

ANNEX B

Florasulam

B.7 Environmental fate and behaviour

B.7.1 Route and rate of degradation in soil (Annex IIA 7.1.1; Annex IIIA 9.1.1)

B.7.1.1 Route of degradation (Annex IIA 7.1.1.1)

B.7.1.1.1 Aerobic degradation in soil (Annex IIA 7.1.1.1.1)

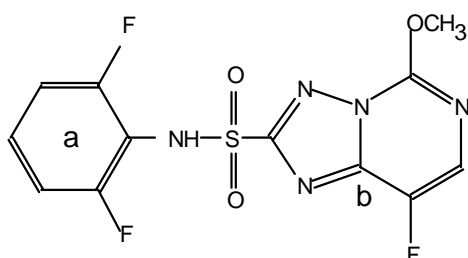


Figure B.7.1.1.1-1 : Structure of florasulam showing the positions of the ^{14}C labels
(a : phenyl; b : triazolopyrimidine)

The aerobic degradation of XDE-570 in soil (Jackson, 1997)

Guidelines :

BBA Guideline Part IV, Section 4.1 \equiv SETAC

GLP :

Yes

Material and Methods :

Test substance : Florasulam

^{14}C phenyl (PH) label, radiochemical purity > 97 %

^{14}C triazolopyrimidine (TP) label, radiochemical purity > 97 %

Soils:

Table B.7.1.1-1-1 : Characteristics of the soils - Aerobic soil metabolism of the a.s.

	Speyer 2.2	Marcham	Kenslow	Andover
Country of origin	Germany	UK	UK	UK
Soil type	loamy sand	sandy clay loam	humus silt loam	silt loam
Textural analysis (%): sand (2000-63 μm) silt (63-2 μm) clay (<2 μm)	76.3 15.4 8.2	48.2 23.0 28.8	13.5 62.4 24.1	18.5 59.2 22.3
pH (KCl)	7.3	7.7	5.6	7.6
Organic carbon (%)	3.9	2.0	6.8	3.1
Cation exchange capacity (mEq/100g)	8.7	9.0	16.0	12.0
Moisture holding capacity (%)	64.1	54.5	95.1	64.3
Microbial biomass: start ($\mu\text{g C/g}$) end	1355 872	354 422	588 771	550 590

Experimental design :

Samples (50g dry weight equivalent) of one soil (Speyer 2.2) were treated separately with ^{14}C -Ph and ^{14}C -TP labelled florasulam at a rate equivalent to 15 g a.s./ha (15 μg a.s./kg soil) . Samples of sterile Speyer 2.2 soil and the three agricultural soils were treated with ^{14}C -TP- florasulam also at 15 g a.s./ha. The soils were maintained at 40% moisture holding capacity under aerobic conditions in the dark at 20 $^{\circ}\text{C}$ for up to 100 days.

Analytical methods :

Evolved $^{14}\text{CO}_2$ was continuously trapped and, at pre-determined time points, samples of soil were removed and extracted first with aqueous 0.01M calcium chloride solution then with acetone: water: acetic acid (90:9:1) and finally with acetonitrile: water: ortho-phosphoric acid (80:19:1). The radioactivity in trapping solutions, soil extracts and soil residues was quantified by liquid scintillation counting (LSC). Soil extracts were analysed by high performance liquid chromatography (HPLC) and thin layer chromatography (TLC) to determine the proportion of parent compound and degradation products present at each time point. Degradation products which accounted for greater than 10% of applied radioactivity were identified by mass spectroscopy and co-chromatography with reference standards.

Findings :

Table B.7.1.1.1-2 : Distribution of applied radioactivity (mean %) after application of ^{14}C triazolopyrimidine florasulam at 15 g a.s./ha to Speyer 2.2 soil under aerobic conditions (HPLC)

	Sampling Interval (Days)							
	0	1	3	7	14	28	59	100
<u>Extractable-</u>	96.41	94.12	88.13	84.26	73.44	55.52	49.43	39.35
florasulam	91.8	39.6	22.6	4.6	1.2	0.6	-	-
5-OH	3.8	49.9	55.8	61.1	41.0	21.9	11.8	3.5
DFP-TSA	-	-	0.9	-	2.2	2.7	1.1	0.4
metabolite M2	-	0.4	2.0	0.6	-	-	0.3	-
DFP-ASTCA	-	1.1	3.2	9.5	13.1	14.8	17.0	8.4
ASTCA	-	0.8	-	3.8	7.0	8.0	11.2	19.7
metabolite M5	-	-	-	-	1.0	2.4	1.3	0.9
TSA	-	-	-	-	-	-	4.5	6.5
metabolite M7	-	-	-	-	-	-	1.1	-
Unresolved Background	0.8	2.3	3.6	4.7	8.0	5.1	1.2	-
$^{14}\text{CO}_2$	-	-	-	0.01	0.10	0.63	1.10	4.86
<u>'Bound'</u>	2.08	4.00	7.56	13.70	23.09	39.59	41.63	48.27
<u>Recovery</u>	98.49	98.12	95.69	97.97	96.63	95.74	92.16	92.48

Table B.7.1.1.1-3 : Distribution of applied radioactivity (mean %) after application of ^{14}C phenyl florasulam at 15 g a.s./ha to Speyer 2.2 soil under aerobic conditions (HPLC)

	Sampling Interval (Days)							
	0	1	3	7	14	28	59	100
<u>Extractable</u>	96.67	95.15	87.27	78.93	65.43	47.40	30.83	20.79
florasulam	96.2	42.2	19.6	4.7	2.6	0.3	0.2	-
5-OH	-	52.7	65.3	64.2	44.5	23.7	9.2	5.5
DFP-TSA	0.2	-	-	-	1.5	2.7	3.6	2.5
metabolite M2	-	-	-	-	0.3	-	0.5	-
DFP-ASTCA	-	-	1.8	8.3	15.1	17.8	15.0	12.7
metabolite M5	-	-	-	1.1	1.3	1.2	1.5	-
metabolite M7	-	-	-	-	-	-	-	-
Unresolved Background	0.3	0.3	0.5	0.6	0.2	1.8 (*)	0.9 (*)	0.2
$^{14}\text{CO}_2$	-	0.09	0.44	1.78	3.10	7.14	11.03	13.58
<u>'Bound'</u>	1.82	3.43	9.60	15.75	26.53	40.43	48.37	57.11
<u>Recovery</u>	98.49	98.67	97.31	96.46	95.06	94.97	90.23	91.48

(*) : sum of polar and unresolved radioactivity

Table B.7.1.1.1-4 : Distribution of applied radioactivity (mean %) after application of ^{14}C triazolopyrimidine florasulam at 15 g a.s./ha to sterile soil under aerobic conditions (HPLC)

	Sampling Interval (Days)			
	1	8	28	106
<u>Extractable</u>	96.73	94.46	88.84	77.90
florasulam	79.3	77.3	66.5	44.1
5-OH	12.4	7.9	8.4	17.6
DFP-TSA	-	-	-	0.5
metabolite M2	0.4	1.8	1.6	2.0
DFP-ASTCA	0.	-	1.2	1.9
ASTCA	-	-	0.7	1.1
Other	4.3	7.5	10.5	10.8
$^{14}\text{CO}_2$	nd	nd	0.02	0.10
<u>'Bound'</u>	3.20	5.75	7.13	17.44
<u>Recovery</u>	99.93	100.21	95.99	95.43

Table B.7.1.1.1-5 : Distribution of applied radioactivity (mean %) after application of ^{14}C triazolopyrimidine florasulam at 15 g a.s./ha to Marcham soil under aerobic conditions (HPLC)

	Sampling Interval (Days)							
	0	1	3	7	14	28	59	100
<u>Extractable</u>	96.87	95.98	89.40	87.28	81.82	66.58	55.47	47.81
florasulam	95.6	62.9	40.7	20.0	10.0	5.5	1.1	1.1
5-OH	-	28.5	38.8	49.9	37.0	24.6	7.0	5.8
DFP-TSA	-	-	0.5	0.6	1.9	2.9	0.4	0.4
metabolite M2	-	0.6	1.8	0.6	1.0	0.4	0.1	-
DFP-ASTCA	-	0.9	2.3	4.6	7.0	5.9	4.8	2.6
ASTCA	-	0.2	-	4.6	14.6	18.4	33.1	29.4
metabolite M5	-	-	-	-	-	4.5	2.9	2.6
TSA	-	-	-	-	5.3	3.4	4.9	5.0
metabolite M7	-	-	-	-	-	0.9	1.1	0.8
Unresolved Background	1.3	2.9	5.3	7.1	5.1	0.1	0.1	0.1
$^{14}\text{CO}_2$	-	nd	0.05	0.16	0.73	2.49	5.68	5.94
<u>'Bound'</u>	0.99	3.63	9.16	9.58	14.79	24.98	33.48	35.60
<u>Recovery</u>	97.86	99.61	98.61	97.02	97.34	94.05	94.63	89.35

Table B.7.1.1.1-6 : Distribution of applied radioactivity (mean %) after application of ^{14}C triazolopyrimidine florasulam at 15 g a.s./ha to Kenslow soil under aerobic conditions (HPLC)

	Sampling Interval (Days)							
	0	1	3	7	14	28	59	100
<u>Extractable</u>	92.71	89.23	88.42	78.84	68.38	53.09	43.01	35.66
florasulam	88.3	27.6	3.8	1.1	0.8	0.6	-	-
5-OH	4.0	55.0	71.6	60.4	40.7	28.1	10.1	8.7
DFP-TSA	-	-	-	1.3	2.4	2.3	1.0	-
metabolite M2	-	2.1	1.6	-	-	0.3	-	-
DFP-ASTCA	-	1.2	2.2	2.8	4.2	1.6	0.9	0.2
ASTCA	-	1.2	0.8	2.3	3.9	14.7	24.8	22.0
metabolite M5	-	-	-	-	0.5	0.9	0.5	-
TSA	-	-	-	3.9	10.0	2.9	4.4	4.6
metabolite M7	-	-	-	1.0	1.5	1.2	0.8	-
Unresolved Background	0.5	2.1	8.4	6.0	4.4	0.4	0.6	0.2
$^{14}\text{CO}_2$	-	nd	nd	0.31	1.35	2.70	7.92	9.83
<u>'Bound'</u>	4.12	6.09	7.08	16.20	25.18	32.64	41.06	43.88
<u>Recovery</u>	96.83	95.32	95.50	95.35	94.91	88.43	91.99	89.37

Table B.7.1.1.1-7 : Distribution of applied radioactivity (mean %) after application of ^{14}C triazolopyrimidine florasulam at 15 g a.s./ha to Andover soil under aerobic conditions (HPLC)

	Sampling Interval (Days)							
	0	1	3	7	14	28	59	100
Extractable	97.61	95.60	93.28	80.80	74.89	67.46	60.07	55.70
florasulam	95.2	48.9	57.5	3.8	0.5	0.4	-	-
5-OH	1.6	36.1	29.3	41.2	21.2	7.1	1.1	0.5
DFP-TSA	-	-	-	1.2	1.9	1.7	-	0.1
metabolite M2	-	1.8	1.6	1.3	0.7	-	-	-
DFP-ASTCA	-	1.2	2.0	6.7	7.1	5.1	1.7	0.2
ASTCA	-	1.1	0.6	10.5	25.1	35.4	40.0	37.9
metabolite M5	-	-	-	-	0.7	4.3	1.2	1.0
TSA	-	-	-	1.7	7.8	11.5	15.2	15.9
metabolite M7	-	-	-	-	-	0.9	0.9	-
Unresolved Background	0.8	6.5	2.3	14.4	9.9	1.1	-	-
$^{14}\text{CO}_2$	-	-	-	0.36	1.24	3.34	6.31	4.80
'Bound'	1.45	4.52	5.01	11.98	18.10	23.91	26.15	29.63
Recovery	99.06	100.12	98.29	93.14	94.23	94.71	92.53	90.13

Table B.7.1.1.1-8 : Degradation rate of florasulam in soil at 20°C under aerobic conditions

Soil	first order		Timme-Frehse	
	DT ₅₀ (days)	DT ₉₀ (days)	DT ₅₀ (days)	DT ₉₀ (days)
Speyer 2.2 (triazolopyrimidine label)	1.7	5.7	0.5	5.0
Speyer 2.2 (phenyl label)	1.7	5.6	0.4	4.5
sterile Speyer 2.2 (triazolopyrimidine label)	116	-	119	-
Marcham (triazolopyrimidine label)	4.5	15	1.4	16
Kenslow (triazolopyrimidine label)	0.7	2.2	< 0.1	0.7
Andover (triazolopyrimidine label)	1.5	5.1	1.9	6.2

Table B.7.1.1.1-9 : Degradation rate of metabolite 5-hydroxy in soil at 20°C under aerobic conditions

Soil	first order		Timme-Frehse	
	DT ₅₀ (days)	DT ₉₀ (days)	DT ₅₀ (days)	DT ₉₀ (days)
Speyer 2.2 (triazolopyrimidine label)	24	79	13	70
Speyer 2.2 (phenyl label)	26	88	15	80
Marcham (triazolopyrimidine label)	29	95	16	90
Kenslow (triazolopyrimidine label)	31	102	15	97
Andover (triazolopyrimidine label)	10	34	7.1	33

Conclusions :

- HPLC retention times of the metabolites were very variable between sets of analysis (e.g. in Hypercarb system used as HPLC confirmatory system)
- The active substance is degraded into to the major metabolite 5-hydroxy and then into the metabolite DFP-ASTCA (M3). The degradation of DFP-ASTCA occurred via the cleavage of the sulphonamide bridge to form ASTCA (M4). The decarboxylation of DFP-ASTCA to DFP-TSA (M1) and ASTCA (M4) to TSA (M6) should be considered as artefacts due to the extraction procedure. (See Jackson, et al., 1998)
- The metabolites 5 hydroxy, DFP-ASTCA, ASTCA and TSA were found, at least in one soil, at level > 10%.
- The DT₅₀ of florasulam in four soils under aerobic conditions at 20°C were in the range 0.7 - 4.5 days. The metabolite 5-hydroxy was degraded with an estimated DT₅₀ of 7 to 31 days.
- The mineralization reached 4.8 - 13.5 % after 100 days. Bound residues reached 29.6 - 57.1 % after 100 days.

The degradation of DFP-ASTCA and ASTCA (Two metabolites of DE-570) in soil (Jackson, et al., 1998)

Guidelines :

The study design is based on SETAC-Europe guidelines.

GLP :

Yes

Material and Methods :

Test substances :

DFP-ASTCA, ¹⁴C triazole label, radiochemical purity = 96.7%

ASTCA, ¹⁴C triazole label, radiochemical purity = 100%

Soils:

Table B.7.1.1.1-9 : Characteristics of the soils - aerobic soil degradation of metabolites DFP-ASTCA and ASTCA

	Marcham	Cuckney
Country of origin	UK	UK
Soil type	sandy clay loam	sand
Textural analysis (%):		
sand (2000-63 µm)	51.37	88.01
silt (63-2 µm)	21.21	5.08
clay (<2 µm)	27.42	6.91
pH (water)	7.9	7.2
Organic carbon (%)	3.4	1.5
Cation exchange capacity (mEquiv/100g)	25.0	9.0
Moisture holding capacity (%)	60.6	33.2
Microbial biomass (µg C/g)	-	156.1

Experimental design :

Samples (50g dry weight equivalent) of 2 soils were treated separately with ¹⁴C-Triazole labelled DFP-ASTCA and ASTCA at a rate equivalent to 15 g a.s. equivalent/ha (20 µg a.s./kg soil). Actual initial concentrations were 18.2 µg/kg soil and 11 µg/kg soil, respectively for DFP-ASTCA and ASTCA. The soils were maintained at 40% moisture holding capacity under aerobic conditions in the dark at 20 °C for up to 118 days.

Analytical methods :

Evolved ¹⁴CO₂ was continuously trapped. Several extraction mixtures were experimented in order to determine the mixture with the best extractability and the stability of the test compounds. Soils were extracted with various extraction solvents (acetonitrile: 0.5 M HCl (80:20 v/v); 0.05M borate buffer depending on the soil and the analyzed test compound) followed by solid phase extraction. The radioactivity in trapping solutions, soil extracts and soil residues was quantified by liquid scintillation counting (LSC). Soil extracts were analysed by high performance

liquid chromatography (HPLC) to determine the proportion of degradation products (DFP-ASTCA, ASTCA, DFP-TSA and TSA) present at each time point.

Findings :

Table B.7.1.1.1-11 : Degradation rate of metabolites DFP-ASTCA and ASTCA

	Soil Type	DT ₅₀ (days)
DFP-ASTCA	Marcham sandy clay loam	25
	Cuckney sand	8
ASTCA	Marcham sandy clay loam	502
	Cuckney sand	158

Conclusions :

This study confirmed that DFP-ASTCA is a transient metabolite which is further degraded to DFP-TSA and ASTCA and finally to TSA (minor route of degradation). The degradation rate of this metabolite was 8-25 days.

The degradation rate of the metabolite ASTCA was 158-502 days. The 'degradation' is based on the increase in bound residue (15.7 and 41.7% after 118 d, respectively in Cuckney and Marcham soils). The metabolite TSA is an artefact resulting from the extraction procedure (9.2 and 0% after 118 d, respectively in Cuckney and Marcham soils). No mineralization of this metabolite occurred.

"In previous studies, significant levels of de-carboxylated forms of DFP-ASTCA and ASTCA were formed but, from the results of this study, it seems likely that these products were formed as artefacts of the carboxylic acid metabolites since the previous studies used acidic organic solvents for extraction"

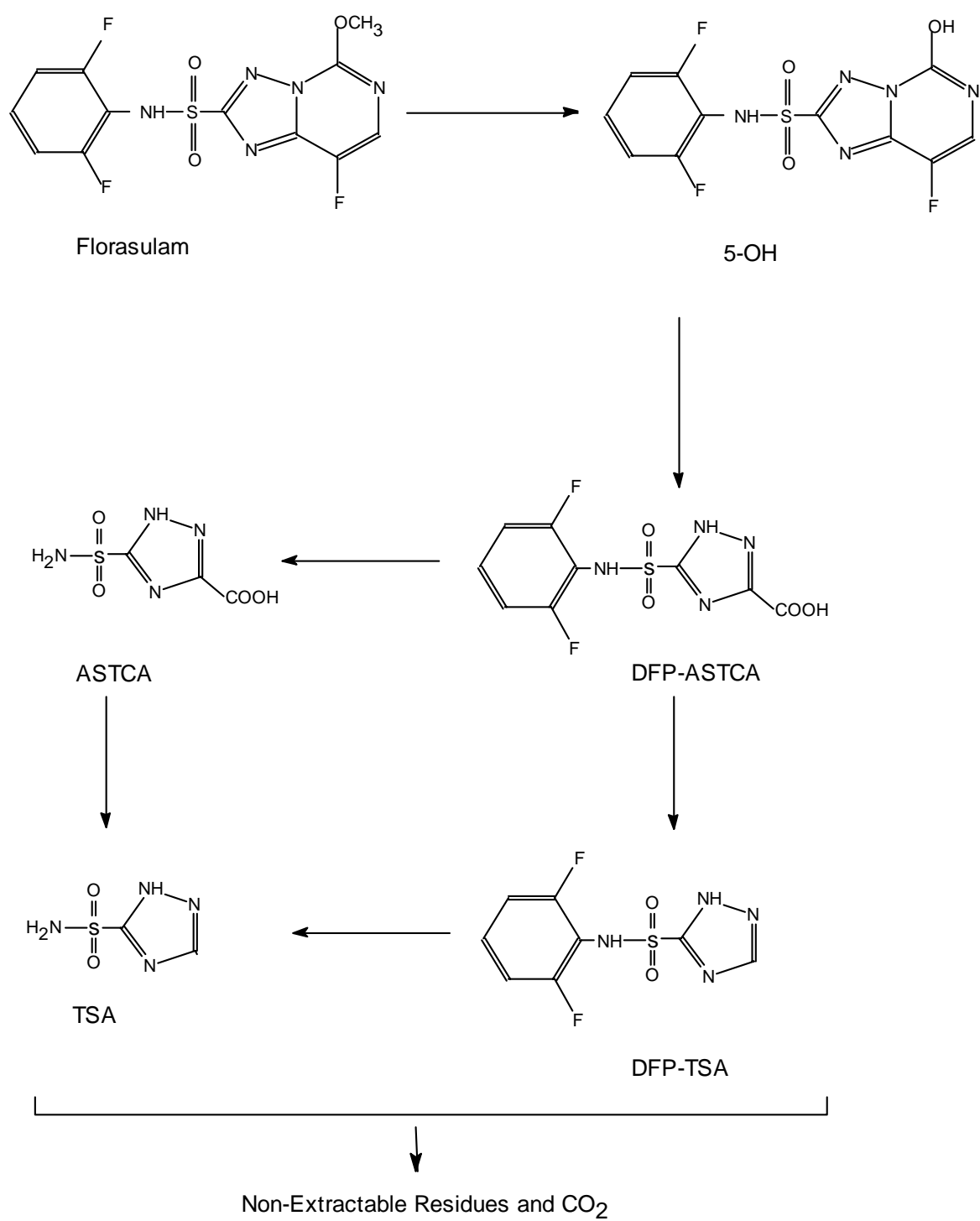


Figure B.7.1.1.1-1 : Proposed route of degradation of florasulam in soil

B.7.1.1.2 Anaerobic degradation in soil (Annex IIA 7.1.1.1.2)

Anaerobic aquatic metabolism study of XDE-570 (Cleveland, et al., 1997)

Guidelines :

SETAC \equiv FIFRA Guideline No. 162-2 and 163-3

GLP :

Yes

Material and Methods :

Test substance : Florasulam

^{14}C phenyl label, radiochemical purity = 98.5%

^{14}C triazolopyrimidine label, radiochemical purity = 99%; 98.8%

Soil:

Table B.7.1.1.2-1 : Characteristics of the soil - Anaerobic soil metabolism of the a.s.

	Speyer 2.2
Country of origin	Germany
Soil type	loamy sand
Textural analysis (%): sand (2000-63 μm) silt (63-2 μm) clay (<2 μm)	75.2 18.0 6.8
pH	5.8
Organic carbon (%)	2.66
Cation exchange capacity (mEquiv/100g)	5.33
Moisture holding capacity at 1/3 bar (%)	10.67

Experimental design :

The soil was flooded with HPLC-grade water (50 g dry soil plus enough water to bring the total volume to 100 ml) and incubated under anoxic conditions for approximately 30 days prior to fortification. The results of a similar experiment which was performed with pond sediment and overlying water were not taken into consideration for the final evaluation.

Samples were treated separately with ^{14}C -TP labelled florasulam at 17 μg a.s./l water. The samples were maintained under anaerobic conditions in the dark at 20 °C for up to 368 days. Redox potential, oxygen concentration and pH were checked.

Analytical methods :

Evolved $^{14}\text{CO}_2$ was continuously trapped in caustic solution. Water fractions were analyzed by LSC and HPLC. Samples of soil were extracted with acetone: water: acetic acid (80:20:1) and quantified LSC and HPLC. The confirmation of florasulam and the metabolite 5-OH was conducted by TLC.

Findings :

Table B.7.1.1.1-8 : Degradation rate of florasulam in soil at 20°C under anaerobic conditions

Soil	first order		Graphical estimation	
	DT ₅₀ (days)	DT ₉₀ (days)	DT ₅₀ (days)	DT ₉₀ (days)
Speyer 2.2 (triazolopyrimidine label)	11	38	18	<45
Speyer 2.2 (phenyl label)	14	46	18	<45

Conclusions :

Under anaerobic conditions, florasulam is degraded into 5-OH metabolite with a DT₅₀ of 11-14 days. The metabolite 5-OH underwent almost no degradation during the whole study duration. 5-OH metabolite levels remain constant from day 30 to day 368 : 69.4 to 85.1% RR and 63.4 to 87% RR respectively for the phenyl and the triazolopyrimidine labeled a.s.

A secondary metabolite recovered at maximum level of 8.2 and 6.4% after 368 days was tentatively identified as triazolo-methyl/carboxylic acid.

The mineralization was low (maximum level of 0.1 and 1.3% RR). Maximum bound residue levels were 11.2 and 7.2% after 368 days. The average mass balances in the 2 sets of samples were 95.2 and 96.5%.

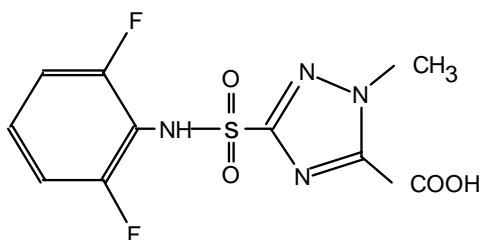


Figure B.7.1.1.2-1 : A secondary metabolite tentatively identified as triazolo-methyl/carboxylic acid.

B.7.1.1.3 Soil photolysis (Annex IIA 7.1.1.1.2)

Photolysis of XDE-570 on soil (Kreger, et al., 1996)

Guidelines :

FIFRA Guideline No. 161-3

Deviations from the SETAC recommendations :

The study was realized with moist and air-dried soils. The results of the trial with moist soil were not taken into consideration in the final evaluation of the study (and not reported in the monograph).

The study should be realized at 20 ± 3 °C and not 25°C. Artificial light source should be used.

GLP :

Yes

Material and Methods :

Test substance : Florasulam

¹⁴C phenyl label, radiochemical purity = 98.3%

Soil:

Table B.7.1.1-1- : Characteristics of the soil - Soil photolysis of the a.s.

	Catlin
Country of origin	USA
Soil type	silt loam
Textural analysis (%):	
sand (2000-63 µm)	10.4
silt (63-2 µm)	62.8
clay (<2 µm)	26.8
pH (KCl)	6.8
Organic carbon (%)	1.70
Cation exchange capacity (mEquiv/100g)	15.62
Moisture potential (1/3 bar)	31.84

Experimental design :

Florasulam was applied at the rate of 0.133 mg/kg to approximately 2 g of soil which were placed into quartz flasks. The photolysis samples were placed in a water bath on the roof of a building (Latitude 39.9°N). Soil temperature was maintained at $25 \pm 1^\circ\text{C}$. Extensive weather data and actinometer data were collected during the whole experiment duration.

Analytical methods :

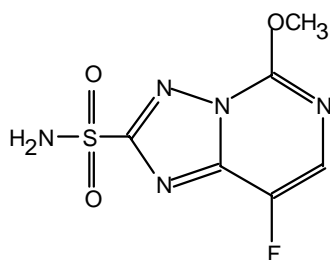
Soil samples were extracted with 90: 10 acetone/ 0.1 N HCL and analyzed by HPLC. Bound residues were determined by LSC.

Findings :

Table B.7.1.1.1-3 : Distribution of applied radioactivity (mean %) after application of ^{14}C phenyl florasulam at 0.133 mg a.s./kg to Catlin air-dried soil - Photolysis study (HPLC)

	Sampling Interval (Days)				
	0	3	10	22	30
Irradiated samples					
<u>Extractable</u>	88.7	86.2	79.2	67.6	64.4
florasulam	85.7	83.7	73.9	58.4	55.7
5-OH	-	-	1.6	2.0	2.1
Unknown 1	-	-	1.3	2.7	2.8
<u>'Bound'</u>	0.6	11.3	17.2	27.7	27.4
<u>Recovery</u>	89.3	97.5	96.5	95.3	91.8
Dark samples					
<u>Extractable</u>	88.7	94.8	90.9	88.2	79.7
florasulam	85.7	92.0	88.9	84.9	76.5
5-OH	-	0.5	0.5	1.1	1.4
Unknown 1	-	0.5	-	-	-
<u>'Bound'</u>	0.6	4.4	7.2	9.2	15.3
<u>Recovery</u>	89.3	99.1	98.1	97.4	95.0

The Unknown 1 peak should be identified as 8-fluoro-5-methoxy(1,2,4)triazolo(1,5c)pyrimidine-2-sulphonamide.



Conclusions :

Florasulam is degraded in air-dried soil with DT_{50} of 44 and 158 days, respectively under light, and in the dark. The photolytic DT_{50} of florasulam is 62 days.

Metabolite 5-hydroxy and Unknown 1 were detected at maximum level of 2.1 and 2.8%. High level of radioactivity was recovered as bound residue (27.7% after 22 days)

B.7.1.2 Rate of degradation (Annex IIA 7.1.1.2.1; Annex IIIA 9.1.1.1.1)

B.7.1.2.1 Aerobic degradation

Effects of temperature on the degradation of DE-570 in soil (Pillar, 1997c)

Guidelines :

The procedure followed the relevant principles of BBA Guideline Part IV, Section 4.1 \equiv SETAC

GLP :

Yes

Material and Methods :

Test substance : Florasulam

^{14}C triazolopyrimidine label, radiochemical purity > 98%

Soils:

Table B.7.1.2.1-1- : Characteristics of the soils - aerobic soil degradation

	Marcham	Cuckney
Country of origin	UK	UK
Soil type	sandy clay loam	sand
Textural analysis (%):		
sand (2000-63 μm)	53.75	78.51
silt (63-2 μm)	20.35	12.06
clay (<2 μm)	25.90	9.43
pH (KCl)	7.6	6.9
Organic carbon (%)	1.4	1.4
Cation exchange capacity (mEquiv/100g)	23.50	14.1
Moisture holding capacity (%)	53.64	30.89
Microbial biomass ($\mu\text{g C/g}$)	265	117

Experimental design :

Samples (50g dry weight equivalent) of 2 soils were treated separately with ^{14}C -TP labelled florasulam at a rate equivalent to 15 g a.s./ha (15 μg a.s./kg soil). The soils were maintained at 40% moisture holding capacity under aerobic conditions in the dark at 5, 15 and 25 $^{\circ}\text{C}$ for up to 97 days.

Analytical methods :

Evolved $^{14}\text{CO}_2$ was continuously trapped and, at pre-determined time points, samples of soil were removed and extracted first with aqueous 0.01M calcium chloride solution then with acetone: water: acetic acid (90:9:1) and finally with acetonitrile: water: ortho-phosphoric acid (80:19:1). The radioactivity in trapping solutions, soil extracts and soil residues was quantified by liquid scintillation counting (LSC). Soil extracts were analysed by high performance liquid chromatography (HPLC) to determine the proportion of parent compound and degradation products (5-hydroxy, DFP-TSA and DFP-ASTCA) present at each time point.

Findings :

Degradation rates were calculated for the active substance and the metabolite 5-hydroxy.

Table B.7.1.2.1-2 : Degradation rate of florasulam in soil under various temperature conditions (HPLC)

Soil Type	Temperature	DT ₅₀ (days)	DT ₉₀ (days)
Marcham sandy clay loam	5	45	149
	15	15	50
	25	5.2	17
Cuckney sand	5	19	64
	15	3.5	11
	25	1.1	36

Table B.7.1.2.1-3 : Degradation rate of metabolite 5-hydroxy in soil under various temperature conditions (HPLC)

Soil Type	Temperature	DT ₅₀ (days)	DT ₉₀ (days)
Marcham sandy clay loam	5	nc	nc
	15	42	141
	25	18	60
Cuckney sand	5	nc	nc
	15	33	109
	25	21	70

nc : not calculated

Conclusions :

The rate of degradation was found to be a strong function of temperature : approximate 3-fold and 4-fold decrease in DT₅₀ with every 10°C rise in Marcham soil and Cuckney soil, respectively.

Effects of moisture on the degradation of DE-570 in soil (Pillar, 1997b)

Guidelines :

The procedure followed the relevant principles of BBA Guideline Part IV, Section 4.1 = SETAC

GLP :

Yes

Material and Methods :

Test substance : Florasulam

¹⁴C triazolopyrimidine label, radiochemical purity > 98%

Soils:

See table B.7.1.2.1-1

Experimental design :

Samples (50g dry weight equivalent) of 2 soils were treated separately with ¹⁴C-TP labelled florasulam at a rate equivalent to 15 g a.s./ha (15 µg a.s./kg soil) . The soils were maintained at 3 different moisture content (saturation point, field capacity, wilt point) under aerobic conditions in the dark at 20 °C for up to 99 days.

Analytical methods :

Evolved ¹⁴CO₂ was continuously trapped and, at pre-determined time points, samples of soil were removed and extracted first with aqueous 0.01M calcium chloride solution then with acetone: water: acetic acid (90:9:1) and finally with acetonitrile: water: ortho-phosphoric acid (80:19:1). The radioactivity in trapping solutions, soil extracts and soil residues was quantified by liquid scintillation counting (LSC). Soil extracts were analysed by high performance liquid chromatography (HPLC) to determine the proportion of parent compound and degradation products (5-hydroxy, DFP-TSA and DFP-ASTCA) present at each time point.

Findings :

Degradation rates were calculated for the active substance and the metabolite 5-hydroxy.

Table B.7.1.2.1-4 : Degradation rate of florasulam in soil under various moisture conditions (HPLC)

Soil Type	Moisture content	Bar	DT ₅₀ (days)	DT ₉₀ (days)
Marcham sandy clay loam	Saturation point	0	5.7	19
	Field capacity	0.05	7.4	25
	Wilt point	15	8.1	27
Cuckney sand	Saturation point	0	6.1	20
	Field capacity	0.05	10	34
	Wilt point	15	8.2	27

Table B.7.1.2.1-5 : Degradation rate of metabolite 5-hydroxy in soil under various moisture conditions (HPLC)

Soil Type	Moisture content	Bar	DT ₅₀ (days)	DT ₉₀ (days)
Marcham sandy clay loam	Saturation point	0	72	238
	Field capacity	0.05	36	119
	Wilt point	15	46	154
Cuckney sand	Saturation point	0	54	179
	Field capacity	0.05	42	141
	Wilt point	15	86	286

Conclusions :

The soil moisture does not play a role in altering the degradation rate of florasulam.

Early stage environmental fate probes for XR-566 and XR-570 (Baloch, et al., 1992)

Probe experiments into the soil sorption, column leaching, behaviour and stability in soil have been conducted. The results of these experiments were not taken in consideration during the final evaluation of the active substance. They were however in line with the results of the other studies.

B.7.1.2.2 Anaerobic degradation

See point B.7.1.1.2.

B.7.1.3 Field studies (Annex IIA 7.1.1.2.2; Annex IIIA 9.1.1.2)

B.7.1.3.1 Soil dissipation testing

The dissipation of XDE-570 and its 5-hydroxy metabolite in soil following a single application of EF-1343, Northern France - 1995; Germany - 1995; UK - 1995; UK - 1996; Southern France - 1996; Greece - 1996 (Maycock, 1995)

Guidelines :

The reports comply with the SETAC recommendations and BBA guidelines

GLP :

Yes

Material and methods :

Test substance :

EF-1343 (SC formulation containing 50 g a.s./l florasulam)

Soils :

See summary tables in the appendix.

Experimental design :

In each location, the test substance was applied to 4 adjacent sub plots (each 7-15 m X 3 m) at a rate of 15 g a.s./ha (twice the maximum commercial rate). The application was made on bare soil when cereals in neighbouring fields were at stage BBCH 32 (March, April) . Soil cores were taken at intervals of 0 to 220-372 days following application. The soil cores were 5 x 50 cm. Soil cores will be cut so as to create five 10 cm horizons . The samples were analysed for florasulam and its major degradation product 5-hydroxy by electrospray liquid chromatography with mass spectrometry (ESP LC/MS-MS) with a limit of determination of 0.05 µg/kg. The DT₅₀ and DT₉₀ values for florasulam and metabolite 5-hydroxy were calculated using the Timme/Frehse model (first order kinetics).

Weather conditions (soil and air temperatures, rainfall) were recorded daily. Irrigation was used.

Findings :

Table B.7.1.3.1-6 : Field soil dissipation DT₅₀ and DT₉₀ values (days) for florasulam and its metabolite 5-OH

Test soil				Test system - temp. (*)	Results (days)				References
Soil type Location	pH	CEC meq/ 100g	O.C. (%)		florasulam		metabolite 5- OH		
					DT ₅₀	DT ₉₀	DT ₅₀	DT ₉₀	
Silty clay loam Tours, N France, 1995	7.4	29.1	1.2	10 °C	9	31	17	55	Maycock, 1995
Silty loam Wetterfeld, Germany, 1995	6.1	13.9	1.2	18 °C **	15	50	14	59	Maycock, 1995
Sandy clay loam Marcham, UK, 1995	7.7	25.8	2.0	10 °C	5	50	42	209	Maycock, 1995
Sand Elvedon, UK, 1996	7.6	11.8	1.1	1 °C	18	61	95	182	Maycock, 1995
Clay loam St Livrade, S France, 1996	8.2	25.2	1.2	10 °C	8	28	9	41	Maycock, 1995
Sandy silt loam Valtothori, Greece, 1996	8.5	10.0	0.9	11 °C	2	23	13	61	Maycock, 1995

* : average soil temperature during the first 7 days after application

** : based on few data

Conclusion:

Florasulam is degraded in soil under field conditions following spring application with DT₅₀ values in the range 2-18 days. Its major metabolite 5-OH is degraded with DT₅₀ values of 9-95 days. Florasulam and its major metabolite were recovered in deeper horizons at following concentrations :

Maximum concentrations of florasulam at 10-20 cm : <0.05-0.75 µg/kg; 20-30 cm : < 0.05-0.96 µg/kg; 30-40 cm : <0.05-0.29; 40-50 cm : <0.05-0.29 µg/kg

Maximum concentrations of metabolite 5-OH at 10-20 cm : <0.10-0.35 µg/kg; 20-30 cm : < 0.05-0.19 µg/kg; 30-40 cm : <0.05-0.22; 40-50 cm : <0.05-0.11 µg/kg

Residues of DE-570 and its 5-hydroxy metabolite in soil at normal harvest following application of EF-1343 to wheat and barley - Europe: 1995 -1996 (Gambie, 1997a)

Guidelines :

SETAC recommendations

GLP :

No

Material and methods :

Test substance :

EF-1343 (SC formulation containing 50 g a.s./l florasulam)

Soils :

See table

Experimental design :

“Soil samples were taken at harvest from supervised residue trials in both Northern and Southern Europe. The trials were performed in spring using EF-1343, an SC formulation containing 50 g as/L, at rates of either 5 or 7.5g as/ha to winter cereals. A single application was made at either cereal growth stage BBCH 30-32, which would represent the majority of commercial applications, or the later growth stage of BBCH 49. Some trial plots received applications at both these growth stages.

Representative soil cores were taken from each of the trial plots at normal harvest without any tillage. Additional soil cores were taken from two of the plots at a time corresponding to the normal sowing of an autumn following crop. The soil cores were divided into two horizons (0 - 10 cm and 10 - 20 cm) except from one location where the soil was kept as a single 0 - 20 cm core. The samples were analysed by either organic extraction or, to determine the bioavailable residues, by aqueous extraction. Both florasulam and the 5-OH metabolite are quantified in these two methods. Results were calculated on a dry-weight soil basis. “

Findings :

Table 7.1.1.2.2-9 : Residues of florasulam in soil 'at harvest' following application of EF-1343 (SC containing 50 g/l florasulam)

Trial No	Country	Year	Soil Texture	pH	% OC	Rate g as/ha	PH I (d)	µg a.s./kg soil			
								0-10 cm		0-10 cm	
								Org	Aqu	Org	Aqu
R96-114A ²	France - N	1996	Sandy silt loam	7.6	0.9	7.5 ^a	64	<0.05	na	ND	na
R96-116A ¹	France - N	1996	Silt loam	6.8	0.9	7.5 ^a	90	<0.05	na	ND	na
R96-112A ¹	France - S	1996	Silty clay loam	6.5	1.0	5 ^a	86	ND	na	ND	na
R95-015A ²	France - S	1995	Clay loam	8.1	1.7	5 ^{**a}	63	<0.05	ND	ND	ND
						7.5 ^{**a}	63	<0.05	<0.05	ND	ND
							140	ND	ND	ND	ND
						7.5 ^{**c}	57	<0.05	0.07	<0.05	ND
							134	ND	ND	ND	ND
R96-112A ¹	France - S	1996	Silty clay loam	6.5	1.0	7.5 ^a	86	<0.05	na	ND	na
RF96-011A ₁	Germany	1996	Silty clay loam	8.2	1.6	6.5, 7.5 ^c	69	<0.05	na	ND	na
						7.1 ^a	94	ND	na	ND	na
						7.5 ^b	69	ND	na	ND	na
RF95-006A ₁	Germany	1995	Silty clay loam	4.5	1.35	7.5 ^{*a}	89	ND	ND	ND	ND
						7.5 ^a	89	ND	ND	ND	ND
RF95-009A ₂	Germany	1995	Clay loam	6.2	1.56	7.5 ^{*a}	78	ND	na	ND	na
						7.5 ^a	78	ND	ND	ND	ND
R95-011A ³	Italy	1995	Clay	7.9	1.9	5 ^{*a}	96	ND	ND	ND	ND
R96-010A ¹	Italy	1996	Clay	8.5	2.3	5 ^{*a}	86	ND	na	ND	na
						5 ^a	86	<0.05	na	ND	na
R95-012A ²	Italy	1995	Silty Clay	7.3	1.5	5 ^a	96	ND	ND	ND	ND
R96-006A ²	Spain	1996	Silty clay	8.6	1.1	4.2 ^a	119	<0.05	na	<0.05	na
						4.6 ^{*a}	119	<0.05	na	<0.05	na
R95-046A ¹	Spain	1995	Sandy clay loam	8	0.9	5 ^{*a}	84	0.18	0.22	<0.05	<0.05
R95-045A ²	Spain	1995	Sand	8.3	0.4	5 ^a	74	0.12	0.10	0.06	0.05
R95-049A ¹	UK	1995	Clay loam	6.7	1.5	7.5 ^a	115	na	0.08 [#]	na	na
						7.5 ^b	69	na	0.07 [#]	na	na
						7.5 ^c	69	na	0.11 [#]	na	na
R95-049B ¹	UK	1995	Sandy clay loam	6.6	1.1	7.5 ^a	107	0.06	0.07	ND	ND
						7.5 ^b	76	0.12	0.12	<0.05	<0.05
						7.5 ^c	76	0.14	0.14	ND	<0.05
R96-002A ²	UK	1996	Sandy loam	7.4	2.3	7.5 ^a	94	0.05	na	ND	na
R96-003A ¹	UK	1996	Sandy loam	6.2	1.5	7.5 ^a	96	0.38	0.24	<0.05	na
						7.5 ^b	65	0.46	0.26	0.06	na
						7.5 ^c	65	1.03	0.68	<0.05	na
R95-051A ²	UK	1995	Sandy loam	7.7	1.4	7.5 ^b	72	<0.05	<0.05	ND	ND
						7.5 ^c	72	0.06	<0.05	ND	ND
R96-002A ²	UK	1996	Sandy loam	7.4	2.3	7.5 ^b	70	<0.05	na	ND	na
						7.5 ^c	70	0.12	<0.05	<0.05	na

¹ = Winter Wheat, ² = Winter Barley, ³ = Durum Wheat na = not analysed, ND = not detected (<0.01 µg/kg)

a = single application at GS 32, b = single application at GS 49 c = two applications at GS 32 and 49

* = applied with 0.1% polyglcol adjuvant, ** = applied with 0.1% Agral adjuvant, # = 0 - 20 cm horizon

Residues of metabolite 5-hydroxy in soil 'at harvest' following application of EF-1343 (SC containing 50 g/l florasulam)

Trial No	Country	Year	Soil Texture	pH	% OC	Rate g as/ha	PHI (d)	µg a.s./kg soil			
								0-10 cm		0-10 cm	
								Org	Aqu	Org	Aqu
R96-114A ²	France - N	1996	Sandy silt loam	8	0.9	7.5 ^a	64	0.13	na	<0.05	na
R96-116A ¹	France - N	1996	Silt loam	7	0.9	7.5 ^a	90	0.36	na	<0.05	na
R96-112A ¹	France - S	1996	Silty clay loam	7	1.0	5 ^a	86	0.07	na	<0.05	na
R95-015A ²	France - S	1995	Clay loam	8	1.7	5** ^a	63	0.07	0.10	ND	ND
						7.5** ^a	63	0.16	0.22	<0.05	ND
							140	ND	ND	ND	ND
						7.5** ^c	57	0.16	0.28	<0.05	<0.05
							134	ND	<0.05	ND	ND
R96-112A ¹	France - S	1996	Silty clay loam	7	1.0	7.5 ^a	86	0.12	na	<0.05	na
RF96-011A ¹	Germany	1996	Silty clay loam	8	1.6	6.5, 7.5 ^c	69	0.08	na	<0.05	na
						7.1 ^a	94	0.05	na	ND	na
						7.5 ^b	69	0.1	na	<0.05	na
RF95-006A ¹	Germany	1995	Silty clay loam	5	1.4	7.5** ^a	89	0.08	0.05	ND	ND
						7.5 ^a	89	0.11	0.06	<0.05	<0.05
RF95-009A ²	Germany	1995	Clay loam	6	1.6	7.5** ^a	78	0.06	na	ND	na
						7.5 ^a	78	0.09	na	ND	na
R95-011A ³	Italy	1995	Clay	8	1.9	5** ^a	96	ND	ND	ND	ND
R96-010A ¹	Italy	1996	Clay	9	2.3	5** ^a	86	ND	na	ND	na
						5 ^a	86	ND	na	ND	na
R95-012A ²	Italy	1995	Silty Clay	7	1.5	5 ^a	96	ND	ND	ND	ND
R96-006A ²	Spain	1996	Silty clay	9	1.1	4.2 ^a	119	0.05	na	na	na
						4.6** ^a	119	0.05	na	ND	na
R95-046A ¹	Spain	1995	Sandy Clay loam	8	0.9	5** ^a	84	0.15	0.19	<0.05	<0.05
R95-045A ²	Spain	1995	Sand	8	0.4	5 ^a	74	0.26	0.16	0.12	0.10
R95-049A ¹	UK	1995	Clay loam	7	1.5	7.5 ^a	115	na	0.46 [#]	na	na
						7.5 ^b	69	na	0.59 [#]	na	na
						7.5 ^c	69	na	1.15 [#]	na	na
R95-049B ¹	UK	1995	Sandy clay loam	7	1.1	7.5 ^a	107	0.34	0.76	ND	<0.05
						7.5 ^b	76	0.45	0.66	0.06	0.06
						7.5 ^c	76	0.72	1.04	0.10	0.10
R96-002A ²	UK	1996	Sandy loam	7	2.3	7.5 ^a	94	0.42	na	<0.05	na
R96-003A ¹	UK	1996	Sandy loam	6	1.5	7.5 ^a	96	1.18	1.05	0.09	na
						7.5 ^b	65	0.74	0.52	0.16	na
						7.5 ^c	65	2.1	1.79	0.10	na
R95-051A ²	UK	1995	Sandy loam	8	1.4	7.5 ^b	72	0.08	0.07	<0.05	<0.05
						7.5 ^c	72	0.14	0.10	<0.05	<0.05

R96-002A ²	UK	1996	Sandy loam	7	2.3	7.5 ^b	70	0.46	na	<0.05	na
						7.5 ^c	70	1.09	1.00	0.06	na

¹ = Winter Wheat, ² = Winter Barley, ³ = Durum Wheat na = not analysed, ND = not detected (<0.01 µg/kg)

a = single application at GS 32, b = single application at GS 49 c = two applications at GS 32 and 49

* = applied with 0.1% polyglcol adjuvant, ** = applied with 0.1% Agral adjuvant, # = 0 - 20 cm horizon

Conclusions :

Active substance and metabolite 5-OH concentrations measured in soil during the supervised field trials are confirming the results obtained in the field dissipation studies.

B.7.1.3.2 Soil residue testing - soil accumulation testing

The study is not required.

B.7.2 Adsorption, desorption and mobility in soil (Annex IIA 7.1.2 and 7.1.3; Annex IIIA 9.1.2)

B.7.2.1 Adsorption and desorption of the active substance and relevant metabolites (Annex IIA 7.1.2)

Mobility studies of XDE-570 and 5-hydroxy-XDE-570 (Ostrander, 1996)

Guidelines :

OECD Guideline No. 106 \equiv FIFRA Guideline No. 163-1

GLP :

Yes

Material and methods :

Test substance :

¹⁴C phenyl labeled florasulam, radiochemical purity : 98.5%

¹⁴C phenyl labeled 5-hydroxy metabolite, radiochemical purity : 97.6%

Soils :

Table B.7.2.1-1 : Characteristics of the soils - Adsorption /desorption of florasulam and metabolite 5-hydroxy

	Catlin	Hanford	Pewamo	Fuquay	Kenslow	Speyer
Country of origin	USA	USA	USA	USA	UK	Germany
Soil type	silty clay loam	sandy loam	clay	sand	silt loam	sandy loam
Textural analysis (%):						
sand (2000-63 μ m)	10.8	60.0	14.8	90.8	18.4	75.4
silt (63-2 μ m)	62.0	31.2	32.0	8.0	58.6	14.9
clay (<2 μ m)	27.2	8.8	53.2	1.2	23.1	9.7
pH (KCl)	7.0	7.4	5.7	4.7	6.1	7.3
Organic carbon (%)	2.2	1.0	2.4	6.4	6.8	3.9
Cation exchange capacity (mEquiv/100g)	15.3	5.1	22.5	2.0	16.0	8.7

Experimental design :

The adsorption and desorption of [¹⁴C] florasulam and its main metabolite in 6 soils was determined by the equilibrium method.

For the a.s., 2 soil samples/conc./soil type (1/5 soil/CaCl₂ ratio) were shaken in calcium chloride solutions (four dilutions: 1.3, 0.13, 0.013, 0.0067 mg/kg soil) continuously for 2 hours at 25°C to measure adsorption. Desorption equilibration time was 2 hours. 2 replicates /soil/concentration

For the metabolite 5-hydroxy, 2 soil samples/conc./soil type (1/2 soil/CaCl₂ ratio) were shaken in calcium chloride solutions (four dilutions: 1.4, 0.14, 0.014, 0.007 mg/kg soil) continuously for 24 \pm 2 hours at 25°C to measure adsorption. Desorption equilibration time was 18 \pm 2 hours. 2 replicates /soil/concentration

The recovery of the radioactivity was 101.2 \pm 1.8 % and 96.9 \pm 4%, for the a.s. and its main metabolite respectively. HPLC analysis of the supernatant confirmed that <5% a.s. degradation occurred during the 2-h adsorption and desorption times. Analyses confirmed that <3% and < 6% metabolite degradation took place during the 24-h adsorption and 18h desorption times .

The Freundlich adsorption constants K_f and 1/n, and the K_{oc} values, were calculated for each soil.

Findings :

Table B.7.2.1-2 : Adsorption/desorption of florasulam

Soil	Adsorption					Desorption				
	K _f	1/n	R ²	Mean K _d (ml/g)	Mean K _{oc}	K _f	1/n	R ²	Mean K _d (ml/g)	Mean K _{oc}
Catlin	0.89	0.88	0.992	0.33	15	2.19	0.88	0.97	1.05	49
Hanford	0.22	0.86	0.943	0.08	8	3.18	0.64	0.79	0.49	50
Pewamo	1.88	0.92	0.995	0.94	38	4.25	0.89	0.98	2.00	82
Fuquay	0.35	1.00	0.978	0.35	54	1.31	0.96	0.89	1.24	194
Kenslow	1.47	0.94	0.998	0.90	13	2.33	0.94	0.99	1.45	21
Speyer 2.2	0.13	0.95	0.810	0.14	4	3.94	0.64	0.78	0.50	13

Table B.7.2.1-3 : Adsorption/desorption of 5-hydroxy metabolite

Soil	Adsorption					Desorption				
	K _f	1/n	R ²	Mean K _d (ml/g)	Mean K _{oc}	K _f	1/n	R ²	Mean K _d (ml/g)	Mean K _{oc}
Catlin	0.69	0.88	0.994	0.23	11	1.49	0.79	0.971	0.30	14
Hanford	0.21	0.95	0.892	0.16	16	0.47	0.94	0.770	0.49	49
Pewamo	1.73	0.90	0.998	0.72	30	2.54	0.85	0.998	0.76	31
Fuquay	0.24	0.98	0.986	0.20	32	0.08	1.16	0.920	0.35	55
Kenslow	1.55	0.90	0.999	0.66	10	1.15	0.94	0.997	0.72	11
Speyer 2.2	0.07	1.10	0.827	0.28	7	0.75	0.92	0.958	0.42	11

Conclusions :

Florasulam has 'very high mobility' according to Mc Call classification.

The metabolite 5-hydroxy has 'very high mobility' according to Mc Call classification.

The soil sorption of DFP-ASTCA and ASTCA (two metabolites of DE-570) (Jackson, et al., 1999)

Guidelines :

OECD Guideline No. 106

GLP :

Yes

Material and methods :

Test substance :

DFP-ASTCA, ¹⁴C triazole label, radiochemical purity = 90.4%

ASTCA, ¹⁴C triazole label, radiochemical purity = 99%

Soils :

Table B.7.2.1-4 : Characteristics of the soils - Adsorption /desorption of DFP-ASTCA and ASTCA

	Marcham	Cuckney	Sutton	Thessaloniki	Elvedon	Toulouse	Tours	Wetterfield	Catlin	Hanford
Country of origin	UK	UK	UK	Greece	UK	France	France	Germany	USA	USA
Soil type	sandy clay loam	sand	sandy clay loam	sandy silt loam	sand	clay	silty clay	silty clay loam	silty loam	sandy loam
Textural analysis (%):										
sand (2000-63 µm)	51.4	88.0	44.6	38.5	91.6	26.0	6.9	3.9	11.2	60.0
silt (63-2 µm)	21.2	5.1	34.1	49.8	4.7	36.5	63.5	79.4	62.0	31.2
clay (<2 µm)	27.4	6.9	21.3	11.7	3.7	37.5	29.6	16.7	26.8	8.8
pH (KCl)	7.9	7.2	7.5	8.5	7.6	8.2	7.4	6.1	6.5	7.4
Organic carbon (%)	3.4	1.5	2.1	0.9	1.1	1.2	1.2	1.2	1.7	1.0

Experimental design :

The adsorption and desorption of [¹⁴C]DFP-ASTCA and ASTCA in 10 soils was determined by the equilibrium method. For both metabolites, 2 soil samples/soil type (1/5 soil/CaCl₂ ratio) were shaken in calcium chloride solutions (concentrations of 63 and 40 µg/l for DFP-ASTCA and ASTCA, respectively) continuously for 1 hour at 25°C to measure adsorption. Desorption equilibration time was 1 hour. 2 replicates /soil.

No degradation of either substance in either of the soil/solutions mixtures

The Freundlich adsorption constants K_d and K_{oc} values, were calculated for each soil.

Findings :

Table B.7.2.1-5 : Adsorption/desorption of DFP-ASTCA

Soil	Adsorption		Desorption	
	Mean K _d (ml/g)	Mean K _{oc}	Mean K _d (ml/g)	Mean K _{oc}
Marcham	0.94	28	1.22	36
Cuckney	0.39	26	0.63	42
Sutton	0.68	32	0.90	43
Thessaloniki	0.90	100	1.24	138
Elvedon	0.26	24	0.50	46
Toulouse	0.64	53	0.80	67
Tours	0.65	54	0.89	74
Wetterfield	0.63	52	0.88	73
Catlin	0.88	52	1.30	76
Hanford	1.10	110	1.69	169

Table B.7.2.1-6 : Adsorption/desorption of ASTCA

Soil	Adsorption		Desorption	
	Mean K_d (ml/g)	Mean K_{oc}	Mean K_d (ml/g)	Mean K_{oc}
Marcham	1.65	49	1.88	55
Cuckney	0.42	28	0.57	38
Sutton	1.87	89	2.26	108
Thessaloniki	1.00	111	1.30	144
Elvedon	0.30	27	0.52	47
Toulouse	0.89	74	1.08	90
Tours	1.78	148	2.00	167
Wetterfield	0.60	50	0.73	61
Catlin	1.62	95	2.13	125
Hanford	1.59	159	2.22	222

Conclusions :

Koc of 24-110 indicate that DFP-ASTCA has 'very high to high mobility' according to Mc Call classification.
Koc of 27-159 indicate that ASTCA has 'very high to high mobility' according to Mc Call classification.

The sorption of XDE-570 in soil (Pillar, 1997a)

This exploratory study evaluated the adsorption of florasulam in 9 European soils at only one test concentration : 0.07 mg a.s. /l calcium chloride solution.

The resulting Koc (2, 6, 7, 7, 8, 10, 10, 15, 69) cannot be taken into account in the final evaluation of the dossier.
They however confirm the results of the previous study (Ostrander, 1996)

B.7.2.2 Column leaching studies with the active substance and relevant metabolites (Annex IIA 7.1.3.1; Annex IIIA 9.1.2.1)

The Non-aged Soil Column Leaching of DE-570 (Pillar, 1997d)

Guidelines :

German BBA Guidelines for the Official Examination of Plant Protection Agents, Part IV, Section 4.2.

GLP :

No

Material and methods :

Test substance :

¹⁴C triazolopyrimidine **labeled** florasulam, radiochemical purity > 97%

Soils :

Table B.7.2.2-1 : Characteristics of the soils - Column leaching with florasulam fresh residue

	Cuckney	Elvedon	Marcham
Country of origin	UK	UK	UK
Soil type	sand	sand	sandy clay loam
Textural analysis (%):			
sand (2000-63 µm)	85.42	91.60	57.30
silt (63-2 µm)	7.18	4.68	18.22
clay (<2 µm)	7.40	3.72	18.22
pH (KCl)	6.6	7.6	7.7
Organic carbon (%)	0.8	1.1	2.0
Cation exchange capacity (mEq/100g)	-	-	-
Moisture holding capacity (%)	-	-	-
Microbial biomass (µg C/g)	-	-	-

Experimental design :

The mobility of [¹⁴C-triazolopyrimidine] florasulam was investigated in 3 soils using glass columns (30 cm length, 5 cm internal diameter, 5 cm segments). The test substance was applied to the top of 1 pre-conditioned soil column for each soil type at a rate equivalent to 15 g a.s./ha (2.94 µg/ column) and leached with 393 cm³ 0.01 M calcium chloride solution (200 mm rainfall) over a period of two days at room temperature . At the end of the leaching period the radioactivity in the leachate was determined by liquid scintillation counting (LSC). Soil segments were determined by combustion and LSC. All leachates were analysed by high performance liquid chromatography (HPLC).

Findings :

Table B.7.2.2-2 : Mean distribution of radioactivity (% of applied) in soil columns and leachates from columns treated with florasulam

Soil	Cuckney Sand (pH 6.6)	Elvedon Sand (pH 7.6)	Marcham Sandy Clay Loam (pH 7.7)
Soil Horizon			
0 - 5 cm	1.3	0.4	3.3
5 - 10 cm	1.6	0.2	2.1
10 - 15 cm	4.0	0.4	3.0
15 - 20 cm	3.7	0.6	3.4
20 - 25 cm	5.0	1.2	8.1
25 - 30 cm	5.9	2.5	9.4
Total in Soil	21.5	5.5	29.3
Leachate			
0 - 18 hours	nd	nd	0.1
18 - 26.5 hours	8.5	0.1	9.1
26.5 - 43 hours	56.4	82.6	44.1
43 - 48 hours	12.0	9.4	14.4
Total in Leachate	76.9	92.1	67.7
Total Recovery	98.4	97.6	97.0

The major components of the leachate were florasulam (41-54% applied radioactivity) and metabolite 5-hydroxy (22-39%) A third component was found at low level (<5% applied radioactivity) in 2 soils

Conclusions :

This study confirms the high leaching potential of florasulam and metabolite 5-hydroxy. Significant degradation of the a.s. occurred during the 2-day leaching period.

B.7.2.3 Aged residue column leaching (Annex IIA 7.1.3.2; Annex IIIA 9.1.2.1)

No study with aged residue was performed.

B.7.2.4 Lysimeter and field leaching studies (Annex IIA 7.1.3.3; Annex IIIA 9.1.2.2)

The dissipation of XDE-570 in soil and crops using field lysimeters (Jackson, et al., 1997)

Guidelines :

SETAC \equiv BBA Guideline Part IV, Section 4.3

GLP :

Yes

Material and Methods :

Test substance : ^{14}C triazolopyrimidine **labeled florasulam, radiochemical purity > 97%**

Soil :

Table B.7.2.4-1 : Characteristics of the soils - Lysimeter study

Origin of soil:	Cuckney, UK				Sutton, UK				
Classification:	Sand				Loam				
Depth (cm):	0	29-50	50-73	73-100	0	22-52	52-79	79-92	92-100
Particle size distribution:									
% sand	88	88	90	86	52	49	67	77	90
% silt	6	7	5	2	29	25	15	16	7
% clay	6	5	5	12	19	26	18	7	3
% Organic carbon:	0.6	0.2	0.1	0.1	2.49	0.83	0.55	0.19	0.11
PH (water)	6.2	6.2	5.4	6.0	6.5	7.6	8.0	8.4	8.5

Test conditions :

- The product was applied to the lysimeters (0.8 m² surface area and 1 m depth) filled with undisturbed soil monoliths.

- Nominal and actual application rates as well as the total radioactivity applied were mentioned for each lysimeter.

- The total precipitation of each year were :

year 1 : April 94 - April 95- 1005 mm (including 96 mm irrigation)

year 2 : April 95 - April 96 - 773 mm (including 55 mm irrigation)

year 3 : April 96 - April 97 - 509 mm (including 25 mm irrigation)

- The total amounts leachate were 158.3-213.01 during the first year of the study. They were 137.0-151.61 during the second year of the study.

Analytical methods :

Leachates : Total radioactivity were determined by liquid scintillation counting (LSC). The individual leachate samples of lysimeter 29 were concentrated using a solid phase extraction procedure. The concentrated extracts were stored frozen. HPLC analysis was carried out on the annual pooled leachate.

Soil : All samples were analysed for total radioactivity by combustion and LSC. Samples with sufficient radioactivity were extracted and analysed by TLC and HPLC.

Crop : Total radioactivity in the crops was analysed by combustion and LSC.

Experimental design :

Several lysimeters were installed in order to investigate the leaching of florasulam under different soil/ application rate/ application date conditions. The effect of single application or applications during 2 successive years was also assessed.

Table B.7.2.4-2 : Lysimeter study - Experimental design

Soil	Sand, Cuckney					Loam, Sutton	
Lysimeter identification	25	26	27	28	29	31	33
First crop	Winter barley		Winter wheat			Winter wheat	
First year	(April 95-April 96)		(April 94-April 95)			(April 94-April 95)	
application rate (g a.s./ha)	5	5	5	5	25	5	5
application date	16-2-95	16-2-95	19-4-94	19-4-94	20-4-94	20-4-94	20-4-94
Following crops	winter wheat	winter wheat	winter barley	winter oilseed rape	winter barley	winter barley	winter barley
Second year	(April 96-April 97)		(April 95-April 96)			(April 95-April 96)	
application rate (g a.s./ha)	-	-	5	-	-	5	-
application date	-	-	20-4-95	-	-	20-4-95	-

Table B.7.2.4-3 : Radioactivity recovered in the leachates of the lysimeters - concentrations expressed in µg equivalent florasulam/ l and % applied radioactivity for the total leachate of one year

Lysimeter identification		25	26	27	28	29	31	33
first year	mean conc. µg/l	0.04	0.04	0.05	0.04	0.27	0.01	0.01
	% appl. radioactivity	2.466	2.457	4.389	3.897	4.433	1.043	0.753
second year	mean conc. µg/l	0.03	0.03	0.05	0.03	0.21	0.01	0.01
	% appl. radioactivity	0.982	1.076	1.519	1.79	2.388	0.268	0.758
third year	mean conc. µg/l	-	-	0.04	-	-	-	< 0.01
	% appl. radioactivity	-	-	0.44	-	-	-	0.078

HPLC analysis of the leachates produced by the lysimeter 29 (exaggerated application rate of 25g /ha) revealed that the main components recovered in water were the metabolites 5-hydroxy, ASTCA, DFP-TSA and polar compounds. The active substance was not detected in the leachates (only one occurrence in the leachate of 18/1/95-8/2/95 at 0.03 µg/l (appl. rate of 25 g a.s./ha))

Conclusions :

- The total radioactivity recovered in the combined leachates of one year was detected at level < 0.1 µg/l. The radioactivity was identified as metabolites 5-hydroxy, ASTCA, DFP-TSA and polar compounds.
- Florasulam was not detected in the leachates.

B.7.3 Summary of behaviour in soil

Table B.7.3-1 : Summary of behaviour in soil - Laboratory degradation rates of florasulam and its metabolite 5-OH

Test soil				Test system	Results (days)				References
Soil type Location	pH	CEC meq/ 100g	O.C. (%)		florasulam		metabolite 5-OH		
					DT ₅₀	DT ₉₀	DT ₅₀	DT ₉₀	
Speyer 2.2 loamy sand (TP)	7.3	8.7	3.9	aerobic, 20°C	1.7	5.7	24	79	Jackson, 1997
Speyer 2.2 loamy sand (Ph)	7.3	8.7	3.9		1.7	5.6	26	88	
sterile Speyer 2.2 (TP)	7.3	8.7	3.9		116	-	-	-	
Marcham sandy clay loam (TP)	7.7	9.0	2.0		4.5	15	29	95	
Kenslow humus silt loam (TP)	5.6	16.0	6.8		0.7	2.2	31	102	
Andover silt loam (TP)	7.6	12.0	3.1		1.5	5.1	10	34	
Speyer 2.2 loamy sand (TP)	5.8	5.33	2.66	anaerobic, 20°C	11	38	-	-	Cleveland, et al., 1997
Speyer 2.2 loamy sand (Ph)	5.8	5.33	2.66		14	46	-	-	
Catlin, silt loam	6.8	15.62	1.70	photolysis, 25°C	62	-	-	-	Kreger, et al., 1996
Marcham sandy clay loam (TP)	7.6	23.50	1.4	aerobic, 5°C	45	149	-	-	Pillar, 1997c
				aerobic, 15°C	15	50	42	141	
				aerobic, 25°C	5.2	17	18	60	
Cuckney sand (TP)	6.9	14.1	1.4	aerobic, 5°C	19	64	-	-	
				aerobic, 15°C	3.5	11	33	109	
				aerobic, 25°C	1.1	36	21	70	
Marcham sandy clay loam (TP)	7.6	23.50	1.4	saturation point, 20°C	5.7	19	72	238	Pillar, 1997b
				field capacity, 20°C	7.4	25	36	119	
				wilt point, 20°C	8.1	27	46	154	
Cuckney sand (TP)	6.9	14.1	1.4	saturation point, 20°C	6.1	20	54	179	
				field capacity, 20°C	10	34	42	141	
				wilt point, 20°C	8.2	27	86	286	

Table B.7.3-3 : Summary of behaviour in soil - Laboratory degradation rates of metabolites DFP-ASTCA and ASTCA

Test soil				Test system	Results (days) DT ₅₀	References
Soil type Location	pH	CEC meq/ 100g	O.C. (%)			
Marcham sandy clay loam (TP)	7.9	25.0	3.4	aerobic, 20°C,	DT ₅₀ (DFP-ASTCA) = 25 d DT ₅₀ (ASTCA) = 502 d	Jackson, et al., 1998
Cuckney sand (TP)	6.9	14.1	1.4	aerobic, 20°C,	DT ₅₀ (DFP-ASTCA) = 8 d DT ₅₀ (ASTCA) = 158 d	

Table B.7.3-3 : Summary of behaviour in soil - Field soil dissipation rates of florasulam and its metabolite 5-OH

Test soil				Test system - appl. rate (g a.s./ha) - temperature (*)	Results (days)				References
Soil type Location	pH	CEC meq/ 100g	O.C. (%)		florasulam		metabolite 5-OH		
					DT ₅₀	DT ₉₀	DT ₅₀	DT ₉₀	
Silty clay loam Tours, N France, 1995	7.4	29.1	1.2	- 13.5 g a.s./ha - 10 °C	9	31	17	55	Maycock, 1995
Silty loam Wetterfeld, Germany, 1995	6.1	13.9	1.2	18 °C *	15	50	14	59	
Sandy clay loam Marcham, UK, 1995	7.7	25.8	2.0	10 °C	5	50	42	209	
Sand Elvedon, UK, 1996	7.6	11.8	1.1	1 °C	18	61	95	182	
Clay loam St Livrade, S France, 1996	8.2	25.2	1.2	10 °C	8	28	9	41	
Sandy silt loam Valtothori, Greece, 1996	8.5	10.0	0.9	11 °C	2	23	13	61	

* : average soil temperature during the first 7 days after application

Table B.7.3-3 : Summary of behaviour in soil - Koc determination of florasulam and its metabolite 5-OH

Test soil				Test system	Results						References
Soil type Location	pH	CEC meq/ 100g	O.C. (%)		florasulam			metabolite 5-OH			
					Kf	1/n	Koc	Kf	1/n	Koc	
Catlin, silty clay loam	7.0	15.3	2.2	Laboratory adsorption /desorption	0.89	0.88	15	0.69	0.88	11	Ostrander, 1996
Hanford, sandy loam	7.4	5.1	1.0		0.22	0.86	8	0.21	0.95	16	
Pewamo,clay	5.7	22.1	2.4		1.88	0.92	38	1.73	0.90	30	
Fuquay, sand	4.7	2.0	6.4		0.35	1.00	54	0.24	0.98	32	
Kenslow, silt loam	6.1	16.0	6.8		1.47	0.94	13	1.55	0.90	10	
Speyer sandy loam	7.3	8.7	3.9		0.13	0.95	4	0.07	1.10	7	

Table B.7.3-4 : Summary of behaviour in soil - Koc determination of metabolites DFP-ASTCA and ASTCA

Test soil				Test system	Results				References
Soil type Location	pH	CEC meq/ 100g	O.C. (%)		DFP-ASTCA		ASTCA		
					Kd	Koc	Kd	Koc	
Marcham sandy clay loam	7.9	-	3.4	Laboratory adsorption /desorption	0.94	28	1.65	49	Jackson, et al., 1999
Cuckney sand	7.2	-	1.5		0.39	26	0.42	28	
Sutton sandy clay loam	7.5	-	2.1		0.68	32	1.87	89	
Thessaloniki sandy silt loam	8.5	-	0.9		0.90	100	1.00	111	
Elvedon sand	7.6	-	1.1		0.26	24	0.30	27	
Toulouse clay	8.2	-	1.2		0.64	53	0.89	74	
Tours silty clay	7.4	-	1.2		0.65	54	1.78	148	
Wetterfield silty clay loam	6.1	-	1.2		0.63	52	0.60	50	
Catlin silty loam	6.5	-	1.7		0.88	52	1.62	95	
Hanford sandy loam	7.4	-	1.0		1.10	110	1.59	159	

Table B.7.3-5 : Summary of behaviour in soil - Column leaching

Test soil				Test system	Results % applied radioactivity in the leachate	References
Soil type Location	pH	CEC meq/ 100g	O.C. (%)			
Cuckney sand (TP)	6.6	-	0.8	Column leaching, fresh residue 15 g a.s./ha, 2 days, 200 mm rainfall	76.9	Pillar, 1997d
Elvedon sand (TP)	7.6	-	1.1		92.1	
Marcham sandy clay loam (TP)	7.7	-	2.0		67.7	

Table B.7.3-6 : Summary of behaviour in soil - Outdoor lysimeter study

Test soil				Test system	Results	References
Soil type Location	pH	CEC meq/ 100g	O.C. (%)			
Sand, Cuckney	5.4-6.2	-	0.6 to 0.1	1 application/year at 5 g a.s./ha	Total residue of one year : 0.03 to 0.05 µg/l	Jackson et al., 1997

Sand, Cuckney	5.4-6.2	-	0.6 to 0.1	1 application/year at 25 g a.s./ha (exaggerated rate)	Total residue of one year : 0.21 µg/l - No a.s. in the leachate - Metabolites 5-hydroxy, ASTCA, DFP-TSA and polar compounds were recovered in the leachate.
Loam, Sutton	6.5-8.5	-	2.49 to 0.11	1 application/year at 5 g a.s./ha	Total residue of one year : <0.01 to 0.01 µg/l

Predicted environmental concentration in soil (PECs) Annex IIIA 9.1.3)

The PEC were calculated assuming that

- Florasulam has a DT₅₀ of 18 days (maximum field DT₅₀; very conservative assumption); metabolite 5-OH has a DT₅₀ of 95 days (maximum field DT₅₀; very conservative assumption)
- The active substance is applied once/season, at the maximum rate of 7.5 g a.s./ha, in cereals (2-3 leaves to stem elongation)
- 100% of the spray reaches the soil surface.
- The active substance is distributed in a 5 cm soil layer with a soil density of 1.5 g/cm³

Table B.7.3-7 : PEC soil of the active substance and metabolite 5-OH

Time after applications (days)	Cereals 7.5 g a.s./ha 1 application 100% of applied dose reaching the soil			
	Florasulam		metabolite 5-OH	
	Actual concentration (mg/kg soil)	TWA concentration (mg/kg soil)	Actual concentration (mg/kg soil)	TWA concentration (mg/kg soil)
0	0.0100	0.0100	0.0100	0.0100
1	0.0096	0.0098	0.0099	0.0100
2	0.0093	0.0096	0.0099	0.0099
4	0.0086	0.0093	0.0097	0.0099
7	0.0076	0.0088	0.0095	0.0097
14	0.0058	0.0077	0.0090	0.0095
21	0.0044	0.0069	0.0086	0.0093
28	0.0034	0.0061	0.0081	0.0090
50	0.0015	0.0044	0.0069	0.0084
100	0.0002	0.0025	0.0048	0.0071

B.7.4 Fate and behaviour in water (Annex IIA 7.2.1; Annex IIIA 9.2)

B.7.4.1 Hydrolysis rate of relevant metabolites, degradation and reaction products (Annex IIA 7.2.1.1)

2 hydrolysis studies were performed with the a.s.. These studies are described under point B.2.1.15

The Determination of the Hydrolytic Stability of Radiolabelled XDE-570 (Phillips, 1996)

The Aqueous Hydrolysis of XR-570 (Jackson, et al., 1993)

[phenyl-UL-¹⁴C]- and [9-triazolopyrimidine-¹⁴C]- labelled a.s., radiochemical purity resp. 99.4 and 98.4% :

Findings :

Tests at 25 °C at pH 5, 7 and 9 :

pH 5 : no degradation observed after 30 d

pH 7 : no degradation observed after 30 d

pH 9 : $k = 0.00692 \text{ d}^{-1}$; $t_{1/2} = 100 \text{ d}$ (phenyl-label)

$k = 0.00706 \text{ d}^{-1}$; $t_{1/2} = 98 \text{ d}$ (triazole-label)

Further test at 20 °C at pH 9 :

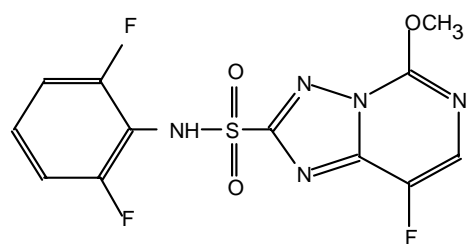
$k = 0.00316 \text{ d}^{-1}$; $t_{1/2} = 219 \text{ d}$ (phenyl-label)

$k = 0.00306 \text{ d}^{-1}$; $t_{1/2} = 226 \text{ d}$ (triazole-label)

Major hydrolysis products : metabolite 5-hydroxy and an intermediate compound formed by addition of water to the a.s.

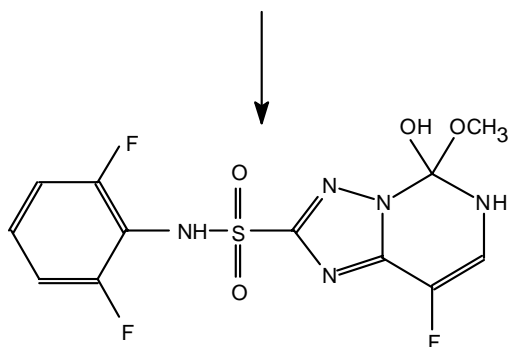
Conclusions :

Florasulam is hydrolytically stable at 20-25°C at pH 5-9. No hydrolysis study with metabolites is necessary.

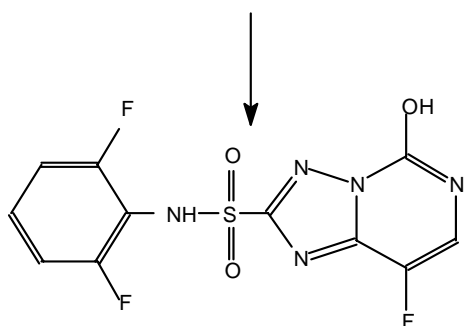


Florasulam

Figure B.7.4.1-1 : Proposed hydrolysis pathway of florasulam in water



Structure formed by addition of water



5-OH

B.7.4.2 Direct phototransformation of relevant metabolites, degradation and reactions products in water (Annex IIA 7.2.1.2)

Photodegradation studies were performed with the a.s. These studies are described under point B.2.1.16

Aqueous photolysis of XDE-570 in natural sunlight (Yoder, 1996)

[aniline-UL-¹⁴C]- and [9-triazolopyrimidine-¹⁴C]- labelled a.s. (AN and TP resp.), radiochemical purity resp. 97.7 and 99.0% :

Findings :

at 25°C, buffered at pH 5 (sterile conditions, acetonitrile as cosolvent) :

Mass balance :

- recovery in exposed samples ranged from 96.7 to 109.7% (AN) and from 96.6 to 101.1% (TP) of applied radioactivity
- (dark controls : from 88.5 to 103.3% (AN) and from 96.7 to 101.1% (TP))
- material balance after 32 d incubation (TP) :

	<u>% of applied radioactivity</u>
parent florasulam	75.5
TPSA	17.0

(dark-control : after 32 d, florasulam accounted for 95% of applied radioactivity)

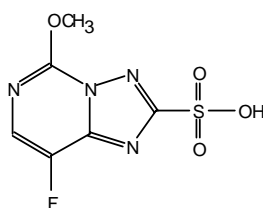
Photolysis rate :

$k = 0.00311 \text{ d}^{-1}$; $t_{1/2} = 223 \text{ d}$ (AN)

$k = 0.00791 \text{ d}^{-1}$; $t_{1/2} = 88 \text{ d}$ (TP)

TP samples received more sunlight than AN samples

Major photolysis product :



triazolosulfonic acid (TPSA) of florasulam (max. 17% of applied radioactivity)

Conclusions :

Method concurs with SETAC-procedures, except for use of natural sunlight instead of artificial light source. Results are thus difficult to compare to those obtained for other substances.

Degradation of DE-570 in a natural lakewater (Gibson, 1997)

The study evaluates the photodegradation rate of florasulam in natural lake water outdoors. The study does not concur with the SETAC requirements. This study was not taken into account in the final evaluation.

B.7.4.3 Ready biodegradability of the active substance (Annex IIA 7.2.1.3.1)

XDE-570 (pure) : Assessment of ready biodegradability (Jenkins, 1994)

Guidelines :

EEC Method C4

GLP :

Yes

Material and Methods :

Test substance :

Florasulam; purity : 99.7%

Experimental design :

A five-day bacterial inhibition assay was performed under the conditions of the Closed Bottle Test.

In the modified Sturm Test, florasulam was added to two vessels containing inoculated mineral salts medium, to give a nominal test concentration of 10 mg C/l.

Test and control vessels were aerated for 29 days with air that had been treated to remove carbon dioxide (CO₂). The CO₂ produced by each culture was trapped in a series of Drechsel bottles containing barium hydroxide.

Findings :

Closed bottle test : Florasulam at a nominal concentration of 10 mg C/l did not inhibit degradation of the reference material sodium benzoate. In this preliminary test, degradation of florasulam alone was insignificant.

Sturm test : The mean cumulative CO₂ production by the mixtures containing florasulam at 10 mgC/l was equivalent to 2 % of the TCO₂ (106.4 mg CO₂). Sodium benzoate was degraded by 71 % after seven days and 94 % after 29 days.

Conclusions :

Florasulam is not readily biodegradable.

XDE-570 5-hydroxy metabolite : Assessment of ready biodegradability (Jenkins, 1995)

Guidelines :

EEC Method C4

GLP :

Yes

Material and Methods :

Test substance :

XDE-570 5-hydroxy metabolite; purity : 97.0%

Experimental design :

A five-day bacterial inhibition assay was performed under the conditions of the Closed Bottle Test.

In the modified Sturm Test, 5-hydroxy metabolite was added to two vessels containing inoculated mineral salts medium, to give a nominal test concentration of 10 mg C/l.

Test and control vessels were aerated for 29 days with air that had been treated to remove carbon dioxide (CO₂). The CO₂ produced by each culture was trapped in a series of Drechsel bottles containing barium hydroxide.

Findings :

Closed bottle test : 5-hydroxy metabolite at a nominal concentration of 10 mg C/l did not inhibit degradation of the reference material sodium benzoate. In this preliminary test, degradation of 5-hydroxy metabolite was insignificant.

Sturm test : The mean cumulative CO₂ production by the mixtures containing 5-hydroxy metabolite at 10 mgC/l was equivalent to 2 % of the TCO₂ (106.4 mg CO₂). Sodium benzoate was degraded by 70 % after seven days and 93 % after 29 days.

Conclusions :

The 5-hydroxy metabolite is not readily biodegradable.

B.7.4.4 Water/sediment study (Annex IIA 7.2.1.3.2)

The aerobic degradation of radiolabelled XDE-570 in natural waters and associated sediments (Phillips, 1997)

Guidelines :

SETAC \equiv BBA Guideline Part IV, Section 5-1 (1990)

GLP :

Yes

Material and Methods :

Test substances :

^{14}C triazolopyrimidine labeled florasulam, radiochemical purity = 99%

^{14}C phenyl labeled florasulam, radiochemical purity = 99%

Water and sediment :

Samples were collected in 2 streams (15-25 cm depth) in UK. 'No pesticides have been applied to either site in the last 3 years.'

Table B.7.4.4-1 : Characteristics of the sediments

	Brown Carrick Hill	Auchingilsie
Redox potential at collection (mv)	30 mv	34 mv
pH at collection	5.4	5.9
Soil Type (UK Classification)	sandy loam	clay loam
Organic carbon (%)	2.2	2.9
Cation exchange capacity (meq/100g)	23.4	24.3
Particle size distribution (%)		
clay (< 2 μm)	13.12	25.71
silt (2 μm to 63 μm)	21.83	29.84
sand (63 μm to 2 mm)	65.04	44.46
Total nitrogen (mg/kg)	1900	1000
Total phosphorus (mg/kg)	656.9	615.8

Table B.7.4.4-2 : Characteristics of the waters

	Brown Carrick Hill	Auchingilsie
Temperature at collection	4°C	3.5°C
pH at collection	7.6	6.6
Oxygen concentration (%)		
- surface	101	102
- above the sediment	97	97
Redox potential	137 mv	138 mv
Total nitrogen (mg/l)	4.2	2.
Total phosphorus (mg/l)	0.1	< 0.05
Dissolved organic carbon (mg/l)	-	-
Water hardness (mg/l as CaCO_3)	61.0	10.0
Suspended solids (mg/l)	-	-

Oxygen concentration, pH and redox potential of water and sediment were monitored during the experiment.

Experimental design :

[^{14}C] Florasulam was applied at concentrations of 7.8 and 8.4 $\mu\text{g/l}$ to glass cylinders containing two equilibrated water/sediment systems at a depth of 2.5 cm of sediment and 6 cm of water and maintained at 20°C. The radioactivity in the water was determined by liquid scintillation counting and high performance liquid chromatography (HPLC) after elution through a C_{18} solid phase extraction (SPE) cartridge. Sediment was extracted twice with

acetone:water:acetic acid (90:9:1). Extracts were analyzed by LSC and by HPLC after elution through a C₁₈ SPE cartridge. Analysis was confirmed by TLC.

Findings :

Table B.7.4.4-3 : Distribution of applied radioactivity (mean %) after application of [¹⁴C-phenyl] florasulam to Brown Carrick Hill ditch water/sandy loam sediment system under aerobic conditions (HPLC)

	Sampling Interval									
	0 h	6 h	1d	2 d	7 d	14 d	30 d	60 d	100 d	182 d
<u>Volatile traps (CO₂)</u>	N.A.	-	-	-	-	-	-	-	3.73	8.05
<u>Water extract</u>	101.83	91.66	93.30	86.54	78.88	79.53	65.19	63.94	47.00	43.69
Florasulam	101.83	91.66	93.30	77.31	41.35	22.40	8.98	-	-	-
5-hydroxy	-	-	-	9.23	37.53	57.13	53.00	63.94	36.60	16.84
ΣUnknown 1, 2, 3	-	-	-	-	-	-	-	-	-	26.85
<u>Sediment extract</u>	2.78	10.91	9.54	16.28	21.61	20.02	33.82	35.23	32.67	32.54
Florasulam	n.a.	8.73	4.60	7.42	4.39	1.32	1.10	-	-	-
5-hydroxy	n.a.	2.18	4.94	8.86	17.22	17.47	24.71	35.06	23.64	11.47
ΣUnknown 1, 2, 3	n.a.	-	-	-	-	1.23	-	-	8.86	20.83
<u>Sediment residue</u>	-	1.11	-	1.18	2.54	5.36	5.78	7.28	11.25	11.49
<u>Apparatus wash</u>	-	-	-	-	-	-	-	-	-	-
<u>Mass balance</u>	104.61	103.68	102.84	104.00	103.03	104.91	104.79	106.45	94.65	95.77

Table B.7.4.4-4 : Distribution of applied radioactivity (mean %) after application of [¹⁴C-triazolpyrimidine] florasulam to Brown Carrick Hill ditch water/sandy loam sediment system under aerobic conditions (HPLC)

	Sampling Interval									
	0 h	6 h	1d	2 d	7 d	14 d	30 d	60 d	100 d	182 d
<u>Volatile traps (CO₂)</u>	-	-	-	-	-	-	-	-	1.93	1.93
<u>Water extract</u>	96.24	95.83	99.83	87.25	83.15	78.10	67.48	64.36	64.54	54.60
Florasulam	96.24	95.83	99.83	79.53	65.28	41.30	9.12	-	-	-
5-hydroxy	-	-	-	7.72	11.87	36.80	58.36	64.36	56.58	39.79
ΣUnknown 1, 2, 3	-	-	-	-	-	-	-	-	7.96	14.82
<u>Sediment extract</u>	6.40	5.18	4.14	12.54	15.44	19.47	24.50	27.55	34.33	36.44
Florasulam	n.a.	n.a.	n.a.	4.22	3.72	1.41	-	-	-	-
5-hydroxy	n.a.	n.a.	n.a.	8.32	11.72	18.06	24.50	27.55	32.84	26.35
ΣUnknown 1, 2, 3	n.a.	n.a.	n.a.	-	-	-	-	-	1.37	6.9
<u>Sediment residue</u>	-	-	-	-	-	6.18	8.73	9.28	7.34	8.52
<u>Apparatus wash</u>	-	-	-	-	-	-	-	-	-	-
<u>Mass balance</u>	102.64	101.01	103.97	99.79	98.59	103.75	100.71	101.19	108.14	101.49

Table B.7.4.4-5 : Distribution of applied radioactivity (mean %) after application of [¹⁴C-phenyl] florasulam to Auchingilsie ditch water/clay loam sediment system under aerobic conditions (HPLC)

	Sampling Interval									
	0 h	6 h	1d	2 d	7 d	14 d	30 d	60 d	100 d	182 d
<u>Volatile traps (CO₂)</u>	-	-	-	-	-	-	-	-	-	3.35
<u>Water extract</u>	106.12	103.40	91.82	104.24	94.63	89.47	70.11	58.75	63.15	50.83
Florasulam	106.12	103.40	91.82	104.24	94.63	79.39	44.23	7.62	-	-
5-hydroxy	-	-	-	-	-	10.08	25.88	51.13	63.15	43.07
ΣUnknown 1, 2, 3	-	-	-	-	-	-	-	-	-	7.76
<u>Sediment extract</u>	0.77	1.84	5.09	1.95	10.38	12.19	26.16	37.87	35.16	35.26
Florasulam	n.a.	n.a.	n.a.	n.a.	5.22	4.30	3.60	-	-	1.60
5-hydroxy	n.a.	n.a.	n.a.	n.a.	5.16	7.89	22.56	36.08	35.06	27.39
ΣUnknown 1, 2, 3	n.a.	n.a.	n.a.	n.a.				1.59		6.14
<u>Sediment residue</u>	-	-	-	-	-	3.03	6.68	12.44	4.53	11.13
<u>Apparatus wash</u>	-	-	-	-	-	-	-	-	-	-
Mass balance	106.89	105.24	96.91	106.19	105.01	104.69	102.95	109.06	102.84	100.57

Table B.7.4.4-6 : Distribution of applied radioactivity (mean %) after application of [¹⁴C-triazolpyrimidine] florasulam to Auchingilsie ditch water/clay loam sediment system under aerobic conditions (HPLC)

	Sampling Interval									
	0 h	6 h	1d	2 d	7 d	14 d	30 d	60 d	100 d	182 d
<u>Volatile traps (CO₂)</u>	-	-	-	-	-	-	-	-	-	-
<u>Water extract</u>	101.0	96.55	98.19	95.63	88.73	83.91	72.33	65.99	56.37	55.67
Florasulam	101.0	96.55	98.19	95.63	79.16	69.56	43.09	8.95	-	-
5-hydroxy	-	-	-	-	9.57	14.35	29.24	57.04	46.26	49.66
ΣUnknown 1, 2, 3	-	-	-	-	-	-	-	-	10.11	6.01
<u>Sediment extract</u>	1.56	5.02	4.07	5.63	11.96	14.44	22.13	32.81	36.61	38.94
Florasulam	n.a.	n.a.	n.a.	n.a.	6.12	4.15	3.55	-	-	0.94
5-hydroxy	n.a.	n.a.	n.a.	n.a.	5.84	10.29	18.58	32.54	29.65	32.52
ΣUnknown 1, 2, 3	n.a.	n.a.	n.a.	n.a.	-	-	-	-	4.58	5.31
<u>Sediment residue</u>	-	-	-	-	2.66	3.77	6.96	3.65	6.35	8.06
<u>Apparatus wash</u>	-	-	-	-	-	-	-	-	-	-
Mass balance	102.56	101.57	102.26	101.26	103.35	102.12	101.42	102.45	99.33	102.67

Table B.7.4.4-7 : Degradation rate of florasulam and metabolite 5-hydroxy in water/sediment systems in days (DT whole system)

	florasulam	metabolite 5-hydroxy
--	------------	----------------------

	DT ₅₀	DT ₉₀	DT ₅₀
Sandy loam (phenyl label)	8.7	28.7	68.59
Sandy loam (triazolpyrimidine label)	8.9	29.6	243.96
Clay loam (phenyl label)	16.6	55.3	Not calculated
Clay loam (triazolpyrimidine label)	18.0	59.7	Not calculated

Conclusions :

The a.s. is degraded with DT₅₀ values of 8.7-18 days (whole system) . The metabolite 5-hydroxy is degraded slowly with DT₅₀ up to 244 days (whole system).

Unknown 1 accounted for a maximum of 39 % (phenyl label) and 14% (triazolpyrimidine label) of applied radioactivity in the sandy loam system at 182 days and 10% (phenyl label) or 11% (triazolpyrimidine label) in the clay loam system at 182 or 100 days. Unknown 3 reached at maximum of 15% at 100 days in the sandy loam system with phenyl label, but was less than 5% in the other 3 treatment groups.

The active substance and metabolites were recovered in both water and sediment phases.

The mineralization reached levels of 0-3.7 % after 100 days depending of test system. Bound residue reached a maximum of 4.5-11.2% applied radioactivity after 100 days.

B.7.4.5 Degradation in the saturated zone of active substance, metabolites, degradation and reaction products (Annex IIA 7.2.1.4)

The study is not required.

B.7.5 Impact on water treatment procedures (Annex IIIA 9.2.2)

Due to its use pattern (herbicide in cereals), it is not expected that the a.s. would contaminate water treatment plants.

B.7.6 Summary of behaviour in water and predicted environmental concentrations in surface water and in ground water (PEC_{sw}, PEC_{gw}) (Annex IIIA 9.21, 9.2.3)

Hydrolysis

Hydrolysis rate ($t_{1/2}$) of florasulam is = 219-226 d at pH 9, 20°C. No hydrolysis occurs at pH 5, 7. The main hydrolysis product is the 5-OH.

Photodegradation

Photolysis rate ($t_{1/2}$) of florasulam under natural sunlight conditions is 88 and 223 d, respectively for the aniline and triazolpyrimidine labeled a.s. The main photodegradation product of florasulam is the triazolosulfonic acid of florasulam (TPSA)

Ready biodegradability

Florasulam and the metabolite 5-OH are not ready biodegradable.

Water sediment study

The a.s. is degraded with DT_{50} values of 8.7-18 days (whole system) . The metabolite 5-hydroxy is degraded slowly with DT_{50} up to 244 days (whole system).

Unknown 1 accounted for a maximum of 39 % (phenyl label) and 14% (triazolpyrimidine label) of applied radioactivity in the sandy loam system at 182 days and 10% (phenyl label) or 11% (triazolpyrimidine label) in the clay loam system at 182 or 100 days. Unknown 3 reached at maximum of 15% at 100 days in the sandy loam system with phenyl label, but was less than 5% in the other 3 treatment groups.

The active substance and metabolites were recovered in both water and sediment phases.

The mineralization reached levels of 0-3.7 % after 100 days depending of test system. Bound residue reached a maximum of 4.5-11.2% applied radioactivity after 100 days.

PEC surface water

The PEC_{sw} were calculated assuming that :

- The a.s. is degraded with DT₅₀ of 18 d (maximum value in water/sediment study); the metabolite 5-OH is degraded with DT₅₀ of 244 d (maximum value in water/sediment study);
- Drift scenarios according to Ganzelmeier (1992) is applied.
- The waterbody is 30 cm deep
- The application rate is 7.5 g a.s./ha
- Calculations according to a first order kinetics

Table B.7.6-1 : PEC sw of the active substance and metabolite 5-OH

Time after applications (days)	Cereals 7.5 g a.s./ha 1 application, 1m drift 4% of applied dose reaching the water body			
	florasulam		metabolite 5-OH	
	Actual concentration (µg/l)	TWA concentration (µg/l)	Actual concentration (µg/l)	TWA concentration (µg/l)
0	0.1	0.1	0.1	0.1
1	0.096	0.098	0.1	0.1
2	0.093	0.096	0.099	0.1
4	0.086	0.093	0.099	0.099
7	0.076	0.088	0.098	0.099
14	0.058	0.077	0.096	0.098
21	0.045	0.069	0.094	0.097
28	0.034	0.061	0.092	0.096
42	0.020	0.050	0.089	0.094

PEC groundwater

Calculation of PEC (groundwater) for DE-750 Metabolites Using PELMO 3.0 (Mc Reath A., 1999)

To assess potential concentrations in ground water, simulations were performed with the computer model PELMO (version 3.0) using the following scenarios : Borstel and Parabraunerde soils; Hamburg average climate; 1 application of florasulam to winter wheat at the maximum rate of 3.75 g a.s./ha (7.5 g a.s./ha, 50% crop interception) on 15th April; Oilseed rape was taken as following crop.

Table B.7.6-2 : Physico-chemical properties of florasulam, metabolites 5-OH, ASTCA, DFP-ASTCA used for modelling

	Florasulam	5-OH	ASTCA	DFP-ASTCA
Molecular weight (g mol ⁻¹)	359	345	302	192
Koc value (cm ³ /g)	18	18	53	83
Degradation rate (DT ₅₀ days)	2.1	24	10.5	335

Findings :

Table B.7.6-3 : Predicted annual average concentrations

Soil	Year	$\mu\text{g/L}$			
		Florasulam	5-Hydroxy	DFP-ASTCA	ASTCA
Borstel	1	0.000	0.038	0.004	0.002
	2	0.000	0.021	0.009	0.082
	3	0.000	0.000	0.000	0.224
	4	0.000	0.000	0.000	0.054
	5	0.000	0.000	0.000	0.001
Parabraunerde	1	0.000	0.017	0.002	0.001
	2	0.000	0.060	0.019	0.071
	3	0.000	0.001	0.002	0.273
	4	0.000	0.000	0.000	0.062
	5	0.000	0.000	0.000	0.001

Conclusions :

No parent florasulam was predicted to leach over five years .The 5-hydroxy metabolite showed a maximum average annual concentration of 0.038 $\mu\text{g/L}$ during year 1 in Borstel soil and 0.060 $\mu\text{g/L}$ during year 2 in the Parabraunerde. No 5-hydroxy metabolite was predicted to leach in year 3. Predicted annual concentrations of DFD-ASTCA were very low with a maximum of 0.019 $\mu\text{g/L}$ during year 2 in the Parabraunerde. The highest predicted concentrations in leachate were seen for the ASTCA metabolite : maximum average annual concentrations were 0.224 $\mu\text{g/L}$ during year 3 Borstel soil and 0.273 $\mu\text{g/L}$ during year 3 in Parabraunerde. Average annual concentrations of ASTCA were less than 0.1 $\mu\text{g/L}$ during all other years.

B.7.7 Fate and behaviour in air (Annex IIA, 7.2.2; Annex IIIA 9.3)

Investigation of the Volatilization of DE-570 formulated as 50 g as/l SC from Soil and Dwarf Runner Bean (Knoch, et al., 1997)

Guidelines :

BBA Guideline Part IV, Section 6-1

GLP :

Yes

Material and Methods :

Test substance :

Formulation EF 1343 (SC containing 50 g/l ¹⁴C triazolopyrimidine labeled florasulam, radiochemical purity = 99%)

Experimental design :

The test solution was applied with a syringe on 2 leaves/plant. The bean plants in full blossom were then placed in the wind tunnel.

The test solution was similarly applied to petri dishes containing German standard soil 2.1 (sand). The plates were then placed in the wind tunnel.

Leaf and soil samples were collected after 0, 1, 3, 6 and 24 hours, extracted with acetonitrile: water: phosphoric acid (80: 19: 1) and analyzed by LSC. Samples taken after 0 and 24 hours were further analyzed by radio-TLC/HPLC.

Test conditions :

Table B.7.7-1 : Volatilization of florasulam - Tests conditions in the wind tunnel

Parameter	Experimental part	Actual value of parameter
soil temperature	soil	19.3-21.5 °C
soil humidity	soil	60% MWHC ± 1%
relative air humidity	plant soil	37.2-41.6 % 39.4-51.9%
air flow	plant soil	1.1-2.2 m/s 1.3-1.6 m/s
air temperature	plant soil	21.0-22.3 °C 20.3-21.6 °C

Findings and conclusion:

No volatilization (1.3%) of florasulam from soil occurred within 24 hours.

No volatilization (-0.8%) of florasulam from plant occurred within 24 hours.

The figures are below the BBA trigger values of 20%.

B.7.8 Summary of behaviour in air and predicted environmental concentrations in air (PECa) (Annex IIIA 9.3)

- The Henry constant reveals that the a.s. is moderately volatile.

vapour pressure at 20°C = 0.77×10^{-5} Pa

water solubility at 20°C = 0.121 g/L

⇒ H at 20°C = 2.29×10^{-5} Pa.m³/mol

- No volatilization from plant or from soil occurs .

B.7.9 Definition of the residue (Annex IIA 7.3)

Definition of the residue in soil : florasulam and metabolite 5-OH

Definition of the residue in water : florasulam and metabolite 5-OH

B.7.10 References relied on

Environmental fate and behaviour of the active substance (Annex IIA 7)

Annex point(s) 91/414/EEC	Author, Title, Test institute, Report number/Study ID, Date of report For publications: reference	Dow AgroScience s Report No.	GLP GEP	Published Protected
IIA 7.1.1.1.1 IIA 7.1.3.1 IIA 7.1.3.2	Baloch, R, Yon, DA, Early Stage Environmental Fate Probes for XR-566 and XR-570 DowElanco Europe, Letcombe Regis, Oxon, UK -, September 1992	GHE-P-2790	No	Unpublishe dProtected
IIA 7.1.1.1.2 IIA 7.1.1.2.1	Cleveland, CB, Gilbert, JR, Sanders, LT, Anaerobic Aquatic Metabolism Study of XDE-570 DowElanco, Indianapolis, Indiana, USA ENV95137, April 1997	GH-C 4366	Yes	Unpublishe dProtected
IIA 7.1.1.2.2	Gambie, A, The stability of DE-570 and its 5-hydroxy metabolite in soil under frozen storage conditions. (Letter report). DowElanco Europe, Letcombe Regis, Oxon. UK -, December 1997b	-	Yes	Unpublishe dProtected
IIA 7.1.1.2.2	Gambie, A, Residues of DE-570 and its 5-hydroxy metabolite in soil at normal harvest following application of EF-1343 to wheat and barley - Europe: 1995 -1996 DowElanco Europe, Letcombe Regis, Oxon. UK GHE-P-6833, December 1997a	GHE-P-6833	Yes	Unpublishe dProtected
IIA 7.2.1.2	Gibson, R, Degradation of DE-570 in a Natural Lakewater DowElanco Europe, Letcombe Regis, Oxon, UK E97-060, November 1997	GHE-P-6732	Yes	Unpublishe dProtected
IIA 7.1.1.1.1 IIA 7.1.1.2.1 IIA 7.1.2	Jackson, R, Ghosh, D, The Aerobic Degradation of XDE-570 in Soil DowElanco Europe, Letcombe Regis, Oxon, UK 3U, October 1997	GHE-P-4710	Yes	Unpublishe dProtected
IIA 7.1.1.1.1	Jackson R., Massart J. The Degradation of DFP-ASTAC and ASTAC (two metabolites of DE-570) in soil Dow AgroSciences, Letcombe Laboratory, Letcombe Regis, Wantage, Oxon, UK E98-025, December 1998	GHE-P-7522	Yes	Unpublishe dProtected
IIA 7.1.2	Jackson R., Massart J. The Soil Sorption of DFP-ASTAC and ASTAC (two metabolites of DE-570) Dow AgroSciences, Letcombe Laboratory, Letcombe Regis, Wantage, Oxon, UK E98-082, January 1999	GHE-P-7622	Yes	Unpublishe dProtected
IIA 7.1.3.3	Jackson, R, Paterson, G, The Dissipation of XDE-570 in Soil and Crops using Field Lysimeters	GHE-P-6751	Yes	Unpublishe dProtected

Annex point(s) 91/414/EEC	Author, Title, Test institute, Report number/Study ID, Date of report For publications: reference	Dow AgroScience s Report No.	GLP GEP	Published Protected
	DowElanco Europe, Letcombe Regis, Oxon, UK 2U, December 1997			dProtected
IIA 7.2.1.1	Jackson, R, Portwood, D, The Aqueous Hydrolysis of XR-570 DowElanco Europe, Letcombe Regis, Oxon, UK -, December 1993	GHE-P-3326	No	Unpublishe dProtected
IIA 7.2.1.3.1	Jenkins, WR, XDE-570 (Pure): Assessment of Ready Biodegradability Pharmaco::LSR, Eye, Suffolk, UK 94/DES180/0468, July 1994	GHE-P-3736	Yes	Unpublishe dProtected
IIA 7.2.1.3.1	Jenkins, WR, XDE-570 5-Hydroxy Metabolite: Assessment of Ready Biodegradability Pharmaco::LSR, Eye, Suffolk, UK 95/DES284/0692, October 1995	GHE-P-4552	Yes	Unpublishe dProtected
IIA 7.2.2	Knoch, E, Investigation of the Volatilization of DE-570 Formulated as 50 g as/l SC from Soil and Dwarf Runner Bean Institut Fresenius, 45699 Herten, Germany E97-049, November 1997	GHE-P-6747	Yes	Unpublishe dProtected
IIA 7.1.1.1.2 IIA 7.1.1.2.1	Krieger, MS, Yoder, RN, Photolysis of XDE-570 on Soil DowElanco, Indianapolis, USA ENV95083, October 1996	GH-C 4123	Yes	Unpublishe dProtected
IIIA 9.1.3	Mc Reath A., Comments on draft monograph - Calculation of PEC (groundwater) for DE-750 Metabolites Using PELMO 3.0 Dow AgroSciences, European Registration Departement, Letcombe Regis, Wantage, Oxon, UK. September 1999	-	No	-
IIA 7.1.1.2.2	Maycock, R, Wright, D, The Dissipation of XDE-570 and its 5-Hydroxy Metabolite in Soil Following a Single Application of EF-1343, Northern France -1995 DowElanco Europe, Letcombe Regis, Oxon, UK RL95-055, October 1997a	GHE-P-6367	Yes	Unpublishe dProtected
IIA 7.1.1.2.2	Maycock, RM, Wright, D, The Dissipation of XDE-570 and its 5-Hydroxy Metabolite in Soil at Intervals Following a Single Application of EF-1343, Southern France - 1996 DowElanco Europe, Letcombe Regis, Oxon, UK RL96-098, October 1997b	GHE-P-6369	Yes	Unpublishe dProtected
IIA 7.1.1.2.2	Maycock, RM, Wright, D, The Dissipation of XDE-570 and its 5-Hydroxy Metabolite in Soil Following a Single Application of EF-1343, Germany -1995 DowElanco Europe, Letcombe Regis, Oxon, UK RL95-053, November 1997c	GHE-P-6366	Yes	Unpublishe dProtected

Annex point(s) 91/414/EEC	Author, Title, Test institute, Report number/Study ID, Date of report For publications: reference	Dow AgroScience s Report No.	GLP GEP	Published Protected
IIA 7.1.1.2.2	Maycock, R, Wright, D, The Dissipation of XDE-570 and its 5-Hydroxy Metabolite in Soil Following a Single Application of EF-1343, UK -1995 DowElanco Europe, Letcombe Regis, Oxon, UK RL95-054, November 1997d	GHE-P-6381	Yes	Unpublishe dProtected
IIA 7.1.1.2.2	Maycock, RM, Wright, D, The Dissipation of XDE-570 and its 5-Hydroxy Metabolite in Soil Following a Single Application of EF-1343, UK -1996 DowElanco Europe, Letcombe Regis, Oxon, UK RL96-001, November 1997e	GHE-P-6368	Yes	Unpublishe dProtected
IIA 7.1.1.2.2	Maycock, RM, Wright, D, The Dissipation of XDE-570 and its 5-Hydroxy Metabolite in Soil Following a Single Application of EF-1343, Greece - 1996 DowElanco Europe, Letcombe Regis, Oxon. UK RL96-147, November 1997f	GHE-P-6370	Yes	Unpublishe dProtected
IIA 7.1.2	Ostrander, JA, Mobility Studies of XDE-570 and 5-Hydroxy-XDE-570 DowElanco, Indianapolis, Indiana 46268, USA ENV95020, January 1996	GH-C 3868	Yes	Unpublishe dProtected
IIA 7.2.1.1	Phillips, M, The Determination of the Hydrolytic Stability of Radiolabelled XDE-570 Inveresk Research International Ltd, Tranent, Scotland, UK 386209, September 1996	GHE-P-4986	Yes	Unpublishe dProtected
IIA 7.2.1.3.2	Phillips, M, The Aerobic Degradation of XDE-570 in Natural Waters and Associated Sediments Inveresk Research International Ltd, Tranent, Scotland, UK 385362, April 1997	GHE-P-5039	Yes	Unpublishe dProtected
IIA 7.1.2	Pillar, F, The Sorption of XDE-570 in Soil DowElanco Europe, Letcombe Regis, Oxon, UK E97-001, July 1997a	GHE-P-6325R	Yes	Unpublishe dProtected
IIA 7.1.1.2.1	Pillar, F, Effects of Moisture on the Degradation of DE-570 in Soil DowElanco Europe, Letcombe Regis, Oxon, UK E97-003, November 1997b	GHE-P-6750	Yes	Unpublishe dProtected
IIA 7.1.1.2.1	Pillar, F, Effects of Temperature on the Degradation of DE-570 in Soil DowElanco Europe, Letcombe Regis, Oxon, UK E97-002, December 1997c	GHE-P-6749	Yes	Unpublishe dProtected
IIA 7.1.3.1	Pillar, F, The Non-Aged Soil Column Leaching of DE-57- DowElanco Europe, Letcombe Regis, Oxon, UK GHE-P-6785, December 1997d	GHE-P-6785	No	Unpublishe dProtected
IIA 7.1.1.1.1	Watson, D, Certificate of Analysis XDE-570: Confirmation of the Identity of an Isolated Soil Degradate of XDE-570 using Liquid Chromatography - Mass Spectroscopy Huntingdon Research Centre, Huntingdon, Cambridgeshire,	GHE-P-4504	Yes	Unpublishe dProtected

Annex point(s) 91/414/EEC	Author, Title, Test institute, Report number/Study ID, Date of report For publications: reference	Dow AgroSciences Report No.	GLP GEP	Published Protected
	UK DWC 723/950881, July 1995			
IIA 7.2.1.2	Yoder, RN, Aqueous Photolysis of XDE-570 in Natural Sunlight DowElanco, Indianapolis, Indiana 46268, USA ENV95023, March 1996	GH-C 3951	Yes	Unpublished Protected

Environmental fate and behaviour of the formulation EF-1343 (Annex IIIA 9)

Annex point(s) 91/414/EEC	Author, Title, Test institute, Report number/Study ID, Date of report For publications: reference	Dow AgroSciences Report No.	GLP GEP	Published Protected
IIIA 9.1.2.1 (see IIA 7.1.1.1.1)	Baloch, R, Yon, DA, Early Stage Environmental Fate Probes for XR-566 and XR-570 DowElanco Europe, Letcombe Regis, Oxon, UK -, September 1992	GHE-P-2790	No	Unpublished Protected
IIIA 9.1.1.1 (see IIA 7.1.1.1.2)	Cleveland, CB, Gilbert, JR, Sanders, LT, Anaerobic Aquatic Metabolism Study of XDE-570 DowElanco Indianapolis, Indiana, USA ENV95137, April 1997	GH-C 4366	Yes	Unpublished Protected
IIIA 9.1.1.2 (see IIA 7.1.1.2.2)	Gambie, A, Residues of DE-570 and its 5-hydroxy metabolite in soil at normal harvest following application of EF-1343 to wheat and barley - Europe: 1995 -1996 DowElanco Europe, Letcombe Regis, Oxon. UK GHE-P-6833, December 1997a	GHE-P-6833	Yes	Unpublished Protected
IIIA 9.2.3 (see IIA 7.2.1.2)	Gibson, R, Degradation of DE-570 in a Natural Lakewater DowElanco Europe, Letcombe Regis, Oxon, UK E97-060, November 1997	GHE-P-6732	Yes	Unpublished Protected
IIIA 9.1.1.1 IIIA 9.1.2.1 IIIA 9.1.3 (see IIA 7.1.1.1.1)	Jackson, R, Ghosh, D, The Aerobic Degradation of XDE-570 in Soil DowElanco Europe, Letcombe Regis, Oxon, UK 3U, October 1997	GHE-P-4710	Yes	Unpublished Protected
IIIA 9.1.1.2 IIIA 9.1.2.2 IIIA 9.2.1 (see IIA 7.1.3.3)	Jackson, R Paterson, G, The Dissipation of XDE-570 in Soil and Crops using Field Lysimeters DowElanco Europe, Letcombe Regis, Oxon, UK 2U, December 1997	GHE-P-6751	Yes	Unpublished Protected
IIIA 9.3 (see IIA 7.2.2)	Knoch, E, Investigation of the Volatilization of DE-570 Formulated as 50 g as/l SC from Soil and Dwarf Runner Bean Institut Fresenius, 45699 Herten, Germany E97-049, November 1997	GHE-P-6747	Yes	Unpublished Protected

Annex point(s) 91/414/EEC	Author, Title, Test institute, Report number/Study ID, Date of report For publications: reference	Dow AgroSciences Report No.	GLP GEP	Published Protected
IIIA 9.1.1.1 (see IIA 7.1.1.1.2)	Krieger, MS, Yoder, RN, Photolysis of XDE-570 on Soil DowElanco, Indianapolis, USA ENV95083, October 1996	GH-C 4123	Yes	Unpublished Protected
IIIA 9.1.1.2 (see IIA 7.1.1.2.2)	Maycock, RM, Wright, D, The Dissipation of XDE-570 and its 5-Hydroxy Metabolite in Soil Following a Single Application of EF-1343, Northern France -1995 DowElanco Europe, Letcombe Regis, Oxon, UK RL95-055, October 1997a	GHE-P-6367	Yes	Unpublished Protected
IIIA 9.1.1.2 (see IIA 7.1.1.2.2)	Maycock, RM, Wright, D, The Dissipation of XDE-570 and its 5-Hydroxy Metabolite in Soil at Intervals Following a Single Application of EF-1343, Southern France - 1996 DowElanco Europe, Letcombe Regis, Oxon, UK RL96-098, October 1997b	GHE-P-6369	Yes	Unpublished Protected
IIIA 9.1.1.2 (see IIA 7.1.1.2.2)	Maycock, RM, Wright, D, The Dissipation of XDE-570 and its 5-Hydroxy Metabolite in Soil Following a Single Application of EF-1343, Germany -1995 DowElanco Europe, Letcombe Regis, Oxon, UK RL95-053, November 1997c	GHE-P-6366	Yes	Unpublished Protected
IIIA 9.1.1.2 (see IIA 7.1.1.2.2)	Maycock, RM, Wright, D, The Dissipation of XDE-570 and its 5-Hydroxy Metabolite in Soil Following a Single Application of EF-1343, UK -1996 DowElanco Europe, Letcombe Regis, Oxon, UK RL96-001, November 1997d	GHE-P-6368	Yes	Unpublished Protected
IIIA 9.1.1.2 (see IIA 7.1.1.2.2)	Maycock, RM, Wright, D, The Dissipation of XDE-570 and its 5-Hydroxy Metabolite in Soil Following a Single Application of EF-1343, Greece - 1996 DowElanco Europe, Letcombe Regis, Oxon. UK RL96-147, November 1997e	GHE-P-6370	Yes	Unpublished Protected
IIIA 9.1.1.2 (see IIA 7.1.1.2.2)	Maycock, RM, Wright, D, The Dissipation of XDE-570 and its 5-Hydroxy Metabolite in Soil Following a Single Application of EF-1343, UK -1995 DowElanco Europe, Letcombe Regis, Oxon, UK RL95-054, November 1997f	GHE-P-6381	Yes	Unpublished Protected
IIIA 9.1.2.1 (see IIA 7.1.2)	Ostrander, JA, Mobility Studies of XDE-570 and 5-Hydroxy-XDE-570 DowElanco, Indianapolis, Indiana 46268, USA ENV95020, January 1996	GH-C 3868	Yes	Unpublished Protected
IIIA 9.2.3 (see IIA 7.2.1.3.2)	Phillips, M, The Aerobic Degradation of XDE-570 in Natural Waters and Associated Sediments Inveresk Research International Ltd, Tranent, Scotland, UK 385362, April 1997	GHE-P-5039	Yes	Unpublished Protected
IIIA 9.1.2.1 IIIA 9.2.1 (see IIA 7.1.2)	Pillar, F, The Sorption of XDE-570 in Soil DowElanco Europe, Letcombe Regis, Oxon, UK E97-001, July 1997a	GHE-P-6325R	Yes	Unpublished Protected

Annex point(s) 91/414/EEC	Author, Title, Test institute, Report number/Study ID, Date of report For publications: reference	Dow AgroSciences Report No.	GLP GEP	Published Protected
7.1.2)				
IIIA 9.1.1.1 (see IIA 7.1.1.2.1)	Pillar, F, Effects of Moisture on the Degradation of DE-570 in Soil DowElanco Europe, Letcombe Regis, Oxon, UK E97-003, November 1997b	GHE-P-6750	Yes	UnpublishedProtected
IIIA 9.1.2.1 (see IIA 7.1.3.1)	Pillar, F, The Non-Aged Soil Column Leaching of DE-570 DowElanco Europe, Letcombe Regis, Oxon, UK GHE-P-6785, December 1997c	GHE-P-6785	No	UnpublishedProtected
IIIA 9.1.1.1 IIIA 9.2.1 (see IIA 7.1.1.2.1)	Pillar, F, Effects of Temperature on the Degradation of DE-570 in Soil DowElanco Europe, Letcombe Regis, Oxon, UK E97-002, December 1997d	GHE-P-6749	Yes	UnpublishedProtected
IIIA 9.1.3 IIIA 9.2.1 IIIA 9.2.3	Yon, DA, Calculation of Predicted Environmental Concentrations (PEC's) for DE-570 DowElanco Europe, Letcombe Regis, Oxon, UK E97-072, December 1997	GHE-P-6812	Yes	UnpublishedProtected

ANNEX B

Florasulam

Appendix F : Soil dissipation testing

Annex Point: IIA 7.1.1.2.2/01

FIELD TRIALS, SOIL RESIDUE SUMMARY

Responsible body for reporting (name & address):	Dow AgroSciences, Letcombe Laboratories, Letcombe Regis, Wantage, Oxon. OX12 9JT, UK	Active substance (common name):	Florasulam
Country:	Northern France	Matrix:	Soil
Content of active substance (g/kg or g/l):	50 g/l	Page:	
Formulation (e.g. WP):	SC	Indoor/outdoor:	Outdoor
Commercial Product (name):	EF-1343	Other active substance in the formulation (common name and content):	None
Producer of commercial product:	Dow AgroSciences		
Residues calculated as:	µg/kg Florasulam	Masterfile Reference:	K03

1	2	3	4		5	6	7	8	9	10	11
Report No. Location including Postal Code	Matrix	Soil characteristics 1) soil texture 0-10cm 2) pH 3) % organic C 4) meq/100 g	Application rate per treatment		Application date (a)	days after application	soil layer	Residues (µg/kg) (c)	DT ₅₀ days	DT ₉₀ days	Remarks: (b)
			kg a.s/ha	Water (l/ha)							
GHE-P-6367 41310 Bezay- 41, Tours, Northern France	Soil	1) Silty clay loam 2) pH 7.4 3) 1.2% 4) 29.1	0.0135	260	03 Apr 95	0 3 7 15 29 60 122 210	0-10 cm only	6.60 3.85 6.76 3.32 0.92 0.08 <0.05 <0.05	9	31	Residues of florasulam Analytical Method ERC 96.21

Remarks:

- (a) Year must be indicated.
- (b) Remarks may include: climatic conditions; references to analytical method; information concerning the metabolites included.
- (c) Results at or below the limit of determination should be identified, e.g. <0.01.

Note: All entries to be filled as appropriate. N/A - Not applicable. ND - Not detected.

Annex Point: IIA 7.1.1.2.2/01

FIELD TRIALS, SOIL RESIDUE SUMMARY

Responsible body for reporting (name & address):	Dow AgroSciences, Letcombe Laboratories, Letcombe Regis, Wantage, Oxon. OX12 9JT, UK	Active substance (common name):	Florasulam
Country:	Northern France	Matrix:	Soil
Content of active substance (g/kg or g/l):	50 g/l	Page:	
Formulation (e.g. WP):	SC	Indoor/outdoor:	Outdoor
Commercial Product (name):	EF-1343	Other active substance in the formulation (common name and content):	None
Producer of commercial product:	Dow AgroSciences		
Residues calculated as:	µg/kg 5-hydroxy Florasulam	Masterfile Reference:	K03

1	2	3	4		5	6	7	8	9	10	11
Report No. Location including Postal Code	Matrix	Soil characteristics 1) soil texture 0-10cm 2) pH 3) % organic C 4) meq/100 g	Application rate per treatment kg a.s/ha Water (l/ha)		Application date (a)	days after application	soil layer	Residues (µg/kg) (c)	DT ₅₀ days	DT ₉₀ days	Remarks: (b)
GHE-P-6367 41310 Bezay- 41, Tours, Northern France	Soil	1) Silty clay loam 2) pH 7.4 3) 1.2% 4) 29.1	0.0135	260	03 Apr 95	0 3 7 15 29 60 122 210	0-10 cm only	1.97 2.98 1.94 1.98 1.57 0.12 <0.05 <0.05	17	55	Residues of 5-hydroxy Florasulam metabolite Analytical Method ERC 96.21

Remarks:

- (a) Year must be indicated.
- (b) Remarks may include: climatic conditions; references to analytical method; information concerning the metabolites included.
- (c) Results at or below the limit of determination should be identified, e.g. <0.01.

Note: All entries to be filled as appropriate. **N/A** - Not applicable. **ND** - Not detected.

Annex Point: IIA 7.1.1.2.2/02

FIELD TRIALS, SOIL RESIDUE SUMMARY

Responsible body for reporting (name & address):	Dow AgroSciences, Letcombe Laboratories, Letcombe Regis, Wantage, Oxon. OX12 9JT, UK	Active substance (common name):	Florasulam
Country:	Germany	Matrix:	Soil
Content of active substance (g/kg or g/l):	50 g/l	Page:	
Formulation (e.g. WP):	SC	Indoor/outdoor:	Outdoor
Commercial Product (name):	EF-1343	Other active substance in the formulation (common name and content):	None
Producer of commercial product:	Dow AgroSciences		
Residues calculated as:	Florasulam µg/kg	Masterfile Reference:	K05

1	2	3	4		5	6	7	8	9	10	11
Report No. Location including Postal Code	Matrix	Soil characteristics 1) soil texture 0- 10cm 2) pH 3) % organic C 4) meq/100 g	Application rate per treatment kg a.s/ha Water (l/ha)		Application date (a)	days after application	soil layer	Residues (µg/kg) ©	DT ₅₀ days	DT ₉₀ days	Remarks: (b)
GHE-P-6366 35321 Wetterfeld, Laubach, Hessen, Germany	Soil	1) Silt loam 2) pH 6.1 3) 1.2% 4) 13.9	0.015	412	25 Apr 95	0 3 8 15 28 62 122 217	0-10 cm only	3.81 2.37 2.03 2.45 1.15 <0.05 <0.05 <0.05	15	50	Residues of florasulam Analytical Method ERC 95.1

Remarks:

- (a) Year must be indicated.
- (b) Remarks may include: climatic conditions; references to analytical method; information concerning the metabolites included.
- © Results at or below the limit of determination should be identified, e.g. <0.01.

Note: All entries to be filled as appropriate. **N/A** - Not applicable. **ND** - Not detected.

Annex Point: IIA 7.1.1.2.2/02

FIELD TRIALS, SOIL RESIDUE SUMMARY

Responsible body for reporting (name & address):	Dow AgroSciences, Letcombe Laboratories, Letcombe Regis, Wantage, Oxon. OX12 9JT, UK	Active substance (common name):	Florasulam
Country:	Germany	Matrix:	Soil
Content of active substance (g/kg or g/l):	50 g/l	Page:	
Formulation (e.g. WP):	SC	Indoor/outdoor:	Outdoor
Commercial Product (name):	EF-1343	Other active substance in the formulation (common name and content):	None

Producer of commercial product: Dow AgroSciences

Residues calculated as: 5-hydroxy µg/kg

Masterfile Reference:

K05

1	2	3	4		5	6	7	8	9	10	11
Report No. Location including Postal Code	Matrix	Soil characteristics 1) soil texture 0- 10cm 2) pH 3) % organic C 4) meq/100 g	Application rate per treatment kg a.s/ha Water (l/ha)		Application date (a)	days after application	soil layer	Residues (µg/kg) (c)	DT ₅₀ days	DT ₉₀ days	Remarks: (b)
GHE-P-6366 35321 Wetterfeld, Laubach, Hessen, Germany	Soil	1) Silt loam 2) pH 6.1 3) 1.2% 4) 13.9	0.015	412	25 Apr 95	0 3 8 15 28 62 122 217	0-10 cm only	0.86 0.77 0.30 0.79 2.52 0.24 0.048 0.010	14	59	Residues of 5-hydroxy metabolite Analytical Method ERC 95.1 At 217 days, the value 0.010 µg/kg was assigned to the result <0.05 µg/kg in order to produce kinetics

Remarks: (a) Year must be indicated.

(b) Remarks may include: climatic conditions; references to analytical method; information concerning the metabolites included.

(c) Results at or below the limit of determination should be identified, e.g. <0.01.

Note: All entries to be filled as appropriate. N/A - Not applicable. ND - Not detected.

Annex Point: IIA 7.1.1.2.2/03

FIELD TRIALS, SOIL RESIDUE SUMMARY

Responsible body for reporting (name & address):	Dow AgroSciences, Letcombe Laboratories ,Letcombe Regis, Wantage, Oxon. OX12 9JT, UK	Active substance (common name):	Florasulam
Country:	UK	Matrix:	Soil
Content of active substance (g/kg or g/l):	50 g/l	Page:	
Formulation (e.g. WP):	SC	Indoor/outdoor:	Outdoor
Commercial Product (name):	EF-1343	Other active substance in the formulation (common name and content):	None
Producer of commercial product:	Dow AgroSciences	Masterfile Reference:	K04
Residues calculated as:	µg/kg Florasulam		

1	2	3	4		5	6	7	8	9	10	11
Report No. Location including Postal Code	Matrix	Soil characteristics 1) soil texture 0- 10cm 2) pH 3) % organic C 4) meq/100 g	Application rate per treatment kg a.s/ha Water (l/ha)		Application date (a)	days after application	soil layer	Residues (µg/kg) (c)	DT ₅₀ days	DT ₉₀ days	Remarks: (b)
GHE-P- Orchard Farm, Marcham, Abingdon, Oxfordshire, UK	Soil	1) Sandy clay loam 2) 7.7 3) 2.0 4) 25.8	0.015	200	04 Apr 95	0 2 7 14 28 56 111 195	0-20 cm soil horizons combined mean residue	3.510 2.225 1.240 1.673 1.173 0.428 0.115 0.038	5	50	Residues of florasulam Method ERC 96.21. Results <LOQ, >LOD ≡ 0.025 µg/kg. Results at ND ≡ 0.01 µg/kg.

Remarks:

- (a) Year must be indicated.
- (b) Remarks may include: climatic conditions; references to analytical method; information concerning the metabolites included.
- (c) Results at or below the limit of determination should be identified, e.g. <0.01.

Note: All entries to be filled as appropriate. N/A - Not applicable. ND - Not detected.

Annex Point: IIA 7.1.1.2.2/03

FIELD TRIALS, SOIL RESIDUE SUMMARY

Responsible body for reporting (name & address):	Dow AgroSciences, Letcombe Laboratories, Letcombe Regis, Wantage, Oxon. OX12 9JT, UK	Active substance (common name):	Florasulam
Country:	UK	Matrix:	Soil
Content of active substance (g/kg or g/l):	50 g/l	Page:	
Formulation (e.g. WP):	SC	Indoor/outdoor:	Outdoor
Commercial Product (name):	EF-1343	Other active substance in the formulation (common name and content):	None
Producer of commercial product:	Dow AgroSciences	Masterfile Reference:	K04
Residues calculated as:	µg/kg 5-hydroxy		

1	2	3	4		5	6	7	8	9	10	11
Report No. Location including Postal Code	Matrix	Soil characteristics 1) soil texture 0- 10cm 2) pH 3) % organic C 4) meq/100 g	Application rate per treatment kg a.s/ha Water (l/ha)		Application date (a)	days after application	soil layer	Residues (µg/kg) (c)	DT ₅₀ days	DT ₉₀ days	Remarks: (b)
GHE-P- Orchard Farm, Marcham, Abingdon, Oxfordshire, UK	Soil	1) Sandy clay loam 2) 7.7 3) 2.0 4) 25.8	0.015	200	04 Apr 95	0 2 7 14 28 56 111 195	0-20 cm soil horizons combined mean residue	0.180 0.078 0.130 0.270 0.568 0.408 0.148 0.080	39	206	Residues of 5- hydroxy metabolite Method ERC 96.21. Results <LOQ, >LOD ≡ 0.025 µg/kg. Results at ND ≡ 0.01 µg/kg.

Remarks:

- (a) Year must be indicated.
- (b) Remarks may include: climatic conditions; references to analytical method; information concerning the metabolites included.
- (c) Results at or below the limit of determination should be identified, e.g. <0.01.

Note: All entries to be filled as appropriate.

N/A - Not applicable.

ND - Not detected.

Annex Point: IIA 7.1.1.2.2/04

FIELD TRIALS, SOIL RESIDUE SUMMARY

Responsible body for reporting (name & address):	Dow AgroSciences, Letcombe Laboratories, Letcombe Regis, Wantage, Oxon. OX12 9JT, UK	Active substance (common name):	Florasulam
Country:	UK	Matrix:	Soil
Content of active substance (g/kg or g/l):	50 g/l	Page:	
Formulation (e.g. WP):	SC	Indoor/outdoor:	Outdoor
Commercial Product (name):	EF-1343	Other active substance in the formulation (common name and content):	None
Producer of commercial product:	Dow AgroSciences	Masterfile Reference:	K06
Residues calculated as:	µg/kg Florasulam		

1	2	3	4		5	6	7	8	9	10	11
Report No. Location including Postal Code	Matrix	Soil characteristics 1) soil texture 0-10cm 2) pH 3) % organic C 4) meq/100 g	Application rate per treatment		Application date (a)	Days after application	soil layer	Residues (µg/kg) ©	DT ₅₀ days	DT ₉₀ days	Remarks: (b)
			kg a.s/ha	Water (l/ha)							
GHE-P-6368 Elveden, Thetford, Norfolk, UK	Soil	1) Sand 2) 7.6 3) 1.1 4) 11.8	0.015	200	08 Mar 96	0 3 7 14 27 61 122 230	Mean of the combined residues in 0-30 cm soil horizon	1.620 1.700 1.222 1.650 0.705 0.357 0.015 0.010	18	61	Residues of florasulam. Method ERC 96.21. Results <LOQ, >LOD ≡ 0.025 µg/kg. Results at ND ≡ 0.01 µg/kg.

Remarks:

- (a) Year must be indicated.
- (b) Remarks may include: climatic conditions; references to analytical method; information concerning the metabolites included.
- © Results at or below the limit of determination should be identified, e.g. <0.01.

Note: All entries to be filled as appropriate. **N/A** - Not applicable. **ND** - Not detected.

Annex Point: IIA 7.1.1.2.2/04

FIELD TRIALS, SOIL RESIDUE SUMMARY

Responsible body for reporting (name & address):	Dow AgroSciences, Letcombe Laboratories, Letcombe Regis, Wantage, Oxon. OX12 9JT, UK	Active substance (common name):	Florasulam
Country:	UK	Matrix:	Soil
Content of active substance (g/kg or g/l):	50 g/l	Page:	
Formulation (e.g. WP):	SC	Indoor/outdoor:	Outdoor
Commercial Product (name):	EF-1343	Other active substance in the formulation (common name and content):	None
Producer of commercial product:	Dow AgroSciences		
Residues calculated as:	µg/kg 5-hydroxy	Masterfile Reference:	K06

1	2	3	4		5	6	7	8	9	10	11
Report No. Location including Postal Code	Matrix	Soil characteristics 1) soil texture 0-10cm 2) pH 3) % organic C 4) meq/100 g	Application rate per treatment		Application date (a)	days after application	soil layer	Residues (µg/kg) (c)	DT ₅₀ days	DT ₉₀ days	Remarks: (b)
			kg a.s/ha	Water (l/ha)							
GHE-P-6368 Elveden, Thetford, Norfolk, UK	Soil	1) Sand 2) 7.6 3) 1.1 4) 11.8	0.015	200	08 Mar 96	0 3 7 14 27 61 122 230	Mean of the combined residues in 0-20 cm soil horizon	0.255 0.450 0.193 0.408 0.933 0.680 0.440 0.025	95	182	Residues of 5-hydroxy florasulam metabolite. Method ERC 96.21. Results <LOQ, >LOD ≡ 0.025µg/kg.

Remarks:

- (a) Year must be indicated.
- (b) Remarks may include: climatic conditions: references to analytical method; information concerning the metabolites included.
- (c) Results at or below the limit of determination should be identified, e.g. <0.01.

Note: All entries to be filled as appropriate. **N/A** - Not applicable. **ND** - Not detected.

Annex Point: IIA 7.1.1.2.2/05

FIELD TRIALS, SOIL RESIDUE SUMMARY

Responsible body for reporting (name & address):	Dow AgroSciences, Letcombe Laboratories, Letcombe Regis, Wantage, Oxon. OX12 9JT, UK	Active substance (common name):	Florasulam
Country:	Southern France	Matrix:	Soil
Content of active substance (g/kg or g/l):	50 g/l	Page:	
Formulation (e.g. WP):	SC	Indoor/outdoor:	Outdoor
Commercial Product (name):	EF-1343	Other active substance in the formulation (common name and content):	None
Producer of commercial product:	Dow AgroSciences	Masterfile Reference:	K07
Residues calculated as:	µg/kg Florasulam		

1	2	3	4		5	6	7	8	9	10	11
Report No. Location including Postal Code	Matrix	Soil characteristics 1) soil texture 0- 10cm 2) pH 3) % organic C 4) meq/100 g	Application rate per treatment kg a.s/ha Water (l/ha)		Application date (a)	Days after application	soil layer	Residues (µg/kg) ©	DT ₅₀ days	DT ₉₀ days	Remarks: (b)
GHE-P-6369 31530 St Livrade, Toulouse, Southern France	Soil	1) Clay loam 2) 8.2 3) 1.2 4) 25.2	0.015	260	05 Apr 96	0 4 7 14 27 62	Mean of the combined residues in 0-30 cm soil horizon	2.130 2.293 1.038 0.803 0.158 0.015	8	28	Residues of florasulam Method ERC 96.21. Results <LOQ, >LOD ≡ 0.025 µg/kg. Results at ND ≡ 0.01 µg/kg.

Remarks:

- (a) Year must be indicated.
- (b) Remarks may include: climatic conditions; references to analytical method; information concerning the metabolites included.
- © Results at or below the limit of determination should be identified, e.g. <0.01.

Note: All entries to be filled as appropriate. N/A - Not applicable. ND - Not detected.

Annex Point: IIA 7.1.1.2.2/05

FIELD TRIALS, SOIL RESIDUE SUMMARY

Responsible body for reporting (name & address):	Dow AgroSciences, Letcombe Laboratories, Letcombe Regis, Wantage, Oxon. OX12 9JT, UK	Active substance (common name):	Florasulam
Country:	Southern France	Matrix:	Soil
Content of active substance (g/kg or g/l):	50 g/l	Page:	
Formulation (e.g. WP):	SC	Indoor/outdoor:	Outdoor
Commercial Product (name):	EF-1343	Other active substance in the formulation (common name and content):	None
Producer of commercial product:	Dow AgroSciences	Masterfile Reference:	K07
Residues calculated as:	µg/kg 5-hydroxy		

1	2	3	4		5	6	7	8	9	10	11
Report No. Location including Postal Code	Matrix	Soil characteristics 1) soil texture 0- 10cm 2) pH 3) % organic C 4) meq/100 g	Application rate per treatment kg a.s/ha Water (l/ha)		Application date (a)	days after application	soil layer	Residues (µg/kg) (c)	DT ₅₀ days	DT ₉₀ days	Remarks: (b)
GHE-P-6369 31530 St Livrade, Toulouse, Southern France	Soil	1) Clay loam 2) 8.2 3) 1.2 4) 25.2	0.015	260	05 Apr 96	0 4 7 14 27 62 122 222	0-10 cm only	0.54 0.29 0.34 0.66 0.46 0.025 0.01 0.01	9	41	Residues of 5- hydroxy metabolite Method ERC 96.21. Results <LOQ, >LOD ≡ 0.025 µg/kg. Results at ND ≡ 0.01 µg/kg.

Remarks:

(a) Year must be indicated.

(b) Remarks may include: climatic conditions; references to analytical method; information concerning the metabolites included.

(c) Results at or below the limit of determination should be identified, e.g. <0.01.

Note: All entries to be filled as appropriate. N/A - Not applicable. ND - Not detected.

Annex Point: IIA 7.1.1.2.2/06

FIELD TRIALS, SOIL RESIDUE SUMMARY

Responsible body for reporting (name & address):	Dow AgroSciences, Letcombe Laboratories, Letcombe Regis, Wantage, Oxon. OX12 9JT, UK	Active substance (common name):	Florasulam
Country:	Greece	Matrix:	Soil
Content of active substance (g/kg or g/l):	50 g/l	Page:	
Formulation (e.g. WP):	SC	Indoor/outdoor:	Outdoor
Commercial Product (name):	EF-1343	Other active substance in the formulation (common name and content):	None
Producer of commercial product:	Dow AgroSciences	Masterfile Reference:	K08
Residues calculated as:	µg/kg Florasulam		

1	2	3	4		5	6	7	8	9	10	11
Report No. Location including Postal Code	Matrix	Soil characteristics 1) soil texture 0- 10cm 2) pH 3) % organic C 4) meq/100 g	Application rate per treatment		Application date (a)	Days after application	soil layer	Residues (µg/kg) (c)	DT ₅₀ days	DT ₉₀ days	Remarks: (b)
			kg a.s/ha	Water (l/ha)							
GHE-P-6370 Valtothori, Thessaloniki, Greece	Soil	1) Sandy silt loam 2) 8.5 3) 0.9 4) 10.0	0.015	200	02 Apr 96	0 3 7 14 28 59 122 213	Mean of the combined residues in 0-30 cm soil horizon	2.833 1.383 1.235 0.665 0.253 0.090 0.015 0.010	2	23	Residues of florasulam. Method ERC 96.21. Results <LOQ, >LOD ≡ 0.025 µg/kg. Results at ND ≡ 0.01 µg/kg.

Remarks:

(a) Year must be indicated.

(b) Remarks may include: climatic conditions; references to analytical method; information concerning the metabolites included.

(c) Results at or below the limit of determination should be identified, e.g. <0.01.

Note: All entries to be filled as appropriate. N/A - Not applicable. ND - Not detected.

Annex Point: IIA 7.1.1.2.2/06

FIELD TRIALS, SOIL RESIDUE SUMMARY

Responsible body for reporting (name & address):	Dow AgroSciences, Letcombe Laboratories, Letcombe Regis, Wantage, Oxon. OX12 9JT, UK	Active substance (common name):	Florasulam
Country:	Greece	Matrix:	Soil
Content of active substance (g/kg or g/l):	50 g/l	Page:	
Formulation (e.g. WP):	SC	Indoor/outdoor:	Outdoor
Commercial Product (name):	EF-1343	Other active substance in the formulation (common name and content):	None
Producer of commercial product:	Dow AgroSciences	Masterfile Reference:	K08
Residues calculated as:	µg/kg 5-hydroxy		

1	2	3	4		5	6	7	8	9	10	11
Report No. Location including Postal Code	Matrix	Soil characteristics 1) soil texture 0- 10cm 2) pH 3) % organic C 4) meq/100 g	Application rate per treatment		Application date (a)	days after application	soil layer	Residues (µg/kg) (c)	DT ₅₀ days	DT ₉₀ days	Remarks: (b)
			kg a.s/ha	Water (l/ha)							
GHE-P-6370 Valtothori, Thessaloniki, Greece	Soil	1) Sandy silt loam 2) 8.5 3) 0.9 4) 10.0	0.015	200	02 Apr 96	0 3 7 14 28 59 122 213	Mean of the combined residues in 0-30 cm soil horizon	0.323 0.153 0.327 0.505 0.198 0.088 0.015 0.010	13	61	Residues of 5-hydroxy metabolite. Method ERC 96.21. Results <LOQ, >LOD ≡ 0.025µg/kg. Results at ND ≡ 0.01 µg/kg.

Remarks:

- (a) Year must be indicated.
- (b) Remarks may include: climatic conditions; references to analytical method; information concerning the metabolites included.
- (c) Results at or below the limit of determination should be identified, e.g. <0.01.

Note: All entries to be filled as appropriate. N/A - Not applicable. ND - Not detected.