

US EPA ARCHIVE DOCUMENT

TEXT SEARCHABLE DOCUMENT

Data Evaluation Record on the terrestrial field dissipation of florasulam

PMRA Submission Number {.....}

EPA MRID Number 46808202

Data Requirement: PMRA Data Code:
EPA DP Barcode: D331183
OECD Data Point:
EPA Guideline: 164-1

Test material: Florasulam

End Use Product name: EF-1343
Formulation type: Suspension concentrate

Concentration of a.i.: 50 g a.i./L

Test material:

Common name: Florasulam.

Chemical name:

IUPAC name: 2',6',8-Trifluoro-5-methoxy[1,2,4]triazolo[1,5-c]pyrimidine-2-sulfonanilide.

CAS name: N-(2,6-Difluorophenyl)-8-fluoro-5-methoxy[1,2,4]triazolo[1,5-c]pyrimidine-2-sulfonamide.
N-(2,6-Difluorophenyl)-8-fluoro-5-methoxy-(1,2,4)-triazolo-(1,5-c)-pyrimidine-2-sulfonamide.
N-(2,6-Difluorophenyl)-8-fluoro-5-methoxy(1,2,4)triazolo(1,5-c)pyrimidine-2-sulphonamide.

CAS No: 145701-23-1.

Synonyms: DE-570; XDE-570; XR-570.

SMILES string: C3(F)C=CC=C(F)C=3NS(=O)(=O)C1N=C2N(N=1)C(OC)=NC=C2F
(EPI Suite v3.12 SMILES string from ISIS .MOL).

Primary Reviewer: Dan Hunt
Cambridge Environmental

Signature: *Dan Hunt*
Date: 07/19/07

Secondary Reviewer: Joan Harlin
Cambridge Environmental

Signature: *Joan Harlin*
Date: 07/19/07

QC/QA Manager: Joan Gaidos
Cambridge Environmental

Signature: *JG*
Date: 07/19/07

Final Reviewer: Cheryl Sutton
EPA Reviewer

Signature: *Cheryl Sutton*
Date: 4/28/2008

Company Code
Active Code
Use Site Category
EPA PC Code: 129108

US EPA ARCHIVE DOCUMENT



Data Evaluation Record on the terrestrial field dissipation of florasulam

PMRA Submission Number {.....}

EPA MRID Number 46808202

CITATION: Maycock, R. 1997. The dissipation of XDE-570 and its 5-hydroxy metabolite in soil at intervals following a single application of EF-1343, Greece-1996. Unpublished study performed by DowElanco Europe, Wantage, Oxon, United Kingdom (laboratory phase) and DowElanco Export SA, Thessaloniki, Greece (field management and field station); and submitted by DowElanco Europe. Report No.: GHE-P-6370. Experiment initiation April 2, 1996 (test application) and completion August 18, 1997 (final chemical analysis date; Table 2, pp. 14-15). Final report issued November 13, 1997.

US EPA ARCHIVE DOCUMENT

Data Evaluation Record on the terrestrial field dissipation of florasulam

PMRA Submission Number {.....}

EPA MRID Number 46808202

EXECUTIVE SUMMARY

Soil dissipation/accumulation of florasulam (XDE-570; 2',6',8-trifluoro-5-methoxy[1,2,4]triazolo[1,5-c]pyrimidine-2-sulfonanilide) under European field conditions was conducted in a bare plot of sandy silt loam soil (ADAS classification) in Greece. The experiment was carried out in accordance with Commission Directive 91/414/EEC, Annex III, Point 7.1.1, as amended by Commission Directive 95/36/EC, and in compliance with the OECD Principles of Good Laboratory Practice. Florasulam was broadcast once at a target rate of 0.015 kg a.i./ha (0.013 lb a.i./A) to a 66 x 3 m plot divided into four subplots. The timing of the application coincided with crops in adjacent fields at BBCH 22-23, which is typical of the timing of the test application to cereals in Europe. The applied rate corresponds to 200% of the maximum proposed use rate of EF-1343 on cereals in Europe, and is three times the proposed maximum application rate (0.0045 lb ai/A or 0.005 kg ai/ha) for use in the United States. Total rainfall during the 213-day study period was 220.6 mm (8.7 inches). The plots were not irrigated and the historical average precipitation amount was not reported. A control plot was not established.

The application rate was not verified using application monitoring devices. Field spikes were not prepared to determine the stability of the parent and transformation products during transport and storage.

Soil samples were collected at 0, 3, 7, 14, 28, 59, 122, and 213 days following the test application to a depth of 0-50 cm. Soil samples were extracted twice by shaking with acetone:1% aqueous acetic acid (9:1, v:v). The combined extracts were concentrated and the analytes were purified by a non-polar (C18) solid phase extraction column, followed by a strong anion exchange (SAX) Bond Elut solid phase extraction column, followed by partitioning into ethyl acetate. Extracts were analyzed for florasulam and 5-hydroxy XDE-570 (N-(2,6-difluorophenyl)-8-fluoro-5-hydroxy-[1,2,4]triazolo[1,5-c]pyrimidine-2-sulfonamide) by LC/MS/MS. The LOQ was 0.05 µg/kg and the LOD was 0.01 µg/kg for both florasulam and 5-hydroxy XDE-570. The storage interval of the test samples prior to analysis was not reported.

The measured zero-time recovery of florasulam in the 0-10 cm soil layer was 8.20 µg/kg (91.1% of the theoretical), decreased to 3.94 µg/kg by 3 days, 1.61 µg/kg by 14 days, 0.67 µg/kg by 28 days, and was last detected above the LOQ at 0.24 µg/kg at 59 days posttreatment. Florasulam was detected in the 10-20 cm soil depth at 0.11-0.12 µg/kg from 0 to 3 days, was a maximum of 0.27 µg/kg at 7 days, and was last detected at or above the LOQ at 0.05 µg/kg at 28 days posttreatment. Florasulam was detected in the 20-30 cm soil depth at 0.05-0.29 µg/kg from 0 to 28 days, in the 30-40 cm soil depth at 0.05-0.22 µg/kg from 0 to 28 days, and in the 40-50 cm soil depth at 0.06-0.29 µg/kg from 7 to 28 days posttreatment. The transformation product **5-hydroxy XDE-570** was initially detected in the 0-10 cm soil depth at 0.94 µg/kg, ranged from 0.42 to 0.73 µg/kg from 3 to 7 days, was a maximum of 1.03 µg/kg at 14 days (11.4% of the applied florasulam), decreased to 0.35 µg/kg by 28 days, and was last detected above the LOQ at 0.22 µg/kg at 59 days posttreatment. 5-Hydroxy XDE-570 was detected in the 10-20 cm soil depth at 0.11-0.30 µg/kg from 7 to 28 days, in the 20-30 cm soil depth at 0.14-0.19 µg/kg from 7

Data Evaluation Record on the terrestrial field dissipation of florasulam

PMRA Submission Number {.....}

EPA MRID Number 46808202

to 28 days, in the 30-40 cm soil depth at 0.08-0.22 µg/kg from 7 to 28 days, and in the 40-50 cm soil depth at 0.06-0.11 µg/kg from 14 to 28 days posttreatment. The reviewer-calculated half-life of 5-hydroxy XDE-570 using linear regression was 22.3 days ($r^2 = 0.8179$), based on linear regression analysis of residues in the 0- to 10-cm soil depth and following the maximum detection at 14 days posttreatment. The registrant-calculated DT90 was 61.1 days.

Under field conditions at the test site, florasulam had an observed DT50 of <3 days; dissipation was biphasic, with a rapid decline to <50% of the day-0 residues by 3 days posttreatment. The DT90 was 22.7 days (registrant-calculated). Residues were not detected at the end of the 213-day study period for carryover.

The major route of dissipation of florasulam under terrestrial field conditions was transformation.

RESULTS SYNOPSIS

Location/soil type: Valtohori, Greece/Sandy silt loam soil (ADAS classification).

Half-life: <3 days (observed).

DT90: 22.7 days (registrant-calculated).

Major transformation products detected: 5-Hydroxy XDE-570.

Dissipation routes: Transformation.

Study Acceptability: This study is classified supplemental. No significant deviations from good scientific practices were noted. However, soils were sampled only to the 50-cm depth rather than to 90-cm, and sampling was not conducted in the two 10-cm increments below the lowest depth of leaching (50 cm). The stability of florasulam and the transformation product 5-hydroxy XDE-570 could not be determined in the test soil. The test substance was applied at three times the proposed maximum label rate. Sampling was not performed to a sufficient depth to define leaching of the test chemical. Soil samples were excessively composited. The study was conducted at a foreign site. The soil was not adequately characterized using the USDA classification system.

I. MATERIALS AND METHODS

GUIDELINE FOLLOWED:

The study was conducted according to Commission Directive 91/414/EEC, Annex III, Point 7.1.1, as amended by Commission Directive 95/36/EC (p. 8). The study author also stated that the study was conducted in accordance with BBA Guideline Part IV, 4-1, December 1986 and IVA Guidelines 'Residue Studies, Part V: Studies on degradation in soil, 1993'

Data Evaluation Record on the terrestrial field dissipation of florasulam

PMRA Submission Number {.....}

EPA MRID Number 46808202

(Appendix 1, p. 23). Deviations from USEPA Subdivision N Guideline 164-1 include:

Storage stability data were not available for review to determine the stability of the parent and 5-hydroxy XDE-570 in the test soil during the storage interval.

The test substance was applied at twice the proposed maximum label rate.

Sampling was not performed to a sufficient depth to define leaching of the test chemical.

Soil samples were excessively composited, providing only one sample for analysis at each sampling interval.

The study author did not establish comparability between the test site soil and U.S. soils, and the soil was not adequately characterized using the USDA classification system.

COMPLIANCE:

The study was conducted in compliance with OECD Principles of Good Laboratory Practice (pp. 3-3a). Signed and dated Data Confidentiality, GLP compliance and Quality Assurance statements were provided (pp. 2-4).

A. MATERIALS:

1. Test Material

Florasulam (XDE-570).

Chemical Structure of the active ingredient(s):

See DER Attachment 1.

Description:

White crystalline solid (Appendix 3, p. 108).

Storage conditions of test chemicals:

The florasulam and 5-hydroxy XDE-570 analytical standards were stored refrigerated (nominally 4°C; Appendix 3, pp. 90-91).

Data Evaluation Record on the terrestrial field dissipation of florasulam

PMRA Submission Number {.....}

EPA MRID Number 46808202

Physico-chemical properties of the active ingredient(s):

Parameter	Value	Comment
Chemical formula		
Molecular mass		
Water Solubility		
Vapor Pressure/Volatility		
UV Absorption		
pKa		
K _{ow} /log K _{ow}		
Stability of compound at room temperature, if provided		

2. Test site: The test site was located in Valtohori, near Thessaloniki in Greece (pp. 8-9). The test site was in an area of mixed agriculture including cereal production, and the area is considered typical of a Southern European cereal growing area. The soil at the test site was classified as a sandy silt loam soil (ADAS classification; Table 2, p. 14). A pesticide use history was not reported for the test site.

Table 1: Geographic location, site description and climatic data at the study sites.

Details		Test site
Geographic coordinates	Latitude	Not reported
	Longitude	Not reported
	Province/State	Valtohor
	Country	Greece
	Ecoregion	Not reported
Slope Gradient		Not reported
Depth to ground water (m)		Not reported
Distance from weather station used for climatic measurements		Weather data were collected from the Valtohori Weather Station, and were supplemented with data from the Thessaloniki Cotton Institute Weather Station when data were not available from the Valtohori Weather Station. The distance of the weather stations from the test site was not reported.
Indicate whether the meteorological conditions before starting or during the study were within 30 year normal levels (Yes/No). If no, provide details.		Total precipitation during the study period (measured from April 2, 1996-November 1, 1996) was 220.6 mm or 8.7 inches. The historical average precipitation amount was not reported.

Data were obtained from p. 9 and Appendix 2, pp. 52-71 of the study report.

US EPA ARCHIVE DOCUMENT

Data Evaluation Record on the terrestrial field dissipation of florasulam

PMRA Submission Number {.....}

EPA MRID Number 46808202

Table 2: Site usage and management history for the previous three years.

Use	Year	Test site
Crops grown	Previous year	Not reported
	2 years previous	Not reported
	3 years previous	Not reported
Pesticides used	Previous year	Not reported
	2 years previous	Not reported
	3 years previous	Not reported
Fertilizers used	Previous year	Not reported
	2 years previous	Not reported
	3 years previous	Not reported
Cultivation methods, if provided (eg., Tillage)	Previous year	Not reported
	2 years previous	Not reported
	3 years previous	Not reported

Prior to the test application, the plot was tilled to a fine tilth (p. 9 of the study report).

3. Soils:

Table 3: Properties of the soil at the test site.

Property	Depth (cm)				
	0-10	10-20	20-30	30-40	40-50
Textural classification	Sandy silt loam ¹				
% sand	38.54	37.64	38.03	44.44	51.45
% silt	49.78	50.30	49.76	46.02	42.23
% clay	11.68	12.06	12.20	9.54	6.32
pH in water	8.5	8.7	8.8	8.7	8.9
Organic matter (%)	1.5	1.4	1.4	0.7	0.5
Total organic carbon (%)	0.9	0.8	0.8	0.4	0.3
CEC (mEq/100 g)	10.0	9.3	9.1	5.2	7.0
Bulk density (g/cm ³)	1.65				
Moisture at 1/3 atm (%)	Not reported	Not reported	Not reported	Not reported	Not reported
Taxonomic classification (e.g., ferro-humic podzol)	Fluvi-calcaric fluvisol				
Soil mapping unit	Not reported				

Data were obtained from pp. 8-9 and Table 2, p. 14 of the study report. Organic matter was calculated by the reviewer from percent organic carbon (% o.m. = % o.c. x 1.72). Water holding capacity was reported as 57-60% (w/w); conditions were not specified.

¹ The textural classification was reported as a sandy silt loam according to the ADAS classification; the reviewer could not determine the USDA textural classification because the two methods use different particle size ranges to classify the sand, silt, and clay fractions.

US EPA ARCHIVE DOCUMENT

Data Evaluation Record on the terrestrial field dissipation of florasulam

PMRA Submission Number {.....}

EPA MRID Number 46808202

B. EXPERIMENTAL DESIGN:

1. Experimental design:

Table 4: Experimental design.

Details		Test site
Duration of study		213 days
Uncropped (bare) or cropped		Bare
Control used (Yes/No)		No
No. of replications	Controls	N/A
	Treatments	One, divided into four subplots
Plot size (L x W m)	Controls	N/A
	Treatments	66 x 3 m
Distance between control plot and treated plot		N/A
Distance between treated plots		N/A
Application rate(s) used (g a.i./ha)		15 g a.i./ha
Was the maximum label rate per ha used in study? (Yes/No)		No, the application rate was three times the reported maximum.
Number of applications		One
Application Date(s) (dd mm yyyy)		02/04/1996
For multiple applications, application rate at Day 0 and at each application time (mg a.i./kg soil)		0.009 mg a.i./kg ¹
Application method (eg., spraying, broadcast etc.)		Broadcast
Type of spray equipment, if used		Azo backpack sprayer with 3 m boom and Lurmark 02F80 nozzles.
Total volume of spray solution applied/plot OR total amount broadcasted/plot		200 L/ha
Identification and volume of carrier (e.g., water), if used		Water
Name and concentration of co-solvents, adjuvants and/or surfactants, if used		None
Indicate whether the following monthly reports were submitted:		
Precipitation:		No, reported daily.
Average minimum and maximum air temperature:		No, reported daily.
Average minimum and maximum soil temperature:		No, reported daily.
Average annual frost-free periods:		No
Indicate whether the Pan evaporation data were submitted		No
Meteorological	Cloud cover	20%

US EPA ARCHIVE DOCUMENT

Data Evaluation Record on the terrestrial field dissipation of florasulam

PMRA Submission Number {.....}

EPA MRID Number 46808202

Details		Test site
conditions during application	Temperature	18°C
	Humidity	62%
	Wind speed and direction	0-1 km/hr, South
	Sunlight (hr)	Not available
Pesticides used during study:		None
Name of product/a.i conc.: Amount applied: Application method:		
Supplemental irrigation used (Yes/No)		No
If yes, provide the following details: No. of irrigation: Interval between irrigation: Amount of water added each time: Method of irrigation:		
Indicate whether water received through rainfall + irrigation equals the 30 year average rainfall (Yes/No)		Could not be determined.
Were the application concentrations verified?		No
Were field spikes used?		No
Good agricultural practices followed (Yes or No)		Not reported
Indicate if any abnormal climatic events occurred during the study (eg., drought, heavy rainfall, flooding, storm etc.)		The plots received only 2.2 mm (0.09 inches) of rainfall from June 1 st through mid-July, and only 26.6 mm (1.0 inches) from June 1 st through the end of August.
If cropped plots are used, provide the following details: Plant - Common name/variety: Details of planting: Crop maintenance:		N/A
Volatilization included in the study (Yes/No)		No
Leaching included in the study (Yes/No)		Yes
Run off included in the study (Yes/No)		No

Data were obtained from pp. 8-9; Table 2, pp. 14-15; and Appendix 2, pp. 52-71 of the study report.

1 The theoretical day-0 recovery of florasulam in the 0-10 cm depth was calculated by the reviewer using the reported site bulk density value of 1.65 g/cm³ and the target application rate of 0.013 lb a.i./A.

2. Application Verification: The application rate was not verified using application monitoring devices.

3. Field Spiking: Field spikes were not prepared.

4. Volatilization: Volatilization was not measured.

Data Evaluation Record on the terrestrial field dissipation of florasulam

PMRA Submission Number {.....}

EPA MRID Number 46808202

5. Leaching: Twenty cores (five from each subplot) were collected from the test plot to a depth of 50 cm, at 0, 3, 7, 14, 28, 59, 122, and 213 days following the test application, to determine the mobility of the test substance in the soil profile (p. 9; Table 1, p. 13). Additional samples were collected at 365 days posttreatment but were not analyzed (Table 2, p. 15).

6. Run off: Run off was not studied.

7. Supplementary Study: A storage stability study using soil from the test site was not conducted.

8. Sampling:

Table 5: Soil sampling

Details	Test site
Method of sampling (random or systematic)	Systematic, along predefined lines incremented by 100 cm.
Sampling intervals	0, 3, 7, 14, 28, 59, 122, 213, and 365 days ¹
Method of soil collection (eg., cores)	Cores
Sampling depth	50 cm
Number of cores collected per plot	20 (5/subplot)
Number of segments per core	Five
Length of soil segments (after sectioning)	10 cm
Core diameter	5 cm
Method of sample processing, if any	Composited by depth (all subplots comprising one sample) and sieved (4 mm).
Storage conditions	Frozen
Storage length (days)	Not reported

Data were obtained from p. 9 and Tables 1-2, pp. 13 and 15 of the study report.

¹ Samples collected at 365 days were not analyzed.

9. Analytical Procedures:

Number of soil samples analysed per treatment or composite sample: One, analyzed in duplicate (pp. 9-10; Appendix 3, p. 92).

Extraction, clean up and concentration of soil samples: Soil samples were extracted twice by shaking with acetone:1% aqueous acetic acid (9:1, v:v), and the combined extracts were concentrated by evaporation (p. 10). Following the addition of water, the analytes were purified by a non-polar (C18) solid phase extraction column, followed by a strong anion exchange (SAX) Bond Elut solid phase extraction column, followed by partitioning into ethyl acetate. Following the addition of aqueous acetic acid, the organic phase was evaporated and the remaining aqueous solution was further diluted with aqueous acetic acid.

Data Evaluation Record on the terrestrial field dissipation of florasulam

PMRA Submission Number {.....}

EPA MRID Number 46808202

Identification and quantification of parent compound: Extracts were analyzed for florasulam by LC/MS/MS (parameters were not reported; p. 10). Analysis employed the analytical standard Reference CHD No 779/94-295 Lot 3, Sponsor Batch No TSN 100381 (purity 99.7%; Appendix 3, pp. 90 and 108).

Identification and quantification of transformation products: Extracts were analyzed for 5-hydroxy XDE-570 (N-(2,6-difluorophenyl)-8-fluoro-5-hydroxy-[1,2,4]triazolo[1,5-c]pyrimidine-2-sulfonamide; Appendix 1, p. 39) by LC/MS/MS (parameters were not reported; p. 10). Analysis employed the analytical standards Reference HD No 0060/95-295 Lot 1, Sponsor Batch No DK1-B550-16 (purity 98.7%) and Reference CHD No 0060/95-295 Lot 4, Sponsor Batch No TSN101151 (purity 98.1%; Appendix 3, pp. 90-91 and 109-111).

Detection limits (LOD, LOQ) for the parent compound in soil: The LOQ was 0.05 µg/kg and the LOD was set at 20% of the LOQ or 0.01 µg/kg (p. 9; Appendix 3, p. 91).

Detection limits (LOD, LOQ) for the transformation products in soil: The LOQ was 0.05 µg/kg and the LOD was set at 20% of the LOQ or 0.01 µg/kg (p. 9; Appendix 3, p. 91).

II. RESULTS AND DISCUSSION

APPLICATION MONITORS: The application rate was not verified using application monitoring devices.

2. RECOVERY FROM FIELD SPIKES: Field spikes were not prepared to determine the stability of the parent and transformation products during transport and storage.

3. MASS ACCOUNTING: A mass balance was not determined for the test site.

Data Evaluation Record on the terrestrial field dissipation of florasulam

PMRA Submission Number {.....}

EPA MRID Number 46808202

Table 6. Concentration of florasulam residues expressed as µg/kg in soil from the test site.

Compound	Soil depth (cm)	Sampling times (days)							
		0	3	7	14	28	59	122	213
Florasulam	0-10	8.20	3.94	3.14	1.61	0.67	0.24	<0.05	ND
	10-20	0.12	0.11	0.27	0.20	0.05	<0.05	ND	ND
	20-30	0.18	0.09	0.29	0.18	0.05 ¹	ND	ND	ND
	30-40	0.06	0.06	0.22	0.21	0.05 ¹	NA	NA	NA
	40-50	<0.05	<0.05	0.29	0.08	0.06 ¹	NA	NA	NA
5-Hydroxy XDE-570	0-10	0.94	0.42	0.73	1.03	0.35	0.22	<0.05	ND
	10-20	ND	ND	0.11	0.30	0.11	<0.05	ND	ND
	20-30	<0.05	<0.05	0.14	0.19	0.14	<0.05	ND	ND
	30-40	ND	ND	0.08	0.22	0.19	NA	NA	NA
	40-50	ND	ND	<0.05	0.06	0.11	NA	NA	NA

Data were obtained from Table 1, p. 13 of the study report. Total extractable and non-extractable residues were not determined. Reported residues are means of duplicate analysis unless otherwise noted; duplicate values were reported in Appendix 3, Table 1, pp. 96-97 of the study report. ND = Not detected. NA = Not analyzed.

¹ One of duplicate analysis (the duplicate was <0.05 µg/kg).

4. PARENT COMPOUND: The measured zero-time recovery of florasulam in the 0-10 cm soil layer was 8.20 µg/kg, which is 91.1% of the theoretical (reviewer-calculated based on a theoretical day-0 value of 9 µg/kg; see footnote 1 to DER Table 4; Table 1, p. 13). Following day 0, florasulam decreased to 3.94 µg/kg by 3 days, 1.61 µg/kg by 14 days, 0.67 µg/kg by 28 days, and was last detected above the LOQ at 0.24 µg/kg at 59 days posttreatment. Florasulam was detected in the 10-20 cm soil depth at 0.11-0.12 µg/kg from 0 to 3 days, was a maximum of 0.27 µg/kg at 7 days, and was last detected at or above the LOQ at 0.05 µg/kg at 28 days posttreatment. Florasulam was detected in the 20-30 cm soil depth at 0.05-0.29 µg/kg from 0 to 28 days, in the 30-40 cm soil depth at 0.05-0.22 µg/kg from 0 to 28 days, and in the 40-50 cm soil depth at 0.06-0.29 µg/kg from 7 to 28 days posttreatment (Appendix 3, Table 1, p. 97). Samples were not collected below 50 cm.

HALF-LIFE: Under field conditions at the test site, florasulam had a half-life value of 12.5 days in soil ($r^2 = 0.9227$), calculated using linear regression and the equation $t_{1/2} = \ln 2 / k$, where k is the rate constant. The half-life was based on all available data from the 0- to 10 cm soil depth. However, the reviewer notes that the dissipation was biphasic, with a rapid decline to <50% of the day-0 residues by 3 days posttreatment. The reviewer-calculated half-life of florasulam in the total soil profile (0- to 50-cm) using linear regression was 11.9 days ($r^2 = 0.9469$), calculated using the entire residue data set. The DT90 was 22.7 days (registrant-calculated; pp. 11-12; Figure 2, p. 19).

5. TRANSFORMATION PRODUCTS: The transformation product 5-hydroxy XDE-570 (N-(2,6-difluorophenyl)-8-fluoro-5-hydroxy-[1,2,4]triazolo[1,5-c]pyrimidine-2-sulfonamide) was

Data Evaluation Record on the terrestrial field dissipation of florasulam

PMRA Submission Number {.....}

EPA MRID Number 46808202

initially detected in the 0-10 cm soil depth at 0.94 µg/kg, ranged from 0.42 to 0.73 µg/kg from 3 to 7 days, was a maximum of 1.03 µg/kg at 14 days (11.4% of the applied florasulam; reviewer-calculated based on a theoretical day-0 florasulam value of 9 µg/kg; the 5-hydroxy XDE-570 concentration was not converted into parent equivalents), decreased to 0.35 µg/kg by 28 days, and was last detected above the LOQ at 0.22 µg/kg at 59 days posttreatment (Table 1, p. 13). 5-Hydroxy XDE-570 was detected in the 10-20 cm soil depth at 0.11-0.30 µg/kg from 7 to 28 days, in the 20-30 cm soil depth at 0.14-0.19 µg/kg from 7 to 28 days, in the 30-40 cm soil depth at 0.08-0.22 µg/kg from 7 to 28 days, and in the 40-50 cm soil depth at 0.06-0.11 µg/kg from 14 to 28 days posttreatment. Samples were not collected below 50 cm.

The reviewer-calculated half-life of 5-hydroxy XDE-570 using linear regression was 22.3 days ($r^2 = 0.8179$), based on linear regression analysis and residue data from the 0- to 10-cm depth following the maximum detection at 14 days posttreatment. The study author calculated a DT50 and DT90 of 13.3 days and 61.1 days, respectively, for 5-hydroxy XDE-570 using first-order kinetics (pp. 11-12; Figure 3, p. 20); kinetics were performed on the residues in the 0-30 cm soil horizon and using the day-14 maximum concentration as the initial time-point.

Table 7: Chemical names and CAS numbers for the transformation products of florasulam.

Applicants Code Name	CAS Number	Chemical Name	Chemical Formula	Molecular Weight (g/mol)	Smiles String
5-Hydroxy XDE-570		N-(2,6-difluorophenyl)-8-fluoro-5-hydroxy-[1,2,4]triazolo[1,5-c]pyrimidine-2-sulfonamide			

Data were obtained from Appendix 1, p. 39 of the study report.

6. EXTRACTABLE AND NON-EXTRACTABLE RESIDUES: Non-extractable residues were not measured.

Table 8: Dissipation routes of florasulam under field conditions.

Route of dissipation		% of applied amount
Accumulation (residues) in soil/ carry over		0% ¹
Transformation (% of transformation products)		11.4% (day 14) ²
Leaching, if measured (maximum depth detected)	Florasulam	40-50 cm ³
	5-OH XDE-570	40-50 cm ³
Volatilization, if measured		Not measured
Plant uptake, if measured		N/A
Run off, if measured		Not measured
Total		

Data were obtained from Table 1, p. 13 of the study report. Percentages of the applied values are reviewer-calculated based on the reviewer-calculated theoretical florasulam expected in the 0-10 cm soil depth at day 0 based on the target application rate (0.009 mg a.i./kg; see footnote 1 to DER Table 4).

¹ Residues of florasulam and 5-hydroxy XDE-570 were not detected at the end of the 213-day study period.

US EPA ARCHIVE DOCUMENT

Data Evaluation Record on the terrestrial field dissipation of florasulam

PMRA Submission Number {.....}

EPA MRID Number 46808202

2 Maximum concentration of 5-hydroxy XDE-570 during the study period.

3 Maximum depth sampled.

N/A = Not applicable.

7. VOLATILIZATION: The concentration of applied florasulam lost through volatilization was not determined at the test site.

8. PLANT UPTAKE: N/A.

9. LEACHING: Residues of florasulam and 5-hydroxy XDE-570 were detected in the soil at levels above the LOQ in the lowest depth collected, the 40-50 cm depth (Table 1, p. 13).

10. RUN OFF: Run off was not studied at the test site.

11. RESIDUE CARRYOVER: The DT90 value of florasulam was 22.7 days (registrant-calculated; pp. 11-12; Figure 2, p. 19). Residues were not detected at the end of the 213-day study period for carryover (Table 1, p. 13).

12. SUPPLEMENTARY STUDY RESULTS: A storage stability study was not conducted using soil from the test site.

III. STUDY DEFICIENCIES:

1. The stability of florasulam and 5-hydroxy XDE-570 in the test soil could not be determined because a storage stability study (using either spiked field or spiked laboratory samples) was not conducted. Additionally, the length of storage of the test samples was not reported. Subdivision N Guidelines require that a storage stability study be conducted to determine the stability of the analytes under typical laboratory storage conditions.
2. The test substance was applied at an exaggerated target application rate of 0.015 kg a.i./ha (0.30 L/ha) or three times the proposed maximum label rate in the U.S. The reviewer notes that application at the maximum label rate is required for kinetic studies because dissipation rates can vary unpredictably at different application rates.
3. Sampling was not performed to a sufficient depth to define leaching of the test chemical. Residues of the parent and transformation product, 5-hydroxy XDE-570, were detected above the LOQ in the lowest soil depth collected, the 40-50 cm depth (Table 1, p. 13). The USEPA Pesticide Registration Rejection Rate Analysis (reference 3) calls for sample cores to be collected to a depth of 90 cm, divided into segments and analyzed until one residue-free core is found. The study author stated that residues present at depths >30 cm within hours of application were observed in other study trials, and that the phenomenon was investigated in Report No. GHE-P-6718 and shown to be related to the use of the corer (p. 10).

Data Evaluation Record on the terrestrial field dissipation of florasulam

PMRA Submission Number {.....}

EPA MRID Number 46808202

4. Soil samples were excessively composited, providing only one sample for analysis at each sampling interval. The study author stated that the five soil cores from each of the four subplots were combined to give a single sample of 20 cores and that after sectioning into 10 cm horizons, the replicate horizons from all 20 cores were bulked, sieved, and analyzed in duplicate (pp. 9-10). Subdivision N Guidelines recommend that a minimum of three composites be generated per plot.
5. The field study was conducted in Greece. Subdivision N Guidelines require that terrestrial field dissipation studies be conducted domestically. Additionally, the textural classification was reported according to the ADAS classification (Table 2, p. 14); Subdivision N Guidelines require that the soil be adequately characterized using the USDA classification system.

IV. REVIEWER'S COMMENTS:

1. Historical average rainfall data were not reported to allow for comparison with the total water input from rainfall received during the study period. As a result, the reviewer could not determine whether the rainfall received during the study period was typical for the region, and could not assess whether sufficient water was available to facilitate leaching of the test material.
2. The study author calculated the DT50 of florasulam using first-order kinetics (Timme Frehse software, version 2.00) and the residue data from the 0-30 cm soil horizon (p. 10). Using this model, the author calculated a DT50 of 2.1 days and a DT90 of 22.7 days (pp. 11-12; Figure 2, p. 19).
3. The reviewer noted that certain site-specific details were not reported, such as slope of the test plot, depth to groundwater and plot history. The reviewer also noted that pre-treatment soil samples did not contain background residues of the test compound (Table 1, p. 13).
4. The reported analytical methodology was not complete; LC/MS/MS parameters were not reported. Additionally, a method validation study was not performed.
5. Concurrent recoveries of florasulam and 5-hydroxy XDE-570 were determined by fortifying control soil samples at fortification levels of 0.05, 0.50, 1.00, and 5.00 ppb (Appendix 3, p. 93). Mean recoveries across all fortification levels (\pm relative standard deviation) were $90 \pm 10.0\%$ for florasulam (range of 72 to 109%) and $83 \pm 8.3\%$ for 5-hydroxy XDE-570 (range of 70 to 96%; Appendix 3, p. 93).
6. The study author stated that soil biomass values taken at pre-treatment and 4 and 12 months posttreatment indicated a viable soil (p. 12).

Data Evaluation Record on the terrestrial field dissipation of florasulam

PMRA Submission Number {.....}

EPA MRID Number 46808202

V. REFERENCES:

1. U.S. Environmental Protection Agency. 1982. Pesticide Assessment Guidelines, Subdivision N, Chemistry: Environmental Fate, Section 164-1, Terrestrial Field Dissipation Studies. Office of Pesticide and Toxic Substances, Washington, DC. EPA 540/9-82-021.
2. U.S. Environmental Protection Agency. 1993. Pesticide Registration Rejection Rate Analysis - Environmental Fate. Office of the Prevention, Pesticides, and Toxic Substances, Washington, DC. EPA 738-R-93-010.
3. U.S. Environmental Protection Agency. 1989. FIFRA Accelerated Reregistration, Phase 3 Technical Guidance. Office of the Prevention, Pesticides, and Toxic Substances, Washington, DC. EPA 540/09-90-078.

US EPA ARCHIVE DOCUMENT

Data Evaluation Record on the terrestrial field dissipation of florasulam

PMRA Submission Number {.....}

EPA MRID Number 46808202

Attachment 1: Structures of Parent Compound and Transformation Products

US EPA ARCHIVE DOCUMENT

Data Evaluation Record on the terrestrial field dissipation of florasulam

PMRA Submission Number {.....}

EPA MRID Number 46808202

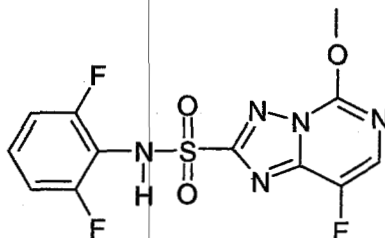
Florasulam [DE-570; XDE-570; XR-570]

IUPAC Name: 2',6',8-Trifluoro-5-methoxy[1,2,4]triazolo[1,5-c]pyrimidine-2-sulfonamide.

CAS Name: N-(2,6-Difluorophenyl)-8-fluoro-5-methoxy[1,2,4]triazolo[1,5-c]pyrimidine-2-sulfonamide.
N-(2,6-Difluorophenyl)-8-fluoro-5-methoxy-(1,2,4)-triazolo-(1,5-c)-pyrimidine-2-sulfonamide.
N-(2,6-Difluorophenyl)-8-fluoro-5-methoxy(1,2,4)triazolo(1,5-c)pyrimidine-2-sulphonamide.

CAS Number: 145701-23-1.

SMILES String: C3(F)C=CC=C(F)C=3NS(=O)(=O)C1N=C2N(N=1)C(OC)=NC=C2F (EPI Suite v3.12 SMILES string from ISIS .MOL).



Data Evaluation Record on the terrestrial field dissipation of florasulam

PMRA Submission Number {.....}

EPA MRID Number 46808202

Identified Compounds

US EPA ARCHIVE DOCUMENT

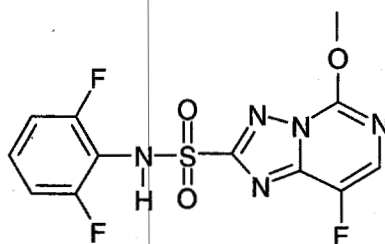
Data Evaluation Record on the terrestrial field dissipation of florasulam

PMRA Submission Number {.....}

EPA MRID Number 46808202

Florasulam [DE-570; XDE-570; XR-570]

IUPAC Name: 2',6',8-Trifluoro-5-methoxy[1,2,4]triazolo[1,5-c]pyrimidine-2-sulfonanilide.
CAS Name: N-(2,6-Difluorophenyl)-8-fluoro-5-methoxy[1,2,4]triazolo[1,5-c]pyrimidine-2-sulfonamide.
N-(2,6-Difluorophenyl)-8-fluoro-5-methoxy-(1,2,4)-triazolo-(1,5-c)-pyrimidine-2-sulfonamide.
N-(2,6-Difluorophenyl)-8-fluoro-5-methoxy(1,2,4)triazolo(1,5-c)pyrimidine-2-sulphonamide.
CAS Number: 145701-23-1.
SMILES String: C3(F)C=CC=C(F)C=3NS(=O)(=O)C1N=C2N(N=1)C(OC)=NC=C2F (EPI Suite v3.12 SMILES string from ISIS .MOL).



5-Hydroxy DE-570 [5-OH; 5-OH-XDE-570; 5-OH DE-570; 5-Hydroxy-XDE-570]

IUPAC Name: Not reported.
CAS Name: N-(2,6-Difluorophenyl)-8-fluoro-5-hydroxy(1,2,4)triazolo(1,5c)pyrimidine-2-sulphonamide.
N-(2,6-Difluorophenyl)-8-fluoro-5-hydroxy(1,2,4)triazolo(1,5c)pyrimidine-2-sulfonamide.
CAS Number: Not reported.
SMILES String: C3(F)C=CC=C(F)C=3NS(=O)(=O)C1N=C2N(N=1)C(O)=NC=C2F (EPI Suite v3.12 SMILES string from ISIS .MOL).

