date Study No.
	Form	Kg ai/ha	kg ai/hl	Water, l/ha	No.			
1999 (Kerman)	WG 62.5	0.24 - 0.25	0.017	1383 - 1423	4	7	0.08, 0.07 (0.08)	Thompson 2001 IR-4 07336 1278-99
1999 (Kerman/Peter)	WG 62.5	0.24	0.017	1412	4	7	0.04, 0.03 (0.04)	Thompson 2001 IR-4 07336 1278-99
1999 (Kerman)	WG 62.5	0.24	0.017	1405	4	7	0.06, 0.04 (0.05)	Thompson 2001 IR-4 07336 1278-99

Oil seeds

Table 111. Fludioxonil residues in rape seed after seed treatment from supervised trials in France, the UK, Sweden, and Germany.

Country Year (variety)	Form	g ai/100 kg seed	Sample	PHI (days)	Fludioxonil residue mg/kg	Author Date Study No. Syn No
France, North 1997 (Navajo)	FS 321.3	12	Seeds	300	<0.02	Mair 1999 1176/97 4957
France, North 1997 (Navajo)	FS 321.3	12	Seeds	293	<0.02	Mair 1999 1177/97

Country Year (variety)	Form	g ai/100 kg seed	Sample	PHI (days)	Fludioxonil residue mg/kg	Author Date Study No. Syn No
						4958
UK 1997 (Apex)	FS 321.3	12	Seeds	323	<0.02, <0.02, <0.02	Mair 1999 1025/98 4970
UK 1997 (Apex)	FS 321.3	12	Seeds	322	<0.02, <0.02, <0.02	Mair 1999 1026/98 4971
Sweden 1998 (Sponsor)	FS 321.3	12	Seeds	145	<0.02	Smith 1999 gr 68098 5045
Germany 1998 (Laser)	FS 321.3	12.	Seeds	330	<0.02	Smith 2000 gr 61299 1197
Germany 1998 (Laser)	FS 321.3	12.	Seeds	319	<0.02	Smith 2000 gr 62499 1198
Germany 1999 (Evita)	FS 321.3	12.	Seeds	133	<0.02	Smith 2000 gr 71199 1199
Germany 1998 (Licosmos)	FS 321.3	12	Seeds	130	<0.02	Smith 1999 gr 66298 5043
Germany 1998 (Licosmos)	FS 321.3	12	Seeds	124	<0.02	Smith 1999 gr 65498 5044
France, South 1997 (Goeland)	FS 321.3	12	Seeds	278	<0.02	Mair 1999 1178/97 4959
France, South 1997 (Bristol)	FS 321.3	12	Seeds	282	<0.02	Mair 1999 1179/97 4960
France, South 1998 (Capitol)	FS 321.3	12	Seeds	274	<0.02, <0.02	Mair 2000 1110/98 5201
France, South 1998 (Columbus)	FS 321.3	13	Seeds	277	<0.02, <0.02	Mair 2000 1112/98 5202
France, South 1998 (Goeland)	FS 321.3	12	Seeds	285	<0.02, <0.02	Mair 2000 1113/98 5203

Table 112. Fludioxonil residues in harvested cotton seed after seed treatment from supervised trials in the USA and Greece.

Country Year (Variety)	Form	g ai/ 100 kg seed	Sample	PHI (days)	Fludioxonil residue mg/kg	Author Date Study No. Syn No
USA, CA	FS 4	15	Undelinted seed Hulls	189 189	<u><0.05</u> <0.05	Vincent 1998 ABR-97111 59-96
USA, MS	FS 4	8.3	Field trash Gin trash Undelinted seed	152 152 152	<0.05 <0.05 <u><0.05</u>	Vincent 1998 ABR-97111 59-96
USA, TX	FS 4	13	Gin trash Undelinted seed	165 165	<0.05 <u><0.05</u>	Vincent 1998 ABR-97111 59-96
USA, TX	FS 4	4.9	Field trash Gin trash Undelinted seed	132 132 132	<0.05 <0.05 <u><0.05</u>	Vincent 1998 ABR-97111 59-96
USA, OK	FS 4	6.0	Field trash Gin trash Undelinted seed	174 174 174	<0.05 <0.05 <u><0.05</u>	Vincent 1998 ABR-97111 59-96
USA, NM	FS 4	4.4	Field trash Gin trash Undelinted seed	188 188 188	<0.05 <0.05 <u><0.05</u>	Vincent 1998 ABR-97111 59-96
Greece 1991 (S80)	FS 100	10.	Hulls Seeds, dehulled	175 175	<0.02, <0.02 <0.02, <u><0.02</u>	Mair 1993 2119/91 0252
Greece 1991 (S80)	FS 100	20.	Hulls Seeds, dehulled	175 175	<0.02, <0.02 <0.02, <u><0.02</u>	Mair 1993 2120/91 0253
Greece 1997 (Eva)	ES 104	2.5	Hulls Seeds, dehulled	149 149	<0.05, <0.05 <0.02, <0.02	Kühne 1999 2309/97 1673
Greece 1997 (Eva)	ES 104	2.5	Hulls Seeds, dehulled	165 165	<0.05, <0.05 <0.02, <0.02	Kühne 1999 2310/97 1674
Greece 1997 (Eva)	ES 104	2.5	Hulls Seeds, dehulled	159 159	<0.05, <0.05 <0.02, <0.02	Kühne 1999 2311/97 1675

Herbs and Spices

Herbs

Table 113. Residues of fludioxonil in chives from supervised trials in the USA.

Location Year (variety)	Application					PHI days	Fludioxonil residue, mg/kg	Author Date Study No.
	Form	kg ai/ha	kg ai/hl	Water, l/ha	No.			
MD 2000 (Fancy)	WG 62.5	0.392- 0.396	0.100	392-397	4	6	Fresh 2.2, 1.8	Hong Chen 2002

Location Year (variety)	Application					PHI days	Fludioxonil residue, mg/kg	Author Date Study No.
	Form	kg ai/ha	kg ai/hl	Water, l/ha	No.			
							(2.0)	07126
OR 2000 (Standard open)	WG 62.5	0.245- 0.250	0.047	520-528	4	7	Fresh 1.8, 1.7 (1.8)	Hong Chen 2002 07126
CA 2000 (Common)	WG 62.5	0.245- 0.259	0.167	147-154	4	7	Fresh 4.5, 3.3 (3.9) Dry 31.	Hong Chen 2002 07126

Table 114. Residues of fludioxonil in basil from supervised trials in the USA.

Location Year (variety)	Application					PHI days	Fludioxonil residue, mg/kg	Author Date Study No.
	Form	kg ai/ha	kg ai/hl	Water, l/ha	No.			
NY 2000 (Genovese basil)	WG 62.5	0.392- 0.411	0.123	311-344	4	8	Fresh 13.1, 10.8 (12)	Hong Chen 2002 07123
FL 2000 (HBA 105)	WG 62.5	0.242- 0.249	0.088	277-286	4	7	Fresh 2.0, 1.8 (1.9)	Hong Chen 2002 07123
CA 2000 (Italian large leaf basil)	WG 62.5	0.243- 0.245	0.064	374-387	4	7	Fresh[1] 3.4, 2.7 (3.0) Dry[1] 23.	Hong Chen 2002 07123

[1] Fresh and dry control samples contained apparent residues at 0.02 mg/kg

Straw, fodder, and forage of cereal grains and grasses

Table 115. Fludioxonil residues in wheat forage and straw after seed treatment from supervised trials in France, Switzerland, and Germany.

Country Year (variety)	Form	No.	g ai/ 100 kg seed	PHI (days)	Sample	Fludioxonil residue mg/kg	Author, Date Study No. Syn No
France, North 1998 (Ritmo)	FS 287.5	1	6.1	262	straw	<0.05; <0.05	Pointurier 2000 9940201 5191
France, North 1997 (Vivant)	FS 312.5	1	5.3	276	straw	<0.05	Pointurier 1999 9840301 1927
France, North 1997 (Sidéral)	FS 312.5	1	5.4	251	straw	<0.05	Pointurier 1999 9840302 1928
France, North 1997 (Vivant)	FS 206.25	1	5.5	276	straw	<0.05	Pointurier 1999 9840401 1957

Country Year (variety)	Form	No.	g ai/ 100 kg seed	PHI (days)	Sample	Fludioxonil residue mg/kg	Author, Date Study No. Syn No
France, North 1997 (Sidéral)	FS 206.25	1	5.7	251	straw	<0.05	Pointurier 1999 9840402 1956
France, North 1998 (Forence Aurore)	FS 206.25	1	5.4	65 91 156	whole plant whole plant straw	<0.02 <0.02 <0.05; <0.05	Pointurier 2000 9840502 2013
France, North 1998 (Furio)	FS 312.5	1	5.3	151	straw	<0.05; <0.05	Pointurier 2000 9840601 2016
France, North 1998 (Prinqual)	FS 312.5)	1	5.2	156	straw	<0.05; <0.05	Pointurier 2000 9840602 2017
France, North 1989 (Maris Huntsman)	FS 25	1	5.0	267	straw	<0.02	Tournayre 1992 04/92 0189
France, North 1993 (Soissons)	FS 300	1	5.0	240	straw	<0.05	Argento 1995 OS94405 1116
France, North 1993 (Soissons)	FS 150	1	5.0	240	straw	<0.02	Maffezzoni 1994 OS94402 539
France, North 1996 (Sidéral)	FS 320	1	5.0	251	straw	<0.04	Pointurier 1998 OS97401/SJ99 1587
France, North 1996 (Tremie)	FS 320	1	5.0	270	straw	<0.04	Pointurier 1998 OS97401/KJ99 1586
France, South 1996 (Soissons)	FS 320	1	5.0	223	straw	<0.04	Pointurier 1998 OS97401/AC99 1585
France, South 1993 (Sidéral)	FS 150	1	5.0	212	straw	<0.02	Maffezzoni 1994 OS94402 0539
France, South 1997 (Soissons)	FS 206.25	1	5.6	217	straw	<0.05	Pointurier 1999 9840403 1955
France, South 1997 (Scipion)	FS 206.25	1	5.6	223	straw	<0.05	Pointurier 1999 9840404 1954
France, South 1993 (Fidel)	FS 150	1	5.0	223	straw	<0.02	Maffezzoni 1994 OS94402 0539
France, South 1998 (Sidéral)	FS 312.5	1	6.0	242	straw	<0.05; <0.05	Pointurier 2000 9940402 2034

Country Year (variety)	Form	No.	g ai/ 100 kg seed	PHI (days)	Sample	Fludioxonil residue mg/kg	Author, Date Study No. Syn No
France, South 1998 (Florence Aurore)	FS 206.25	1	5.4	64 92 147	whole plant whole plant straw	<0.02 <0.02 <0.05; <0.05	Pointurier 2000 9840503 2014
France, South 1998 (Furio)	FS 206.25	1	5.5	60 101 145	whole plant whole plant straw	<0.02 <0.02 <0.05; <0.05	Pointurier 2000 9840504 2015
France, North 1998 (Furio)	FS 206.25	1	5.3	71 92 153	whole plant whole plant straw	<0.02 <0.02 <0.05, <0.05	Pointurier 2000 9840501 2012
France, South 1998 (Orqual)	FS 312.5	1	6.0	238	straw	<0.05, <0.05	Pointurier 2000 99-40-401 2033
France, South 1998 (Florence Aurore)	FS 312.5	1	5.1	147	straw	<0.05; <0.05	Pointurier 2000 9840603 2018
France, South 1997 (Furio)	FS 312.5	1	5.2	145	straw	<0.05, <0.05	Pointurier 2000 984064 2019
France, South 1998 (Sidéral)	FS 287.5	1	7.4	242	straw	<0.05; <0.05	Pointurier 2000 9940202 5192
France, South 1993 (Sidéral)	FS 300	1	5.0	212	straw	<0.05	Argento 1995 OS94405 1116
France, South 1993 (Fidel)	FS 300	1	5.0	223	straw	<0.05	Argento 1995 OS94405 1116
France, South 1989 (Maris Huntsman)	FS 25	1	5.0	211	straw	<0.02	Tournayre 1992 03/92 0188
Switzerland 1989 (Albis)	FS 25	1	5.0	128	straw	<0.05; <0.05	Altenburger 1991 2030/89 1171
Switzerland 1989 (Arina)	FS 25	1	5.0	276	straw	<0.05; <0.05	Altenburger 1991 2028/89 0075
Germany 1991	FS 25	1	5.0	59 126	whole plant straw	<0.04 <0.04	Brandl 1993 gr 40191 0677
Germany 1991 (Max)	FS 25	1	5.0	66 142	whole plant straw	<0.04 <0.04	Brandl 1993 gr 10191 0676

Country Year (variety)	Form	No.	g ai/ 100 kg seed	PHI (days)	Sample	Fludioxonil residue mg/kg	Author, Date Study No. Syn No
Germany 1991 (Max)	FS 25	1	5.0	68 131	whole plant straw	<0.04 <0.04	Brandl 1993 gr 70191 0678
Germany 1992 (Star)	FS 125	1	5.0	61 112	whole plant straw	<0.04 <0.04	Brandl 1993 gr 6292 1145
Germany 1992 (Star)	FS 125	1	5.0	66 128	whole plant straw	<0.04 <0.04	Brandl 1993 gr 6292 1145
Germany 1991 (Herzog)	FS 125	1	5.0	210 272	whole plant straw	<0.04 <0.04	Brandl 1993 gr 6292 1145
Germany 1992 (Star)	FS 125	1	5.0	56 116	whole plant straw	<0.04 <0.04	Brandl 1993 gr 6292 1145
Germany 1991 (Herzog)	FS 125	1	5.0	209 284	whole plant straw	<0.04 <0.04	Brandl 1993 gr 6292 1145
Germany 1991 (Max)	FS 125	1	5.0	66 142	whole plant straw	<0.04 <0.04	Brandl 1993 gr6291 1144
Germany 1991 (Max)	FS 125	1	5.0	59 126	whole plant traw	<0.04 <0.04	Brandl 1993 gr6291 1144
Germany 1991 (Max)	FS 125	1	5.0	68 131	whole plant straw	<0.04 <0.04	Brandl 1993 gr6291 1144
Germany 1993 (Star)	FS 050	1	5.0	56 128	whole plant straw	<0.02 <0.02	Kühl 1994 gr6893 0546
Germany 1993 (Star)	FS 050	1	5.0	59 147	whole plant straw	<0.02 <0.02	Kühl 1994 gr6893 0546
Germany 1993 (Star)	FS 050	1	5.0	71 159	whole plant straw	<0.02 <0.02	Kühl 1994 gr6893 0546

Table 116. Fludioxonil residues in rye forage and straw from supervised trials (seed treatment) in Denmark.

Year (Variety)	Form	No.	g ai/ 100 kg seed	PHI days	Sample	Fludioxonil residue mg/kg	Author Date Study No. Syn No
1991 (Petkus II)	FS 25	1	5.00	264 277 291 305	whole plant stalks stalks straw	<0.05 <0.05 <0.05 <0.05	Mair 1993 2020/91 0333

Table 117. Fludioxonil residues in barley forage and straw after seed treatment from supervised trials in France, Switzerland, and Germany.

Country Year (variety)	Form.	No.	g ai/ 100 kg seed	PHI, days	Sample	Fludioxonil residue mg/kg	Author Date Study No. Syn No
France, North 1989 (Astrix)	FS 25	1	5.0	256	straw	<0.02	Maffezzoni 1992 05/92 0190
Germany 1992 (Gaulois)	FS 60	1	5.00	213 287	whole plant straw	<0.05 <0.05	Kühl 1995 gr 6793 0692
Germany 1993 (Sissy)	FS 60	1	5.0	79 156	whole plant straw	<0.05 <0.05	Kühl 1995 gr 6793 0692
Germany 1993 (Sissy)	FS 60	1	5.0	60 143	whole plant straw	<0.05 <0.05	Kühl 1995 gr 6793 0692
Switzerland 1989 (Cornel)	FS 25	1	5.0	115	straw	<0.05; <0.05	Altenburger 1990 2029/89 0074
Switzerland 1999 (Meltan)	FS 25	1	3.5	112	straw	<0.02; <0.02	Tribolet 2000 4000/99 6895
Switzerland 1999 (Meltan)	FS 25	1	3.5	112	straw	<0.02; <0.02	Tribolet 2000 4001/99 6896
France, North 1999 (Scarlett, Spring)	FS 425	1	2.8	48 133	whole plant straw	<0.05; n/a <0.05	Pointurier 2000 9940801 5303
France, North 1998 (Nevada)	FS 425	1	2.3	151	straw	<0.05, <0.05	Pointurier 2000 9840701 5155
France, North 1998 (Prisma)	FS 425	1	2.6	65 91 156	whole plant whole plant straw	<0.02 <0.02 <0.05, <0.05	Pointurier 2000 9840801 5157

Country Year (variety)	Form.	No.	g ai/ 100 kg seed	PHI, days	Sample	Fludioxonil residue mg/kg	Author Date Study No. Syn No
France, North 1998 (Esterel)	FS 425	1	2.9	262	straw	<0.05	Pointurier 2000 9940501 5284
France, North 1994 (Clarine)	FS 302	1	2.5	217	straw	<0.02, <0.02	Maffezzoni 1996 OS95400/SJ99 0987
France, North 1994 (Intro)	FS 30	1	2.5	217	straw	<0.02, <0.02	Maffezzoni 1996 OS95400/SJ98 0988
France, North 1994 (Express)	FS 302	1	2.5	237	straw	<0.02, <0.02	Maffezzoni 1996 OS95400/KJ02 0989
France, North KJ01 1994 (Labea)	FS 302	1	2.5	237	straw	<0.02, <0.02	Maffezzoni 1996 OS95400/KJ01 0822
France, South 1998 (Pastoral, Winter)	FS 425	1	2.9	224	straw	<0.05, <0.05	Pointurier 2000 9940601 5211
France, South 1998 (Maeva, Winter)	FS 425	1	2.9	212	straw	<0.05, <0.05	Pointurier 2000 9940602 5210
France, South 1998 (Plaisant, Winter)	FS 425	1	2.9	216	straw	<0.05	Pointurier 2000 9940603 5209
France, South 1998 (Gaelic, winter)	FS 425	1	2.8	242	straw	<0.05, <0.05	Pointurier 2000 9940604 5208
France 1999 (Scarlett, Winter)	FS 425	1	2.7	48	whole plant	<0.05, <0.05	Pointurier 2000 9940801 5303
France, South 1998 (Prisma)	FS 425	1	2.6	125	straw	<0.05, <0.05	Pointurier 2000 9840702 5156
France, South 1998 (Nevada)	FS 425	1	2.3	53 89 124	whole plant whole plant straw	<0.02 <0.02 <0.05, <0.05	Pointurier 2000 9840802 5158
France, South 1994 (Intro)	FS 302	1	2.5	208	straw	0.02, 0.02	Maffezzoni 1996 OS95400/AC98 0986
France, South 1994 (Plaisant)	FS 302	1	2.5	204	straw	0.02, 0.02	Maffezzoni 1996 OS95400/AC99 0985

Country Year (variety)	Form.	No.	g ai/ 100 kg seed	PHI, days	Sample	Fludioxonil residue mg/kg	Author Date Study No. Syn No
France, South 1994 (Pastoral)	FS 302	1	2.5	209	straw	<0.02, <0.02	Maffezzoni 1996 OS95400/LD02 0984
France, South 1994 (Plaisant)	FS 302	1	2.5	209	straw	<0.02, <0.02	Maffezzoni 1996 OS95400/LD01 0983
Germany 1999 (Krona, Spring)	FS 43.75	1	2.6	118	straw	<0.02	Smith 2000 gr 67299 6902
Germany 1999 (Krona, Spring)	FS 43.75	1	2.5	115	straw	≤0.02 <0.02	Smith 2001 gr 68499 5189
Germany 1998 (Cabria)	FS 43.75	1	2.6	285	straw	<0.02	Smith 2000 gr 63299 6900

Table 118. Fludioxonil residues in maize and sweet corn forage and fodder after seed treatment from supervised trials in France, Germany, Greece, Hungary, South Africa, Spain, and the USA.

Country Year (Variety)	Form	g ai/ 100 kg seed	Sample	PHI	Fludioxonil residue mg/kg	Author Date Study No. Syn No
USA-MS 1992 Field corn (6138X)	4FS	25	Forage Forage Silage-stage forage Fodder	30 60 106 147	<0.01, <0.01 <0.01, <0.01 ≤0.01, <0.01 ≤0.01, <0.01	Selman 1993 ABR-93030 31-92
USA-IL 1992 Field corn (G4385)	4FS	25	Forage Forage Silage-stage forage Fodder	31 60 116 166	<0.01, <0.01 <0.01, <0.01 ≤0.01, <0.01 ≤0.01, <0.01	Selman 1993 ABR-93030 31-92
USA-IA 1992 Field corn (2073X)	4FS	15	Forage Forage Silage-stage forage Fodder	32 60 124 167	<0.01, <0.01 <0.01, <0.01 ≤0.01, <0.01 ≤0.01, <0.01	Selman 1993 ABR-93030 31-92
USA-MO 1992 Field corn (2073X)	4FS	15	Forage Forage Silage-stage forage Fodder	31 61 125 179	0.01[1], 0.01[1], <0.01[1] 0.01[2], <0.01[2], <0.01[2] <0.01, <0.01 ≤0.01, <0.01 ≤0.01, <0.01	Selman 1993 ABR-93030 31-92
USA-PA 1992 Field corn (2073X)	4FS	15	Forage Forage Silage-stage forage Fodder	32 60 134 169	<0.01, <0.01 <0.01, <0.01 ≤0.01. <0.01 ≤0.01, <0.01	Selman 1993 ABR-93030 31-92
USA-FL 1992 Sweet corn (Revere)	4FS	15	Forage Forage	29 82	<0.01, <0.01 <0.01, <0.01	Selman 1993 ABR-93030 31-92
USA-WA 1992 Sweet corn (Terminator)	4FS	25	Forage Forage	30 98	<0.01, <0.01 ≤0.01, <0.01	Selman 1993 ABR-93030 31-92
USA-WI 1992	4FS	25	Forage Forage	38 97	<0.01, <0.01 ≤0.01, <0.01	Selman 1993

Country Year (Variety)	Form	g ai/ 100 kg seed	Sample	PHI	Fludioxonil residue mg/kg	Author Date Study No. Syn No
Sweet corn (Terminator)						ABR-93030 31-92
Germany 1992 (Garant)	FS 200	5.0	whole plant whole plant whole plant whole plant	58 86 118 147	0.06 0.07 <0.02 <0.02	Heyer 1994 RCC 463306 4011
Germany 1992 (Garant)	FS 200	5.0	whole plant whole plant whole plant whole plant	59 91 119 153	<0.02 <0.02 <0.02 <0.02	Heyer 1994 RCC 463307 4033
France, North 1998 (Bahia)	FS 424.6	3.4	whole plant cobs remainder (stem & leaves)	57 111 111	<0.02, <0.02 <0.02, <0.02 <0.02, <0.02	Pointurier 1999 9841301 5056
France North, 1998 (Antares)	FS 424.6	3.4	cobs remainder (stem & leaves)	105 105	0.02, 0.02 <0.02, <0.02 <0.02, <0.02	Pointurier 1999 9841601 5057
France, North 1999 (Anjou 285)	FS 424.6	2.6	cobs remainder (stem & leaves)	106 106	<0.02, <0.02 <0.05, <0.05	Pointurier 2000 9941201 5241
France, North 1997 (Antares)	FS 035	2.5	cobs remainder (stem & leaves) cobs remainder (stem & leaves)	125 125 133 133	<0.02 <0.02 <0.02 <0.02	Kühne 1998 2321/97 1082
France, North 1997 (Bahia)	FS 035	2.5	whole plant cobs remainder (stem & leaves)	119 135 135	<0.02 <0.02 <0.02	Kühne 1998 2322/97 1083
Germany 1997 (Antares)	FS 035	2.5	whole plant cobs remainder (stem & leaves) cobs remainder (stem & leaves)	70 124 124 141 141	<0.02 <0.02 <0.02 <0.02 <0.02	Smith 1998 gr 44497 1088
Germany 1997 (Antares)	FS 035	2.5	whole plant cobs remainder (stem & leaves) cobs remainder (stem & leaves)	80 118 118 142 142	<0.02 <0.02 <0.02 <0.02 <0.02	Smith 1998 gr 43197 1089
France, South 1998 (Furio)	FS 424.6	3.4	whole plant cobs remainder (stem & leaves)	63 92 92	<0.02, <0.02 <0.02, <0.02 <0.02, <0.02	Pointurier 1999 9841302 5058
France, South 1998 (Occitan)	FS 424.6	3.3	cobs remainder (stem & leaves)	92 92	<0.02, <0.02 <0.02, <0.02	Pointurier 1999 9841602 5059
France, South 1999 (Occitan)	FS 424.6	2.6	cobs remainder (stem & leaves)	86 86	<0.02, <0.02 <0.05, <0.05	Pointurier 2000 9941202 5243
France, South 1997 (Occitan)	FS 035	2.5	cobs remainder (stem & leaves) cobs	105 105 132 132	<0.02 <0.02 <0.02 <0.02	Kühne 1998 2323/97 1084

Country Year (Variety)	Form	g ai/ 100 kg seed	Sample	PHI	Fludioxonil residue mg/kg	Author Date Study No. Syn No
			remainder (stem & leaves)			
France, South 1997 (Furio)	FS 035)	2.5	cobs remainder (stem & leaves) cobs remainder (stem & leaves)	92 92 109 109	<0.02 <0.02 <0.02 <0.02	Kühne 1998 2324/97 1085
Greece 1997 (Tundra)	FS 035	2.5	cobs remainder (stem & leaves)	106 106	<0.02, <0.02 <0.02, <0.02	Kühne 1998 2306/97 1186
Italy 1997 (Tundra)	FS 035	2.5	cobs whole plant	111 111	<0.02, <0.02 <0.02, <0.02	Kühne 1998 2036/97 1087
Spain 1997 (Miguel)	FS 035	2.5	cobs remainder (stem & leaves, fodder)	112 163	<0.02, <0.02 <0.02, <0.02	Kühne 1998 2025/97 1086
Spain 1997 (Miguel)	FS 035	2.5	whole plant	142	<0.02, <0.02	Kühne 1998 2024/97 1153

Table 119. Fludioxonil residues in sorghum forage, hay, fodder, and silage after seed treatment from supervised trials in the USA.

Country, Year (Variety)	Form	No.	g ai/100 kg seed	PHI days	Sample	Fludioxonil residue mg/kg	Author Date Study No.
KA 1992 (Funks G1711)	4FS	1	16	30	Forage	<0.01, <0.01	Boyette 1993 US 33-92
				30	Hay	<0.01, <0.01	
				64	Forage	<0.01, <0.01	
				64	Hay	<0.01, <0.01	
				95	Silage	<0.01, <0.01	
				127	Fodder	<0.01, <0.01	
			26	30	Forage	<0.01, <0.01	
				30	Hay	<0.01, <0.01	
				64	Forage	<0.01, <0.01	
				64	Hay	<0.01, <0.01	
				95	Silage	<0.01, <0.01	
				127	Fodder	<0.01, <0.01	
TX 1992	4FS	1	14	31	Forage	<0.01, <0.01	Boyette 1993 US 33-92
				31	Hay	<0.01, <0.01	
				59	Forage	<0.01, <0.01	
				59	Hay	<0.01, <0.01	
				97	Silage	<0.01, <0.01	
				145	Fodder	<0.01, <0.01	
			23	31	Forage	<0.01, <0.01	
				31	Hay	<0.01, <0.01	
				59	Forage	<0.01, <0.01	
				59	Hay	<0.01, <0.01	
				97	Silage	<0.01, <0.01	
				145	Fodder	<0.01, <0.01	

Miscellaneous fodder and forage crops

Table 120. Fludioxonil residues in rape forage and straw after seed treatment from supervised trials in France, UK, Sweden, and Germany.

Country Year (variety)	Form	g ai/100 kg seed	Sample	PHI (days)	Fludioxonil residue mg/kg	Author Date Study No. Syn No
France, North 1997 (Navajo)	FS 321.3	12	Whole plant	184	<0.05	Mair 1999 1176/97 4957
France, North 1997 (Navajo)	FS 321.3	12	Whole plant	173	<0.05	Mair 1999 1177/97 4958
UK 1997 (Apex)	FS 321.3	12	Whole plant	177	<0.05, <0.05, <0.05	Mair 1999 1025/98 4970
UK 1997 (Apex)	FS 321.3	12	Whole plant	176	<0.05, <0.05, <0.05	Mair 1999 1026/98 4971
Sweden 1998 (Sponsor)	FS 321.3	12	Whole plant Straw	34 145	<0.05 <0.05	Smith 1999 gr 68098 5045
Germany 1998 (Laser)	FS 321.3	12.4	Whole plant Straw	217 330	<0.05 <0.05	Smith 2000 gr 61299 1197
Germany 1998 (Laser)	FS 321.3	12.4	Whole plant Straw	219 319	<0.05 <0.05	Smith 2000 gr 62499 1198
Germany 1999 (Evita)	FS 321.3	12.5	Whole plant Straw	56 133	<0.05 <0.05	Smith 2000 gr 71199 1199
Germany 1998 (Licosmos)	FS 321.3	12	Whole plant Straw	46 130	<0.05 <0.05	Smith 1999 gr 66298 5043
Germany 1998 (Licosmos)	FS 321.3	12	Whole plant Straw	51 124	<0.05 <0.05	Smith 1999 gr 65498 5044
France, South 1997 (Goeland)	FS 321.3	12	Whole plant	162	<0.05	Mair 1999 1178/97 4959
France, South 1997 (Bristol)	FS 321.3	12	Whole plant	176	<0.05	Mair 1999 1179/97 4960

FATE OF RESIDUES IN STORAGE AND PROCESSING

In processing

In hydrolysis experiments designed to simulate typical processing operations (Reischmann, 2000) [pyrrole-4-^{14}C]fludioxonil was incubated in aqueous buffer solutions at a concentration of 0.9 mg/l at 90°C (pH 4, 20 min.), 100°C (pH 5 60 min.) and 120°C (pH 6, 20 min.) (Table 121). Suitable aqueous buffer solutions were prepared at concentrations ≤ 0.01 M to keep the pH constant and to avoid any possible catalytic effects of the buffer.

Sterilised test solutions were heated and then allowed to cool before neutralisation. The total recovered radioactivity was measured for each test solution and the identity of the radioactive components checked by both HPLC and 2D-TLC against reference standards.

Table 121. Representative hydrolysis conditions.

Temperature (°C)	pH	Incubation time (min)	Process represented
90	4	20	Pasteurization
100	5	60	Baking, brewing, boiling
120	6	20	sterilisation

After incubation, the radioactivity in the neutralised buffer solutions represented unchanged fludioxonil (96.2–106.3% of the applied radioactivity), demonstrating that no significant hydrolytic degradation had taken place under the simulated processing conditions.

Processing studies on plums, strawberries, grapes, citrus, tomato, potato, and cotton seed were reported.

Residues are incurred in plums both as a result of foliar treatment and post-harvest applications. The latter treatments are applied to plums in the USA intended for supply directly to the retail trade, according to the manufacturer. Plums harvested after foliar treatments may be further processed however.

In a trial in Germany fludioxonil, formulated as WG 62.5, was applied to plum trees three times during the season as a foliar treatment (0.22 g ai/ha/application) at one test location. Fruit was harvested 14 days after the final treatment and residues measured in plums, washed plums, washing water, plum purée and prunes. A flow chart for processing is given in Figure 4. The results are shown in Table 122.

In two French and one Swiss trials (Maffezzoni, Report 9812203, 1999, Report 9812204, 1999; Salvi, Report 2012/00, 2002) fludioxonil, formulated as WG 62.5, was applied to plum trees as a foliar spray three times during the season at 0.15-0.16 kg ai/ha/application. The fruit were harvested 14 days after the final treatment. Residues were measured in the fresh plums and in the dried plums (prunes).

Figure 4. Flow chart for plum processing.

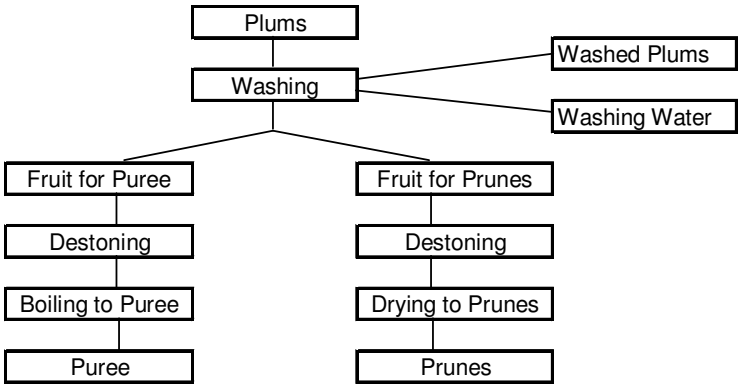


Table 122. Fludioxonil residues in plums and processed commodities from trials in France, Germany, and Switzerland.

Country Year (variety)	Application				Commodity	Fludioxonil mg/kg	Processing factor	Author Date Study No. Syn No.
	Form	kg ai/ha	water, l/ha	PHI days				
Germany 2000 (Hauszwetsche (Schäfer))	WG 62.5	0.225	1500	14	Plums Plums Washed plums Dried plums Plum jam Plum juice Plum pomace Plum preserves Plum purée	<0.02 control 0.06 0.05 0.16 0.04 0.006 0.13 0.03 0.07	 0.83 2.67 0.67 0.10 2.16 0.50 1.17	Smith, 2000 gr 90898 5205
France (South) 1998 (Plum d'Ente)	WG 62.5	0.15	1028-1047	14	Plums Dried plums	0.05* 0.09*	1.80	Maffezzoni 1999 9812203 4988
France (South) 1998 (Plum d'Ente)	WG 62.5	0.15	1000-1022	14	Plums Dried plums	0.03* 0.06*	2.00	Maffezzoni 1999 9812204 4989
Switzerland 2001 (Fellenberg)	WG 62.5	0.15	930	14	Plums Plum purée Prunes	0.036 0.016** 0.043**	 0.44 1.19	Salvi 2002 2012/00 5497
							Means: **n** Prunes 1.92 4 Jam 0.67 1 Juice 0.10	

Country Year (variety)	Application				Commodity	Fludioxonil mg/kg	Processing factor	Author Date Study No. Syn No.
	Form	kg ai/ha	water, l/ha	PHI days				
							1 Preserves 0.50 1 Purðe 0.80 2	

* mean of two determinations
** mean of four determinations

In one trial in France and three in Germany (Pointurier, Report 011101, 2001; Beinhauer, Report GR02196, 1996, Report GR02296, 1996) fludioxonil formulated as WG 62.5 was applied to protected strawberries (France) or field-grown strawberries (Germany) three times during the season at 250 g ai/ha. The fruit were harvested 3 or 7 days (France) or 10 days (Germany) after the final treatment. Residues were measured in the washed strawberries, washing water, strawberry preserves, strawberry jam and juice. Results are shown in Table 123.

Table123. Fludioxonil residues in strawberries and processed commodities from trials in France and Germany.

Country Year (Variety)	Application				Commodity	Fludioxonil, mg/kg	Processing factor	Author Date Study No. Syn No
	Form	kg ai/ha	water, l/ha	PHI days				
France (North) 2001 (Chandler)	WG 62.5	0.250	400	3	Strawberries Washed berries Washing water Preserves Jam Juice	0.19 0.09 0.07 0.08, 0.08, 0.12, 0.13 (0.10*) 0.06, 0.05, 0.07, 0.06 (0.06*) 0.03, 0.03, 0.04, 0.03 (0.03*)	 0.47 0.53 0.32 0.16	Pointurier 2002 0011101 5337
Germany 1996 (Senga-Sengana)	WG 62.5	0.250	2000	10	Strawberries Washed berries Preserves Jam	0.14, 0.13** 0.12, 0.11** 0.11, 0.10** 0.07, 0.07**	 0.86 0.79 0.50	Beinhauer 1996 gr02196 0877
Germany 1996 (Korona)	WG 62.5	0.250	2000	10	Strawberries Washed berries Preserves Jam	0.39, 0.37** 0.17, 0.17** 0.21, 0.20** 0.08, 0.08**	 0.45 0.55 0.21	Walser 1996 2188/96 0878
							Means: Washed fruit 0.59 (n = 3) Preserves 0.62 (n = 3 Jam 0.34 (n = 1) Juice 0.16 (n = 1)	

* mean of four separately processed samples

** duplicate analyses, mean used to calculate processing factors

Three grape trials in Greece were used to obtain field-incurred residues for processing grapes into raisins (Kuhne, Report 02-2111, 2003, Report 02-2112, Report 02-2113, 2003). Fludioxonil was applied to grape vines as a foliar spray, formulated as WG 62.5, twice during the season at 0.250 kg ai/ha/application. Grapes harvested 7 days after the final treatment were sun-dried on the ground for 40 days. The crop was turned over twice during the drying period. Residues measured in grapes and raisins are shown in Table 124.

Table 124. Fludioxonil residues in grapes and raisins from trials in Greece.

Year (variety)	Application				PHI days	Commodity	Fludioxonil mg/kg,	Author Date Study No. Syn No
	Form	No	g/l	kg ai/ha				
2002 (Black Corinth)	WG 62.5	2	0.250	0.250	0 7 15 7 Processing factor	Berries Berries Berries Raisin --- 1.4	0.34 0.41, 0.22 (0.32) 0.38 0.50, 0.43 (0.46) --- 1.4	Kühne 2003 02-2111 5574
2002 (Sultana)	WG 62.5	2	0.250	0.250	0 7 14 7 Transfer factor	Berries Berries Berries Raisin 1.2	0.62 0.42, 0.39 (0.40) 0.35 0.51, 0.43 (0.47) 1.2	Kühne 2003 02-2112 5575
2002 (Sultana)	WG 62.5	2	0.250	0.250	0 7 14 7 Processing factor	Berries Berries Berries Raisin 1.2	0.59 0.37, 0.36 (0.36) 0.26 0.46, 0.44 (0.45) 1.2	Kühne 2003 02-2113 5576

In twelve trials in Chile, fludioxonil formulated as WG 62.5 was applied to grape vines as a foliar spray twice during the season at 0.193-0.517 kg ai/ha (Walser, Reports 2218/95, 1996, 2119/95, 1996, 2220/95, 1996, 2221/95, 1996, 2222/95, 1996, 2223/95, 1996, 2224/95, 1996, 2225/95, 1996, 2226/95, 1996, 2227/95, 1996, 2228/95, 1996, 2229/95, 1996). The fruit were harvested 7 or 21 days after the final treatment. Grapes were processed into raisins by one of two procedures. The first was a local practice of spreading the grapes (15 kg) on cardboard in the sun for about 20 days. The mixture was turned twice each day. The other was an industrial procedure. The grapes (16 kg) were washed with 1% aqueous sodium hydroxide at 80°C, followed by a cold water rinse, then treated with "sulfur gas" for 8 hours and subsequently dried in an oven at 65°C and 35% relative humidity for 35 hours. The final moisture content was about 14%. Finally, the raisins were air-dried for five days. In all cases, juice was produced in Switzerland by pressing grapes (10 kg) in a hydraulic press. Residues were measured in grapes, juice, and raisins by HPLC (REM 133). Residues and processing factors are summarized in Table 125.

Table 125. Fludioxonil residues in grapes, raisins and juice from trials in Chile.

Year (variety)	Application				PHI Days	Commodity	Residues, mg/kg	Processing factor, raisin	Processing factor, juice	Author Date Study No. Syn No
	Formu-lation	No.	g/l	kg ai/ha						
1995 (Thompson Seedless)	WG 62.5	2	0.16 - 0.17	0.196 - 0.253	7 29 230	Berries Raisins Juice	0.27, 0.22 (0.24) 0.14, 0.13 (0.14) 0.17, 0.16 (0.16)	0.58	0.67	Walser 1996 2218/95 0840
1995 (Thompson Seedless)	WG 62.5	2	0.17 - 0.20	0.195 - 0.249	7 29 230	Berries Raisins Juice	0.21, 0.16 (0.18) 0.17, 0.15 (0,16) 0.18, 0.17 (0.18)	0.89	1.0	Walser 1996 2219/95 0841
1995 (Thompson Seedless)	WG 62.5	2	0.17 - 0.20	0.196 - 0.252	21 43 244	Berries Raisins Juice	0.21, 0.16 (0.18) 0.13, 0.11 (0.12) 0.14, 0.14 (0.14)	0.67	0.78	Walser 1996 2220/95 0842
1995 (Thompson Seedless)	WG 62.5	2	0.17 - 0.20	0.193 - 0.248	21 43 244	Berries Raisins Juice	0.16, 0.13 (0.14) 0.10, 0.09 (0.10) 0.12, 0.12 (0.12)	0.71	0.86	Walser 1996 2221/95 0843
1995 (Thompson Seedless)	WG 62.5	2	0.32 - 0.36	0.394 - 0.495	7 29 230	Berries Raisins Juice	0.29, 0.27 (0.28) 0.30, 0.27 (0.28) 0.24, 0.23 (0.24)	1.0	0.86	Walser 1996 2222/95 0844
1995 (Thompson Seedless)	WG 62.5	2	0.30 - 0.32	0.394 - 0.517	21 43 244	Berries Raisins Juice	0.32, 0.36 (0.34) 0.41, 0.39 (0.40) 0.28, 0.26 (0.27)	1.2	0.79	Walser 1996 2223/95 0845
1995 (Thompson Seedless)	WG 62.5	2	0.20 - 0.25	0.196 - 0.247	7 18 181	Berries Raisins[1] Juice	0.25, 0.32 (0.28) 0.54, 0.43 (0.48) 0.29, 0.25 (0.27)	1.7	0.96	Walser 1996 2224/95 0846
1995 (Thompson Seedless)	WG 62.5	2	0.20 - 0.23	0.200 - 0.233	21 32 195	Berries Raisins[1] Juice	0.25, 0.23 (0.24) 0.31, 0.28 (0.30) 0.16, 0.16 (0.16)	1.2	0.67	Walser 1996 2225/95 0847
1995 (Thompson Seedless)	WG 62.5	2	0.20 - 0.25	0.198 - 0.249	7 18 181	Berries Raisins[1] Juice	0.30, 0.27 (0.28) 0.34, 0.29 (0.32) 0.28, 0.19 (0.24)	1.1	0.86	Walser 1996 2226/95 0848
1995 (Thompson Seedless)	WG 62.5	2	0.20 - 0.25	0.199 - 0.244	21 32 195	Berries Raisins[1] Juice	0.33, 0.27 (0.30) 0.36, 0.31 (0.34) 0.26, 0.17 (0.22)	1.1	0.73	Walser 1996 2227/95 0849
1995 (Thompson Seedless)	WG 62.5	2	0.40 - 0.49	0.396 - 0.494	7 18 181	Berries Raisins[1] Juice	0.76, 0.76 (0.76) 0.98, 0.86 (0.92) 0.80, 0.79 (0.80)	1.2	1.0	Walser 1996 2228/95 0850
1995 (Thompson Seedless)	WG 62.5	2	0.40 - 0.49	0.398 - 0.463	21 32 195	Berries Raisins[1] Juice	0.74, 0.43 (0.58) 0.64, 0.62 (0.63) 0.64, 0.56 (0.60)	1.1	1.0	Walser 1996 2229/95 0851
							Mean	1.1	0.92	
							Mean combined with Greek processing factors (Table 124)	1.1		

[1] Commercial process.

Fifteen grape trials in Germany, Italy, Spain, and Switzerland were used to obtain field-incurred residues for processing grapes into wine (Ipach, Reports gr 51295, 1997, gr 51195, 1997; gr 51095, 1997; Lefevre, Report gr 5094, 1996; Walser, Reports 205095, 1996, 2049/95, 1996, 2066/96,

1997, 2007/96, 1997, 2008/96, 1997; Kissling, Reports 2057/94, 1995, 2058/94, 1995, 2059/94, 1995, 2109/94, 1995, 2101/94, 1995; Maffezzoni, Reports OF 94143, 1995, OF 95123/KJ46, 1996, OF95122, 1996, OF 95123/TP14, 1996, OF 95123/BY87, 1996). Fludioxonil was applied to grape vines as a foliar spray, formulated as WG 62.5, twice during the season at between 0.25 and 0.30 kg ai/ha, i.e. within 25% of the application rate of 25 g ai/ha in the typical European GAP. The fruit were harvested for processing 21 to 50 days after the final treatment.

A typical wine-making procedure was described briefly in several of the reports. The grapes were crushed and stems removed. Approximately 50 mg of SO_2 1 l was added, equivalent to 100 mg 1 l of potassium disulfide, and the crush mixture was heated to 60°C, poured into a steel vat and cooled overnight. The following day Trenolin red (20 mg/100 l) was added to assist cell splitting, and the mixture was pressed. Typically, 40 kg of grapes yielded 28 l of must. The must was allowed to stand until solids settled. Sugar was added to the separated must liquid, which was transferred to 25 l glass balloon flasks, to which were added a yeast inoculaton (5 g/100 l). The young wine was separated from the yeast and sampled. To the remaining young wine was added 100 mg SO_2/l and 2g Bentonite. After additional maturation, the wine was filtered. The temperature in the wine cellar ranged from 8 to 15°C. The entire process occurred over 1–14 months (first or young wine, about 9-120 days), with processing initiated immediately after harvest. Samples were stored frozen before analysis.

Residues were measured in grapes, juice, must, pomace, young wine, and wine. The results and processing factors are shown in Table 126**.**

Table 126. F ludioxonil residues in grapes and wine from trials in Germany, Italy, Spain, France, and Switzerland.

Country Year (variety)	Application				PHI days	Sample	Fludioxonil mg/kg or mg/l	Transfer factor	Author Year Study No. Syngenta No
	Form	No.	g/l	kg ai/ha					
Germany 1995 (Dornfelder)	WG 62.5	2	0.38	0.30	0	Berries	1.15		Ipach 1999 gr 51295 0717
					13	Berries	0.50		
					28	Berries	0.36		
					36	Berries	*0.28*		
					38	Must	0.029; 0.029	0.10	
					41	Berries	0.24		
					157	Young wine	0.005; 0.005	0.018	
					435	Wine	0.008; 0.007	0.028	
Germany 1995 (Scheurebe)	WG 62.5	2	0.38	0.30	0	Berries	0.67		Ipach 1997 gr 51195 0718
					13	Berries	0.47		
					28	Berries	0.33		
					34	Berries	*0.17*		
					35	Must	0.076; 0.067 (0.072)	0.42	
					41	Berries	0.20		
					157	Young wine	0.008; 0.007	0.044	
					435	Wine	0.008; 0.007	0.044	

Country Year (variety)	Application				PHI days	Sample	Fludioxonil	Transfer factor	Author Year Study No. Syngenta No
	Form	No.	g/l	kg ai/ha			mg/kg or mg/l		
Germany 1995 (Müller-Thurgau)	WG 62.5	2	0.38	0.30	0	Berries	0.95		Ipach 1997 gr 51095
					13	Berries	0.53		
					28	Berries	0.34		
					34	Berries	*0.31*		0719
					35	Must	0.075; 0.075	0.24	
					41	Berries	0.24		
					157	Young wine	<0.005; <0.005	0.016	
					435	Wine	0.006; 0.006	0.019	
Germany gr 52694 1994 (Kerner)	WG 62.5	2	0.38	0.30	0B	Berries	0.16		Lefevre 1996 gr 5094 0813
					0	Berries	0.62		
					14	Berries	0.36		
					29	Berries	0.25		
					34	Berries	*0.17*		
					36	Must	0.054	0.32	
					43	Berries	0.16		
					147	Young wine	0.007	0.041	
					302	Wine	0.005	0.029	
Germany gr 52794 1994 (Dornfelder)	WG 62.5	2	0.38	0.30	0B	Berries	0.27		Lefevre 1996 gr 5094 0813
					0	Berries	0.68		
					14	Berries	0.32		
					29	Berries	0.2		
					34	Berries	*0.24*		
					36	Must	0.005	0.021	
					43	Berries	0.21		
					147	Young wine	0.016	0.067	
					302	Wine	<0.005	0.021	
Switzerland 1995 (Pinot Noir)	WG 62.5	2	0.38	0.30	0B	Berries	0.14		Walser 1996 2050/95 1093
					0	Berries	3.26		
					14	Berries	1.43		
					28	Berries	1.22		
					35	Berries	*1.64*		
					35	Juice	1.84	1.1	
					42	Berries	1.0		
					44	Wine, 1st fermentation	0.18	0.11	
					210	Wine, 2nd fermentation	0.089	0.054	
Switzerland 1995 (Chasselas)	WG 62.5	2	0.38	0.30	0B	Berries	0.07		Walser 1996 2049/95 1092
					0	Berries	1.49		
					14	Berries	1.19		
					28	Berries	0.79		
					35	Berries	*0.99*		
					35	Juice	0.27	0.27	
					42	Berries	0.41		
					44	Wine, 1st fermentation	0.31	0.31	
					210	Wine, 2nd fermentation	0.011	0.011	
Switzerland 1994 (Pinot Noir)	WG 62.5	2	0.38	0.30	0	Berries	5.2		Kissling 1995 2057/94 1136
					35	Berries	1.4		
					42	Berries	1.2		
					50	Berries	*1.6*		
					50	Juice	2.0	1.2	
					61	Wine, 1st fermentation	0.1	0.062	
					222	Wine, 2nd fermentation	0.054	0.034	

Country Year (variety)	Application				PHI days	Sample	Fludioxonil mg/kg or mg/l	Transfer factor	Author Year Study No. Syngenta No
	Form	No.	g/l	kg ai/ha					
Switzerland 1994 (Chasselas)	WG 62.5	2	0.38	0.30	0 35 42 49 49 61 222	Berries Berries Berries Berries Juice Wine, 1st fermentation Wine, 2nd fermentation	3.8 0.66 0.9 *0.6* 0.24 0.2 0.022	 0.40 0.33 0.037	Kissling 1995 2058/94 1137
Switzerland 1994 (Chasselas)	WG 62.5	2	0.38	0.30	0 36 43 49 49 62 223	Berries Berries Berries Berries Juice, Wine, 1st fermentation Wine, 2nd fermentation	2.7 1.3 1.1 *1.4* 0.39 0.15 0.012	 0.28 0.11 0.0086	Kissling 1995 2059/94 1138
Italy 1996 (Moscato)	WG 62.5	2	0.36	0.25	0B 0 14 21 28 29 29 42 42 220 220	Berries Berries Berries Berries Berries Must, analysis1 Must, analysis2 Young wine, analysis1 Young wine, analysis2 Wine, analysis1 Wine, analysis2	0.1 0.88 0.27 0.31 *0.41* 0.186 0.245 0.074 0.067 0.047 0.045	 0.45 0.60 avg 0.52 0.18 0.16 avg 0.17 0.11 0.11 avg 0.11	Walser 1997 2066/96 0971
Spain 1996 (Mazuelo)	WG 62.5	2	0.30 0.50	0.25	0B 0 7 14 21 28 28 28 45 45	Berries Berries Berries Berries Berries Berries Must, specimen 1 Must, specimen 2 Wine, specimen 1 Wine, specimen 2	<0.02 0.6 0.51 0.12 0.22 *0.31* 0.245 0.229 0.071 0.066	 0.79 0.74 avg 0.76 0.23 0.22 avg 0.22	Walser 1997 2007/96 0928

Country Year (variety)	Application				PHI days	Sample	Fludioxonil mg/kg or mg/l	Transfer factor	Author Year Study No. Syngenta No
	Form	No.	g/l	kg ai/ha					
Italy 1994 (Trebbiano Romagnolo)	WG 62.5	2	0.25	0.25	0 7 14 21 21 	Berries Berries Berries Berries Must Young wine Wine	0.59 0.55 0.63 *0.43* 0.12 0.089 <0.005	 0.28 0.21 0.012	Kissling 1995 2109/94 0658
Spain 1994 (Macabeo)	WG 62.5	2	0.16	0.25	0 12 19 22 26 26 49	Berries Berries Berries Juice Berries Must Wine	1.28 0.77 *0.53* 0.59 0.56 0.55 0.12	 1.1 1.1 1.0 0.23	Kissling 1995 2101/94 1134
Spain 1996 (Macabeo)	WG 62.5	2	0.38 - 0.39	0.24 - 0.26	0B 0 7 14 21 28 28 28 71 71	Berries Berries Berries Berries Berries Berries Must, specimen 1 Must, specimen 2 Wine, specimen 1 Wine, specimen 2	0.1 1.62 1.32 0.76 0.41 *0.3* 0.640 0.685 0.070 0.072	 2.1 2.3 avg 2.2 0.23 0.24 avg 0.24	Walser 1997 2008/96 0962
France South 1994 (Cabernet Franc)	WG 62.5	1	2.0	0.30	70	Berries grape juice must wine	0.07 0.05 0.07 0.04	 0.71 1.0 0.57	Maffezzoni 1995 OF94143 0595
France South 1994 (Ugni Blanc)	WG 62.5	1	2.0	0.30	89	Berries Grape juice must wine	0.03 0.01 0.05 0.02	 0.33 1.7 0.67	Maffezzoni 1995 OF94143 0595
France 1994 (Tranpram-ilo)	WG 62.5	1	3.0	0.30	55	Berries Grape juice must wine	0.07 0.05 0.09 0.06	 0.71 1.3 0.86	Maffezzoni 1995 OF94143 0595
France 1994 (Pinot Noir)	WG 62.5	1	2.7	0.30	73	Berries Grape juice must wine	0.02 0.01 <0.01 <0.01	 0.59 0.50 0.50	Maffezzoni 1995 OF94143 0595
France, North 1996 (Gamay)	WG 62.5	1	1.5	0.30	70	Berries Wine	0.08 0.02	 0.25	Maffezzoni 1996 OF95123 0783
France, North 1995 (Gamay)	WG 62.5	1	1.5	0.30	70	Berries Wine	0.09 0.02	 0.22	Maffezzoni 1996 OF95122 0707

Country Year (variety)	Application				PHI days	Sample	Fludioxonil mg/kg or mg/l	Transfer factor	Author Year Study No. Syngenta No
	Form	No.	g/l	kg ai/ha					
France, South 1995 (Cabernet Franc)	WG 62.5	1	1.5	0.30	66	Berries Wine	0.11 0.03	0.27	Maffezzoni 1996 OF95123 0782
France, South 1995 (Carignan/ Monticola	WG 62.5	1	2.3	0.30	72	Berries Wine	0.05 0.01	0.20	Maffezzoni 1996 OF95123 0784
AVERAGE & RANGE							Wine (<100 day) Wine (>100 day)	**0.30±**0.22 0.012-0.86 **0.036±**0.028 0.0086-0.11	n = 17 n=11

0B: before final treatment
Must: pressed grape juice used for wine fermentation

Lemons treated with fludioxonil by a packing line spray with storage wax (Deco 202) at 930 g ai per 250,000 kg fruit were taken through a small batch processing operation simulating normal commercial processing (Thompson, Report 07947, 2003; see Table 64). The fruit were washed (brushwasher and spray) and surface-abraded for oil recovery (abrasion peeler and spray). The oil emulsion was treated with commercial pectinase, separated, and the oil fraction centrifuged for recovery, freeze/thawed, dried and filtered. The abraded fruit was juiced (Juice Tree juice extractor), the peels being collected and the juice finished by screening to remove peel and coarse pulp. A sample of juice was pasteurized at 93.3°C (200°F) for canning. The peel was shredded (Rietz grinder) and the solid fractions neutralised with lime, pressed and dried. Samples of unwashed fruit, juice, oil and dried pulp were frozen before analysis.

Residues are presented in Table 127.

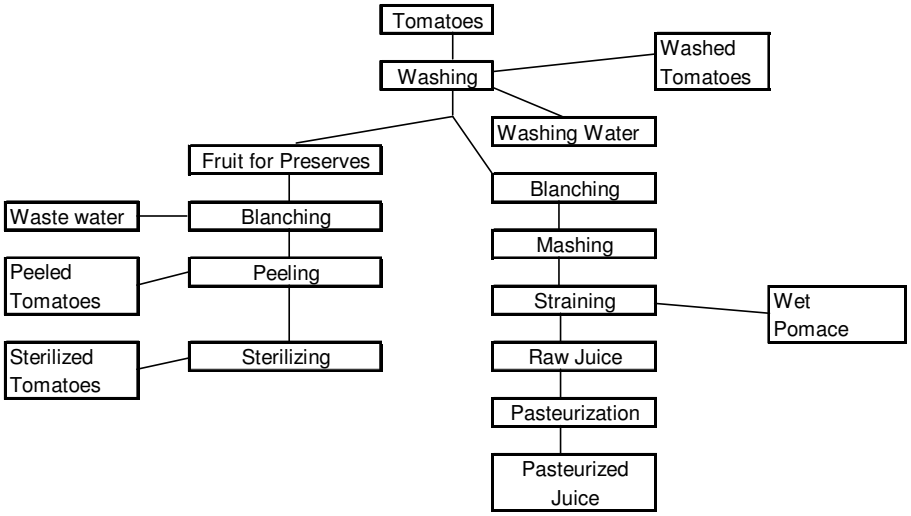
Table 127. Fludioxonil residues in lemons and processed commodities from a trial in California, USA.

Year (variety)	Form	Application rate (g as/liter)	No	PHI (days).	Commodity	Fludioxonil, mg/kg[4]	Processing factor	Author Date Study No. Syn No
Packing line spray with storage wax[1]								
2001 (Eureka)	50 WP	10.[1]	1	0	Unwashed fruit Juice Oil Pulp	0.65 <0.02 39.7 1.39	 0.031 61 2.1	Thompson 2003 IR4-07947

[1] 0.93 kg ai/250,000 kg fruit

A tomato trial in Switzerland was used to obtain field-incurred residues for processing (Tribolet, Report 2126/99, 2000). Fludioxonil formulated as WG 62.5 was applied to tomato plants as a foliar spray three times during the season at 0.250 kg/ha. The tomatoes were harvested 7 days after the final treatment. Two studies were carried out to produce juice, paste and preserves. Residues were measured in raw fruit, washed fruit, washing water, wet pomace, raw juice, pasteurised juice, raw paste, pasteurised paste, peeled fruit, washing water from peeling and preserves. Two follow-on processing studies were also carried out in which residues were measured in raw fruit, pasteurised juice, pasteurised paste and preserves. A flow diagram outlining the processing is given in Figure 5.

Figure 5. Flow chart for tomato processing.



The results from these processing studies are shown in Table 128.

Table 128. Fludioxonil residues in tomatoes and processed commodities from a trial in Switzerland.

Year (variety)	Application					PHI days.	Commodity	Fludioxonil mg/kg	Transfer factor	Author Date Study No. Syn No
	Form.	kg ai/ha	kg ai/hl	water l/ha	no.					
1999 (Petula) Study 1	WG 62.5	250	0.017	1500	3	B* 7	Tomato	<0.02		
							Tomato	0.05	1.0	
							Washed fruit	0.020	0.40	
							Washing water	0.019		
							Wet pomace	0.182	3.6	
							Raw juice	0.010		
							Pasteurized juice	0.011	0.22	
							Raw paste	0.081		
							Pasteurized paste	0.078	1.6	
							Peeled tomatoes	<0.02	0.40	
							Washing water from peeling	0.010		
							Preserves	<0.01	0.20	

Year (variety)	Application Form.	kg ai/ha	kg ai/hl	water l/ha	no.	PHI days.	Commodity	Fludioxonil mg/kg	Transfer factor	Author Date Study No. Syn No
1999 (Petula) Study 2						7	Tomato	0.05	1.0	Tribolet 2000 2126/99 5345
							Washed fruit	0.15	3.0	
							Washing water	0.023		
							Wet pomace	0.149	3.0	
							Raw juice	0.015		
							Pasteurized juice	0.011	0.22	
							Raw paste	0.076		
							Pasteurized paste	0.054	1.1	
							Peeled tomatoes	<0.02	0.40	
							Washing water from peeling	0.011		
							Preserves	<0.01	0.20	
1999 (Petula) Follow-on Study 1						7	Tomato	0.05	1.0	Tribolet 2000 2126/99 5345
							Pasteurized juice	0.012	0.24	
							Pasteurized paste	0.076	1.5	
							Preserves	<0.01	0.20	
1999 (Petula) Follow-on Study 2						7	Tomato	0.05	1.0	Tribolet 2000 2126/99 5345
							Pasteurized juice	0.010	0.20	
							Pasteurized paste	0.059	1.2	
							Preserves	<0.01	0.20	
AVERAGE/RANGE										
							Peeled tomato		0.4	n=2
							Pasteurized juice		0.22 0.20-0.24	n=4
							Pasteurized paste		1.4 1.1-1.6	n=4
							Pomace (wet)		3.3 3.0-3.6	n=2

* before treatment

A processing study on potatoes was carried out in the USA in which seed pieces were treated with fludioxonil at target rates of 5, 15 and 25 g ai/100 kg and planted at 12 sites in 11 states (Selman, Report 426006, 1996). Samples from trials in California and Idaho (PHI 99 and 143 days respectively) were used to determine residues in processed commodities. The Idaho potatoes had no residues (<0.01 mg/kg), whereas one of two samples analysed from the California trial, conducted at a threefold rate, had a value of 0.019 mg/kg; the other was <0.01 mg/kg. See Table 101. See Table 129 for results of the processing study, and Table 130 the processing factors for the animal feed items culls and peel and trimmings.

Table 129. Fludioxonil residues in potatoes and processed fractions after application as a seed treatment in the USA.

Location Year (variety)	Application Form	No.	g ai/100 kg seed	Residues mg/kg PHI days	Commodity	Fludioxonil mg/kg	Author Date Study No. Syn No

Location Year (variety)	Application Form	No.	g ai/100 kg seed	Residues mg/kg PHI days	Commodity	Fludioxonil mg/kg	Author Date Study No. Syn No
CA 1992 (Red La Soda)	DP 0.5%	1	5	99	Tubers before processing Culls Wet peel and trimmings Potatoes peeled and rinsed Potato chips Potato granules	<0.01 <0.01 0.010 <0.01 <0.01 <0.01	Selman 1996 ABR-93027 0747
CA 1992 (Red La Soda)	DP 1.5%	1	15	99	Tubers before processing Culls Wet peel and trimmings Potatoes peeled and rinsed Potato chips Potato granules	<0.01 0.022 0.016 <0.01 <0.01 <0.01	Selman 1996 ABR-93027 0747
CA 1992 (Red La Soda)	DP 2.5%	1	25	99	Tubers before processing Culls Wet peel and trimmings Potatoes peeled and rinsed Potato chips Potato granules	<0.01 <0.01 0.031 <0.01 <0.01 <0.01	Selman 1996 ABR-93027 0747
ID 1992 (Russet Burbank)	DP 0.5%	1	5	143	Tubers before processing Culls Wet peel and trimmings Potatoes sliced and peeled Potato chips Potato granules	<0.01 <0.01 <0.01 <0.01 <0.01 <0.01	Selman 1996 ABR-93027 0747
ID 1992 (Russet Burbank)	DP 1.5%	1	15	143	Tubers before processing Culls Wet peel and trimmings Potatoes sliced and peeled Potato chips Potato granules	<0.01 <0.01 0.015 <0.01 <0.01 <0.01	Selman 1996 ABR-93027 0747
ID 1992 (Russet Burbank)	DP 2.5%	1	25	143	Tubers before processing Culls Wet peel and trimmings Potatoes sliced and peeled Potato chips Potato granules	<0.01 <0.01 0.017 <0.01 <0.01 <0.01	Selman 1996 ABR-93027 0747

* 2 samples combined for processing

Residues were below the limit of quantification (<0.01 mg/kg) in tubers before processing, but were quantified in potato culls from one trial in which potatoes were seed-treated at 15 g ai/100 kg seed and in wet peel and trimmings from two trials in which potatoes were treated at 5, 15 and 25 g ai/100 kg seed. In cases where residues were measurable in processed fractions, transfer values were calculated by taking residues in tubers before processing as equal to the LOQ (0.01 mg/kg).

Table 130. Processing factors for potato processed fractions.

Commodity	Seed treatment (g ai/100 kg seed)	Fludioxonil from CA trial (mg/kg)	Fludioxonil from ID trial (mg/kg)	Processing factor (CA trial)	Processing factor (ID trial)
Tubers before processing	5 (1X)	<0.01	<0.01		
Tubers before processing	15 (3X)	<0.01	<0.01		
Tubers before processing	25 (5X)	<0.01	<0.01		
Culls	5	<0.01	<0.01	nc	nc
Culls	15	0.022	<0.01	2.2	nc
Culls	25	<0.01	<0.01	nc	nc
Wet peel and trimmings	5	0.010	<0.01	1.0	nc
Wet peel and trimmings	15	0.016	0.015	1.6	1.5

Wet peel and trimmings	25	0.031	0.017	3.1	1.7
Mean wet peel and trimmings				1.8	
Culls				<2.2	

nc: not calculable

Residues in cereal grain (wheat, barley, maize, rye, etc) harvested after seed treatment with fludioxonil at rates of 2.3–10 g ai/100 g seed were below the LOQ (0.02 or 0.04 mg/kg depending on analytical method). Some processing data are included with the field trials summarized above. However, the residues in both the raw agricultural commodity and the processed commodity, e.g. flour, were below the limit of quantification (<0.02-<0.05) so processing factors could not be calculated. It is unlikely that exaggerated treatment rates on the seeds (up to tenfold) would have yielded quantifiable residues in the crops.

A similar situation exists with the seed treatment of oilseed crops. Six supervised trials were conducted in the USA to determine the residues in dehulled cotton seed, field trash and processing and ginning fractions at harvest following use of seed treated with fludioxonil (4 FS, 48% fludioxonil w/w) at target rates of 5 and 15 g ai/100 kg seed (Vincent, Report ABR-9711, 1998). However, residues were not quantifiable in either the seed or the processed products, so processing factors could not be calculated.

Table 131. Processing of cotton seed in the USA into refined oil, meal, and hulls.

Location Year (Variety)	Form	g ai/ 100 kg seed	Sample	PHI (days)	Fludioxonil mg/kg	Author Date Study No. Syn No
CA	FS 4	14.6	Undelinted seed Hulls Meal Refined oil	189 189 189 189	<0.05 <0.05 <0.05 <0.05	Vincent 1998 ABR-97111 59-96
MS	FS 4	8.33	Field trash Gin trash Undelinted seed	152 152 152	<0.05 <0.05 <0.05	Vincent 1998 ABR-97111 59-96
TX	FS 4	12.6	Gin trash Undelinted seed Hulls Meal Refined oil	165 165 165 165 165	<0.05 <0.05 <0.05 <0.05 <0.05	Vincent 1998 ABR-97111 59-96
TX	FS 4	4.89	Field trash Gin trash Undelinted seed	132 132 132	<0.05 <0.05 <0.05	Vincent 1998 ABR-97111 59-96
OK	FS 4	5.96	Field trash Gin trash Undelinted seed	174 174 174	<0.05 <0.05 <0.05	Vincent 1998 ABR-97111 59-96
NM	FS 4	4.44	Field trash Gin trash Undelinted seed	188 188 188	<0.05 <0.05 <0.05	Vincent 1998 ABR-97111 59-96
Greece 1991 (S80)	FS 100	10.0	Hulls Seeds, dehulled	175 175	<0.02, <0.02 <0.02, <0.02	Mair 1993 2119/91 0252
Greece 1991 (S80)	FS 100	20.0	Hulls Seeds, dehulled	175 175	<0.02, <0.02 <0.02, <0.02	Mair 1993 2120/91 0253
Greece 1997 (Eva)	ES 104	2.50	Hulls Seeds, dehulled	149 149	<0.05, <0.05 <0.02, <0.02	Kühne 1999 2309/97

Location Year (Variety)	Form	g ai/ 100 kg seed	Sample	PHI (days)	Fludioxonil mg/kg	Author Date Study No. Syn No
						1673
Greece 1997 (Eva)	ES 104	2.50	Hulls Seeds, dehulled	165 165	<0.05, <0.05 <0.02, <0.02	Kühne 1999 2310/97 1674
Greece 1997 (Eva)	ES 104	2.50	Hulls Seeds, dehulled	159 159	<0.05, <0.05 <0.02, <0.02	Kühne 1999 2311/97 1675

RESIDUES IN ANIMAL COMMODITIES

Farm animal feeding studies

A ruminant feeding study was reported. No study was available on poultry feeding.

A feeding study on cows was carried out at three dosing levels equivalent to 0.55 ppm (0.017-0.019 mg/kg bw) (1x), 1.62 ppm (0.052–0.060 mg/kg bw) (3x) and 5.5 ppm (0.173–0.200 mg/kg bw) (10x) fludioxonil in the diet together with a control Holstein cow (Boyette, Reports BIOL-94016, 1996, BIOL-94010, 1996). There were 3 cows in each of the treatment groups. After acclimatization, fludioxonil was administered daily to the cows in gelatine capsules for 28-30 consecutive days. Milk samples were collected before dosing on days 0, 1, 3, 7, 14, 21 and 26 and the cows were killed on days 28-30, all within 24 hours of the final dose. Omental fat, perirenal fat, round muscle, tenderloin muscle, liver and kidney were collected. Milk and tissue samples were analysed for residues of fludioxonil and metabolites via oxidation to CGA-192155, method AG-616 (See Residue Analysis section). The LOQ for fludioxonil was 0.01 mg/kg for milk and muscle and 0.05 mg/kg for all other tissues.

The results are shown in Tables 132 and 133. Residues of fludioxonil were found in milk samples taken at days 3, 7, 14 and 21 from cows fed at the highest dose rate (5.5 ppm in the diet), the maximum residue (0.019 mg/kg) being found in the sample taken from cow 4A at 14 days. The day 14 samples also gave the highest mean residue (0.010 mg/kg). Fludioxonil residues in milk samples taken from cows fed at the 1x and 3x dose rates were all below the limit of quantification (<0.01 mg/kg).

No residues of fludioxonil were found at or above the limit of quantification (0.01 mg/kg for muscle, 0.05 mg/kg for other tissues) in any of the tissues analysed.

Table 132. Residues of fludioxonil and metabolites (converted to CGA-192155) found in milk from ruminant feeding study.

Animal number	Dose level in diet	Residues (mg/kg) at dosing (day)						
		0 (pre-dosing)	1	3	7	14	21	26
2A	1x	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
2B	0.55 ppm	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
2C		<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
3A	3x	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
3B	1.62 ppm	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
3C		<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
4A	10x	<0.01	<0.01	<0.01	<0.01	**0.019**	**0.012**	<0.01
4B	5.5 ppm	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
4C		<0.01	<0.01	**0.016**	**0.011**	**0.010**	**0.014**	<0.01

Table 133: Residues of fludioxonil and metabolites (converted to CGA-192155) found in tissues from ruminant feeding study.

Animal number	Dose level in diet	Residues (mg/kg)					
		Round muscle	Tenderloin muscle	Liver	Kidney	Perirenal fat	Omental fat
2A	1x	na	na	na	na	na	na
2B	0.55 ppm	na	na	na	na	na	na
2C		na	na	na	na	na	na
3A	3x	na	na	na	na	na	na
3B	1.62 ppm	na	na	na	na	na	na
3C		na	na	na	na	na	na
4A	10x	<0.01	<0.01	<0.05	<0.05	<0.05	<0.05
4B	5.5 ppm	<0.01	<0.01	<0.05	<0.05	<0.05	<0.05
4C		<0.01	<0.01	<0.05	<0.05	<0.05	<0.05

na: not analysed

NATIONAL MAXIMUM RESIDUE LIMITS

The various national maximum residues limits (MRLs) are listed in Table134. The information was supplied by the manufacturer.

Table 134. National MRLs.[1]

	Country	MRL (mg/kg)	Commodity	Notes
FRUIT				
Stone fruits	Switzerland	0.5		
Stone fruits	USA	5		
Apple	USA	(5)		IR-4 proposed
Apricot	Austria	(0.5)		Draft publication
Apricot	Canada	2		
Apricot	France	0.5		
Apricot	Germany	0.5		IT, German conciliation procedure
Apricot	Italy	0.5		
Apricot	Japan	0.5		
Blackberry	Slovenia	1		
Blackberry	Switzerland	1		
Bushberry	USA	2		
Caneberry	USA	5		
Cherry	Austria	(0.5)		Draft publication
Cherry	France	0.3		
Cherry	Italy	0.5		
Cherry	Japan	0.5		
Citrus	USA	(10)		IR-4 proposed
Citrus, dried pulp	USA	(20)		IR-4 proposed
Elder	Austria	2		Draft publication
Grape	Australia	2		
Grape	Austria	2		
Grape	Belgium	2		
Grape	Bolivia	2		
Grape	Bolivia	0.2	Wine	

	Country	MRL (mg/kg)	Commodity	Notes
Grape	Canada	1		
Grape	Canada	1		
Grape	Croatia	0.02		
Grape	Cyprus	0.5	Wine	
Grape	Cyprus	0.2		
Grape	France	0.5		
Grape	France	0.2	Wine	
Grape	Germany	2		
Grape	Israel	1		
Grape	Italy	2		
Grape	Italy	0.5	Wine	
Grape	Japan	5		
Grape	Lebanon	3		
Grape	Luxembourg	0.5		
Grape	Luxembourg	0.2	Wine	
Grape	Netherlands	2		
Grape	Paraguay	0.5		
Grape	Paraguay	0.2	Wine	
Grape	Portugal	2		
Grape	Portugal	0.3	Wine	
Grape	Romania	0.02		
Grape	Serbia+Montenegro	0.02		
Grape	Slovenia	0.02		
Grape	South Africa	0.05		
Grape	South Africa	0.05	Wine	
Grape	Spain	1		
Grape	Spain	1	Fruit juice	
Grape	Spain	0.2	Wine	
Grape	Switzerland	3		
Grape	Switzerland	0.5	Wine	
Grape	Taiwan	0.5		
Grape	Turkey	0.5		
Grape	Turkey	0.2	Wine	
Grape	USA	1		
Grape	Uruguay	2		
Grape	Uruguay	0.2	Wine	
Grape	Yugoslavia	2		
Juneberry	USA	2		
Kiwi	USA	(20)		IR-4 proposed
Lingonberry	USA	2		
Longan	USA	1		
Lychee	USA	1		
Mango	Taiwan	2		
Nectarine	Canada	2		
Nectarine	Japan	0.5		
Nectarine	USA	5		
Peach	Austria	0.5		Draft publication
Peach	Canada	2		
Peach	France	0.5		
Peach	Germany	0.5		IT, German conciliation procedure
Peach	Italy	0.5		
Peach	Japan	0.5		
Peach	USA	5		
Pear	Austria	0.5		Draft publication
Pear	Italy	0.5		

	Country	MRL (mg/kg)	Commodity	Notes
Pear	Spain	0.5		
Pear	USA	(5)		IR-4 proposed
Plum	Austria	(0.5)		Draft publication
Plum	Canada	2		
Plum	France	0.2		
Plum	Italy	0.5		
Plum	Japan	0.5		Including prune
Pomegranate	USA	(2)		IR-4 proposed. A time-limited tolerance exists at 5 mg/kg.
Pululan	USA	1		
Rambutan	USA	1		
Raspberry	Germany	1		
Raspberry	Slovenia	1		
Raspberry	Switzerland	1		
Raspberry	USA	2		
Spanish lime	USA	1		
Strawberry	Austria	1		
Strawberry	Belgium	0.5		
Strawberry	Bolivia	1		
Strawberry	Canada	2		
Strawberry	Finland	0.5		
Strawberry	France	1		
Strawberry	Germany	1		
Strawberry	Israel	0.5		
Strawberry	Italy	2		
Strawberry	Japan	5		
Strawberry	Korea (South)	2		
Strawberry	Luxembourg	0.5		
Strawberry	Netherlands	2		
Strawberry	Norway	0.5		
Strawberry	Paraguay	1		
Strawberry	Portugal	1		Published in Portaria 1101/99 on 21/12/99
Strawberry	Spain	1		
Strawberry	Switzerland	1		
Strawberry	USA	2		
Strawberry	Uruguay	1		
VEGETABLES				
Brassica, leafy greens	USA	10		
Bulb vegetable Group (except onion)	USA	0.02		
Cucurbit Group	Spain	0.3		
Cucurbit Group	USA	0.01		
Fruiting vegetable Group	USA	0.01		Excluding cucurbits
Leafy Vegetable Group	USA	0.01		
Legume vegetable Group (except Brassica)	USA	0.01	Foliage	
Legume vegetable Group	USA	0.01	Whole plant	
Root/tuber vegetable Group	USA	0.02	Foliage and root	
Bean	Austria	(0.5)		Draft publication
Bean	Brazil	0.04		
Bean	France	0.2		
Bean	Germany	0.2	Pod	
Bean	Japan	0.2		Including kidney bean, cow pea
Bean	Spain	0.2		

	Country	MRL (mg/kg)	Commodity	Notes
Bean	Switzerland	0.1		
Pepper (Bell pepper)	Austria	1		
Pepper (Bell pepper)	Germany	1		
Pepper (Bell pepper)	Italy	1		
Pepper	Paraguay	1		
Pepper	Spain	1		
Brassica, head and stem	USA	2		
Broad bean	Japan	0.1		
Carrot	USA	0.75		
Cucumber	Austria	0.2		Draft publication
Cucumber	Bolivia	0.5		
Cucumber	Germany	0.2		
Cucumber	Greece	0.3		
Cucumber	Italy	1		
Cucumber	Japan	2		
Cucumber	Slovenia	1		
Cucumber	Spain	0.3		
Cucumber	Switzerland	0.5		
Cucumber	Uruguay	1		
Egg plant (aubergine)	Austria	0.2		
Egg plant (aubergine)	Germany	1		
Egg plant (aubergine)	Greece	0.4		
Egg plant (aubergine)	Italy	1		
Egg plant (aubergine)	Japan	2		
Egg plant (aubergine)	Slovenia	1		
Egg plant (aubergine)	Spain	0.5		
Egg plant (aubergine)	Switzerland	0.5		
Lettuce	Austria	2		Draft publication
Lettuce	Bolivia	1		
Lettuce	France	10		
Lettuce	Italy	10		
Lettuce	Japan	1		Including Tisya and Salad-na
Lettuce	Paraguay	1		
Lettuce	Slovenia	10		
Lettuce	Spain	2		
Lettuce	Switzerland	3		
Lettuce	Uruguay	1		
Onion	Canada	0.2	Dry bulb	
Onion	Canada	7	Green	
Onion	Germany	0.3		
Onion	Switzerland	0.05		
Onion	USA	0.2	Dry bulb	
Onion	USA	7	Green	
Onion	Japan	0.1		
Pea	France	0.05		Green peas and protein peas
Pea	Germany	0.5	Pod	fresh pea with pod
Pea	Japan	0.1		
Pea	Japan	0.1		
Pea	UK	(0.05)	Seed	NCP proposed UK name: peas, pulses
Pea,	UK	(0.05)	Pea	NCP proposed: UK name: peas, green
Potato	Australia	0.02		Seed treatment
Potato	Brazil	0.02	tuber	Seed treatment
Potato	Canada	0.02		Seed treatment
Potato	Czech Republic	0.05		Seed treatment
Potato	Japan	0.02		Seed treatment
Potato	Korea (South)	0.1		Seed treatment
Potato	Russia	0.02		

	Country	MRL (mg/kg)	Commodity	Notes
Summer squash (Zucchini)	Austria	(0.2)		Draft publication
Summer squash (Zucchini)	Italy	1		
Summer squash (Zucchini)	Slovenia	1		
Tomato	Austria	(0.5)		Draft publication
Tomato	Bolivia	1		
Tomato	Greece	0.4		
Tomato	Israel	0.3		
Tomato	Italy	1		
Tomato	Japan	2		
Tomato	Paraguay	1		
Tomato	Portugal	1		Published in Portaria 1101/99 on 21/12/99
Tomato	Slovenia	1		
Tomato	Spain	0.5		
Tomato	Switzerland	0.5		
Tomato	Turkey	1		
Tomato	Uruguay	1		
Turnip greens	USA	10		
Watercress	USA	7		
OILSEED				
Cotton	Brazil	0.04		Seed treatment
Cotton	USA	0.05	By-products	Seed treatment
Cotton Group	Venezuela	0.05		Seed treatment
Cotton, undelinted seed	USA	0.05		Seed treatment
Mustard	Canada	0.05		
Peanut	Brazil	0.02	Nut	Seed treatment
Peanut	Japan	0.1		
Peanut	USA	0.01	Nut	
Rape	Canada	0.01		
Rape	Estonia	0.05		Seed treatment
Rape	USA	0.01	Seed	Seed treatment
Safflower, seed	USA	0.01		
Soya	Brazil	0.04		
Soya	Japan	0.1		
Sunflower	USA	0.01	Seed	Seed treatment
Yam	USA	(8)		IR-propsed
CEREALS				
Cereals	Austria	0.05		Seed treatment
Cereals	Belarus	0.02		Seed treatment
Cereals	Czech Republic	0.05		Seed treatment
Cereals	Japan	0.02		Seed treatment
Cereals	Russia	0.02		Seed treatment
Cereals	Switzerland	0.02		Seed treatment
Cereals	USA	0.01		Seed treatment
Cereals	USA	0.02		Seed treatment
Barley	Estonia	0.05		Seed treatment
Barley	Italy	0.05		Seed treatment
Barley	Japan	0.02		Seed treatment
Barley	Slovenia	0.02		Seed treatment
Barley	UK	0.02		Seed treatment
Buckwheat	Japan	0.02		Seed treatment
Corn, sweet	France	0.05		
Grass	USA	0.01	Foliage	
Maize	Brazil	0.04		Seed treatment
Maize	France	0.05		Seed treatment
Maize	Italy	0.05		Seed treatment
Maize	Japan	0.02		Seed treatment

	Country	MRL (mg/kg)	Commodity	Notes
Maize	Moldavia	0.02		Seed treatment
Maize	Russia	0.02		Seed treatment
Maize	Spain	0.05		Seed treatment
Maize	Venezuela	0.05		Seed treatment
Oat	UK	0.02		Seed treatment
Rice	Italy	0.05		Seed treatment
Rice	Japan	0.02		Seed treatment
Rice	Korea (South)	0.1		
Rice	Venezuela	0.05		
Rye	Denmark	0.02		Seed treatment
Rye	Japan	0.02		Seed treatment
Rye	Norway	0.1		Seed treatment
Sorghum	Venezuela	0.05		Seed treatment
Wheat	Denmark	0.02		Seed treatment
Wheat	France	0.02		Seed treatment
Wheat	Italy	0.05		Seed treatment
Wheat	Japan	0.02		Seed treatment
Wheat	Norway	0.1		Seed treatment
Wheat	Slovenia	0.2		Seed treatment
Wheat	Slovenia	0.2		Seed treatment
Wheat	UK	0.02		Seed treatment
TREE NUTS				
Peanut, meat (hulls removed)	USA	0.01		
Pistachio	USA	0.1		
HERBS AND SPICES				
Spice Group	USA	0.02		
Herb, fresh	USA	10		
Herb, dried	USA	65		
Salal	USA	2		
MAMMALIAN PRODUCTS				
Cow	Australia	0.05	Edible offal	
Cow	Australia	0.01	Meat	
Cow	Australia	0.01	Milk	
ANIMAL FEED				
Forage, fodder & straw of cereals	USA	0.01		
Forage, fodder and hay of grass	USA	0.01		
Non-grass animal feed	USA	0.01		
Peanut hay	USA	0.01		
Rape seed forage	USA	0.01		

[1]As provided by the manufacturer. Values in parentheses have not been finalized as of 06/2004.

APPRAISAL

Fludioxonil, or 4-(2,2-difluorobenzo[1,3]dioxol-4-yl)-1*H*-pyrrole-3-carbonitrile, is a fungicide that belongs to the chemical class phenylpyrroles. It functions by blocking the protein kinase which catalyses the phosphorylation of a regulatory enzyme of glycerol synthesis. It is specific for a limited number of fungi. It was evaluated for the first time by the 2004 Joint Meeting.

Metabolism

Animals

The metabolism of ^{14}C-pyrrole-labelled fludioxonil was studied in goats and laying hens. Two goats were given radiolabelled fludioxonil orally at a level equivalent to 100 ppm in the feed for 4 consecutive days. The levels of radioactive residue, calculated as fludioxonil, were: 0.07 mg/kg in tenderloin muscle, 0.19 mg/kg in fat, 5.8 mg/kg in liver, 2.9 mg/kg in kidney and 2.2 mg/kg in milk on day 4. Organic solvents released 35% of the TRR in liver, 76% in muscle, 50% in kidney, 35% in liver, 87% in fat and 90% in milk. Protease treatment of the solid residues from solvent extraction of liver, kidney and muscle released 75–91% of the remaining activity. Less than half of this released activity was characterised as proteins by derivatization with 2,4-dinitrofluorobenzene.

The main component identified in muscle was fludioxonil, representing 24% and 43% of the TRR in the two goats. Likewise, fludioxonil was the main component of the residue in omental fat, representing 83% TRR. The main identified metabolite in muscle was the sulfate conjugate of the 2-hydroxy or 5-hydroxy derivative of fludioxonil (22% or 2% TRR). Minor metabolites identified in muscle (<10% TRR) included the 2-*O*-glucuronide derivative of fludioxonil and the 5-*O*-glucuronide derivative of fludioxonil. (The position numbers refer to the pyrrole ring.) About 50% of the residue in muscle and 83% of the residue in fat were identified.

Multiple components were found in kidney and liver. The following were identified in kidney: 2-*O*-glucuronide derivative of fludioxonil (23% TRR); 7´-*O*-glucuronide derivative (8% TRR); 5-*O*-glucuronide derivative (15% TRR); fludioxonil (2% TRR); and 2- or 5-*O*-sulfate ester (0.7% TRR), for a total identification of 48%. In liver, only fludioxonil was identified (14% TRR). Two labile compounds (24% TRR) were also encountered. No compounds without the pyrrole–phenyl linkage were identified.

On the basis of the identified and characterised residues, the Meeting concluded that the metabolism of fludioxonil via the oral route in goats involves oxidation of the pyrrole ring at the 2 and 5 positions, followed by rapid conversion to sulfate and glucuronide conjugates. A minor route involves oxidation of the benzodioxol ring at the 7´ position and conversion to the glucuronide conjugate. Evidence was also found for substantial incorporation into natural products, including proteins, in kidney and liver.

Five laying hens were given gelatin capsules containing [^{14}C-pyrrole]fludioxonil for 8 consecutive days at a rate equivalent to about 89 ppm in the feed. The vast majority of the radiolabelled residue was eliminated in the excreta (88–102% of the total administered dose). The levels of radioactive residues, calculated as fludioxonil, in the tissues and eggs were as follows: liver, 8.9 mg/kg; muscle, 0.12 mg/kg; skin with fat, 0.25 mg/kg; peritoneal fat, 0.17 mg/kg; egg yolk, 1.8 mg/kg (day 7); egg white, 0.054 mg/kg (day 7).

A series of organic solvent extractions released 61% TRR in liver, 33% in kidney, 62% in muscle, 42% in skin with fat, 74% in egg white and 83% in egg yolk. The solids remaining after solvent extraction of liver (33% TRR), kidney (54%) and muscle (34%) were solubilized with protease and characterised by treatment with 2,4-dinitrofluorobenzene. Protease solubilized 54% of the unextracted activity in liver, 63% of that in kidney and 67% of that in muscle. About 25% of the released radioactivity (<10% TRR) was derivatized by 2,4-dinitrofluorobenzene at pH 2, indicating the terminal amino group of amino acids.

Alkaline hydrolysis (15% KOH, 95 ^{o}C) released all the remaining radioactivity from the solvent-extracted liver (33% TRR), but it could be characterised only as acidic, polar compounds.

About 69% of the TRR in eggs, 24% in liver, 14% in kidney, 44% in muscle and 29% in skin with fat were identified. The main metabolites identified in eggs were the sulfate conjugate of the 1-hydroxy derivative of fludioxonil (40% TRR) , the succinamic acid derivative (10% TRR) and the sulfate conjugate of the 2-hydroxy or 5-hydroxy derivative (13% TRR). Fludioxonil was a minor component (2.1% TRR) in eggs. The succinamic acid derivative was the only significant metabolite identified in liver, at about 6% TRR. The metabolites identified in kidney were the glucuronide conjugate of the 2-hydroxy or 5-hydroxy derivative (4.7% TRR), fludioxonil (2.6% TRR) and the 7´-

hydroxy derivative (2.8% TRR). The main components identified in breast muscle were fludioxonil (29% TRR) and the sulfate conjugate of the 1-hydroxy derivative. A similar situation existed for skin with attached fat, which contained fludioxonil (9.8%) and the sulfate conjugate of the 1-hydroxy derivative (14%).

On the basis of the characterisations and identifications made in the study of metabolism in hens, the Meeting concluded that metabolism in poultry involves oxidation at the C-2, C-5 and N-1 positions in the pyrrole ring and at the C-7´ of the benzodioxol ring. This is followed by the formation of sulfate or glucuronide conjugates. The C-2 hydroxypyrrole further oxidizes to the 2,5-dioxo-2,5-dihydro pyrrole and succinamic acid derivatives. The last two compounds are unique to poultry. The remaining metabolites found in the hen and all the metabolites in ruminants were also found in rats. The studies of metabolism in rats were reviewed by the WHO Expert Group of the 2004 JMPR.

Plants

The metabolism of radiolabelled fludioxonil resulting from its foliar application has been studied in grape, tomato, peach, green onion and head lettuce. Grape vines were sprayed three times at 3-week intervals with [pyrrole-4-^{14}C] fludioxonil at a rate of 500 g ai/ha per application. Samples of grapes and leaves were taken at intervals, immediately after the first application, up to grape maturity 35 days after the final application. Grapes at maturity contained 2.5–2.8 mg/kg of radiolabelled residue, calculated as fludioxonil. About 57% of the TRR was a surface residue, released by a methanol–water rinse; another 32% of the TRR was released by solvent extraction. The leaves at maturity contained 5.2 mg/kg of radiolabelled residue, 52% as a surface residue and 44% solvent extracted.

The residues in grapes and leaves were extensively identified. In grapes at maturity, seven compounds were identified, but only fludioxonil at 70% TRR exceeded 2% TRR. The metabolites included the succinamic acid derivative (<1% TRR), the 3-hydroxy succinamic acid derivative (<1% TRR), the glucose conjugate of 2-hydroxyacetamide benzodioxol (<1% TRR), 2-hydroxyacetamide benodioxol (<1% TRR), the 2-hydroxy-5-oxo derivative (2% TRR), the 2,5-dioxo derivative (<1% TRR) and the 1-hydroxy-2,5-dioxo derivative (<1% TRR). Similar metabolites were identified in leaves, fludioxonil representing 69% of the TRR; no other metabolite exceeded 6% TRR.

The metabolism of [pyrrole-4-^{14}C]fludioxonil was studied in greenhouse tomato plants that were sprayed three times at 2-week intervals with a wettable powder formulation at a single application rate of 750 g ai/ha. Forty days after the last application, leaves and tomatoes were sampled. The leaves contained a fludioxonil-equivalent radiolabelled residue level of 7.0 mg/kg, and the tomatoes contained 0.28 mg/kg. Of the residue on tomatoes, 41% was on the surface. Rinsing and solvent extraction released 95% of the residues in tomatoes and 95% of those in leaves. About 73% of the tomato residue and 69% of the leaf residue was fludioxonil. Five metabolites, representing 3.6% of the TRR in tomato, were identified. These were the same metabolites as in grapes, except that the 2,5-dioxopyrrole derivative was not found and the benzodioxole-4-carboxylic acid derivative was present but at below the LOQ (<0.001 mg/kg).

The metabolism of [phenyl-U-^{14}C]fludioxonil was studied in peaches. Three foliar applications at 30-day intervals were made at 130 and 1300 g ai/ha, starting at petal fall. Mature fruit was collected 28 days after the second treatment. In a second trial, two applications, 950 and 2860 g ai/ha, were made at a 35-day interval, starting at petal fall. Samples of immature and mature fruits were taken 30 and 114 days after the second application. The radiolabelled residue level, calculated as fludioxonil, was 0.083 mg/kg and 0.98 mg/kg in the first trial after application at 130 and 1300 g ai/ha respectively. The residue level on mature peaches in the second trial was 0.26 mg/kg (114-day PHI).

Extraction with acetonitrile:water:acetic acid (80:20:1) released $\geq$ 88% TRR in all cases. Analyses were conducted on extracts from 28-day peaches treated at 130 and 1300 g ai/ha in the first trial and on 114-day peaches from the second trial. Fludioxonil was the main component in all cases, ranging from 22% TRR to 62% TRR. Eight metabolites were identified in the 114-day PHI peaches, of which four are also grape or tomato metabolites (succinamic acid derivative, 3% TRR; 2-hydroxy-

5-oxo derivative, 1.4% TRR; 2-hydroxy-5-oxo derivative, 1% TRR; benzodioxole-4-carboxylic, 1% TRR). The other metabolites included oxidized fludioxonil glucose conjugates at 7% TRR and an oxirane-2-carboxylic acid derivative at 3% TRR. About 54% of the TRR in peach was identified.

The metabolism of [phenyl-U-^{14}C]fludioxonil on green onions was studied after radiolabelled fludioxonil was applied twice at a 14-day interval at a rate of 560 or 680 g ai/ha and at 2800 or 3380 g ai/ha. Samples were taken at maturity (14-day PHI) and at other intervals. TRR as fludioxonil represented 1.6 mg/kg on the onions given the 560 or 680 kg ai/ha treatment and 10 mg/kg on those given the 2800 or 3380 g ai/ha treatment. After the 2800 or 3380 g ai/ha treatment, 51% of the TRR was souble in organic solvents and 21% in water.

The metabolitic profiles were qualitatively similar at the two treatment levels and at the various sampling intervals. In the onions treated at 2800 or 3380 g ai/ha at mature harvest (14-day PHI), fludioxonil comprised 49% of the TRR. Six metabolites were identified, but none represented >2% TRR. These were the same metabolites identified in the studies of grape, tomato and peach metabolism.

The metabolism of [pyrrole-4-^{14}C]fludioxonil on head lettuce was studied after three foliar treatments at 10-day intervals at 200 g ai/ha. A second experiment was conducted at 600 g ai/ha per application. With a 6-day PHI, the TRR calculated as fludioxonil was 1.3 mg/kg after treatment at 200 g ai/ha and 5.8 mg/kg at 600 g ai/ha treated. Almost 100% of the radioactivity was extracted with methanol:water. Fludioxonil was the main component (68% TRR after the 200 g ai/ha treatment, 80% after the 600 g ai/ha treatment).

Six metabolites, four of which corresponded to metabolites in the studies of tomato, grape and peach, were identified. No metabolite exceeded 3% TRR. Metabolites unique to head lettuce were lactic acid conjugates of fludioxonil (1–2% TRR).

Several studies were also conducted on metabolism after seed treatment. Seed potatoes were treated with [pyrrole-4-^{14}C]fludioxonil at a rate of 2.5 g ai/100 kg seed. The pieces were planted, and mature potatoes were harvested after 95 days. The tuber contained 0.006 mg/kg radiolabelled residue, calculated as fludioxonil. Fludioxonil represented 21% of the TRR.

Rice seeds were soaked in a [pyrrole-4-^{14}C]fludioxonil solution equivalent to 6.5 kg ai/100 kg seed. Rice plants were grown in a glasshouse and harvested at maturity, 152 days after treatment. Stalks, hulls and seeds contained ≤ 0.002 mg/kg radiolabelled residue as fludioxonil equivalents.

The metabolism of [pyrrole-4-^{14}C]fludioxonil was studied in field-grown spring wheat plants treated at 7.4 g ai/100 kg seed. Plants were harvested 48 days (ear emergence), 83 days (milky stage) and 106 days (maturity) after treatment. At 48 days, stalks contained 0.005 mg/kg of radioactive residue (calculated as fludioxonil). At 83 days, stalks contained 0.004 mg/kg and ears contained 0.002 mg/kg. At maturity, stalks contained 0.015 mg/kg, husks contained 0.005 mg/kg, and grain contained 0.003 mg/kg.

Cotton-seed was treated at a rate of 2.5 or 5.0 g ai/100 kg seeds with [pyrrole-4-^{14}C]fludioxonil and then planted in sandy loam soil in pots. Plants were sampled at maturity, 186 days after treatment. Cotton-seed treated at 5.0 g ai/100 kg seed contained 0.003 mg/kg TRR, and those treated at 2.5 g ai/100 kg contained 0.012 mg/kg. Only 20–30% of the radioactivity could be extracted.

Soya bean seeds were treated with [pyrrole-4-^{14}C]fludioxonil at a rate of 5.0 g ai/100 kg seed and grown to mature plants in a greenhouse. The plants were sampled at intervals of 28 days after planting (sixth node stage), 38 days (mid- to full bloom stage) and 133 days (maturity). Soya bean forage (sixth node) contained 0.096 mg/kg TRR, calculated as fludioxonil. Soya bean hay (mid-flowering) contained 0.041 mg/kg. At maturity, stalks contained 0.005 mg/kg, dry beans contained 0.015 mg/kg, and dry hulls contained 0.012 mg/kg. The main tentatively identified component in forage and hay was 6-hydroxy-2H-chromeno[3,4-c]pyrrol-4-one, representing 2% TRR.

The metabolism of fludioxonil in and on plants after foliar and seed treatment is adequately understood. Generally, the residue concentrations resulting from seed treatment were too low to

permit extraction and identification. The numerous studies of foliar application indicate a similar metabolic pathway, showing fludioxonil as the main component of the residue.

The pathway is characterised by the generation of a large number of metabolites and proceeds mainly through oxidation. Each metabolite represents <10% TRR. With the exception of oxidation at the 7´-C of the benzoldioxol ring, the oxidations and conjugations occur at the C-2, C-5 and N-1 positions of the pyrrole ring. Ultimately, cleavage of the pyrrole ring, probably via the formation of succinamic acid derivatives, results in formation of 2,2-difluorobenzo[1,3]dioxole metabolites. In studies with pyrrole- or phenyl-labelled ^{14}C -fludioxonil, no metabolites were found, indicating cleavage of the bond between the phenyl and pyrrole ring.

No information was provided on the degradation of fludioxonil when applied post-harvest. Nevertheless, the use of both short and long PHIs in the trial on metabolism in peaches after foliar application provides some indication of the fate of fludioxonil when applied to fruit post-harvest. The study of metabolism in peach shows that the main constituent in the residue is fludioxonil.

Soil

The degradation of fludioxonil on soil exposed to light is rapid, with a half-life of <1 day for the component of fludioxonil on the surface. On the basis of isolated and identified degradates in studies of radiolabelled compound, it would appear that fludioxonil degrades to 4-(2,2-difluorobenzo[1,3]dioxol-4-yl)-2,5-dioxo-2,5-dihyrdo-1H-pyrrole-3-carbonitrile or the 2,5-dioxo derivative of fludioxonil. This metabolite undergoes epoxidation at the C-3 to C-4 position and pyrrole ring opening to give 3-carbamoyl-2-cyano-3-(2,2-difluorobenzo[1,3]dioxol-4-yl)oxirane-2-carboxylic acid. The latter degrades to 2,2-difluoro-benzo[1,3]-dioxole-4-carboxylic acid, a compound found in the studies of rotational crops (see below).

The breakdown of fludioxonil in soil under aerobic conditions with no exposure to light is slow. Mineralization to carbon dioxide is the main route of breakdown (4–45% of applied radioactivity). Some unextractable residues (8–27%) also form. The half-life in sandy loam soil is approximately 250 days.

Four studies of confined rotational crops were conducted with [pyrrole-4-^{14}C]fludioxonil. In the first study, soil was sprayed with [pyrrole-4-^{14}C]fludioxonil at a rate of 750 g ai/ha, and lettuce, winter wheat, sugar beets and maize were planted after intervals of 90, 140, 320 and 345 days respectively. Lettuce at maturity (152 days post-treatment) contained 0.006 mg/kg radiolabelled residue, winter wheat stems and grain contained 0.008 and 0.002 mg/kg 429 days post-treatment, sugar beet roots and tops contained 0.001 and <0.001 mg/kg respectively, and maize stalks and grain contained 0.005 and <0.001 mg/kg respectively, at maturity (519 days after treatment). The concentrations of residue were too low to pursue isolation and identification.

In a follow-up study, spring wheat, mustard and turnips were planted 33 days after application of [pyrrole-4-^{14}C]fludioxonil to bare ground at a rate of 120 g ai/ha. At maturity, the residue levels were <0.01 mg/kg (TRR) in the turnips and mustard greens and 0.006 mg/kg in wheat grain. In 25% mature wheat forage (109 days post-treatment) and in wheat straw (175 days post-treatment), however, the residue levels were 0.058 and 0.12 mg/kg respectively. The following components were identified in immature wheat forage: fludioxonil (2.4% TRR, 0.001 mg/kg), 6-hydroxy-2H-chromeno[3,4-c]pyrrol-4-one (11% TRR, 0.006 mg/kg, tentative identification), 4-hydroxy-2,5-dione derivative (4.2% TRR, 0.002 mg/kg), 2,5-dioxo derivative (<0.001 mg/kg, tentative identification), 2,2-difluoro-benzo[1,3]dioxole-4-carboxylic acid (2.3% TRR, 0.001 mg/kg, tentative identification) and 2-(2,2-difluorobenzo[1,3]dioxol-4-yl)-2-hydroxyacetamide (<0.001 mg/kg, tentative identification). Fludioxonil (<0.001 mg/kg) and similar metabolites at similar concentrations were detected in wheat straw. This work was confirmed by another experiment conducted at 60 g ai/ha.

In a final trial, [phenyl-U-^{14}C]fludioxonil was sprayed onto bare ground at a rate of 1120 g ai/ha. Rotational crops of spring wheat, mustard and radishes were planted 30, 90 and 210 days after treatment and grown to normal maturity. Radish tubers contained 0.14, 0.019 and 0.019 mg/kg of radiolabelled residue at plant-back intervals of 30, 90 and 210 days, about 50% of which could be extracted with organic solvents and water. Mustard greens contained 0.033, 0.044 and 0.050 mg/kg at

30, 90 and 120 days after treatment. Mature wheat straw contained 0.36, 0.14 and 0.11 mg/kg radiolabelled residues, and grain contained 0.058, 0.021 and 0.019 at 30, 90 and 120 days plant-back, of which about 40% from straw and 20% from grain was extractable.

The main metabolite identified in the various commodities was 2,2-difluorobenzo[1,3]dioxole-4-carboxylic acid, at levels ranging from 4.4% TRR in mature wheat straw (30-day plant-back) to 38% TRR (radish tuber, 90-day plant-back). Fludioxonil generally represented <4% TRR (≤ 0.001 mg/kg) in all matrices except mature radish tuber (30-day plant-back), in which it represented 12% TRR or 0.016 mg/kg.

Field rotational crop studies were conducted in which fludioxonil was applied four times to bare soil at 280 g ai/ha per application, followed at plant-back intervals of 30, 90, 150 and 210 days by sowing of wheat, turnips and leaf lettuce. The mature crops contained no detectable residues of fludioxonil at any plant-back interval, with a LOQ of 0.01 mg/kg.

The nature and extent of the residue in rotational crops after use of fludioxonil on the primary crop is adequately delineated. Similar patterns were observed with pyrrole- and phenyl-labelled ^{14}C - fludioxonil, although somewhat greater concentrations of residue were encountered with the phenyl label. In these trials, fludioxonil was not taken up into rotational crops at plant-back intervals as short as 30 days. The metabolism of fludioxonil in the crops was apparently the same as that seen in target crop studies, but this conclusion is speculative as little or no residue was generally found. Primarily on the basis of the confined study with [phenyl-U-^{14}C]fludioxonil, the metabolism and degradation of this compound is characterised by oxidation and cleavage of the pyrrole ring. No metabolites of cleavage of the bond between the phenyl and the pyrrole ring were observed. The proposed metabolic and degradation pathway is that suggested for foliar application of fludioxonil.

The Meeting concluded that the presence of fludioxonil residues in succeeding (rotational) crops from foliar applications is unlikely.

Methods of analysis

The Committee concluded that adequate analytical methods exist for both monitoring and enforcing MRLs and for gathering data in supervised field trials and processing studies. Methods REM-133/AG631A and AG-597 are suitable for the determination of fludioxonil in samples of plant origin. The methods are fully validated for a range of crops and crop types. In addition, fludioxonil residues can be determined in samples of plant origin by European multi-residue method DG S17.

Method REM-133 involves HPLC with ultraviolet detection (268 nm). Only fludioxonil is determined. Samples are extracted and then placed on a phenyl solid-phase extraction cartridge and eluted with the appropriate solvent. The samples are analysed by HPLC with column switching (C-18 and phenyl). The validated LOQ is 0.01–0.04 mg/kg. In some European field trials, method REM 133 was modified by the use of only one HPLC column (amino) with a fluorescence detector (excitation, 265 nm; emission, 312 nm). The method was radiovalidated. In this method, 89% of the total radioactivity was solubilized, and 66% of the fludioxonil determined in the metabolism study was identified.

Method AG-597 is another HPLC method with ultraviolet detection (268 nm). Only fludioxonil is determined. Samples are extracted and then cleaned up by silica solid-phase extraction. Analysis is usually conducted on an amino or a C18 column. The method was validated with a wide array of commodities, with limits of determination of 0.01–0.02 mg/kg, except for sorghum grain, for which the limit was 0.05 mg/kg. The method was validated by the US Environmental Protection Agency. Liquid chromatography with mass spectrometry can be used for confirmation, with quantification on ion 247.

A European multi-residue method based on DFG S19 was developed for an array of plant commodities. Extracts are separated by gel permeation chromatography and analysed by capillary gas chromatography with a mass selective detector, monitoring ions 248, 154 and 127. The method was

validated for fludioxonil only at 0.02 mg/kg for tomato, orange, wheat and rape and at 0.01 mg/kg for grape wine.

The Meeting concluded that an adequate method exists for the determination of fludioxonil and certain metabolites in livestock commodities (meat, milk, poultry, eggs). In the HPLC method, fludioxonil and metabolites are converted to 2,2-difluoro-1,3-benzodioxole-4-carboxylic acid. The resulting residue is quantified by external calibration against standards of this conversion product, with HPLC and a ultraviolet detector (230 nm). Column switching is used, and alternate columns are specified as a confirmatory procedure. The method was validated at 0.01 mg/kg for muscle and milk and at 0.05 mg/kg for eggs, fat, liver and kidney.

Stability of residues in stored analytical samples

The Meeting concluded that fludioxonil is stable in an array of stored frozen commodities. No degradation of fludioxonil was observed in any frozen commodity throughout the duration of the studies. Fludioxonil is stable for at least 24 months in frozen samples of the following commodities: cereal grains, cereal straw, apple, tomato, grape, pea, rape-seed, maize grain, maize meal, sorghum hay, potato tuber and potato flake. Fludioxonil is stable for at least 12 months in frozen broccoli, cabbage and carrots and for 9 months in frozen chives. Fludioxonil is also stable for at least 3 months in frozen peach, plum, cherry and blueberry.

The Meeting also concluded that fludioxonil and metabolites, determined as 2,2-difluoro-1,3-benzodioxole-4-carboxylic acid, are stable for at least 12 months in frozen muscle and for at least 18 months in frozen liver, milk and eggs.

Definition of the residue

The results of the studies of metabolism after both seed treatment and foliar treatment show that the main identified component of the radiolabelled residue is fludioxonil. The identified metabolites generally represent <10% of the TRR. The toxicological evaluation did not reveal any metabolites of special concern relative to the parent. The Meeting concluded that the residue definition for plant commodities for compliance with MRLs and for estimation of dietary intake is fludioxonil.

In the analytical methods for plant commodities, HPLC with ultraviolet detection or gas chromatography with mass spectrometry detection, only fludioxonil is determined.

The results of the studies of metabolism in goats and hens were similar. In goats, the main identified metabolite in meat, fat and liver was fludioxonil, representing 33%, 83% and 14% TRR respectively. The main metabolite in milk and kidney was the pyrrole carbonitrile-*O*-glucuronide, representing 65% and 31% TRR respectively, and the parent was absent. In hens, fludioxonil was present in muscle (7.9–28% TRR) and skin plus attached fat (9.8%). It accounted for 1.2% of the TRR in liver, 2.6% in kidney and 2.2% in egg yolk (equivalent to 2.1% egg TRR). The main identified component of the radioactive residue in eggs and fat was the sulfate conjugate of 4-(2,2-difluorobenzo[1,3]dioxol-4-yl)-1-hydroxy-1*H*-pyrrole-3-carbonitrile. The benzene–pyrrole linkage was intact in all the identified metabolites. The toxicological evaluation did not reveal any metabolites of particular concern relative to the parent.

The P_{ow} for fludioxonil is 4.1, suggesting that fludioxonil is fat-soluble. In goats, the radioactive residue represented 0.07 mg/kg TRR in muscle and 0.26 mg/kg in fat. The main component in muscle and fat was fludioxonil (24–43% TRR in muscle and 83% in fat). The Meeting concluded that the fludioxonil residue is fat-soluble, but it also noted the lack of information on milk fat from both the metabolism and the feeding study.

In the validated analytical method for fludioxonil, fludioxonil and pyrrole-derivative metabolites are converted to 2,2-difluorobenzo[1,3]dioxole-4-carboxylic acid.

The Meeting concluded that the residue definition of the residue for livestock commodities (for compliance with MRLs and for estimation of dietary intake) is the sum of fludioxonil and its

benzopyrrole metabolites, determined as 2,2-difluoro-benzo[1,3]dioxole-4-carboxylic acid and expressed as fludioxonil.

Results of supervised trials on crops

Supervised trials were conducted with foliar treatment, seed treatment and post-harvest treatment of a variety of crops worldwide.

Citrus fruit

Citrus (orange, lemon, grapefruit) was treated by post-harvest dip (120 g ai/hl) or spray (1000 g ai/250 000 kg fruit) in 28 trials conducted in the USA. GAP specifies a maximum of two treatments, one on entering storage and a second on exit of storage for market distribution, at a single application rate of 500 g ai/250 000 kg fruit (2 mg/kg; 0.85 kg ai/hl for droplet-type applications with a low-volume concentrate, 0.24 kg ai/hl for high-volume jet-type sprays) and 0.06 kg ai/hl for 30-s dip treatments. All trials were conducted at twice GAP in a single-application dip or high-volume spray, and nine of the trials included a second application at twice GAP with a re-treatment interval of 0 days. In the absence of data on residue level decrease during storage of citrus, the Meeting considered application at twice GAP an approximation of the practical situation of two treatments at GAP with a variable interval between applications.

The residue levels on orange (six trials; one treatment at twice GAP), in ranked order, were: 0.48, 0.90, 1, 1.4, 2.2 and 2.8 mg/kg. The levels on lemon (seven trials; one treatment at twice GAP) were: 0.46, 0.54, 1., 1.1 (two), 2.9 and 3.2 mg/kg, and those on grapefruit (six trials; one treatment at twice GAP) were: 0.51, 0.94, 0.95, 1.4, 3.8 and 5.2 mg/kg. The nine trials consisting of two sequential applications, each at twice the GAP application rate, were considered exaggerations and were not used; the residue levels ranged from 0.52 mg/kg to 6.0 mg/kg.

The Meeting decided to combine the data; the residue levels on citrus (19 trials; one treatment at twice GAP single rate), in ranked order, were: 0.46, 0.48, 0.51, 0.54, 0.90, 0.94, 0.95, 1 (two), 1.1 (two), 1.4 (two), 2.2, 2.8, 2.9, 3.2, 3.8 and 5.2 mg/kg. Data on residues in pulp were available from only one trial on oranges, in which flesh and peel contained approximately equal concentrations of fludioxonil. The Meeting estimated a maximum residue level for whole citrus of 7 mg/kg and an STMR of 1.1 mg/kg.

Pome fruit

Apples were treated by post-harvest dip or spray in the USA with a 50% wettable powder formulation. GAP specifies a maximum of two treatments, one on entering storage and a second on exit from storage for market distribution, at a single application rate of 500 g ai/250 000 kg fruit (2 mg/kg; 0.85 kg ai/hl for droplet-type applications with a low-volume concentrate, 0.24 kg ai/hl for high-volume jet-type sprays) and 0.06 kg ai/hl for dip treatments of approximately 30 s. Seven trials were conducted at approximately the GAP rate (single application), and two trials were conducted at the GAP rate with two sequential applications: dip at 0.06 kg ai/hl, followed by packing-line spray at 2.5 mg/kg (125% GAP). As GAP specifies two treatments, the Meeting regarded the two trials conducted with two applications as an approximation of GAP. The residue levels were 2.0 and 2.2 mg/kg. The Meeting considered two trials inadequate for estimating a maximum residue level.

Pears were treated by post-harvest dip or spray treatment in the USA with a 50% wettable powder formulation. GAP is identical to that for apples. Twelve trials were conducted, but only two were conducted with two applications: 0.048 kg ai/hl drench (80% dip GAP), followed by a packing-line spray at 0.2–0.6 kg ai/hl or 2.2–6.6 mg/kg fruit (110–300% GAP). As GAP specifies two treatments, the Meeting regarded the two trials conducted with two applications as an approximation of GAP. The residue levels (with an exaggerated rate for the second application) were 1.6 and 2.8 mg/kg. The Meeting considered two trials insufficient for estimating a maximum residue level.

The Meeting considered combining the post-harvest trials on pear and apple (same GAP) for mutual support, but considered four trials insufficient for these commodities.

Pears received foliar treatment with a 25% water-dispersible granule formulation in seven trials conducted at GAP (three in Italy , three in Spain and one in France). The GAPs are as follows: Italy, 0.02 kg ai/hl, 0.25 kg ai/ha, three applications, 14-day PHI; Spain, 0.025 kg ai/ha, 0.25 kg ai/ha, three applications, 7-day PHI. No GAP was available for France, and the GAP of Spain was applied to all trials (7-day PHI). The residue levels, in ranked order, were: 0.14, 0.15, 0.18, 0.21, 0.28, 0.32 and 0.36 mg/kg. The Meeting estimated a maximum residue level of 0.7 mg/kg and an STMR of 0.21 mg/kg.

Stone fruit

In seven post-harvest treatment trials (spray or dip), *peaches* were treated at the GAP of 0.06 kg ai/hl with a 50% wettable powder formulation. The residue levels on peaches after treatment (no storage interval), in ranked order, were: 0.37, 0.42, 1.6, 2.2, 2.8, 3.4 and 3.6 mg/kg

Trials of foliar application of fludioxonil (62.5% water-dispersible granules, 25% fludioxonil) were conducted in France, Italy and Spain. The relevant GAPs are: France, 0.015 kg ai/hl, 14-day PHI; Italy, 0.015 kg ai/hl, 0.25 kg ai/ha, two applications, 14-day PHI. No GAP was available for Spain, and the GAP of Italy was applied. The residue levels in 11 trials at GAP, in ranked order, were: 0.02, 0.04 (two), 0.08 (two), 0.11, 0.23 (two), 0.29 and 0.33 mg/kg. The data set on post-harvest treatment contained the highest residue values and was used to estimate the maximum residue level and the STMR.

Post-harvest treatment of *plums* was investigated in two trials in the USA. GAP is spray application at 0.06 kg ai/hl of a 50% wettable powder formulation. The results were 0.10 and 0.92 mg/kg. As the results for post-harvest treatment of plums were not statistically significantly different from those for peaches with the same GAP, the populations can be combined for mutual support.

Trials of foliar application of fludioxonil (22.5% water-dispersible granules, 25% fludioxonil) to plums were conducted in France, Italy, Germany and Switzerland. The relevant GAPs are: France, 0.012 kg ai/hl, 0.12 kg ai/ha, three applications, 14-day PHI; Italy, 0.025 kg ai/hl, 0.25 kg ai/ha, two applications, 14-day PHI; Switzerland, 0.3 kg ai/ha, two applications, PHI not specified. GAP in Germany was not available, and the GAP of Italy was applied. In 12 trials at GAP, the residue levels, in ranked order, were: <0.02, 0.03, 0.04, 0.05, 0.06 (two), 0.065, 0.07, 0.09, 0.10, 0.11 and 0.17 mg/kg. The data set on post-harvest treatment contained the highest residue values and was used to estimate the maximum residue level and the STMR.

Post-harvest treatment of *cherries* was investigated in two trials in the USA. GAP is spray application at 0.06 kg ai/hl of a 50% wettable powder formulation. The reside levels were 0.19 and 0.68 mg/kg. As the results for post-harvest treatment of cherries were not statistically significantly different from those for peaches, with the same GAP, the populations can be combined for mutual support.

A 25% water-dispersible granule formulation of fludioxonil was applied as a foliar spray to cherries in Europe. In six trials, the residue levels ranged from 0.16 to 0.43 mg/kg after a treatment rate of 0.019 kg ai/hl and a PHI of 7 days. No GAP was provided for any country in Europe.

The results for post-harvest treatment (GAP, dip or spray at 0.06 kg ai/hl) of peaches, plums and cherries were combined. The residue levels in the 11 trials, in ranked order, were: 0.10, 0.19, 0.37, 0.42, 0.68, 0.92, 1.6, 2.2, 2.8, 3.4 and 3.6 mg/kg. The Meeting estimated a maximum residue level of 5 mg/kg and an STMR of 0.80 mg/kg for stone fruit.

Berries and other small fruit

Grape

Trials on foliar treatment of grape vines were available from Chile, France, Germany, Greece, Italy , South Africa, Spain and Switzerland. The relevant GAPs (25% water-dispersible granules) are: Chile, 0.25 kg ai/ha, two applications, 7-day PHI; France, 0.3 kg ai/ha, two applications, 60-day PHI; Germany, 0.015 kg ai/hl, 0.24 kg ai/ha, two applications, 35-day PHI; Italy, 0.02 kg ai/hl, 0.2 kg ai/ha, two applications, 21-day PHI; Spain, 0.25 kg ai/ha, two applications, 21-day PHI; Switzerland, 0.3 kg

ai/ha, one application, early season. The residue values at the GAP of Chile, in ranked order, were: 0.18, 0.24 and 0.28 (two) mg/kg. The trials in France (northern), Germany and Switzerland were evaluated against the GAP of Germany, resulting in six trials in Germany (0.17, 0.20, 0.21, 0.24, 0.28, 0.31 mg/kg) and five trials in Switzerland (0.90, 0.99, 1.4, 1.6 (two) mg/kg) at GAP and combined: 0.17, 0.20, 0.21, 0.24, 0.28, 0.31, 0.90, 0.99, 1.4 and 1.6 (two) mg/kg. The GAP of Spain was used to evaluate the trials in Greece, Italy and Spain. The residue levels in two trials in Spain and one in Italy at this GAP were 0.22, 0.41 and 0.43 mg/kg. The Meeting combined the 18 values for Chile, Germany and Switzerland, Spain and Italy (same population) and found a ranked order of: 0.17, 0.18, 0.20, 0.21, 0.22, 0.24 (two), 0.28 (three), 0.31, 0.41, 0.43, 0.90, 0.99, 1.4 and 1.6 (two) mg/kg. The Meeting estimated a maximum residue level of 2 mg/kg and an STMR of 0.28 mg/kg.

Strawberry

Foliar applications of a 50% wettable powder formulation were made to strawberries in the USA, and of a 25% water-dispersible granule formulation in Europe (glasshouse and outdoor). The relevant GAPs are: France, 0.25 kg ai/ha, one application, 3-day PHI; Germany, 0.125 kg ai/hl, 0.25 kg ai/ha, three applications, 7-day PHI; Italy, 0.02 kg ai/hl, 0.2 kg ai/ha, three applications, 7-day PHI; Spain, 0.25 kg ai/ha, three applications, 7-day PHI; Switzerland, 0.025 kg ai/hl, 0.3 kg ai/ha, two applications, 14-day PHI; USA, 0.25 kg ai/ha, four applications, 0-day PHI. The values from the eight trials in the USA in ranked order were: 0.22, 0.43, 0.54, 0.62, 1.0, 1.2 (two) and 1.3 mg/kg. At GAP of Spain and Germany (0.25 kg ai/ha, three applications, 7-day PHI), the values from outdoor trials in Germany were 0.04 and 0.05 (two) mg/kg; those in Switzerland were 0.13 (two) mg/kg; those in France were 0.09, 0.25, 0.61 and 0.77 mg/kg; that in Italy was 0.14 mg/kg; those in Spain were 0.64 and 0.83 mg/kg; and that in the UK was 0.11 mg/kg. These 13 values may be combined: 0.04, 0.05 (two), 0.09, 0.11, 0.13 (two), 0.14, 0.25, 0.61, 0.64, 0.77 and 0.83 mg/kg. When the European and US populations were combined, the residue levels, in ranked order, were: 0.04, 0.05 (two), 0.09, 0.11, 0.13 (two), 0.14, 0.22, 0.25, 0.43, 0.54, 0.61, 0.62, 0.64, 0.77, 0.83, 1.0, 1.2 (two) and 1.3 mg/kg.

Indoor trials were also conducted in France, Italy, Spain and Switzerland. The ranked order of residue values evaluated against the GAP of Italy was: 0.11, 0.21, 0.27 and 1.9 mg/kg.

When the results of the indoor and outdoor trials were combined, the residue levels in the 25 trials, in ranked order, were: 0.04, 0.05 (two), 0.09, 0.11 (two), 0.13 (two), 0.14, 0.21, 0.22, 0.25, 0.27, 0.43, 0.54, 0.61, 0.62, 0.64, 0.77, 0.83, 1.0, 1.2 (two), 1.3 and 1.9 mg/kg. The Meeting estimated a maximum residue level of 3 mg/kg and an STMR of 0.27 mg/kg.

Raspberry

Foliar applications of a 25% water-dispersible granule formulation of fludioxonil were made to raspberries in Germany and the USA. The relevant GAPs are: Switzerland, 0.025 kg ai/hl, 0.32 kg ai/ha, two applications, 14-day PHI; and USA, 0.25 kg ai/ha, four applications, 0-day PHI. The residue levels, in ranked order, were: 0.19, 0.24 (two) and 0.30 mg/kg in Germany and 0.96, 1.0 (three) and 3.6 mg/kg in the USA.

The Meeting estimated a maximum residue level of 5 mg/kg and an STMR of 1.0 mg/kg for raspberries and extrapolated the values to blackberry and dewberry on the basis of the trials in the USA, which had the highest values.

Blueberry

Foliar applications of a 25% water-dispersible granule formulation of fludioxonil were made to blueberries in Germany and the USA. The relevant GAP is: USA, 0.25 kg ai/ha, four applications, 0-day PHI. No GAP was available for Germany or other European countries. The residue levels in ranked order at GAP in the USÂ were: <0.05, 0.14, 0.26, 0.52, 0.68, 0.84, 0.90 and 1.4 mg/kg. The Meeting estimated a maximum residue level of 2 mg/kg and an STMR of 0.60 mg/kg.

Black and red currant

Foliar application of a 25% water-dispersible granule formulation of fludioxonil was made to black currants in four trials and to red currants in one trial in Germany. As no GAP is available for Germany or other European countries, the Meeting could not estimate an STMR or maximum residue level.

Assorted tropical andsubtropical fruits

Lychee

Fludioxonil (25% water-dispersible granules) was applied as a foliar spray to lychee in the USA, where GAP is: 0.25 kg ai/ha, four applications, 0-day PHI. The residue levels in ranked order were: 0.81, 0.92 and 1.4 mg/kg. The Meeting noted that five or seven applications were made at about 7-day intervals and that the extra one or three applications would have been made ≤ 21 days before harvest. On the basis of studies of decline in other fruit crops, they might have made a significant contribution (about 25%) to the final residue level. Therefore, the Meeting did not estimate a maximum residue level or an STMR.

Kiwi

Kiwi fruit in the USA were treated post-harvest at 0.06 kg ai/hl with a wettable powder formulation. GAP specifies application of a 50% wettable powder formulation as a dip at 0.06 kg ai/hl for 30 s or as a low-volume application with a control droplet-type application at 0.24 kg ai/hl or 2.5 mg/kg fruit. Trials were conducted, with two methods (dip, spray) at two locations and a single method (dip) at a third. The ranked order of residue levels in the five trials was: 1.6, 5.2, 7.2, 8.6 and 9.0 mg/kg. The Meeting estimated mg/kg a maximum residue level of 15 mg/kg and an STMR of 7.2 mg/kg for kiwi fruit.

Pomegranate

Pomegranate in the USA were treated post-harvest at 0.06 kg ai/hl with a wettable powder formulation. The residue levels were 0.65 and 0.95 mg/kg; however, there is no GAP, and the Meeting could not estimate a maximum residue level or an STMR.

Bulb vegetables

Green (spring) onions

Fludioxonil was applied as a foliar spray of a wettable powder formulation to green onions in the USA. The relevant GAP is 0.25 kg ai/ha, four applications, 7-day PHI. The residue levels in ranked order were 0.14, 0.59 and 3.0 mg/kg. The Meeting estimated a maximum residue level of 5 mg/kg and an STMR of 0.59 mg/kg.

Bulb onion

Fludioxonil (wettable powder formulation) was applied as a foliar spray to onions in France, Italy, Germany and Switzerland and in the USA. The relevant GAPs are: Austria, 0.25 kg ai/ha, three applications, 7-day PHI; Switzerland, 0.25 kg ai/ha, two applications, unspecified PHI; and USA, 0.25 kg ai/ha, four applications, 7-day PHI. GAP in Switzerland (assumed 0-day PHI) was applied to the other European countries in the absence of a GAP for southern Europe. The residue levels in trials on bulb onions (fresh) at the Swiss GAP were: France, <0.02, 0.05 and 0.06 mg/kg; and Italy, <0.02, 0.04, 0.07 and 0.34 mg/kg. The levels in three trials on bulb onions (dry) in the USA at US GAP were: <0.02 (three), 0.04 (two) and 0.06 mg/kg. The Meeting combined the data sets for Europe and the USA and found a ranked order of residue levels of: <0.02 (five), 0.04 (three), 0.05, 0.06 (two), 0.07 and 0.34 mg/kg. The Meeting estimated a maximum residue level of 0.5 mg/kg and an STMR of 0.04 mg/kg.

Brassica vegetables

Broccoli

Fludioxonil (water-dispersible granule formulation) was applied as a foliar spray to broccoli in Canada and the USA. The relevant GAP is: 0.25 kg ai/ha, four applications, 7-day PHI. The residue levels in seven trials at US GAP, in ranked order, were: 0.07, 0.10, 0.18, 0.23, 0.26, 0.34 and 0.36 mg/kg. The Meeting estimated a maximum residue level of 0.7 mg/kg and an STMR of 0.23 mg/kg.

Cabbage

Fludioxonil (water-dispersible granule formulation) was applied as a foliar spray to cabbage in the USA. The relevant GAP is: 0.25 kg ai/ha, four applications, 7-day PHI. The residue levels in ranked order on cabbage with wrapper leaves in six trials at GAP were: 0.17, 0.17, 0.21, 0.27, 0.5 and 1.2 mg/kg. The Meeting estimated a maximum residue level of 2 mg/kg and an STMR of 0.24 mg/kg.

Fruiting vegetables

Cucumber

A 25% water-dispersible granule formulation of fludioxonil was applied as a foliar spray (glasshouse and field) to cucumbers in Greece, Spain and Switzerland. The relevant GAPs are: Italy, 0.02 kg ai/hl, 0.20 kg ai/ha, three applications, 7-day PHI; Spain, 0.025 kg ai/hl, three applications, 7-day PHI; Switzerland, 0.025 kg ai/hl, 3-day PHI. GAP for Greece was not available, and that of of Italy and Spain was used. The results from the 10 glasshouse trials (seven in Spain, one in Greece, two in Switzerland) in ranked order were: <0.02, 0.02 (two), 0.06 (two), 0.07, 0.08 (two), 0.11 and 0.14 mg/kg. The results from the field trials (one in Greece, two in Spain) were: <0.02, 0.02 and 0.03 mg/kg. The populations are not statistically significantly different, and the combined results are: <0.02 (two), 0.02 (three), 0.03, 0.06 (two), 0.07, 0.08 (two), 0.11 and 0.14 mg/kg. The Meeting estimated a maximum residue level of 0.3 mg/kg and an STMR of 0.06 mg/kg.

Summer squash (zucchini)

Two indoor trials were conducted on zucchini in Italy. The relevant GAP is: 25% water-dispersible granule, 0.02 kg ai/hl, 0.20 kg ai/ha, three applications, 7-day PHI. The residue levels were 0.05 and 0.06 mg/kg. The Meeting agreed to use the results for cucumber as support for summer squash. The residue levels in ranked order were: <0.02 (two), 0.02 (three), 0.03, 0.05, 0.06 (three), 0.07, 0.08 (two), 0.11 and 0.14 mg/kg. The Meeting estimated a maximum residue level of 0.3 mg/kg and an STMR of 0.06 mg/kg.

Cantaloupe

A 50% wettable powder formulation was applied to cantaloupe vines (three times 0.28 kg ai/ha, 0.84 kg ai/ha total, 14-day PHI) in the USA by drip irrigation. GAP specifies drip irrigation application of a 50% wettable powder formulation at a rate of 0.28 kg ai/ha. The total seasonal application is limited to 0.84 kg ai/ha, and the PHI is 14 days. The residue levels in ranked order were: <0.02 (two) and 0.02 (two) mg/kg. The Meeting estimated a maximum residue level of 0.03 mg/kg and an STMR of 0.02 mg/kg for whole melon. No information was available on the residue in pulp.

Tomato

Fludioxonil (25% water-dispersible granules) was applied as a foliar spray in glasshouses (11 trials) and in the field (two trials) in Greece, Spain, Switzerland and the UK. The relevant GAPs are: Italy, 0.02 kg ai/hl, 0.2 kg ai/ha, three applications, 7-day PHI; Spain, 0.025 kg ai/hl, three applications, 7-day PHI; Switzerland, 0.25 kg ai/ha, two applications, 3-day PHI. GAPs were not available for Greece or the UK. As three applications were used in all trials, the GAPs of Italy and

Spain were used (7-day PHI). The residue levels in the 14 glasshouse trials at this GAP in ranked order were: 0.05, 0.08, 0.09 (two), 0.10 (two), 0.13 (two), 0.14 (two), 0.16, 0.21, 0.28 and 0.32 mg/kg. The levels in the outside trials in Switzerland were: 0.04 and 0.07 mg/kg. The two groups were statistically the same population, and the combined levels from the 16 trials were: 0.04, 0.05, 0.07, 0.08, 0.09 (two), 0.10 (two), 0.13 (two), 0.14 (two), 0.16, 0.21, 0.28 and 0.32 mg/kg. The Meeting estimated a maximum residue level of 0.5 mg/kg and an STMR of 0.12 mg/kg.

Bell pepper

Fludioxonil (25% water-dispersible granules) was applied as a foliar spray to bell (sweet) peppers in eight glasshouse trials and two field trials in Spain and Switzerland. The relevant GAPs are: Austria, 0.025 kg ai/hl, 0.25 kg ai/ha, three applications, 7-day PHI; Italy, 0.02 kg ai/hl, 0.2 kg ai/ha, three applications, 7-day PHI; Spain, 0.25 kg ai/ha, three applications, 7-day PHI. The GAP of Austria and Italy was used. The ranked order of residue levels in the eight glasshouse trials (six in Spain, two in Switzerland) at GAP was: 0.08, 0.10, 0.14, 0.22, 0.29, 0.46, 0.56 and 0.60 mg/kg. The ranked order in field trials at GAP (one in Italy, one in Spain) was: 0.06 and 0.13 mg/kg. As the two groups are from the same population, they were combined to give levels of: 0.06, 0.08, 0.10, 0.13, 0.14, 0.22, 0.29, 0.46, 0.56 and 0.60 mg/kg. The Meeting estimated a maximum residue level of 1 mg/kg and an STMR of 0.18 mg/kg.

Egg plant

Fludioxonil formulated as 25% water-dispersible granule was applied to Egg plant as a foliar spray three times in glasshouse trials in Italy and Spain. The relevant GAPs are: Italy, 0.02 kg ai/hl, 0.2 kg ai/ha, three applications, 7-day PHI; Spain, 0.025 kg ai/hl, three applications, 7-day PHI. The results at GAP in ranked order were: 0.03, 0.06, 0.06 and 0.08 mg/kg. The Meeting estimated a maximum residue level of 0.3 mg/kg and an STMR of 0.06 mg/kg.

Sweet corn (corn-on-the-cob)

Fludioxonil (flowable concentrate) was applied to sweet corn seed in the USA before planting. The relevant GAP is 5 g ai/1000 kg seed. The residue levels were <0.01 in three trials at three to five times GAP. The Meeting recognized the similarity of sweet corn and maize (see below) and decided to translate the field trial data for seed treatment of maize (same GAP as sweet corn) to sweet corn seed treatment. The residue levels in the eight trials were all <0.01 mg/kg. The Meeting estimated a maximum residue level of 0.01 (*) mg/kg and an STMR 0.01 mg/kg.

Leafy vegetables

Lettuce, head

Fludioxonil (25% water-dispersible granules) was applied as a foliar spray to lettuce in 11 glasshouse and 17 field trials in France, Germany, Italy, Spain and Switzerland. The relevant GAPs are: France, 0.15 kg ai/ha, four applications, 14-day PHI; Italy, 0.018 kg ai/hl, 0.18 kg ai/ha, three applications, 14-day PHI; Spain, 0.15 kg ai/ha, three applications, 14-day PHI; Switzerland, 0.12 kg ai/ha, two applications, early season. No GAP was available for Germany. The GAP of Italy was used to evaluate the trials. The ranked order of residue levels in the glasshouse trials was: 0.72, 0.98, 1.1, 2.4, 2.5, 2.7 (two), 3.4 (two), 4.7 and 6.0 mg/kg. The ranked order of residue levels in the field trials was: <0.02 (six), 0.02 (two), 0.04 (three), 0.07, 0.11, 0.17, 0.29, 1.2 and 1.2 mg/kg. The two sets are not from the same population. In the basis of the indoor trials, the Meeting estimated a maximum residue level of 10 mg/kg and an STMR of 2.7 mg/kg.

Watercress

Watercress was treated with fludioxonil (25% water-dispersible granules) as a foliar spray in the USA. The relevant GAP is: 0.25 kg ai/ha, four applications, 0-day PHI. In the two trials at GAP,

the residue levels were 4.2 and 4.5 mg/kg. The OECD York Workshop recommended a minimum of three trials for commodities that are not significant in trade or in the diet. (See mustard greens.)

Mustard greens

Supervised trials were conducted on mustard greens in the USA. The relevant GAP is: 0.24 kg ai/ha, water-dispersible granules, four applications, 7-day PHI. The ranked order of residue levels in the seven trials at GAP was: 0.06, 0.49, 0.54, 0.76, 1.2, 6.6 and 7.1 mg/kg. The Meeting decided to combine the results of the trials on watercress and mustard greens (same GAP) for mutual support. The ranked order of levels in the nine trials was 0.06, 0.49, 0.54, 0.76, 1.2, 4.2. 4.5, 6.6 and 7.1 mg/kg. The Meeting estimated a maximum residue level of 10 mg/kg and an STMR of 1.2 mg/kg for both watercress and mustard greens.

Legume vegetables and pulses

Bean pod with seed (common bean, French bean, edible podded bean)

Fludioxonil (water-dispersible granules) was applied as a foliar spray to beans in pod in 22 field and glasshouse trials in France, Spain and Switzerland. The relevant GAPs are: France, 0.083 kg ai/hl, 0.25 kg ai/ha, number of applications not specified, 14-day PHI; Spain, 0.025 kg ai/hl, three applications, 14-day PHI. No GAP was available for Switzerland, and the GAP of France was applied. The ranked order of residue levels from the 15 field trials was: <0.02, 0.02 (two), 0.03 (five), 0.04 (two), 0.06 (three), 0.09 and 0.13 mg/kg. The ranked order in the seven glasshouse trials was: 0.03, 0.04 (two), 0.06, 0.09, 0.17 and 0.20 mg/kg. The two groups are not from different populations and were therefore combined to give residue levels of: <0.02, 0.02 (two), 0.03 (six), 0.04 (four), 0.06 (four), 0.09 (two), 0.13, 0.17 and 0.20 mg/kg. The Meeting estimated a maximum residue level of 0.3 mg/kg and an STMR of 0.04 mg/kg for beans (pods and/or immature seeds). This maximum residue level and STMR are extended to peas with pod.

Trials were also conducted on the seed treatment of broad bean and French bean seeds (flowable concentrate) at 5 g ai/100 kg seed in Denmark and Germany. The residue levels were <0.02 mg/kg on bean seed in all six trials, but no GAP was available.

Peas (succulent)

Fludioxonil (25% water-dispersible granules) was applied as a foliar spray to pea vines in France and Switzerland. The relevant GAP is that of France for legume vegetables: 0.083 kg ai/hl, 0.25 kg ai/ha, number of applications not specified, 14-day PHI. No GAP was available for Switzerland, and the GAP of France was applied. The ranked order of residue levels in the trials at GAP was: <0.02 (10) and 0.02 mg/kg.

Trials were also conducted of seed treatment of peas with a flowable concentrate or water-dispersible granule formulation in France and the UK. The residue levels were <0.02 mg/kg in the six trials conducted at the GAP of the UK (5% water-dispersible granules, 10 g ai/100 kg seed).

The Meeting estimated a maximum residue level of 0.03 mg/kg and an STMR of 0.02 mg/kg for peas, shelled (succulent seeds) on the basis of the trials with foliar application. Nevertheless, the maximum residue level and STMR also accommodate seed treatment use and are extended to succulent beans without pod.

Pulses (dry bean and dry pea)

A water-dispersible granule formulation of fludioxonil was applied as a foliar spray to pea and bean (kidney) vines in France. The relevant GAP is: Austria and Spain, water-dispersible granules, 0.25 kg ai/ha, two applications, 14-day PHI. No GAP was available for France, and the GAP

of Spain was applied. The ranked order of residue levels in dry pea and bean was: <0.02 (two), 0.04 (two) and 0.05 mg/kg.

Supervised trials on the treatment of pea seed in France were also considered. GAP in the UK is application of a 5% water-dispersible granule formulation of fludioxonil (w/w) at a rate of 10 g ai/100 kg pea seed. The residue levels in the seven trials at this GAP were <0.02 mg/kg in dry seed at harvest.

The Meeting estimated a maximum residue level of 0.07 mg/kg and an STMR of 0.02 mg/kg for dry peas and for dry beans after foliar application of fludioxonil. The Meeting noted that this also accommodates use of fludioxonil for seed treatment.

Root and tuber vegetables

Potato

Fludioxonil (flowable concentrate, dustable powder) was applied to potato pieces as seed treatment in six trials in Australia, three in South Africa and 13 in the USA. The available GAPs are: Australia, flowable concentrate, 10%, 2.5 g ai/100 kg seed; USA, flowable concentrate, 2.5 g ai/100 kg seed. The ranked order of residue levels on mature potatoes in trials at GAP in Australia and the USA was: <0.01 (16) and 0.01 (17) mg/kg. The Meeting estimated a maximum residue level of 0.02 mg/kg and an STMR of 0.01 mg/kg.

Yam

A 50% wettable powder formulation of fludioxonil was applied as post-harvest treatment to yams at 0.06 kg ai/hl in the USA. GAP specifies application of a 50% wettable powder formulation as a single dip application at a rate of 0.06 kg ai/hl for about 30 s. Two trials were conducted at GAP, and in each trial both whole tubers and tuber pieces (cut yams) were tested. The ranked order of residue levels was: 4.6 and 5.0 mg/kg. The Meeting regarded two independent trials as insufficient for estimating a maximum residue level or an STMR.

Carrot

Nine trials were conducted in the USA in which carrot plots were given four foliar applications of fludioxonil at 0.24 kg ai/ha. The relevant GAP is: water-dispersible granules, 0.25 kg ai/ha, four applications, 7-day PHI. The residue levels in seven trials at GAP were: 0.04, 0.16, 0.18, 0.20, 0.20, 0.25 and 0.42 mg/kg. The Meeting estimated a maximum residue level of 0.7 mg/kg and an STMR of 0.20 mg/kg.

Asparagus

In two trials in Germany, asparagus plants were treated with a water-dispersible granule formulation after harvest. This gives a PHI of about 240 days. No GAP was available for Germany. GAP for Austria specifies use of a 25% water-dispersible granule formulation three times with a 14–21-day interval at 0.042 kg ai/hl or 0.25 kg ai/ha per application. No PHI is specified, but treatments are to be made at transplantation from the glasshouse to the field. The residue levels in the two German trials were <0.02 mg/kg. The Meeting considered two trials insufficient for estimating a maximum residue level or an STMR.

Cereal grains

Fludioxonil formulations were applied to wheat in France, Germany and Switzerland as seed treatment. The relevant GAPs are: Austria, Belgium, UK, flowable concentrate formulation, 5 g ai/100 kg seed, one application. In the 48 trials conducted at or above GAP, the residue levels in ranked order were: <0.02 (36) and <0.04 (12) mg/kg.

One trial was reported from Denmark in which rye seed was treated. The relevant GAP is: Austria, flowable concentrate, 5 g ai/100 kg seed. No GAP is available for Denmark. The residue level was <0.02 mg/kg.

Fludioxonil was applied as seed treatment to barley in 30 trials in France, Germany and Switzerland. The relevant GAPs are: Austria, Belgium, UK, flowable concentrate formulation, 5 g ai/100 kg seed. No GAP was available for France, Germany or Switzerland. The residue levels in six trials conducted at or above GAP were <0.02 mg/kg.

Fludioxonil was applied as a seed treatment to maize (field corn) in 27 trials in France, Germany, Greece, Hungary, Italy and Spain, three trials in South Africa and five trials in the USA. The relevant GAP is: USA, flowable concentrate formulation, 5 g ai/100 kg seed. No GAPs were available for Europe or South Africa, and the GAP of the USA was applied. The ranked order of residue values in trials conducted at GAP and at three and five times the GAP rate were <0.01 (five) and <0.02 (seven) mg/kg.

Fludioxonil was applied as seed treatment to sorghum in four trials in the USA. The relevant GAP is flowable concentrate formulation, 5 g ai/100 kg seed. The residue level at three to five times the GAP rate was <0.05 mg/kg.

All residue levels resulting from seed treatment of the five different cereal grains were below the LOQ. The combined results from all the trials, in ranked order, were: <0.01 (five), <u>≤0.02</u> (50), <0.04 (12) and <0.05 (four) mg/kg. The Meeting estimated a maximum residue level of 0.05 (*) mg/kg and an STMR of 0.02 mg/kg for cereal grains.

Pistachio nut

Fludioxonil was applied as a water-dispersible granule formulation to pistachio trees in the USA. The relevant GAP is: 0.25 kg ai/ha, four applications, 7-day PHI. The residue levels, in ranked order, were: 0.04, 0.05 and 0.08 mg/kg. The Meeting estimated a maximum residue level of 0.2 mg/kg and an STMR 0.05 mg/kg.

Rape-seed

Fludioxonil was applied to rape as seed treatment in trials in France, Germany, Sweden and the UK. GAP in Germany is treatment of seed with a flowable concentrate formulation at 12 g fludioxonil per 100 kg seed. The residue levels in the 15 trials at this GAP were <0.02 mg/kg. The Meeting estimated a maximum residue level of 0.02 (*) mg/kg and an STMR of 0.02 mg/kg.

Cotton-seed

Fludioxonil was applied as seed treatment (flowable concentrate and emulsion formulations) to cotton in Greece and the USA. The relevant GAP is: USA, flowable concentrate, 5 g ai/100 kg seed. No GAP was available for Greece (or any other European country), and the GAP of the USA was applied. The ranked order of residue levels in the trials was: <0.02 (two) and <u>≤0.05</u> (six) mg/kg. The Meeting estimated a maximum residue level of 0.05 (*) mg/kg and an STMR of 0.05 mg/kg for cotton-seed.

Herbs

Fludioxonil was applied as a foliar spray (water-dispersible granules) to chives and basil in the USA. The relevant GAP is 0.25 kg ai/ha, four applications, 7-day PHI. The residue levels were: 1.8 and 3.9 mg/kg on chives and 1.9 and <u>3.0</u> mg/kg on basil. The Meeting estimated a maximum residue level of 10 mg/kg and an STMR of 2.4 mg/kg for fresh basil and a maximum residue level of 10 mg/kg and an STMR of 2.8 mg/kg for fresh chives.

For each herb, one trial included drying. The drying factor for chives is 8 (31/3.9), and that for

basil is also 8 (23/3). Application of this factor to the data from field trials with chives yielded a revised ranked order of residue levels: 14 and 31 mg/kg. Therefore, the Meeting estimated a maximum residue level of 50 mg/kg and an STMR of 22 mg/kg for dried chives. Application of the drying factor for basil yield a revised ranked order of 15 and 24 mg/kg. Therefore, the Meeting estimated a maximum residue level of 50 mg/kg and an STMR of 20 mg/kg for dried basil.

Animal feedstuffs

Straw, fodder and forage of cereal grains and grasses

Trials of residue levels in forage, fodder and straw after application of fludioxonil as seed treatment were conducted with wheat, rye, barley, maize, sweet corn and sorghum. Trials on wheat were conducted in Europe according to the following relevant GAP: Austria, Belgium, UK, flowable concentrate formulation, 5 g ai/100 kg seed, one application. In the 45 trials conducted at or above GAP, the ranked order of residue levels in straw was: <0.02 (eight), <0.04 (14) and <0.05 (23) mg/kg. The ranked order of residue levels in forage was: <0.02 (seven) and <0.04 (11) mg/kg.

A trial on rye was conducted in Denmark according to the GAP for Austria: flowable concentrate, 5 g ai/100 kg seed. The residue level was <0.05 mg/kg in straw and <0.05 mg/kg in forage.

Trials on barley were conducted in Europe according to the GAP for Austria, Belgium and the UK: flowable concentrate formulation, 5 g ai/100 kg seed. In five trials conducted at or above GAP, the ranked order of residue levels were: <0.02 and <0.05 (four) mg/kg in straw and <0.05 (three) mg/kg in forage.

Trials on maize and sweet corn were conducted Europe (maize only) and in the USA. The relevant GAP was: USA, flowable concentrate formulation, 5 g ai/100 kg seed, as no GAP was available for any country in Europe. There were no detectable residues in fodder (<0.01 (five) mg/kg) or forage (<0.01 (seven) mg/kg). Using the default moisture content value of 40% for maize forage (*FAO Manual*, Appendix IX), the Meeting estimated a maximum residue level of 0.03 (*) mg/kg (0.01/0.40) and an STMR of 0 mg/kg (0.00/0.40) for maize forage (dry).

Field trials were conducted on sorghum in the USA, the relevant GAP being flowable concentrate formulation, 5 g ai/100 kg seed. The residue levels after exaggerated application rates were <0.01 (four) mg/kg on fodder and <0.01 (four) mg/kg on forage.

The combined values for fodder and straw in ranked order were: <0.01 (nine), <0.02 (nine), <0.04 (four) and <0.05 (28). As no data were available on the moisture content, the default value of 83% for maize fodder was used (*FAO Manual*, Appendix IX). The Meeting estimated an STMR of 0 mg/kg (0.00/0.83) and a maximum residue level of 0.06 (*) mg/kg (0.05/0.83) for fodder (dry) and straw of cereal grains.

Rape forage and straw

Fludioxonil was applied to rape as seed treatment in trials in France, Germany, Sweden and the UK. The GAP in Germany is treatment of seed with an flowable concentrate formulation at 12 g fludioxonil per 100 kg seed. The residue levels were <0.05 mg/kg in forage (12) and straw (six).

Fate of residues during processing

A study of hydrolysis with [pyrrole-4-^{14}C]fludioxonil showed that fludioxonil is stable under the typical conditions of pasteurization, baking, brewing, boiling and sterilization.

The processing (transfer) factors through commercial-type processes for plums, strawberries,

grapes, citrus and tomato are summarized in the table below. Factors could not be calculated for cereal grains, cotton-seed or potatoes because there were no quantifiable residues in the raw agricultural commodities, even in trials with exaggerated treatment rates.

Processing factors and STMR-P values for various commodities

Raw agricultural commodity				Processed commodity			
Commodity	MRL (mg/kg)	STMR (mg/kg)	HR (mg/kg)	Commodity	Processing factor	STMR-P (mg/kg)	HR-P (mg/kg)
Plum[1]	5	0.80	3.6	Prunes (dried plums)	1.91[2]	0.96	4.3
				Juice	0.10	0.080	
				Preserves	0.50	0.40	
				Purée	0.80	0.64	
Strawberry	3	0.38	2.2	Juice	0.16	0.061	
				Preserves	0.62	0.24	
				Jam	0.34	0.13	
Grapes	2	0.28	1.6	Raisins (dried grapes)	1.1[3]	0.31	1.8
				Juice	0.92[4]	0.26	
				Wine	0.30[5]	0.08	
				(<100 days)			
				Wine	0.036[6]	0.010	
				(>100 days			
Lemons				Juice	0.031		
				Oil	61		
				Pulp	2.1		
Tomato	0.5	0.12	0.32	Juice	0.22[7]	0.026	
				Paste	1.4[8]	0.17	
				Pomace (wet)	3.3[9]		

[1] Stone fruit, includes field trial data for cherries and peaches
[2] Four trials, range 1.8–2.7, mean 1.91, median 1.6
[3] 15 trials, range 0.58–1.7, mean 1.1, median 1.1
[4] 12 trials, range 0.58–1.0, mean 0.92, median 0.86
[5] 17 trials, range 0.012–0.86, mean 0.30, median 0.24
[6] 11 trials, range 0.0086–0.11, mean 0.036, median 0.029
[7] Four trials for pasteurized juice, range 0.20–0.24, average 0.22, median 0.22
[8] Four trials for pasteurized paste, range 1.1–1.6, average 1.4, median 1.35
[9]Two trials for wet pomace, 3.0 and 3.6

Residues in animal commodities

A feeding study was conducted in which three groups of three dairy cows received 0.55 ppm, 1.6 ppm or 5.5 ppm fludioxonil in the diet for 28–30 days. Residues of fludioxonil and metabolites, determined as CGA-192155 (2,2-difluorobenzo[1,1]dioxole-4-carboxylic acid), were quantifiable only at the highest feeding level (5.5 ppm). Residues were found in the milk of two of three cows, with maximum values of 0.019 mg/kg and 0.014 mg/kg on days 14 and 21 respectively. At the lowest feeding level, residues were detected in milk on days 3–21 at levels of 0.001–0.004 mg/kg, with maximum detection on day 3.

Only tissue samples from cows fed the 5.5 ppm diet were analysed. No residues of fludioxonil or metabolites were found. The LOQ was 0.01 mg/kg in muscle and 0.05 mg/kg in liver, kidney and fat (perirenal and omental).

The dietary intake of ruminants and poultry can be calculated from the recommended STMRs or HRs and consideration of possible animal feed items. The table below shows the bases for the dietary intake calculation.

Commodity	Group	Maximum or highest residue level (mg/kg)	STMR or STMR-P (mg/kg)	Dry matter (%)	Dietary content (%)			Residue contribution (mg/kg)		
					Beef cattle	Dairy cows	Poultry	Beef cattle	Dairy cows	Poultry
Barley grain	GC	0.05		88	50	40	75			
Barley straw (dry)	AS	0.06			10	60				
Cotton-seed	SO	0.05		88	25	25				
Cotton-seed meal		0.05		89	15	15	20			
Maize grain	GC	0.05		88	80	40	**80**	0.02	0.03	0.05
					25	**40**				
Maize forage (dry)	AF	0.03			40	50				
Maize fodder (dry)	AS	0.06			25	15				
Oat grain	GC	0.05		89	50	40	80			
Oat straw (dry)	AS	0.06		90	10	10				
Potato waste	AB		0.01	15	**75**	**40**		0.05	0.03	
Rape meal		0.02		88	15	15	15			
Rape forage	AM	0.05		30	30	30				
						20				
Rye grain	GC	0.05			40	40	50			
Rye straw (dry)	AS	0.06		88	10	10				
Wheat grain	GC	0.05		89	50	40	80			
Wheat forage	AF	0.05		25						
Wheat straw (dry)	AS	0.06		88	10	10				
Pea seed	VD	0.07		90	20	20	**20**			0.02
Total					**100**	**100**	**100**	**0.07**	**0.06**	**0.07**

The calculated dietary intakes of beef cattle, dairy cows and poultry are 0.07, 0.06 and 0.07 mg/kg respectively.

No quantifiable residue was found in the tissues of ruminants at levels 60 times (cows) and 80 times (beef cattle) the calculated dietary burden. Fludioxonil and metabolites were detected in liver and kidney at concentrations of 0.014–0.017 mg/kg and 0.022–0.025 mg/kg respectively, at the 5.5 ppm feeding level. None was detected in fat or muscle. The Meeting concluded that the maximum residue level is the LOQ, 0.05 (*) mg/kg, for offal and 0.01 (*) mg/kg for muscle and that the STMR values for edible offal and muscle are both 0 mg/kg.

In milk, the highest residue level found was 0.019 mg/kg with the 5.5 ppm diet (60 times).

The Meeting concluded that the maximum residue level is the LOQ, 0.01 (*) mg/kg, and that the STMR value for milk is 0 mg/kg.

No feeding study was available with poultry. The dietary intake calculation shows a possible burden of 0.07 ppm. The study of the nature of the residue in poultry was conducted at 89 ppm (1300 times) for 8 consecutive days. While short of the normal 30-day feeding study, the extreme exaggeration provides some idea of the likelihood of residues of fludioxonil and metabolites occurring in poultry commodities. The identified residue levels in eggs, liver, kidney, muscle and skin with fat were 0.26, 0.046, 0.020, 0.036 and 0.036 mg/kg respectively. This strongly suggests that residues will not be quantifiable in these commodities at a feeding level of 0.07 mg/kg. Therefore, the Meeting estimated MRLs at the LOQ of 0.05 (*) mg/kg for eggs, 0.01 (*) mg/kg for poultry meat and 0.05 (*) mg/kg for poultry offal and STMRs of 0 mg/kg for eggs, poultry meat and poultry offal.

RECOMMENDATIONS

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

Definition of the residue for compliance with MRLs and estimation of dietary intake in plant commodities: fludioxonil

Definition of the residue for compliance with MRLs and estimation of dietary intake in livestock commodities: fludioxonil and metabolites determined as 2,2-difluorobenzo[1,1]dioxole-4-carboxylic acid and calculated as fludioxonil. Fludioxonil is fat-soluble.

Commodity		MRL, mg/kg		STMR or STMR-P, mg/kg
CCN	Name	New	Previous	
HH722	Basil	10		2.4
DH722	Basil (dried)	50		20
VD0071	Beans (dry)	0.07		0.02
VP61	Beans, except broad bean and soya bean (green pods and immature seeds)	0.3		0.04
VP62	Beans, shelled (succulent=immature seeds)	0.03		0.02
FB264	Blackberries	5		1.0
FB20	Blueberries	2		0.60
VB400	Broccoli	0.7		0.23
VB41	Cabbages, head	2		0.24
VR577	Carrot	0.7		0.20
GC80	Cereal grains	0.05(*)		0.02
HH727	Chives	10		2.8
HH727	Chives (dried)	50		22
FC0001	Citrus	7 Po		1.1
SO691	Cotton seed	0.05(*)		0.05
VC424	Cucumber	0.3		0.06

Commodity		MRL, mg/kg		STMR or STMR-P, mg/kg
CCN	Name	New	Previous	
FB266	Dewberries (including Boysenberry and Loganberry)	5		1.0
DF269	Dried grapes (currants, raisins, sultanas)			0.31
MO105	Edible offal	0.05 (*)		0
VO440	Egg plant (aubergine)	0.3		0.06
PE0112	Eggs	0.05 (*)		0
FB269	Grapes	2		0.28
	Grape juice			0.26
FI0341	Kiwifruit	15 Po		7.2
VL482	Lettuce, head	10		2.7
AF645	Maize forage (dry)	0.03 (*)		0
MM95	Meat (from mammals other than marine mammals)	0.01(*)		0
VC0046	Melon	0.03		0.02
ML106	Milks	0.01		0
VL485	Mustard greens	10		1.2
VA385	Onions, bulb	0.5		0.04
VA389	Onions, spring	5		0.59
FP230	Pears	0.7		0.21
VD0072	Peas (dry)	0.07		0.02
VP63	Peas (pods and succulent=immature seeds)	0.3		0.04
VP64	Peas, shelled (immature seed)	0.03		0.02
VO455	Peppers, sweet	1		0.18
TN675	Pistachio nut	0.2		0.05
	Plum juice			0.08
	Plum preserves			0.40
	Plum purée			0.64
VR589	Potato	0.02		0.01
PM0110	Poultry meat	0.01 (*)		0
PO0111	Poultry, edible offal of	0.05 (*)		0
DF14	Prunes (dried plums)			0.96
SO495	Rape seed	0.02(*)		0.02
FB272	Raspberries, red, black	5		1.0
FS12	Stone fruits	5 Po		0.80
AS81	Straw and fodder (dry) of cereal grains	0.06(*)		
FB275	Strawberry	3		0.27
	Strawberry juice			0.06
	Strawberry preserves			0.24
	Strawberry jam			0.13
VC0431	Summer squash (zucchini)	0.3		0.06

Commodity		MRL, mg/kg		STMR or STMR-P, mg/kg
CCN	Name	New	Previous	
VO447	Sweet corn (corn-on-the-cob)	0.01(*)		0.01
VO448	Tomato	0.5		0.12
JF448	Tomato juice			0.026
	Tomato paste			0.17
VL473	Watercress	10		1.2
	Wine (grape)			0.01

DIETARY RISK ASSESSMENT

Long-term intake

The IEDIs of fludioxonil based on the STMRs estimated for 45 commodities for the five GEMS/Food regional diets were 0–1% of the ADI (Annex 3 of the Report). The Meeting concluded that the long-term dietary intake of residues of fludioxonil is unlikely to present a public health concern.

Short-term intake

The 2004 JMPR decided that an ARfD for fludioxinil is unnecessary. The Meeting therefore concluded that the short-term dietary intake of fludioxonil residues is unlikely to present a public health concern.

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