

TEXT SEARCHABLE DOCUMENT

Data Evaluation Report on the aerobic biotransformation of pyrasulfotole (AE 0317309) in water-sediment system

PMRA Submission Number 2006-2445

EPA MRID Number 46801713

Data Requirement: PMRA Data Code: 8.2.3.5.4
EPA DP Barcode: D328639
OECD Data Point: IIA 7.8.1
EPA Guideline: 162-4

Test material:

Common name: Pyrasulfotole.

Chemical name:

IUPAC name: (5-Hydroxy-1,3-dimethylpyrazol-4-yl)(α,α,α -trifluoro-2-mesyl-*p*-tolyl)methanone.

(5-Hydroxy-1,3-dimethyl-1H-pyrazol-4-yl)(2-mesyl-4-trifluoromethylphenyl)methanone.

CAS name: (5-Hydroxy-1,3-dimethyl-1H-pyrazol-4-yl)[2-methylsulfonyl]-4(trifluoromethyl)phenyl]methanone.

Methanone, (5-hydroxy-1,3-dimethyl-1H-pyrazol-4-yl)[2-(methylsulfonyl)-4-(trifluoromethyl)phenyl].

CAS No: 365400-11-9.

Synonyms: AE 0317309; K-1196; K-1267.

SMILES string: FC(c1cc(c(cc1)C(=O)c1c(n(nc1C)C)O)S(=O)(=O)C)(F)F (ISIS v2.3/Universal SMILES).

No EPI Suite, v3.12 SMILES String found as of 6/7/06.

Cc1nn(C)c(O)c1C(=O)c2ccc(C(F)(F)F)cc2S(C)(=O)=O.

CS(=O)(=O)c1c(ccc(c1)C(F)(F)F)C(=O)c1c(n(nc1C)C)O.

Primary Reviewer: Lynne Binari
Cambridge Environmental

Signature:
Date: 6/14/06

Secondary Reviewer: Kathleen Ferguson
Cambridge Environmental

Signature:
Date: 6/14/06

QC/QA Manager: Joan Gaidos
Cambridge Environmental

Signature:
Date: 6/14/06

Final Reviewer: Marietta Echeverria
EPA Reviewer

Signature: *Marietta Echeverria*
Date: 1/23/07

Final Reviewer: JD Whall (Officer No. 1268)

Signature: *J.D. Whall*
5/16/07



Data Evaluation Report on the aerobic biotransformation of pyrasulfotole (AE 0317309) in water-sediment system

PMRA Submission Number 2006-2445

EPA MRID Number 46801713

PMRA Reviewer

Date:

Final Reviewer: David McAdam

Signature:

DEH Reviewer

Date



2-11/07

Company Code: BCZ
Active Code: PSA
Use Site Category: 13,14
EPA PC Code: 000692

CITATION: Allan, J. and C. Cheung. 2006. [Pyrazol-3-¹⁴C]AE 0317309 and [phenyl-UL-¹⁴C]AE 0317309: aerobic aquatic metabolism. Unpublished study performed by Bayer CropScience, Stilwell, Kansas; sponsored and submitted by Bayer CropScience, Research Triangle Park, North Carolina. BCS Study No.: A9042104 and Report No.: MEAIM008. Experimental start date December 19, 2003, and termination date December 15, 2005 (p. 6). Final report issued February 28, 2006.

Data Evaluation Report on the aerobic biotransformation of pyrasulfotole (AE 0317309) in water-sediment system

PMRA Submission Number 2006-2445

EPA MRID Number 46801713

EXECUTIVE SUMMARY

The biotransformation of [phenyl-U-¹⁴C]- and [pyrazole-3-¹⁴C]-labeled (5-hydroxy-1,3-dimethylpyrazol-4-yl)(2-mesyl-4-trifluoromethylphenyl)methanone (pyrasulfotole, AE 0317309; radiochemical purities >99%) was studied in pond water-sandy loam sediment (water pH 4.8, dissolved carbon not reported; sediment pH 4.5-5.4, organic carbon 4.1%) from North Carolina and pond water-silty clay sediment (water pH 7.5, dissolved organic carbon not reported; sediment pH 6.9-7.5, organic carbon 0.81%) from Kansas for 131-132 days under aerobic conditions in darkness at 25 ± 2°C. Based on the water volume, [¹⁴C]pyrasulfotole was applied at a rate of 0.015-0.016 mg a.i./L. The sediment:water ratio used was *ca.* 1:7 (38.4 g dry wt. sediment:283 mL water) for the sandy loam systems and *ca.* 1:4 (65.6 g dry wt. sediment:276 mL water) for the silty clay loam systems. This experiment was conducted in accordance with USEPA Subdivision N Guideline §162-4, and in compliance with USEPA GLP Standards 40 CFR, Part 160. The test system consisted of 500-mL, glass flasks attached to a continuous flow-through (humidified air, *ca.* 10-20 mL/minute) system with traps for the collection of CO₂ (ethanolamine) and volatile organics (ethylene glycol; sandy loam systems only). Sandy loam sediment and water were pre-incubated for 7 days, then following treatment, a single flask per label type was collected after 0, 11, 26, 55, 81, 109 and 132 days of incubation. Silty clay loam sediment and water were pre-incubated for 22 days, then following treatment, a single flask per label type was collected after 0, 21, 42, 64, 83, 104 and 131 days of incubation. Water layers were decanted, filtered (200 µm, glass fiber), then concentrated via rotary evaporation for reverse-phase HPLC analysis. Sediment samples were extracted two to three times with acetonitrile:water [4:1 (v:v) for 0- and 11-day sandy loam sediments; 9:1 (v:v) for all other sediment samples] via shaking, then further extracted with the acetonitrile:water solvent using an Accelerated Solvent Extraction (ASE) system (2 cycles, 80°C, 1,500 psi). Resulting sediment extracts were filtered (200 µm), combined and concentrated in the same manner as the water samples for HPLC analysis. One transformation product, 2-methylsulfonyl-4-trifluoromethylbenzoic acid (AE B197555), was identified via HPLC against reference standard.

The test conditions outlined in the study appear to have been maintained throughout the incubations. In sandy loam systems, conditions were moderately oxidizing in the water layer and moderately reducing to moderately oxidizing in the sediment at 10-25 days posttreatment, then were strongly oxidizing in the total system thereafter. Mean redox potentials in the water and sediment were +464 ± 184 mV and +469 ± 254 mV, respectively. In the water layer, dissolved oxygen levels averaged 5.45 ± 0.36 mg/L, while pH levels decreased from 7.22-8.09 at 10-25 days to 3.39-3.97 at 80-131 days. In silty clay loam systems, conditions were moderately oxidizing in the water and sediment throughout the 131-day incubation with mean redox potentials of +274 ± 33 mV and +275 ± 68 mV, respectively. In the water layer, dissolved oxygen and pH levels averaged 5.69 ± 0.20 mg/L and 7.89 ± 0.25, respectively.

Pyrasulfotole (both labels) dissipated slowly throughout the incubation in the silty clay loam systems; however, in the sandy loam systems, pyrasulfotole dissipated slowly during the initial 55 days of incubation, then levels significantly decreased thereafter, primarily through formation

Data Evaluation Report on the aerobic biotransformation of pyrasulfotole (AE 0317309) in water-sediment system

PMRA Submission Number 2006-2445

EPA MRID Number 46801713

of bound sediment residues. Increased adsorption of [^{14}C]residues to the sandy loam sediment appeared coincident with decreased pH levels (<ca. 4) in the water-sandy loam systems, which also approximated the reported pK_a of 4.2 for pyrasulfotole. No major transformation products were detected for either test substance in either system. One minor product,

- 2-methylsulfonyl-4-trifluoromethylbenzoic acid (AE B197555),

was detected in both phenyl-label treated systems at maximums of 1.6-2.2%, 0.8-1.0% and 2.6-3.0% of the applied in the water, sediment and total system, respectively. For both systems, unidentified [^{14}C]residues (both labels) were detected at total maximums of 2.0%, 3.0% and 3.0% in the water, sediment and total system, respectively. For both systems and labels, volatilized $^{14}\text{CO}_2$ was not detected at any interval. Volatile [^{14}C]organics were detected only in pyrazole-label treated sandy loam systems at a maximum 0.4% of the applied; volatile [^{14}C]organics traps were not used with the silty clay loam systems.

In sandy loam systems (both labels), overall recovery of radiolabeled material averaged $96.7 \pm 3.2\%$ (range 89.9-100.4%) of the applied, with no significant losses during the 132-day study. Following application of [^{14}C]pyrasulfotole to the water layer, [^{14}C]residues partitioned from the water layer to the sediment with average ($n = 2$) distribution ratios (water:sediment) of ca. 100:1 at day 0, 2:1 at 11-55 days, 1:4 at 81 days and were 1:>10 thereafter. [^{14}C]Pyrasulfotole in the total system decreased from a mean 97.6% of the applied at day 0 to 44.5% at 81 days and was 16.3-18.2% thereafter. In the water layer, [^{14}C]pyrasulfotole decreased from a mean 97.6% at day 0 to 61.8-68.3% at 11-55 days, 22.9% at 81 days and was 5.2-5.5% thereafter. In the sediment, [^{14}C]pyrasulfotole increased from a mean 0.9% at day 0 to 25.0% at 26 days, then decreased to 11.1-12.7% at 109-132 days. **Linear half-lives** for pyrasulfotole in the water, sediment and total system were 30, 90 and 48 days, respectively, and **nonlinear half-lives** were 46, 99 and 69 days, respectively. **Observed DT50** values were 55-81 days in the water, 81- \geq 132 days in the sediment and 55-109 days in the total system. However, these fitted transformation models do not adequately fit the observed dissipation pattern of parent residues. Extractable [^{14}C]residues in the sediment increased from a mean 0.9% of the applied at day 0 to a maximum 25.1% at 26 days, then decreased to 11.1-13.1% at 109-132 days. Nonextractable [^{14}C]residues were detected at means of 8.1-15.0% at 11-55 days, then sharply increased to 50.7% at 81 days and were 72.6% at study termination. **Pyrasulfotole does not appear to be transforming in this sandy loam system, but rather rapidly partitions from the water to an unextractable sediment-bound phase between Days 55 – 81. Pyrasulfotole is therefore considered to be stable under conditions of this aerobic aquatic system.**

In silty clay loam systems (both labels), overall recovery of radiolabeled material averaged $100.1 \pm 1.4\%$ (range 98.0-102.4%) of the applied. Following application of [^{14}C]pyrasulfotole to the water layer, [^{14}C]residues partitioned from the water layer to the sediment with average ($n = 2$) distribution ratios (water:sediment) of ca. 100:1 at day 0, 3:1 at 21 days and were 2:1 thereafter. [^{14}C]Pyrasulfotole dissipated slowly in the total system decreasing from a mean 101.4% of the applied at day 0 to 82.3-87.8% at 104-131 days. In the water layer,

Data Evaluation Report on the aerobic biotransformation of pyrasulfotole (AE 0317309) in water-sediment system

PMRA Submission Number 2006-2445

EPA MRID Number 46801713

[¹⁴C]pyrasulfotole decreased from a mean 100.9% of the applied at day 0 to 63.9% at study termination, while increasing in the sediment from 0.5% to 23.9% at the same respective intervals. **Observed DT50** values were >131 days in the water, sediment and total system. Linear/nonlinear half-lives for pyrasulfotole in the sediment and total system were not determined because levels of parent in the sediment were still increasing at study termination, and there was insufficient dissipation of parent in the total system. **Pyrasulfotole is considered to be stable under conditions of this aerobic aquatic system.** Extractable [¹⁴C]residues in the sediment increased from a mean 1.0% of the applied at day 0 to 24.4% at 131 days. Nonextractable [¹⁴C]residues increased from a mean 0.5% at day 0 to 13.1% at 104 days and were 10.6% at study termination.

A transformation pathway was provided that was consistent with the products detected in this study, with the exception that mineralization of [¹⁴C]residues to CO₂ was not evident. Under the conditions used in this study, transformation of pyrasulfotole involved hydrolytic cleavage of the phenyl and pyrazole moieties to yield minor levels 2-methylsulfonyl-4-trifluoromethylbenzoic acid (AE B197555) plus other unidentified minor products, with formation of bound sediment residues the primary dissipation pathway.

In a supplementary experiment, treatment of water-sediment systems at the anticipated field use rate for pyrasulfotole (nominally 0.0038 mg a.i./L) appeared to have no impact on the distribution of [¹⁴C]residues between the water and sediment phases for either system or label as compared to the 0.015-0.016 mg a.i./L application rate; water layers and sediment extracts at the lower application rate were not analyzed for parent [¹⁴C]pyrasulfotole and transformation products.

Results Synopsis:

Test system used: Pond water-sandy loam sediment North Carolina.

Linear half-life in water: 29.6 days ($r^2 = 0.8603$).

Linear half-life in sediment: 89.9 days ($r^2 = 0.7898$).

Linear half-life in the total system: 47.9 days ($r^2 = 0.8082$).

Non-linear half-life in water: 46.2 days ($r^2 = 0.8309$).

Non-linear half-life in sediment: 99.0 days ($r^2 = 0.8116$).

Non-linear half-life in total system: 68.6 days ($r^2 = 0.7544$).

Observed DT50 in water: 55-81 days.

Observed DT50 in sediment: 81-≥132 days.

Observed DT50 in total system: 55-109 days.

Note: Linear and non-linear fitted models do not adequately describe dissipation pattern. Pyrasulfotole is not degrading from the whole system, rather appears to rapidly bind to

Data Evaluation Report on the aerobic biotransformation of pyrasulfotole (AE 0317309) in water-sediment system

PMRA Submission Number 2006-2445

EPA MRID Number 46801713

sediments as unextractable material between 55-81 days. Considered stable in aerobic aquatic environments.

Major transformation products:

None.

Minor identified transformation products:

2-Methylsulfonyl-4-trifluoromethylbenzoic acid (AE B197555, maximum 2.2%, 0.8% and 3.0% of applied in water, soil and total system, respectively).

Test system used: Pond water-silty clay loam sediment Kansas.

Linear half-life in water: 250 days ($r^2 = 0.6871$).

Linear half-life in sediment: ND (not determined).

Linear half-life in the total system: ND.

Non-linear half-life in water: 217 days ($r^2 = 0.6828$).

Non-linear half-life in sediment: ND.

Non-linear half-life in total system: ND.

Observed DT50 in water: >131 days.

Observed DT50 in sediment: >131 days.

Observed DT50 in total system: >131 days.

Note: Considered stable in aerobic aquatic environments.

Major transformation products:

None.

Minor identified transformation products:

2-Methylsulfonyl-4-trifluoromethylbenzoic acid (AE B197555, maximum 1.6%, 1.0% and 2.6% of applied in water, soil and total system, respectively).

Study Acceptability: The EPA and PMRA classify this study as **acceptable**. No significant deviations from good scientific practices were noted. The DEH classifies this study **acceptable** for the Lawrence system and **not usable** for Pikeville systems where there was too large a pH range (see Reviewers' Comments).

I. MATERIALS AND METHODS

GUIDELINE FOLLOWED: This study was conducted in accordance with USEPA Subdivision N Guideline §162-4, PMRA Environmental Chemistry and Fate Guidelines for Registration of Pesticides in Canada (1987) and OECD Guideline for the Testing of Chemicals 308: Aerobic and Anaerobic Transformation in

Data Evaluation Report on the aerobic biotransformation of pyrasulfotole (AE 0317309) in water-sediment system

PMRA Submission Number 2006-2445

EPA MRID Number 46801713

Aquatic Sediment Systems (2002; pp. 18, 41-42). No significant deviations from the objectives of Subdivision N guidelines were noted.

COMPLIANCE: This study was conducted in compliance with USEPA GLP Standards 40 CFR, Part 160 (pp. 3, 42). Signed and dated Data Confidentiality, GLP, Quality Assurance and [study] Certification statements were provided (pp. 2-5).

A. MATERIALS:

1. Test Materials [Phenyl-U-¹⁴C]- and [pyrazole-3-¹⁴C]pyrasulfotole (p. 19).

Chemical Structure: See DER Attachment 1.

Description: Technical; physical state not reported.

[Phenyl-U-¹⁴C]pyrasulfotole

Purity: Radiochemical purity: >99% (p. 20; Figure 3B, p. 61; Figure 3D, p. 63).
Lot/Batch No. C938A/B (p. 20).
Analytical purity: Not reported.
Specific activity: 175,000 dpm/μg (28.6 mCi/mmol).
Location of the radiolabel: Uniformly on phenyl ring.

[Pyrazole-3-¹⁴C]pyrasulfotole

Purity: Radiochemical purity: >99% (p. 20; Figure 3A, p. 60; Figure 3C, p. 62).
Lot/Batch No. C939A/B (p. 20).
Analytical purity: Not reported.
Specific activity: 339,000 dpm/μg (55.3 mCi/mmol).
Location of the radiolabel: At 3-C position on pyrazole ring.

Storage conditions of test chemicals: Maintained in acetonitrile solution at -20°C (p. 20).

Data Evaluation Report on the aerobic biotransformation of pyrasulfotole (AE 0317309) in water-sediment system

PMRA Submission Number 2006-2445

EPA MRID Number 46801713

Physico-chemical properties of pyrasulfotole:

Parameter	Value	Comment
Molecular weight	362.3 g/mol	
Molecular formula	C ₁₄ H ₁₃ F ₃ N ₂ O ₄ S	
Water Solubility	4.2 g/L at pH 3.9 (pH 4 buffer) 69.1 g/L at pH 5.4 (pH 7 buffer)* 49.0 g/L at pH 5.2 (pH 9 buffer)*	At 20°C. *Exceeded buffer capacity. BCS Report No.: PA03/008.
Vapor Pressure	2.7 x 10 ⁻⁷ Pa	At 20°C, extrapolated. Siemens AG Report No.: 20040374.02.
UV Absorption	$\lambda_{max1} = 264 \text{ nm}/\epsilon = 1.11 \times 10^4 \text{ L}/(\text{mol}\cdot\text{cm})$ $\lambda_{max2} = 306 \text{ nm}/\epsilon = 5.93 \times 10^3 \text{ L}/(\text{mol}\cdot\text{cm})$	BCS Report No.: PA03/023.
pK _a	4.2	BCS Report No.: PA03/045.
log K _{ow}	0.276, -1.362 and -1.580 at pH 4, 7 and 9, respectively.	At 23°C. BCS Report No.: PA03/010.
Stability of compound at room temperature	Stable as solid.	At ambient temperature. BCS Report No.: PA03/076.
	Stable in aqueous solution at pH 5, 7 and 9.	At 25°C. BCS Report No.: 200578.

Data obtained from pp. 19-20 of the study report.

Data Evaluation Report on the aerobic biotransformation of pyrasulfotole (AE 0317309) in water-sediment system

PMRA Submission Number 2006-2445

EPA MRID Number 46801713

2. Water-sediment collection, storage and properties

Table 1: Description of water-sediment collection and storage

Description		North Carolina (Pikeville)	Kansas (Lawrence)
Geographic location.		Pikeville, Wayne County, North Carolina. Pond located in agricultural use area.	Lawrence, Jefferson County, Kansas. Pond located in agricultural use area.
Coordinates	Latitude:	N35°29.077'	N39°02.996'
	Longitude:	W78°02.623.	W95°11.353'.
Pesticide use history at the collection sites		No pesticide applications for previous 5 years.	
Collection date		November 12, 2003	January 23, 2004
Collection procedures for:	Water:	Collected into plastic bottles; no further description.	
	Sediment:	Collected into 5-gallon buckets using post-hole digger.	Collected into 5-gallon buckets with shovel.
Sampling depth for:	Water:	0- to 15-cm (0- to 6-inch) water depth.	
	Sediment:	Sampled to a depth of 15-40 cm (6-15 inches) at 15- to 20-cm water depth.	0- to 6-inch sediment layer taken at 15- to 20-cm water depth.
Storage conditions		Water and sediment transported day of collection at ambient temperature, then maintained in walk-in refrigerator at <i>ca.</i> 4°C at test facility.	
Storage length		30 days prior to preparation and 7-day pre-incubation of the water-sediment systems.	19 days prior to preparation and 22-day pre-incubation of the water-sediment systems.
Preparation	Water:	None.	
	Sediment:	Homogenized (method not reported) and sieved (2-mm).	

Data obtained from p. 22; Table 1A, p. 44; Table 1B, p. 46; Appendices 4A-5B, pp. 90-93 of the study report.

Data Evaluation Report on the aerobic biotransformation of pyrasulfotole (AE 0317309) in water-sediment system

PMRA Submission Number 2006-2445

EPA MRID Number 46801713

Table 2: Properties of the waters.

Property	North Carolina (Pikeville)		Kansas (Lawrence)	
Temperature (°C) ¹	19		3.0	
pH ¹	4.8		7.5	
Redox potential (mV)	Initial ¹	Final ²	Initial ¹	Final ²
	+366	+585, +654	+246	+276, +276
Oxygen concentration (mg/L)	Initial ³	Final ²	Initial ³	Final ²
	5.64-5.70	5.63-5.74	6.67-6.68	6.02-6.03
Dissolved organic carbon (mg/L)	Not reported.			
Hardness (mg CaCO ₃ /L)	18		175	
Electrical conductivity (mmhos/cm)	0.15		0.48	
Total dissolved solids (mg/L)	116		324	
Biomass (cells/mL water) ⁵	Initial ⁴	Final	Initial ⁴	Final
	44.0 x 10 ⁶	9.4 x 10 ⁶	8.8 x 10 ⁶	11.3 x 10 ⁶

1 At collection site (Table 1A, p. 45; Table 1B, p. 47).

2 Final at 130-131 days posttreatment; 5x systems (Sample ID 39, 49, 139, 149) only (Appendices 5A-B, pp. 92-94).

3 Initial 1-2 days prior to treatment (Appendices 5A-B, pp. 92-93).

4 Initial at day 0 posttreatment interval of the study and Final at 131-132 days (Table 1A, p. 45; Table 1B, p. 47; Appendix 2, p. 88).

5 Phospholipid fatty acid analysis (PLFA; Table 1A, p. 45; Table 1B, p. 47).

Data obtained from Table 1A, p. 45; Table 1B, p. 47; Appendices 5A-B, pp. 92-94 of the study report.

Data Evaluation Report on the aerobic biotransformation of pyrasulfotole (AE 0317309) in water-sediment system

PMRA Submission Number 2006-2445

EPA MRID Number 46801713

Table 3: Properties of the sediments.

Property		North Carolina (Pikeville)		Kansas (Lawrence)	
Soil texture		Sandy loam		Silty clay loam	
% Sand (2000-50 Φ m):		64		11	
% Silt (50-2 Φ m):		32		50	
% Clay (<2 Φ m):		4		39	
pH	sediment:water (1:1):	5.4		7.5	
	0.01M CaCl ₂ :	4.5		6.9	
Organic carbon (%) ¹		4.1		0.81	
Organic matter (%)		7.0		1.4	
CEC (meq/100 g)		5.0		28.4	
Redox potential (mV)		Initial ²	Final	Initial ²	Final
		+221 to +240	+712, +696	+110 to +115	+320, +306
Moisture (%)	at 1/3 bar:	29.7		36.9	
	at 15 bar:	8.9		22.1	
Bulk density (g/cm ³)		0.76		1.15	
Biomass (cells/g sediment) ⁴		Initial ³	Final	Initial ³	Final
		144 x 10 ⁶	441 x 10 ⁶	171 x 10 ⁶	196 x 10 ⁶

1 As reported in the study, organic carbon (%) = organic matter (%) / 1.72.

2 Initial at 1-2 days prior to treatment and Final at 130-131 days posttreatment (Appendices 5A-B, pp. 92-94).

3 Initial at day 0 posttreatment interval of the study and Final [5x systems (Sample ID 39, 49, 139, 149) only] at 131-132 days (Table 1A, p. 44; Table 1B, p. 46; Appendix 2, p. 88).

4 Phospholipid fatty acid analysis (PLFA; Table 1A, p. 44; Table 1B, p. 46).

Data obtained from Table 1A, p. 44; Table 1B, p. 46; Appendices 5A-B, pp. 92-94 of the study report.

B. EXPERIMENTAL CONDITIONS:

1. Preliminary experiments: None reported.

2. Experimental conditions:

Table 4: Experimental design (5x application rate, Reviewer's Comment No. 1).

Parameter		North Carolina (Pikeville)	Kansas (Lawrence)
Duration of the test		132 days.	131 days.
Water: Filtered/unfiltered water: Type and size of filter used, if any:		Unfiltered.	
Amount of sediment and water per treatment	Water:	283 mL total (225 mL water added plus 58.2 mL associated with the sediment).	276 mL total (225 mL added plus 51.2 mL associated with the sediment).
	Sediment:	38.4 g dry wt. (96.6 g wet wt.).	65.6 g dry wt. (116.8 g wet wt.).

Data Evaluation Report on the aerobic biotransformation of pyrasulfotole (AE 0317309) in water-sediment system

PMRA Submission Number 2006-2445

EPA MRID Number 46801713

Parameter		North Carolina (Pikeville)	Kansas (Lawrence)	
Water/sediment ratios		ca. 7:1 (total mL:g dry wt.).	ca. 4:1 (total mL:g dry wt.)	
		3:1 (v:v, 225 mL water:75 mL sediment).		
Application rates (mg a.i./L)	Nominal:	0.019 mg a.i./L		
	Actual:	phenyl label	0.015 mg a.i./L (4.37 µg a.i./283 mL).	0.016 mg a.i./L (4.35 µg a.i./276 mL).
		pyrazole label	0.015 mg a.i./L (4.34 µg a.i./283 mL).	0.016 mg a.i./L (4.30 µg a.i./276 mL).
Control conditions, if used		No sterile controls were used.		
No. of replications	Control, if used:	No sterile controls were used.		
	Treated:	For each label and system type, ten nonsterile systems were treated with [¹⁴ C]pyrasulfotole to allow for a single replicate at each of seven sampling intervals plus reserves.		
Test apparatus (type/material/volume):		500-mL glass flask (interior height 12.5 cm, diameter 7.2 cm).		
Details of traps for CO ₂ and organic volatile, if any:		Ethanolamine to trap CO ₂ (one trap, 30 mL). Ethylene glycol (one trap, 30 mL) to trap organic volatiles.	Ethanolamine (one trap, 30 mL) to trap CO ₂ .	
If no traps were used, is the system closed?		Continuous flow-through (humidified air, 10-20 mL/minute) volatiles trapping system was used.		
Identity and concentration of co-solvent: acetonitrile (ACN)		Final concentration 0.08% based on water volume [432 µL (phenyl label) or 426 µL (pyrazole label) of ACN:water (1:1, v:v) test solution in 283 mL water].	Final concentration 0.07% based on water volume [370 µL (phenyl label) or 412 µL (pyrazole label) of ACN:water (1:1, v:v) test solution in 276 mL water].	
Test material application method	Volume of the test solution used/treatment:	370-432 µL		
	Application method (eg: mixed/not mixed):	Test solution was applied uniformly to the surface of the water layer using a 250-µL Gilson Microman positive displacement pipette.		
Any indication of the test material adsorbing to the walls of the test apparatus?		Not indicated.		
Microbial biomass/population of controls (units)		Initial	Final	
	Water:	No sterile controls were used.		
	Sediment:			
Microbial biomass/population of treated (units)		Initial	Final	
	Water:	Treated systems were not analyzed for biomass.		
	Sediment:			
Experimental conditions:	Temperature (°C):	25 ± 1-2°C; maintained in a temperature-controlled chamber.		
	Continuous darkness (Yes/No):	Yes.		
Other details, if any		Untreated, nonsterile water-sediment systems were prepared and incubated in the same manner as the treated systems for biomass		

Data Evaluation Report on the aerobic biotransformation of pyrasulfotole (AE 0317309) in water-sediment system

PMRA Submission Number 2006-2445

EPA MRID Number 46801713

Parameter	North Carolina (Pikeville)	Kansas (Lawrence)
	determinations.	

Data obtained from pp. 19, 22-26; Tables 2-3, pp. 48-50; Table 5B, p. 53; Table 5D, p. 55; Figure 2, p. 59; Appendix 1, p. 87; Appendices 4A-B, pp. 90-91 of the study report.

3. Aerobic conditions: Water and sediment were combined in the metabolism flasks and maintained at 25 ± 1 -2EC in darkness for 7 (Pikeville water sandy-loam) or 22 (Lawrence water-silt clay loam) days prior to treatment (pp. 23-24; Table 2, p. 49; Appendices 5A-B, pp. 92-93). Prior to and following treatment, humidified air was continuously bubbled (*ca.* 10-20 mL/minute) into the water layer of the test systems (p. 24; Figure 2, p. 59). In pond water-sandy loam (Pikeville) systems at 1 day prior to treatment, redox potentials and dissolved oxygen in the water layers were +124 to +211 mV and 5.64-5.70 mg/L, respectively, with redox potentials in the sediment of +221 to +240 mV (Appendix 5A, p. 92). In pond water-silty clay loam (Lawrence) systems at 2 days prior to treatment, redox potentials and dissolved oxygen in the water layers were +77 to +87 mV and 6.67-6.68 mg/L, respectively, and redox potentials in the sediment were +110 to +115 mV (Appendix 5B, p. 93).

4. Supplementary experiments: Anticipated use rate (1x) samples (Reviewer's Comment No. 1). Additional water-sediment systems of each type were prepared and incubated as described above, but treated at a nominal application rate of 0.0038 mg a.i./L (pp. 19, 25). This application rate was based on a proposed maximum single use rate for pyrasulfotole of 75 g a.i./ha (p. 19). Assuming direct over-spray of a 1-ha body of water with diffusion to a depth of 2 m, the 75 g a.i./acre field rate converts to a test application rate of 0.0038 mg a.i./L. The actual application rates were lower, 0.0030-0.0031 mg a.i./L, because the amount of [¹⁴C]pyrasulfotole added to each water-sediment system to achieve a concentration of 0.0038 mg a.i./L was based only on the volume of water added (225 mL), rather than on the actual total water (water added plus water associated with the sediment) in each system. Samples resulting from this experiment were analyzed only for total radioactive residues.

Data Evaluation Report on the aerobic biotransformation of pyrasulfotole (AE 0317309) in water-sediment system

PMRA Submission Number 2006-2445

EPA MRID Number 46801713

5. Sampling:

Table 5: Sampling details.

Criteria	North Carolina (Pikeville)	Kansas (Lawrence)
Sampling intervals	0, 11, 26, 55, 81, 109 and 132 days.	0, 21, 42, 64, 83, 104 and 131 days.
Sampling method	Single treated system for each label and system type at each interval.	
Method of collection of CO ₂ and organic volatile compounds	Volatiles trapping solutions were collected at each sampling interval, with all traps replaced at 2 months posttreatment to preclude saturation.	
Sampling intervals/times for:		
Sterility check, if sterile controls are used:	Sterile controls were not prepared.	
Redox potential, dissolved oxygen and pH in water layer and redox potential in sediment:	-7, -1, 10, 25, 53, 80, 108 and 131 days posttreatment.	-22, -9, -2, 14, 20, 41, 63, 82, 104 and 130 days posttreatment.
Sample storage before analysis	<p>Water layers and sediment were separated and the sediment was extracted the day of collection.</p> <p>Water samples and sediment extracts were stored frozen (< -15°C) up to 39 days prior to analysis, with the majority of samples reportedly analyzed within 15 days. However, specific extraction and analysis dates were not provided for review.</p>	
Other details, if any	None reported.	

Data obtained from pp. 24, 26, 40; Table 3, p. 50; Tables 5A-D, pp. 52-55; Appendices 5A-B, pp. 92-94 of the study report.

C. ANALYTICAL METHODS:

Separation of the water and sediment: The water layer was decanted and filtered (Whatman 200 µm, GF/C glass fiber), then triplicate aliquots (1.0 mL) were analyzed for total radioactivity by LSC (p. 27; Appendices 8-9, pp. 98-99).

Extraction/clean up/concentration methods for water and sediment samples: Prior to HPLC analysis, an aliquot (volume not reported) of the water layer was concentrated to near dryness using rotary evaporation (under vacuum, temperature not reported; p. 27); any reconstitution of the resulting residues was not reported.

Sediment was transferred to a 200-mL centrifuge bottle and extracted two to three times with acetonitrile:water [4:1 (v:v) for 0- and 11-day sandy loam (Pikeville) sediments; 9:1 (v:v) for all

Data Evaluation Report on the aerobic biotransformation of pyrasulfotole (AE 0317309) in water-sediment system

PMRA Submission Number 2006-2445

EPA MRID Number 46801713

other sediment samples]; extraction solvent volumes were 100 mL for the initial transfer/extraction and 50 mL thereafter (p. 27; Figure 5, p. 66). Each extraction was done via shaking (mechanical shaker, 15 minutes, speed not reported), then extract and sediment were separated via centrifugation (1,287 g, 15 minutes). Extracts were filtered (Whatman 200 μm , GF/C glass fiber) and analyzed for total radioactivity by LSC. Extracted sediment was combined with diatomaceous earth (*ca.* 5-15 g, Hydromatrix sorbent), then the sediment-sorbent sample was transferred to an extraction cell (3.0 x 15.8 mm) of an Accelerated Solvent Extraction (ASE) system (Model ASE 300, Dionex; Appendix 3, p. 88) and further extracted with acetonitrile:water (9:1, v:v) under the following conditions: 2 cycles, cell temperature 80°C, heating time 5 minutes, static time 5 minutes, flush volume 60%, purge time 120 seconds, pressure 1,500 psi. All sediment extracts were combined and aliquots (1.0 mL) were analyzed for total radioactivity (Appendices 8-9, pp. 98-99). Prior to HPLC analysis, an aliquot (usually 50-100 mL) of the combined extract was concentrated via rotary evaporation (*ca.* 30°C) to near dryness (p. 28). If needed, resulting samples were filtered (0.20 or 0.45 μm Gelman Nylon Acrodisc) and/or further concentrated under a nitrogen stream using a micro-evaporator.

Total ^{14}C measurement: Total ^{14}C residues were determined by summing the concentrations of residues measured in the water layers, sediment extracts, extracted sediment and volatile trapping solutions (p. 31).

Determination of nonextractable residues: Extracted sediment was air-dried and homogenized (method not reported, p. 28). Triplicate aliquots (0.5 g) were analyzed for total radioactivity by LSC following combustion (Appendices 8-9, pp. 98-99).

Determination of volatile residues: Triplicate aliquots (volume not reported) of the ethanalamine and/or ethylene glycol trapping solutions were analyzed for total radioactivity by LSC (p. 24).

Derivatization method, if used: None was reported.

Identification and quantification of parent compound: Concentrated water and sediment extract samples were analyzed by reverse-phase HPLC under the following conditions: Phenomenex Luna C18 (4.6 x 250 mm, 5 μm , 100Å) column, Phenomenex C18 (3 x 4 mm, 10 μm) pre-column, gradient mobile phase combining either (A) 0.1% glacial acetic acid in acetonitrile and (B) 0.1% glacial acetic acid in 0.1mM ammonium acetate (Mobile Phase 1) or (A) 0.1% trifluoroacetic acid in acetonitrile and (B) 0.1% aqueous trifluoroacetic acid (Mobile Phase 2) [percent A:B at 0-5 min. 10:90 (v:v), 10-25 min. 25:75, 30-40 min. 95:5, 45-60 min. 10:90; Gradient 1], injection volume 10 μL -5.0 mL, flow rate 1 mL/minute, UV detector (wavelength not specified), and Beta RAM Model II radioactivity detector (HPLC #1; pp. 28-29). Column recoveries were monitored through the collection and LSC analysis of selected bulk column eluates and averaged $96 \pm 13\%$ and $93 \pm 12\%$ of the applied for Pikeville water and

Data Evaluation Report on the aerobic biotransformation of pyrasulfotole (AE 0317309) in water-sediment system

PMRA Submission Number 2006-2445

EPA MRID Number 46801713

sandy loam sediment extract samples, respectively, and $102 \pm 12\%$ and $108 \pm 22\%$ for Lawrence water and silty clay loam sediment extracts, respectively (DER Attachment 2).

At an unspecified point in the study, a semi-preparative HPLC system was included to reduce interference from matrix effects (p. 29). LC conditions were as described above using Mobile Phase 2 and the following gradient conditions: [percent A:B at 0-5 min. 10:90 (v:v), 10-15 min. 25:70 (this ratio is most likely in error and should either be 25:75 or 30:70), 30-40 min. 95:5, 45-60 min. 10:90; Gradient 2], flow rate 2 mL/minute (HPLC #2, pp. 29-30).

Parent [^{14}C]pyrasulfotole was identified by comparison to the retention time of unlabeled reference standard (Figures 6-7, pp. 67-68). To confirm identification, [^{14}C]pyrasulfotole was isolated from high dose (5x) 132-day water-sandy loam sediment samples and 104-day water-silty clay loam sediment samples via HPLC separation and fraction collection, with the resulting isolated [^{14}C]pyrasulfotole fractions co-chromatographed with unlabeled reference standard (pp. 30-31; Figures 8-11, pp. 69-72). Isolated parent [^{14}C]pyrasulfotole fractions were then combined respective to label and system type and analyzed by LC/MS under the following conditions: Agilent Zorbax C8 (4.6 x 250 mm, 5 μm) LC column, linear gradient mobile phase combining (A) 0.1% aqueous formic acid and (B) methanol [percent A:B at 0 min. 95:5 (v:v), 15 min. 0:100], flow rate 800 $\mu\text{L}/\text{minute}$, post-column split ratio 200:600 $\mu\text{L}/\text{min}$. (MS:radioactivity detector), Raytest Ramona Star radioactivity detector, Finnigan-MAT TSQ 7000 MS, turboionspray interface in multiple reaction monitoring (MRM) mode, positive and negative ion modes, scan range 150-750 amu, scan time 1 second (pp. 16, 30-31). Identification of [^{14}C]pyrasulfotole in isolated samples was made against labeled test substances (p. 39; Figures 4A-B, pp. 64-65; Figures 14-17, pp. 75-78).

Identification and quantification of transformation products: Transformation products were separated and quantified using HPLC as described for the parent compound (pp. 28-31; Figures 6-11, pp. 67-72).

Table 6: Reference compounds available for identifying transformation products of pyrasulfotole (AE 0317309).

Applicant codes	Chemical Name	Purity ¹
AE B197555, K-1198 and K-1367	2-(Methylsulfonyl)-4-(trifluoromethyl)benzoic acid	99.6%
AE 1073910, K-1385	(5-Hydroxy-3-methyl-1H-pyrazol-4-yl)[2-(methylsulfonyl)-4-(trifluoromethyl)phenyl]methanone	99.2%

¹ Purity w/w unless otherwise designated.

Data obtained from p. 21; Figure 1, p. 58 of the study report.

Detection limits (LOD, LOQ) for the parent compound and transformation products: For HPLC analyses, the limit of detection (LOD) was reported as 500 dpm, equivalent to 2.9 ng and

Data Evaluation Report on the aerobic biotransformation of pyrasulfotole (AE 0317309) in water-sediment system

PMRA Submission Number 2006-2445

EPA MRID Number 46801713

1.5 ng for [phenyl-U-¹⁴C]- and [pyrazole-3-¹⁴C]-label treated samples, respectively (p. 34). Sample sizes were selected such that the LOQ was 1% of the applied radioactivity.

For LSC analyses, minimum sensitivities (MSP) for [phenyl-U-¹⁴C]- and [pyrazole-3-¹⁴C]-label treated samples were reported as 3.5-5.2% and 1.8-2.7% of the applied, respectively, for water layer samples, 5.6-8.4% and 2.9-4.3%, respectively, for sediment extracts and 1.1-2.7% and 0.6-1.4%, respectively, for sediment combustions (Appendices 8-9, pp. 98-99).

II. RESULTS AND DISCUSSION

A. TEST CONDITIONS: In pond water-sandy loam (Pikeville) systems, aerobic levels increased with time. At 10-25 days posttreatment, conditions were moderately oxidizing in the water layer (+232 to +249 mV) and moderately reducing to moderately oxidizing in the sediment (+86 to +216 mV). However, from Days 53 to 131 (study termination) conditions in water and sediment were strongly oxidizing (> +548 mV). Average (n = 10) redox potentials in the water and sediment were 464 ± 184 mV (range +232 to +654 mV) and 469 ± 254 mV (+86 to +712 mV), respectively (DER Attachment 2). In the water layer, dissolved oxygen levels averaged 5.45 ± 0.36 mg/L (4.85-5.79 mg/L), while pH levels decreased from 7.22-8.09 at 10-25 days to 3.39-3.97 at 80-131 days.

In pond water-silty clay loam (Lawrence) systems, conditions were moderately oxidizing in the water and sediment throughout the 131-day incubation with average (n = 14) redox potentials of 274 ± 33 mV (+220 to +336 mV) and 275 ± 68 mV (+146 to +352 mV), respectively (DER Attachment 2). In the water layer, dissolved oxygen and pH levels averaged 5.69 ± 0.20 mg/L (5.37-6.03 mg/L) and 7.89 ± 0.25 (7.56-8.29), respectively.

B. MATERIAL BALANCE: Overall recoveries of radiolabeled material averaged (n = 14, both labels) $96.7 \pm 3.2\%$ (range 89.9-100.4%) of the applied for the pond water-sandy loam systems and $100.1 \pm 1.4\%$ (range 98.0-102.4%) for the pond water-silty clay loam systems, with no significant losses in total applied radioactivity for either label or system (DER Attachment 2, Reviewer's Comment No. 2).

Partitioning of [¹⁴C]residues between the water layers and sediment phases was initially similar for the two systems; however, after 55 days posttreatment, [¹⁴C]residues (both labels) adsorbed to the sandy loam sediment at significantly higher levels as compared to the silty clay loam (DER Attachment 2). Increasing adsorption of [¹⁴C]residues to the sandy loam sediment appeared coincident with decreasing pH levels (<ca. 4) in the systems, which also approximated the reported pK_a of 4.2 for pyrasulfotole (p. 20; DER Attachment 2). Following application of [¹⁴C]pyrasulfotole to the water layer, [¹⁴C]residues in the pond water-sandy loam systems partitioned from the water layer to the sediment with average (n = 2, both labels) distribution ratios (water:sediment) of ca. 100:1 at day 0, 2:1 at 11-55 days, 1:4 at 81 days and were 1:>10 at

Data Evaluation Report on the aerobic biotransformation of pyrasulfotole (AE 0317309) in water-sediment system

PMRA Submission Number 2006-2445

EPA MRID Number 46801713

109-132 days. Distribution ratios for the pond water-silty clay loam systems were 72:1 at day 0, 3:1 at 21 days and 2:1 at 42-131 days.

Data Evaluation Report on the aerobic biotransformation of pyrasulfotole (AE 0317309) in water-sediment system

PMRA Submission Number 2006-2445

EPA MRID Number 46801713

Table 7: Biotransformation of [phenyl-U-¹⁴C]- and [pyrazole-3-¹⁴C]-labeled pyrasulfotole (AE 0317309), expressed as percentage of applied radioactivity (mean ± s.d., n = 2¹), in North Carolina (Pikeville) pond water-sandy loam sediment under aerobic conditions.

Compound		Sampling times (days)						
		0	11	26	55	81	109	132
Pyrasulfotole	Water	97.6 ± 0.4	65.8 ± 0.9	61.8 ± 0.6	68.3 ± 1.6	22.9 ± 10.7	5.2 ± 1.1	5.5 ± 0.4
	Sed. ²	0.9 ± 0.1	17.5 ± 0.4	25.0 ± 0.4	22.0 ± 1.0	21.6 ± 12.4	11.1 ± 0.0	12.7 ± 0.7
	System	98.5 ± 0.4	83.3 ± 0.7	86.8 ± 0.4	90.8 ± 1.9	44.5 ± 19.5	16.3 ± 1.1	18.2 ± 0.2
AE B197555 ³	Water	0.0	0.0	1.1	2.2	0.6	1.6	2.2
	Sed.	0.0	0.0	n.d. ⁴	0.1	n.d.	n.d.	0.8
	System	0.0	0.0	1.1	2.3	0.6	1.6	3.0
Unidentified [¹⁴ C]residues ⁵	Water	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	1.9 ± 0.1	0.6 ± 0.8
CO ₂		n.a. ⁶	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Volatile organics ⁷		n.a.	n.d., 0.2	n.d., 0.3	n.d., 0.3	n.d., 0.4	n.d., 0.4	n.d., 0.2
Extractable sediment residues		0.9 ± 0.0	17.5 ± 0.2	25.1 ± 0.3	22.1 ± 0.8	21.6 ± 8.8	11.1 ± 0.0	13.1 ± 0.1
Nonextractable sediment residues		0.0 ± 0.0	15.0 ± 0.6	12.7 ± 1.0	8.1 ± 0.4	50.7 ± 20.0	72.9 ± 2.8	72.6 ± 1.0
Total recovery	Water	97.6 ± 0.4	65.8 ± 0.9	62.3 ± 1.2	69.4 ± 0.6	23.2 ± 10.4	7.9 ± 0.5	7.1 ± 0.9
	Sed.	0.9 ± 0.0	32.5 ± 0.8	37.8 ± 1.3	30.1 ± 1.2	72.3 ± 11.1	84.0 ± 2.7	85.7 ± 0.9
	System	98.5 ± 0.4	98.4 ± 0.1	100.2 ± 0.2	99.6 ± 0.5	95.6 ± 0.6	92.0 ± 2.1	92.9 ± 1.7

1 Reviewer's Comment No. 2.

2 Sediment.

3 2-Methylsulfonyl-4-trifluoromethylbenzoic acid; detectable only in [phenyl-U-¹⁴C]-label treated systems (Figure 1, p. 58).

4 Not detected.

5 Reported as consisting of three [¹⁴C]components detected only in the water layer (p. 38).

6 Not analyzed (Table 5B, p. 53).

7 Detected only in [pyrazole-3-¹⁴C]-label treated systems (Table 5B, p. 53).

Data obtained from p. 38; Table 5B, p. 53; Table 6, p. 56; Figure 1, p. 58 of the study report and DER Attachment 2.

Data Evaluation Report on the aerobic biotransformation of pyrasulfotole (AE 0317309) in water-sediment system

PMRA Submission Number 2006-2445

EPA MRID Number 46801713

Table 8: Biotransformation of [phenyl-U-¹⁴C]- and [pyrazole-3-¹⁴C]-labeled pyrasulfotole (AE 0317309), expressed as percentage of applied radioactivity (mean ± s.d., n = 2¹), in Kansas (Lawrence) pond water-silty clay loam sediment under aerobic conditions.

Compound		Sampling times (days)						
		0	21	42	64	83	104	131
Pyrasulfotole	water	100.9 ± 0.1	75.4 ± 0.1	70.5 ± 0.9	69.2 ± 1.6	68.6 ± 1.4	65.9 ± 0.4	63.9 ± 0.3
	sed. ²	0.5 ± 0.6	17.4 ± 0.4	18.8 ± 1.1	21.7 ± 0.6	17.2 ± 1.2	16.5 ± 0.7	23.9 ± 1.0
	system	101.4 ± 0.4	92.8 ± 0.1	89.3 ± 0.1	88.0 ± 1.9	85.8 ± 0.7	82.3 ± 0.2	87.8 ± 0.4
AE B197555 ³	water	0.0	0.0	0.8	1.3	1.0	1.6	1.0
	sed.	0.0	0.0	0.4	0.6	0.4	1.0	0.6
	system	0.0	0.0	1.2	1.9	1.4	2.6	1.6
Unidentified [¹⁴ C]residues ⁴	water	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.1 ± 0.0
	sed.	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	3.0 ± 0.0	0.2 ± 0.0
	system	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	0.0 ± 0.0	3.0 ± 0.0	0.3 ± 0.0
CO ₂		n.a. ⁵	n.d. ⁶	n.d.	n.d.	n.d.	n.d.	n.a.
Volatile organics		-- ⁷	--	--	--	--	--	--
Extractable sediment residues		1.0 ± 0.1	17.4 ± 0.3	19.0 ± 1.0	19.2 ± 0.1	17.4 ± 0.6	18.5 ± 1.5	24.4 ± 1.2
Nonextractable sediment residues		0.5 ± 0.1	8.5 ± 0.3	9.9 ± 1.6	11.4 ± 0.0	12.9 ± 1.5	13.1 ± 2.8	10.6 ± 0.5
Total recovery	water	100.9 ± 0.1	75.4 ± 0.1	70.9 ± 0.5	69.8 ± 1.0	69.1 ± 2.0	66.7 ± 1.1	64.4 ± 0.2
	sed.	1.4 ± 0.0	25.8 ± 0.0	28.9 ± 0.6	30.6 ± 0.1	30.3 ± 2.2	31.6 ± 1.4	34.3 ± 0.1
	system	102.3 ± 0.1	101.2 ± 0.1	99.8 ± 1.1	100.4 ± 1.0	99.3 ± 0.2	98.3 ± 0.3	99.3 ± 0.8

1 Reviewer's Comment No. 2.

2 Sediment.

3 2-Methylsulfonyl-4-trifluoromethylbenzoic acid; detectable only in [phenyl-U-¹⁴C]-label treated systems (Figure 1, p. 58).

4 Reported as consisting of three [¹⁴C]components detected in the water layers and a single component in [phenyl-U-¹⁴C]-label treated sediment extract (p. 38).

5 Not analyzed (Table 5D, p. 55).

6 Not detected (Table 5D, p. 55).

7 Traps for volatile [¹⁴C]organics were not used (p. 24; Table 2, p. 49).

Data obtained from p. 38; Table 5D, p. 55; Table 7, p. 57; Figure 1, p. 58 of the study report and DER Attachment 2.

C. TRANSFORMATION OF PARENT COMPOUND: In water-sandy loam (Pikeville) systems, [¹⁴C]pyrasulfotole (both labels) in the total system decreased from a mean 97.6% of the applied at day 0 to 44.5% at 81 days and was 16.3-18.2% at 109-132 days (DER Attachment 2). In the water layer, [¹⁴C]pyrasulfotole decreased from a mean 97.6% at day 0 to 61.8-68.3% at 11-55 days, 22.9% at 81 days and was 5.2-5.5% at 109-132 days (DER Attachment 2). In the sediment, [¹⁴C]pyrasulfotole increased from a mean 0.9% at day 0 to a maximum 25.0% at 26 days, then decreased to 11.1-12.7% at 109-132 days (Table 6, p. 56).

In water-silty clay loam (Lawrence) systems, [¹⁴C]pyrasulfotole (both labels) dissipated slowly in the total system decreasing from a mean 101.4% of the applied at day 0 to 82.3-87.8% at 104-

Data Evaluation Report on the aerobic biotransformation of pyrasulfotole (AE 0317309) in water-sediment system

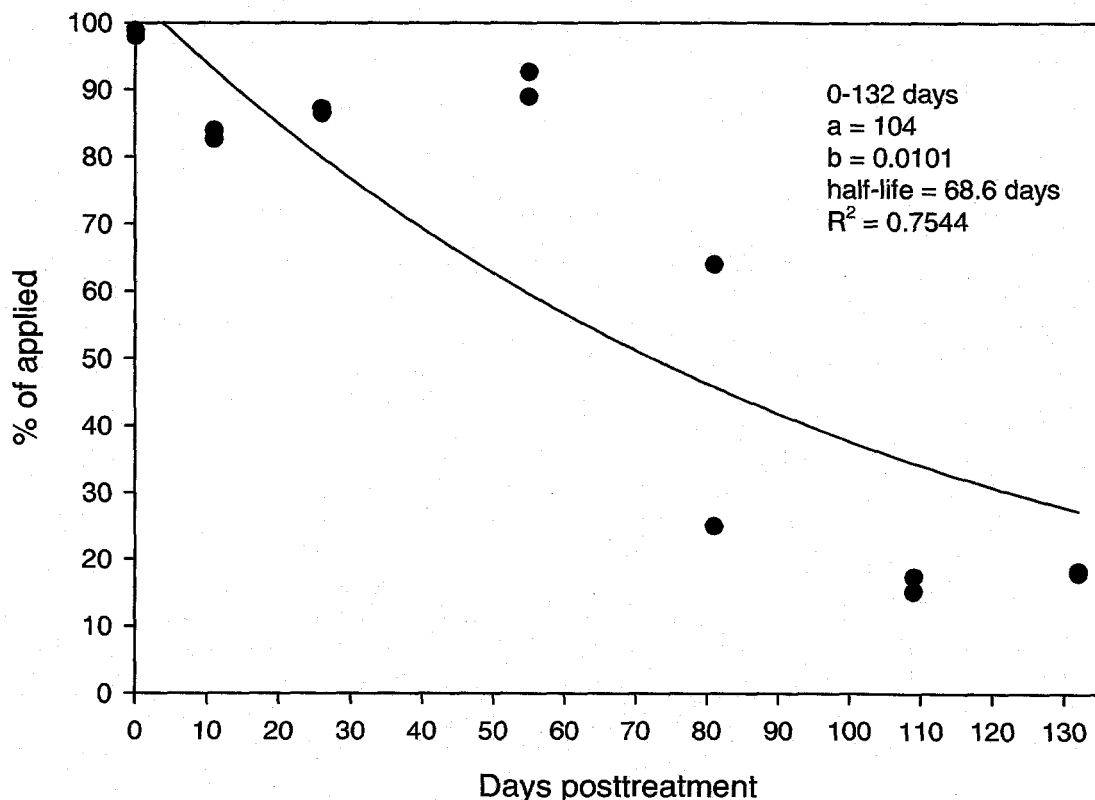
PMRA Submission Number 2006-2445

EPA MRID Number 46801713

131 days (DER Attachment 2). In the water layer, [¹⁴C]pyrasulfotole decreased from a mean 100.9% at day 0 to 63.9% at 131 days (DER Attachment 2). In the sediment, [¹⁴C]pyrasulfotole increased from a mean 0.5% at day 0 to 23.9% at 131 days (Table 7, p. 57).

HALF-LIFE/DT50/DT90: Based on first order linear regression analysis (Excel 2000), the half-lives of pyrasulfotole (both labels) in the water-sandy loam (Pikeville) systems were 30 days in the water (all intervals), 90 days in the sediment (26- to 132-day intervals) and 48 days in the total system (all intervals; DER Attachment 2). Based on nonlinear analysis (SigmaPlot v 8), half-lives were 46, 99 and 69 days in the water, sediment and total system, respectively. Observed DT50 values were 55-81 days in the water, 81-≥132 days in the sediment and 55-109 days in the total system. However, fitted dissipation curves do not adequately describe observed loss of parent material from the whole system (see Fig. 1 below). Pyrasulfotole does not appear to be transforming in the system, rather between Days 55-81 it is rapidly bound to sediments as unextractable material. Because of this apparent lack of transformation, pyrasulfotole is considered stable in this aerobic aquatic system.

Fig. 1. [¹⁴C]Pyrasulfotole (both labels) in aerobic NC pond water-sandy lo total system, nonlinear regression (MRID 46801713)



Data Evaluation Report on the aerobic biotransformation of pyrasulfotole (AE 0317309) in water-sediment system

PMRA Submission Number 2006-2445

EPA MRID Number 46801713

In water-silty clay loam (Lawrence) systems, observed DT50 values were >131 days in the water, sediment and total system. In the water layer, determined linear and nonlinear half-lives (both labels) were 250 and 217 days, respectively. Linear and nonlinear half-lives for pyrasulfotole in the sediment and total system were not determined because levels of [¹⁴C]pyrasulfotole in the sediment were still increasing at study termination, and there was insufficient dissipation of parent in the total system.

The study authors determined half-lives for pyrasulfotole (both labels) of 38 days ($r^2 = 0.85$, nonlinear/double first-order in parallel) in the water layer and 69 days ($r^2 = 0.75$, nonlinear/normal) in the total system for the water-sandy loam (Pikeville) systems; regression analyses were conducted using ModelMaker v 4.0 (pp. 32-33, 39; Figures 21-22, pp. 82-83). Using the same respective regression models, the study authors determined half-lives of 327 days ($r^2 = 0.99$) in the water layer and 578 days ($r^2 = 0.70$) in the total system for the water-silty clay loam (Lawrence) systems (p. 40; Figures 23-24, pp. 84-85).

Data Evaluation Report on the aerobic biotransformation of pyrasulfotole (AE 0317309) in water-sediment system

PMRA Submission Number 2006-2445

EPA MRID Number 46801713

Half-lives/DT50/DT90

Phase	Half-life ¹ (days)	First order regression equation	r ²	DT50 (days)	DT90 ² (days)
North Carolina (Pikeville) pond water-sandy loam sediment					
Phenyl label					
Pond water					
Linear/natural log	28.2	y = -0.0246x + 4.7099	0.8761	--	--
Nonlinear/normal	42.0	--	0.8447	--	--
Observed DT50/90	--	--	--	55-81	≥132
Sandy loam					
Linear/natural log	NA ³	--	--	--	--
Nonlinear/normal	NA	--	--	--	--
Observed	--	--	--	--	--
Total system					
Linear/natural log	44.9	y = -0.0154x + 4.7409	0.8500	--	--
Nonlinear/normal	59.8	--	0.7883	--	--
ObservedDT50/90	--	--	--	55-81	≥132
Pyrazole label					
Pond water					
Linear/natural log	31.1	y = -0.0223x + 4.7604	0.8299	--	--
Nonlinear/normal	50.6	--	0.8238	--	--
Observed DT50/90	--	--	--	55-81	≥132
Sandy loam					
Linear/natural log	NA	--	--	--	--
Nonlinear/normal	NA	--	--	--	--
Observed DT50/90	--	--	--	--	--
Total system					
Linear/natural log	51.2	y = -0.0135x + 4.7882	0.7860	--	--
Nonlinear/normal	77.9	--	0.7383	--	--
Observed DT50/90	--	--	--	81-109	≥132
Both labels					
Pond water					
Linear/natural log	29.6	y = -0.0234x + 4.7352	0.8603	--	--
Nonlinear/normal	46.2	y = 92.91*exp(-0.0029x)	0.8309	--	178
Observed DT50/90	--	--	--	55-81	≥132

Data Evaluation Report on the aerobic biotransformation of pyrasulfotole (AE 0317309) in water-sediment system

PMRA Submission Number 2006-2445

EPA MRID Number 46801713

Phase	Half-life ¹ (days)	First order regression equation	r ²	DT50 (days)	DT90 ² (days)
Sandy loam					
Linear/natural log	89.9	y = -0.0077x + 3.4880	0.7898	--	--
Nonlinear/normal	99.0	y = 31.37*exp(-0.007x)	0.8116	--	--
Observed DT50/90	--	--	--	81-≥132	≥132
Total system					
Linear/natural log	47.9	y = -0.0145x + 4.7645	0.8082	--	--
Nonlinear/normal	68.6	y = 103.9*exp(-0.0101x)	0.7544	--	230
Observed DT50/90	--	--	--	55-109	≥132
Kansas (Lawrence) pond water-silty clay loam sediment					
Phenyl label					
Pond water					
Linear/natural log	254	y = -0.0027x + 4.4568	0.6602	--	--
Nonlinear/normal	217	--	0.6591	--	--
Observed DT50/90	--	--	--	>131	>131
Silty clay loam					
Linear/natural log	NA	--	--	--	--
Nonlinear/normal	NA	--	--	--	--
Observed DT50/90	--	--	--	--	--
Total system					
Linear/natural log	ND ⁴	--	--	--	--
Nonlinear/normal	ND	--	--	--	--
Observed	--	--	--	>131	>131
Pyrazole label					
Pond water					
Linear/natural log	247	y = -0.0028x + 4.4664	0.7152	--	--
Nonlinear/normal	217	--	0.7075	--	--
Observed DT50/90	--	--	--	>131	>131
Silty clay loam					
Linear/natural log	NA	--	--	--	--
Nonlinear/normal	NA	--	--	--	--
Observed DT50/90	--	--	--	--	--

Data Evaluation Report on the aerobic biotransformation of pyrasulfotole (AE 0317309) in water-sediment system

PMRA Submission Number 2006-2445

EPA MRID Number 46801713

Phase	Half-life ¹ (days)	First order regression equation	r ²	DT50 (days)	DT90 ² (days)
Total system					
Linear/natural log	ND	--	--	--	--
Nonlinear/normal	ND	--	--	--	--
Observed DT50/90	--	--	--	>131	>131
Both labels					
Pond water					
Linear/natural log	250	y = -0.0028x + 4.4617	0.6871	--	--
Nonlinear/normal	217	--	0.6828	--	>2 years
Observed DT50/90	--	--	--	>131	>131
Silty clay loam					
Linear/natural log	ND	--	--	--	--
Nonlinear/normal	ND	--	--	--	--
Observed DT50/90	--	--	--	--	--
Total system					
Linear/natural log	ND	--	--	--	--
Nonlinear/normal	ND	--	--	--	>2 years
Observed DT50/90	--	--	--	>131	>131

1 Determined by the primary reviewer using Excel 2000 (linear) and Sigmaplot v 8.0 (nonlinear) and either individual sample data for water layers and total systems (Figures 21-24, pp. 82-85) or mean results (both labels) for sediments (Tables 6-7, pp. 56-57; DER Attachment 2; Reviewer's Comment No. 2).

2 DT90 (90% decline times) values determined by the study author using degradation rate constants (k) obtained via ModelMaker v 4.0 (pp. 32-33; Figures 21-24, pp. 82-85).

3 Not applicable. Individual replicate results for each label were not provided; only the mean of the two labels at each interval were reported.

4 Not determined due to insufficient dissipation.

TRANSFORMATION PRODUCTS: No major transformation products were detected for either test substance in either system. One minor product, 2-methylsulfonyl-4-trifluoromethylbenzoic acid (AE B197555), was detected in both systems treated with [phenyl-U-¹⁴C]pyrasulfotole. Identification of AE B197555 was made via HPLC co-chromatography against reference standard (p. 39; Figure 11, p. 72).

In water-sandy loam (Pikeville) systems, AE B197555 was detected at maximums of 2.2%, 0.8% and 3.0% of the applied in the water, sediment and total system, respectively (Table 6, p. 56). Unidentified [¹⁴C]residues, consisting of three [¹⁴C]components, were detected only in the water layer at a maximum mean 1.9% of applied (p. 38; Table 6, p. 56).

In water-silty clay loam (Lawrence) systems, AE B197555 was detected at maximums of 1.6%, 1.0% and 2.6% in the water, sediment and total system, respectively (Table 7, p. 57). A single

Data Evaluation Report on the aerobic biotransformation of pyrasulfotole (AE 0317309) in water-sediment system

PMRA Submission Number 2006-2445

EPA MRID Number 46801713

unidentified [phenyl-U-¹⁴C]-labeled component was detected at a maximum 3.0% of the applied in the sediment, while unidentified [¹⁴C]residues in the water layers were ≤0.1% (p. 38; Table 7, p. 57).

NONEXTRACTABLE AND EXTRACTABLE RESIDUES: For sandy loam (Pikeville) sediment, extractable [¹⁴C]residues (both labels) increased from a mean 0.9% of the applied at day 0 posttreatment to 25.1% at 26 days, then decreased to 11.1% at 109 days and were 13.1% at 132 days (DER Attachment 2). Nonextractable [¹⁴C]residues (both labels) were detected at means of 8.1-15.0% at 11-55 days, then sharply increased to 50.7% at 81 days and were 72.6% at 132 days (DER Attachment 2).

For silty clay loam (Lawrence) sediment, extractable [¹⁴C]residues (both labels) increased from a mean 1.0% of the applied at day 0 to 17.4% at 21 days, 19.2% at 64 days and were 24.4% at 131 days (DER Attachment 2). Nonextractable [¹⁴C]residues (both labels) increased from a mean 0.5% at day 0 to 13.1% at 104 days and were 10.6% at 131 days (DER Attachment 2).

VOLATILIZATION: For both systems and test substances, volatilized ¹⁴CO₂ (ethanolamine) was not detected at any interval (Table 5B, p. 53; Table 5D, p. 55). For water-sandy loam (Pikeville) systems, volatile [¹⁴C]organics (ethylene glycol) were detected only in [pyrazole-3-¹⁴C]-label treated systems at a maximum 0.4% of the applied (Table 5B, p. 53). Traps for volatile [¹⁴C]organics were not used with the water-silty clay loam (Lawrence) systems (either label; p. 24; Table 2, p. 49).

TRANSFORMATION PATHWAY: The study author provided a transformation pathway that was consistent with the products detected in this study, with the exception that mineralization of [¹⁴C]residues to CO₂ was not evident (p. 40; Figure 25, p. 86). Under the conditions used in this study, transformation of pyrasulfotole involved cleavage of the phenyl and pyrazole moieties to yield 2-methylsulfonyl-4-trifluoromethylbenzoic acid (AE B197555) plus other unidentified minor products, with formation of bound sediment residues the primary dissipation pathway.

Table 9: Chemical names and CAS numbers for the transformation products of pyrasulfotole.

Applicants Code Name	CAS Number	Chemical Name (CAS)	Chemical Formula	MW (g/mol)	Smiles String
AE B197555	142994-06-7	2-Methylsulfonyl-4-(trifluoromethyl)benzoic acid	-- ¹	268.2	CS(=O)(=O)c1cc(C(F)(F)F)ccc1C(=O)O

¹ Information not provided.

Data obtained from Figure 1, p. 58 of the study report.

D. SUPPLEMENTARY EXPERIMENT-RESULTS: Anticipated use rate (1x) samples treated at 0.0030-0.0031 mg a.i./L. The lower application rate appeared to have no impact on the distribution of [¹⁴C]residues between the water and sediment phases throughout the study for either system as compared to the 0.015-0.016 mg a.i./L application rate; water layers and sediment extracts were not analyzed for parent [¹⁴C]pyrasulfotole and transformation products.

Data Evaluation Report on the aerobic biotransformation of pyrasulfotole (AE 0317309) in water-sediment system

PMRA Submission Number 2006-2445

EPA MRID Number 46801713

In water-sandy loam (Pikeville) systems, [¹⁴C]residues partitioned from the water layer to the sediment with average (n = 2, both labels) distribution ratios (water:sediment) of *ca.* 100:1 at day 0, 2:1 at 11-26 days, 1:1 at 55 days, 1:5 at 81 days and were 1:>9 at 109-132 days (DER Attachment 2). At study termination (132 days), 7.2%, 13.8% and 73.9% of the applied radioactivity was associated with the water layers, extractable sediment residues and nonextractable sediment residues, respectively (Table 5A, p. 52). Volatilized ¹⁴CO₂ was not detected at any sampling interval for either label, while volatile [¹⁴C]organics were detected only in [pyrazole-3-¹⁴C]-label treated systems at a maximum 0.4% of applied. In water-silty clay loam (Lawrence) systems, [¹⁴C]residues partitioned from the water layer to the sediment with average (n = 2, both labels) distribution ratios (water:sediment) of *ca.* 100:1 at day 0, 3:1 at 11 days and were 2:1 thereafter (DER Attachment 2). At study termination (131 days), 64.3%, 19.7% and 13.0% of the applied radioactivity was associated with the water layers, extractable sediment residues and nonextractable sediment residues, respectively (Table 5C, p. 54). Volatilized ¹⁴CO₂ was not detected at any sampling interval for either label; traps for volatile [¹⁴C]organics were not used.

Microbial biomass counts in water and sediment from untreated, control systems. For water-sandy loam (Pikeville) systems, biomass counts were 44.0 x 10⁶ cells/mL water and 144 x 10⁶ cells/g sediment, respectively, at day 0, 11.6 x 10⁶ cells/mL and 298 x 10⁶ cells/g, respectively, at 55 days, and 9.4 x 10⁶ cells/mL and 441 x 10⁶ cells/g, respectively, at 132 days (Table 1A, pp. 44-45; Appendix 2, p. 88). For water-silty clay loam (Lawrence) systems, biomass counts were 8.8 x 10⁶ cells/mL water and 171 x 10⁶ cells/g sediment, respectively, at day 0, 5.2 x 10⁶ cells/mL and 255 x 10⁶ cells/g, respectively, at 60 days, and 11.3 x 10⁶ cells/mL and 196 x 10⁶ cells/g, respectively, at 131 days (Table 1B, pp. 46-47; Appendix 2, p. 88).

III. STUDY DEFICIENCIES

No significant deviations from good scientific practices or Subdivision N guidelines were noted.

IV. REVIEWERS' COMMENTS

1. This study was conducted at two nominal application rates; 3.8 µg a.i./L (1x) and 19 µg a.i./L (5x), with the 1x rate equivalent to the anticipated maximum single use rate for pyrasulfotole (p. 25; Table 2, p. 48). The study authors reported that the higher application rate was conducted to "provide sufficient material for analysis" (p. 19) and to "facilitate analytical methodology" (p. 25). Typically this is done to provide sufficient levels of parent and/or transformation products for MS analysis to confirm identifications made via the primary analytical method, such as HPLC. However, in this study, water and sediment samples resulting from the 1x application rate were only analyzed for total radioactive residues, whereas, water and sediment samples from the 5x rate were analyzed for total radioactive residues and also for parent [¹⁴C]pyrasulfotole and possible transformation products via

Data Evaluation Report on the aerobic biotransformation of pyrasulfotole (AE 0317309) in water-sediment system

PMRA Submission Number 2006-2445

EPA MRID Number 46801713

HPLC. Consequently, the portion of this study conducted at the 1x application rate is incomplete and considered a supplementary experiment for the purposes of this review.

2. Excluding results concerning parent [¹⁴C]pyrasulfotole and unidentified [¹⁴C]residues in sediment extracts, mean results and standard deviations presented in this review were determined by the primary reviewer using Microsoft Excel 2000 (9.0.2720) software (DER Attachment 2). Standard deviations were determined using the "biased" or "n" method which determines the standard deviation of the entire sample population. Where possible, mean results and summations reported by the study authors (Tables 5B, p. 53; Table 5D, p. 55; Tables 6-7, pp. 56-57) were verified by the primary reviewer and there was consistent agreement (within ± 0.1% of applied) between the study authors' reported values and those determined by the primary reviewer (DER Attachment 2). Standard deviations presented in the study report differed from those determined by the primary reviewer because the study authors determined standard deviations using the "nonbiased" or "n-1" method which bases the standard deviation on a sample of the population rather than the entire population.

Individual replicate results for parent [¹⁴C]pyrasulfotole in the water layers and total systems were only reported in Figures 21-24, pp. 82-85. Although the labels were not identified, results in other tables presented the pyrazole-label replicate first and the phenyl-label replicate second (Tables 5A-D, pp. 52-55); therefore, the primary reviewer assumed the results in Figures 21-24 were presented in the same manner, but this should be confirmed. The primary reviewer only used individual replicate results from the definitive sampling intervals (see Table 5 of this review) to determine calculated half-life values (DER Attachment 2).

In Table 7 and 8 of this review, mean results for both labels are presented because individual replicate results for [¹⁴C]pyrasulfotole in the sediments were not provided; only the means of both labels (Tables 6-7, pp. 56-57).

3. Observed DT50 values for total residues (days posttreatment).

Phase	Phenyl-U- ¹⁴ C]-pyrasulfotole		[Pyrazole-3- ¹⁴ C]-pyrasulfotole	
	Parent +nonvolatile [¹⁴ C]products ¹	Total [¹⁴ C]residues ²	Parent +nonvolatile [¹⁴ C]products ¹	Total [¹⁴ C]residues ²
North Carolina pond water-sandy loam sediment				
Water	55-81	55-81	55-81	55-81
Sediment	88->132	>132	81->132	>132
Total system	55-81	>132	81-109	>132
Kansas pond water-silty clay loam sediment				
Water	>132	>132	>132	>132
Sediment	>132	>132	>132	>132
Total system	>132	>132	>132	>132

¹ Parent pyrasulfotole plus identified/unidentified [¹⁴C]transformation products.

Data Evaluation Report on the aerobic biotransformation of pyrasulfotole (AE 0317309) in water-sediment system

PMRA Submission Number 2006-2445

EPA MRID Number 46801713

2 All [¹⁴C]residues other than volatilized ¹⁴CO₂.
Data obtained from DER Attachment 2.

4. The study authors suggest that the acidic pH in the water phase of the Pikeville system may have resulted in a greater proportion of pyrasulfotole binding to sediment. An increase in nonextractable residues was also observed in an aerobic soil metabolism study using an acidic soil from the same region (Fleige 2004).
5. The study authors conclude that an aerobic aquatic environment will have limited contribution to the overall degradation of pyrasulfotole. The peer reviewer agrees with this conclusion, and suggests that pyrasulfotole be considered stable and persistent in aerobic water-sediment systems. In the Kansas silty-clay loam system, pyrasulfotole concentrations in the water remained in the 60 – 70% of applied range from day 42 to 131 (study termination), with concentrations in the whole system remaining at 80 – 90% over the same time period. In the North Carolina sandy loam system, the measurable decrease in pyrasulfotole levels between Days 51 and 81 at the same time as there was a rapid rise in unextractable residues in the sediments. Under those conditions, the parent compound appears to be binding tightly to the sediment and is therefore not lost from the system.
6. For the Pikeville system, there were significant changes to redox potential that invalidates this report for use in risk assessment, according to the DEH. The sediment changed from aerobic conditions (+233 mV -1 DAT) to mildly anaerobic (-63 mV at 25 DAT) then back to strongly aerobic conditions (+481 mV, 131 DAT). These changes in redox were also reflected in changes to the pH, from slightly acid, pH 5 (-1 DAT), to alkaline, pH 8.1 (25 DAT), then back to strongly acidic (at least for the environment) by 108 DAT (pH 3.6) and remained so for the rest of the study period. These changes appear to have affected the study with a sharp increase in non-extractable radioactivity. There was a slow increase in non-extractable radioactivity to 17.7% of applied to 55 DAT (mean of both labels, range 15.7-19.7%), which is similar to the Lawrence system, but then over the next 50 days the rate of formation of the non-extractable radioactivity increased sharply to reach 69.9% by 109 DAT (range 69.2-70.5%). This is the same time at which there are the sharp changes to pH and redox potential and reflects formation of the more strong non-ionised form. As the study reports that these changes were due to the laboratory conditions, and these significantly affected the DT50, DEH does not consider the Pikeville system to be usable for risk assessment.

In the Lawrence system the pH was slightly alkaline (pH 8.1, -2 DAT) and remained so during the incubation period (pH range from 7.6 to 8.3). The redox potential started out as mildly anaerobic (-2 DAT +80/112 mV water/sediment; +279/311 corrected to hydrogen reference) and remained mildly anaerobic in the water until termination (range of means of 101 to 34 mV) although the sediment had a large range (140 to -56 mV). DEH calculated DT50s of 248 and 578 days for water and total system, respectively, for Lawrence using first

Data Evaluation Report on the aerobic biotransformation of pyrasulfotole (AE 0317309) in water-sediment system

PMRA Submission Number 2006-2445

EPA MRID Number 46801713

order kinetics (Excel). The degradation in sediment could not be determined as it was still increasing after 131 days.

V. REFERENCES

1. Fleige, R. (2004). [phenyl-U-14C]- and [pyrazole-3-14C]-AE 0317309: Aerobic Metabolism in a Loamy Sand Soil of US Origin under Laboratory Conditions at 25°C. Unpublished study performed, sponsored and submitted by Bayer CropScience, GmbH, Frankfurt, Germany. BCS Study No.: CB 02/011 and Report No.: MEF-386/03. Experimental start date June 24, 2002, and termination date July 31, 2003 (p. 6). Final report issued June 22, 2004. MRID 46801709.
2. U.S. Environmental Protection Agency. 1982. Pesticide Assessment Guidelines, Subdivision N, Chemistry: Environmental Fate, Section 162-4, Aerobic Aquatic Metabolism Studies. Office of Pesticide and Toxic Substances, Washington, DC. EPA 540/9-82-021.
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.
4. 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.
4. Wolfe, N., *et al.* 1990. Abiotic transformations in water, sediments and soil. *In* Pesticides in the Soil Environment, Soil Science Society of America, pp. 103-110.

Data Evaluation Report on the aerobic biotransformation of pyrasulfotole (AE 0317309) in water-sediment system

PMRA Submission Number 2006-2445

EPA MRID Number 46801713

Attachment 1: Structures of Parent Compound and Transformation Products

Data Evaluation Report on the aerobic biotransformation of pyrasulfotole (AE 0317309) in water-sediment system

PMRA Submission Number 2006-2445

EPA MRID Number 46801713

Pyrasulfotole [AE 0317309; K-1196; K-1267]

IUPAC Name: (5-Hydroxy-1,3-dimethylpyrazol-4-yl)(α,α,α -trifluoro-2-mesyl-*p*-tolyl)methanone.

(5-Hydroxy-1,3-dimethyl-1H-pyrazol-4-yl)(2-mesyl-4-trifluoromethylphenyl)methanone.

CAS Name: (5-Hydroxy-1,3-dimethyl-1H-pyrazol-4-yl)[2-methylsulfonyl]-4(trifluoromethyl)phenyl]methanone.

Methanone, (5-hydroxy-1,3-dimethyl-1H-pyrazol-4-yl)[2-(methylsulfonyl)-4-(trifluoromethyl)phenyl].

CAS Number: 365400-11-9.

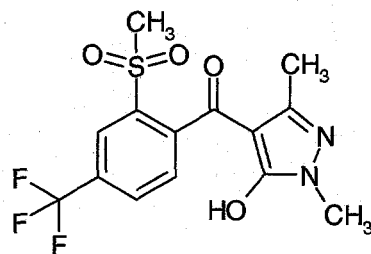
SMILES String: FC(c1cc(c(cc1)C(=O)c1c(n(nc1C)C)O)S(=O)(=O)C(F)F (ISIS v2.3/Universal SMILES).

No EPI Suite, v3.12 SMILES String found as of 6/7/06.

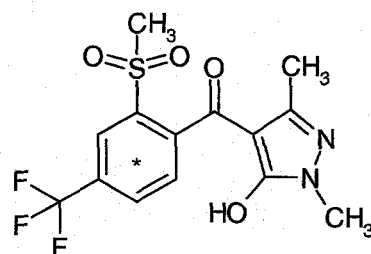
Cc1nn(C)c(O)c1C(=O)c2ccc(C(F)F)cc2S(C)(=O)=O.

CS(=O)(=O)c1c(ccc(c1)C(F)F)C(=O)c1c(n(nc1C)C)O.

Unlabeled



[Phenyl-U-¹⁴C]pyrasulfotole



¹⁴C = Position of radiolabel.

Data Evaluation Report on the aerobic biotransformation of pyrasulfotole (AE 0317309) in water-sediment system

PMRA Submission Number 2006-2445

EPA MRID Number 46801713

Pyrasulfotole [AE 0317309; K-1196; K-1267]

IUPAC Name: (5-Hydroxy-1,3-dimethylpyrazol-4-yl)(α,α,α -trifluoro-2-mesyl-*p*-tolyl)methanone.

(5-Hydroxy-1,3-dimethyl-1H-pyrazol-4-yl)(2-mesyl-4-trifluoromethylphenyl)methanone.

CAS Name: (5-Hydroxy-1,3-dimethyl-1H-pyrazol-4-yl)[2-methylsulfonyl]-4-(trifluoromethyl)phenyl]methanone.

Methanone, (5-hydroxy-1,3-dimethyl-1H-pyrazol-4-yl)[2-(methylsulfonyl)-4-(trifluoromethyl)phenyl].

CAS Number: 365400-11-9.

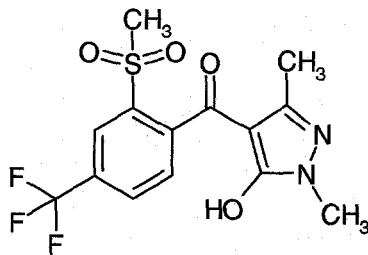
SMILES String: FC(c1cc(c(cc1)C(=O)c1c(n(nc1C)C)O)S(=O)(=O)C(F)F (ISIS v2.3/Universal SMILES).

No EPI Suite, v3.12 SMILES String found as of 6/7/06.

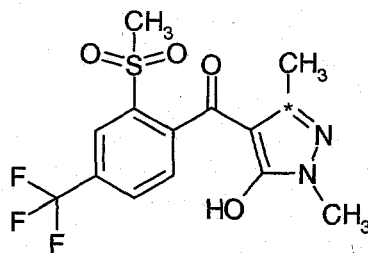
Cc1nn(C)c(O)c1C(=O)c2ccc(C(F)(F)F)cc2S(C)(=O)=O.

CS(=O)(=O)c1c(ccc(c1)C(F)(F)F)C(=O)c1c(n(nc1C)C)O.

Unlabeled



[Pyrazole-3-¹⁴C]pyrasulfotole



¹⁴C = Position of radiolabel.

Data Evaluation Report on the aerobic biotransformation of pyrasulfotole (AE 0317309) in water-sediment system

PMRA Submission Number 2006-2445

EPA MRID Number 46801713

Identified Compounds

Data Evaluation Report on the aerobic biotransformation of pyrasulfotole (AE 0317309) in water-sediment system

PMRA Submission Number 2006-2445

EPA MRID Number 46801713

Pyrasulfotole [AE 0317309; K-1196; K-1267]

IUPAC Name: (5-Hydroxy-1,3-dimethylpyrazol-4-yl)(α,α,α -trifluoro-2-mesyl-*p*-tolyl)methanone.

(5-Hydroxy-1,3-dimethyl-1H-pyrazol-4-yl)(2-mesyl-4-trifluoromethylphenyl)methanone.

CAS Name: (5-Hydroxy-1,3-dimethyl-1H-pyrazol-4-yl)[2-methylsulfonyl]-4(trifluoromethyl)phenyl]methanone.

Methanone, (5-hydroxy-1,3-dimethyl-1H-pyrazol-4-yl)[2-(methylsulfonyl)-4-(trifluoromethyl)phenyl].

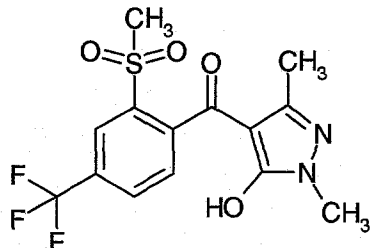
CAS Number: 365400-11-9.

SMILES String: FC(c1cc(c(cc1)C(=O)c1c(n(nc1C)C)O)S(=O)(=O)C(F)F (ISIS v2.3/Universal SMILES).

No EPI Suite, v3.12 SMILES String found as of 6/7/06.

Cc1nn(C)c(O)c1C(=O)c2ccc(C(F)(F)F)cc2S(C)(=O)=O.

CS(=O)(=O)c1c(ccc(c1)C(F)(F)F)C(=O)c1c(n(nc1C)C)O.



Data Evaluation Report on the aerobic biotransformation of pyrasulfotole (AE 0317309) in water-sediment system

PMRA Submission Number 2006-2445

EPA MRID Number 46801713

RPA 203328 [AE B197555-benzoic acid; AE B197555; K-1198; K-1367]

IUPAC Name: 2-Mesyl-4-trifluoromethylbenzoic acid.

CAS Name: Benzoic acid, 2-(methylsulfonyl)-4-(trifluoromethyl)-.

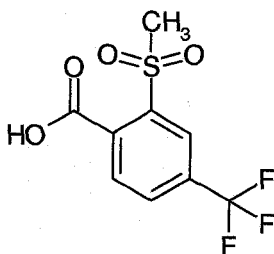
CAS Number: 142994-06-7.

SMILES String: O=C(c1ccc(cc1S(=O)(=O)C)C(F)(F)F)O (ISIS v2.3/Universal SMILES).

No EPI Suite, v3.12 SMILES String found as of 6/7/06.

CS(=O)(=O)c1cc(C(F)(F)F)ccc1C(=O)O.

CS(=O)(=O)c1cc(ccc1C(=O)O)C(F)(F)F.



Data Evaluation Report on the aerobic biotransformation of pyrasulfotole (AE 0317309) in water-sediment system

PMRA Submission Number 2006-2445

EPA MRID Number 46801713

Unidentified Reference Compounds

Data Evaluation Report on the aerobic biotransformation of pyrasulfotole (AE 0317309) in water-sediment system

PMRA Submission Number 2006-2445

EPA MRID Number 46801713

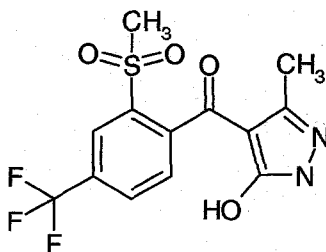
AE 1073910 [AE 0317309 N-Desmethyl; K-1385; K-1197]

IUPAC Name: (5-Hydroxy-3-methyl-1H-pyrazol-4-yl)[2-(methylsulfonyl)-4-(trifluoromethyl)phenyl]methanone.

CAS Name: Methanone, (5-hydroxy-3-methyl-1H-pyrazol-4-yl)[2-(methylsulfonyl)-4-(trifluoromethyl)phenyl].

CAS Number: Not reported.

SMILES String: O=C(C2=C(O)NN=C2C)C1=C(S(=O)(C)=O)C=C(C(F)(F)F)C=C1.CS(=O)(=O)c1cc(ccc1C(=O)c1c([nH]nc1C)O)C(F)(F)F.



Chemical: Pyrasulfotole (AE 0317309)
PC: 000692
MRID: 46801713
Guideline: 162-4

Nonlinear half-lives (exponential decay/single, 2 parameter)

North Carolina pond water-sandy loam sediment

[phenyl-U-¹⁴C]-label:

Phase	water	sediment	system
Half-life (days)	42.0	NA ¹	59.8
R squared	0.8447		0.7883

[pyrazole-3-¹⁴C]-label:

Phase	water	sediment	system
Half-life (days)	50.6	NA	77.9
R squared	0.8238		0.7383

[phenyl-U-¹⁴C]-and [pyrazole-3-¹⁴C]-labels combined:

Phase	water	sediment	system
Half-life (days)	46.2	99.0	68.6
R squared	0.8309	0.8116	0.7544

Kansas pond water-silty clay loam sediment.

[phenyl-U-¹⁴C]-label:

Phase	water	sediment	system
Half-life (days)	216.6	NA	ND ²
R squared	0.6591		

[pyrazole-3-¹⁴C]-label:

Phase	water	sediment	system
Half-life (days)	216.6	NA	ND
R squared	0.7075		

[phenyl-U-¹⁴C]-and [pyrazole-3-¹⁴C]-labels combined:

Phase	water	sediment	system
Half-life (days)	216.6	ND	ND
R squared	0.6828		

¹Individual replicate results not available.

²Calculated half-life not determined.

Chemical: Pyrasulfotole (AE 0317309)

PC: 000692

MRID: 46801713

Guideline: 162-4

Aerobic metabolism of [¹⁴C]pyrasulfotole in two pond water-sediment systems.

Determination of means/standard deviations of treated system parameters (pH, O₂ and redox potentials).

NC (Pikeville) water-sandy loam/both labels.¹

Day	Water layer			Sediment
	pH	O ₂ (mg/L)	Redox E _n (mV)	Redox E _n (mV)
10	7.22	4.85	241	211
	7.33	5.06	249	216
25	8.09	5.04	232	86
	8.04	5.14	242	141
53	8.07			
	5.71			
80	3.97	5.78	548	575
	3.55	5.79	620	672
108	3.39	5.75	630	697
	3.47	5.72	638	679
131	3.72	5.74	585	712
	3.89	5.63	654	696
Mean	5.54	5.45	464	469
std.dev.	1.97	0.36	184	254
max	8.09	5.79	654	712
min	3.39	4.85	232	86
n =	12	10	10	10

KS (Lawrence) water-silty clay loam/both labels.²

Day	Water layer			Sediment
	pH	O ₂ (mg/L)	Redox E _n (mV)	Redox E _n (mV)
14	7.56	5.37	234	166
	7.66	5.45	220	259
20	8.26	5.45	244	146
	8.23	5.58	239	226
41	8.29	5.62	260	190
	8.28	5.72	276	239
63	7.90	5.54	317	338
	7.70	5.69	336	352
82	7.88	5.75	266	311
	7.72	5.70	276	316
104	7.80	5.88	297	337
	7.77	5.80	320	344
130	7.72	6.02	276	320
	7.72	6.03	276	306
Mean	7.89	5.69	274	275
std.dev.	0.25	0.20	33	68
max	8.29	6.03	336	352
min	7.56	5.37	220	146
n =	14	14	14	14

¹Results from Appendix 5A, p. 92 of the study report; 5x systems only ("Sample ID" 32-39 and 42-49).

²Results from Appendix 5B, pp. 93-94 of the study report; 5x systems only ("Sample ID" 132-139 and 142-149).

Means and standard deviations calculated using Microsoft program functions @average(A1:A2) and stdevp(A1:A2).

Chemical: Pyrasulfotole (AE 0317309)
 PC: 000692
 MRID: 46801713
 Guideline: 162-4

**Aerobic metabolism of [¹⁴C]pyrasulfotole in two pond water-sediment systems.
 Confirmation of summations (material balances) and overall means/standard deviations for applied radioactivity.
 North Carolina (Pikeville) pond water-sandy loam sediment/[phenyl-U-¹⁴C]label.**

Day	Water % AR	Sediment				Nonext % AR	Volatiles		Mat bal % AR	Study Mat bal % AR
		Extracts			CO ₂ % AR		Org vol			
		Amb % AR	ASE % AR	Total % AR						
0	97.2	0.8		0.8				98.0	98.0	
11	66.7	14.6	2.6	17.2	14.4			98.3	98.3	
26	63.5	21.9	2.9	24.8	11.7			100.0	100.0	
55	68.8	19.5	3.3	22.8	8.5			100.1	100.1	
81	12.8	9.8	3.0	12.8	70.6			96.2	96.2	
109	7.4	7.7	3.4	11.1	75.6			94.1	94.1	
132	8.0	6.8	6.2	13.0	73.6			94.6	94.6	
Mean								97.3	97.3	
std dev.								2.2	2.2	
maximum								100.1	100.1	
minimum								94.1	94.1	
n =								7	7	

North Carolina (Pikeville) pond water-sandy loam sediment/[pyrazole-3-¹⁴C]label.

Day	Water % AR	Sediment				Nonext % AR	Volatiles		Mat bal % AR	Study Mat bal % AR
		Extracts			CO ₂ % AR		Org vol			
		Amb % AR	ASE % AR	Total % AR						
0	98.0	0.9		0.9				98.9	98.9	
11	64.9	14.8	2.9	17.7	15.6		0.2	98.4	98.4	
26	61.1	21.9	3.4	25.3	13.7		0.3	100.4	100.4	
55	69.9	19.4	1.9	21.3	7.6		0.3	99.1	99.1	
81	33.5	26.5	3.9	30.4	30.7		0.4	95.0	95.0	
109	8.3	7.6	3.5	11.1	70.1		0.4	89.9	89.9	
132	6.2	8.0	5.2	13.2	71.5		0.2	91.1	91.1	
Mean								96.1	96.1	
std dev.								3.9	3.9	
maximum								100.4	100.4	
minimum								89.9	89.9	
n =								7	7	
Both labels										
Mean								96.7	96.7	
std dev.								3.2	3.2	
n =								14	14	

Results from Table 5B, p. 53 of the study report.
 Means and standard deviations calculated using Microsoft
 program functions @average(A1:A2) and stdevp(A1:A2).

Chemical: Pyrasulfotole (AE 0317309)

PC: 000692

MRID: 46801713

Guideline: 162-4

Aerobic metabolism of [¹⁴C]pyrasulfotole in two pond water-sediment systems.

North Carolina (Pikeville) pond water-sandy loam sediment/[phenyl-U-¹⁴C]label.

Total [¹⁴C]residues in sediment.

Day	Sediment		
	Ext.	Nonext	Total
	% AR	% AR	% AR
0	0.8	0.0	0.8
11	17.2	14.4	31.6
26	24.8	11.7	36.5
55	22.8	8.5	31.3
81	12.8	70.6	83.4
109	11.1	75.6	86.7
132	13.0	73.6	86.6

[¹⁴C]Residue water phase:sediment ratios.

Day	Water	Sed	Ratio	Ratio
	% AR	% AR	W:S	S:W
0	97.2	0.8	122	0
11	66.7	31.6	2	0
26	63.5	36.5	2	1
55	68.8	31.3	2	0
81	12.8	83.4	0	7
109	7.4	86.7	0	12
132	8.0	86.6	0	11

North Carolina (Pikeville) pond water-sandy loam sediment/[pyrazole-3-¹⁴C]label.

Total [¹⁴C]residues in sediment.

Day	Sediment		
	Ext.	Nonext	Total
	% AR	% AR	% AR
0	0.9	0.0	0.9
11	17.7	15.6	33.3
26	25.3	13.7	39.0
55	21.3	7.6	28.9
81	30.4	30.7	61.1
109	11.1	70.1	81.2
132	13.2	71.5	84.7

[¹⁴C]Residue water phase:sediment ratios.

Day	Water	Sed	Ratio	Ratio
	% AR	% AR	W:S	S:W
0	98.0	0.9	109	0
11	64.9	33.3	2	1
26	61.1	39.0	2	1
55	69.9	28.9	2	0
81	33.5	61.1	1	2
109	8.3	81.2	0	10
132	6.2	84.7	0	14

Results imported from **Mat bal NC** worksheet.

Chemical: Pyrasulfotole (AE 0317309)

PC: 000692

MRID: 46801713

Guideline: 162-4

Aerobic metabolism of [¹⁴C]pyrasulfotole in two pond water-sediment systems.

Confirmation of summations (material balances) and overall means/standard deviations for applied radioactivity.

Kansas (Lawrence) pond water-silty clay loam sediment/[phenyl-U-¹⁴C]label.

Day	Water % AR	Sediment				Nonext % AR	Volatiles		Mat bal % AR	Study Mat bal % AR
		Extracts			Total % AR		CO ₂ % AR	Org vol		
		Amb % AR	ASE % AR	Total % AR						
0	101.0	1.0		1.0	0.4			102.4	102.4	
21	75.3	17.6		17.6	8.2			101.1	101.1	
42	70.4	19.1	0.9	20.0	8.3			98.7	98.7	
64	68.8	18.2	1.0	19.2	11.4			99.4	99.4	
83	71.0	15.2	1.6	16.8	11.3			99.1	99.1	
104	67.8	18.6	1.3	19.9	10.3			98.0	98.0	
131	64.6	24.3	1.2	25.5	10.0			100.1	100.1	
Mean								99.8	99.8	
std dev.								1.4	1.4	
maximum								102.4	102.4	
minimum								98.0	98.0	
n =								7	7	

Kansas (Lawrence) pond water-silty clay loam sediment/[pyrazole-3-¹⁴C]label.

Day	Water % AR	Sediment				Nonext % AR	Volatiles		Mat bal % AR	Study Mat bal % AR
		Extracts			Total % AR		CO ₂ % AR	Org vol		
		Amb % AR	ASE % AR	Total % AR						
0	100.8	0.9		0.9	0.5			102.2	102.2	
21	75.5	17.1		17.1	8.7			101.3	101.3	
42	71.4	17.1	0.9	18.0	11.5			100.9	100.9	
64	70.8	18.4	0.7	19.1	11.4			101.3	101.3	
83	67.1	16.4	1.6	18.0	14.4			99.5	99.5	
104	65.5	15.7	1.3	17.0	15.9			98.4	98.5	
131	64.2	21.7	1.5	23.2	11.1			98.5	98.5	
Mean								100.3	100.3	
std dev.								1.4	1.4	
maximum								102.2	102.2	
minimum								98.4	98.5	
n =								7	7	
Both labels										
Mean								100.1	100.1	
std dev.								1.4	1.4	
n =								14	14	

Results from Table 5D, p. 55 of the study report.

Means and standard deviations calculated using Microsoft program functions @average(A1:A2) and stdevp(A1:A2).

Chemical: Pyrasulfotole (AE 0317309)
 PC: 000692
 MRID: 46801713
 Guideline: 162-4

**Aerobic metabolism of [¹⁴C]pyrasulfotole in two pond water-sediment systems.
 Kansas (Lawrence) pond water-silty clay loam sediment/[phenyl-U-¹⁴C]label.
 Total [¹⁴C]residues in sediment.**

Day	Sediment		
	Ext.	Nonext	Total
	% AR	% AR	% AR
0	1.0	0.4	1.4
21	17.6	8.2	25.8
42	20.0	8.3	28.3
64	19.2	11.4	30.6
83	16.8	11.3	28.1
104	19.9	10.3	30.2
131	25.5	10.0	35.5

[¹⁴C]Residue water phase:sediment ratios.

Day	Water	Sed	Ratio	Ratio
	% AR	% AR	W:S	S:W
	0	101.0	1.4	72
21	75.3	25.8	3	0
42	70.4	28.3	2	0
64	68.8	30.6	2	0
83	71.0	28.1	3	0
104	67.8	30.2	2	0
131	64.6	35.5	2	1

**Kansas (Lawrence) pond water-silty clay loam sediment/[pyrazole-3-¹⁴C]label.
 Total [¹⁴C]residues in sediment.**

Day	Sediment		
	Ext.	Nonext	Total
	% AR	% AR	% AR
0	0.9	0.5	1.4
21	17.1	8.7	25.8
42	18.0	11.5	29.5
64	19.1	11.4	30.5
83	18.0	14.4	32.4
104	17.0	15.9	32.9
131	23.2	11.1	34.3

[¹⁴C]Residue water phase:sediment ratios.

Day	Water	Sed	Ratio	Ratio
	% AR	% AR	W:S	S:W
	0	100.8	1.4	72
21	75.5	25.8	3	0
42	71.4	29.5	2	0
64	70.8	30.5	2	0
83	67.1	32.4	2	0
104	65.5	32.9	2	1
131	64.2	34.3	2	1

Results imported from **Mat bal KS** worksheet.

Chemical: Pyrasulfotole (AE 0317309)

PC: 000692

MRID: 46801713

Guideline: 162-4

Aerobic metabolism of [¹⁴C]pyrasulfotole in two pond water-sediment systems.

North Carolina (Pikeville) pond water-sandy loam sediment/both labels.

Day	Label	Water			Sediment						Organic volatiles			Material Balance			Study Reported Material Balance		
		% AR	Mean	s.d.	Extracts			Nonextractable			% AR	Mean	s.d.	% AR	Mean	s.d.	% AR	Mean	s.d.
0	PHE	97.2			0.8									98.0			98.0		
	PYR	98.0	97.6	0.4	0.9	0.9	0.0		#DIV/0!	#DIV/0!		#DIV/0!	#DIV/0!	98.9	98.5	0.4	98.9	98.5	0.4
11	PHE	66.7			17.2			14.4						98.3			98.3		
	PYR	64.9	65.8	0.9	17.7	17.5	0.2	15.6	15.0	0.6	0.2	0.2	0.0	98.4	98.4	0.0	98.4	98.4	0.1
26	PHE	63.5			24.8			11.7						100.0			100.0		
	PYR	61.1	62.3	1.2	25.3	25.1	0.3	13.7	12.7	1.0	0.3	0.3	0.0	100.4	100.2	0.2	100.4	100.2	0.2
55	PHE	68.8			22.8			8.5						100.1			100.1		
	PYR	69.9	69.4	0.6	21.3	22.1	0.8	7.6	8.1	0.4	0.3	0.3	0.0	99.1	99.6	0.5	99.1	99.6	0.5
81	PHE	12.8			12.8			70.6						96.2			96.2		
	PYR	33.5	23.2	10.4	30.4	21.6	8.8	30.7	50.7	20.0	0.4	0.4	0.0	95.0	95.6	0.6	95.0	95.6	0.6
109	PHE	7.4			11.1			75.6						94.1			94.1		
	PYR	8.3	7.9	0.5	11.1	11.1	0.0	70.1	72.9	2.8	0.4	0.4	0.0	89.9	92.0	2.1	89.9	92.0	2.1
132	PHE	8.0			13.0			71.5						92.5			94.6		
	PYR	6.2	7.1	0.9	13.2	13.1	0.1	73.6	72.6	1.0	0.2	0.2	0.0	93.2	92.9	0.3	91.1	92.9	1.7

Results from Table 5B, p. 53 of the study report.

Means and standard deviations calculated using Microsoft program functions

@average(A1:A2) and stdevp(A1:A2).

Overall	96.7	3.2		96.7	3.2
maximum	100.4			100.4	
minimum	89.9			89.9	
n =	14			14	

Chemical: Pyrasulfotole (AE 0317309)

PC: 000692

MRID: 46801713

Guideline: 162-4

**Aerobic metabolism of [¹⁴C]pyrasulfotole in two pond water-sediment systems.
Kansas (Lawrence) pond water-silty clay loam sediment/both labels.**

Day	Label	Water			Sediment						CO ₂			Material Balance			Study Reported Material Balance		
		% AR	Mean	s.d.	Extracts ¹			Nonextractable			Organic volatiles			% AR	Mean	s.d.	% AR	Mean	s.d.
					% AR	Mean	s.d.	% AR	Mean	s.d.	% AR	Mean	s.d.						
0	PHE	101.0			1.0			0.4						102.4			102.4		
	PYR	100.8	100.9	0.1	0.9	1.0	0.1	0.5	0.5	0.1		#DIV/0!	#DIV/0!	102.2	102.3	0.1	102.2	102.3	0.1
21	PHE	75.3			17.6			8.2						101.1			101.1		
	PYR	75.5	75.4	0.1	17.1	17.4	0.3	8.7	8.5	0.3		#DIV/0!	#DIV/0!	101.3	101.2	0.1	101.3	101.2	0.1
42	PHE	70.4			20.0			8.3						98.7			98.7		
	PYR	71.4	70.9	0.5	18.0	19.0	1.0	11.5	9.9	1.6		#DIV/0!	#DIV/0!	100.9	99.8	1.1	100.9	99.8	1.1
64	PHE	68.8			19.2			11.4						99.4			99.4		
	PYR	70.8	69.8	1.0	19.1	19.2	0.0	11.4	11.4	0.0		#DIV/0!	#DIV/0!	101.3	100.4	1.0	101.3	100.4	1.0
83	PHE	71.0			16.8			11.3						99.1			99.1		
	PYR	67.1	69.1	2.0	18.0	17.4	0.6	14.4	12.9	1.5		#DIV/0!	#DIV/0!	99.5	99.3	0.2	99.5	99.3	0.2
104	PHE	67.8			19.9			10.3						98.0			98.0		
	PYR	65.5	66.7	1.1	17.0	18.5	1.5	15.9	13.1	2.8		#DIV/0!	#DIV/0!	98.4	98.2	0.2	98.5	98.3	0.3
131	PHE	64.6			25.5			10.0						100.1			100.1		
	PYR	64.2	64.4	0.2	23.2	24.4	1.2	11.1	10.6	0.5		#DIV/0!	#DIV/0!	98.5	99.3	0.8	98.5	99.3	0.8
													Overall	100.1	1.4		100.1	1.4	
													maximum	102.4			102.4		
													minimum	98.0			98.0		
													n =	14			14		

Results from Table 5D, p. 55 of the study report.

¹131-day PHE incorrectly reported as 24.2% in Table 5D.

Means and standard deviations calculated using Microsoft program functions @average(A1:A2) and stdevp(A1:A2).

Chemical: Pyrasulfotole (AE 0317309)
 PC: 000692
 MRID: 46801713
 Guideline: 162-4

**Aerobic metabolism of [¹⁴C]pyrasulfotole in two pond water-sediment systems.
 North Carolina (Pikeville) pond water-sandy loam sediment/both labels.
 Total [¹⁴C]residues in sediment.**

Day	Label	Sediment				
		Ext.	Nonext	Total in Sediment		
		% AR	% AR	% AR	Mean	s.d.
0	PHE	0.8		0.8		
	PYR	0.9		0.9	0.9	0.0
11	PHE	17.2	14.4	31.6		
	PYR	17.7	15.6	33.3	32.5	0.8
26	PHE	24.8	11.7	36.5		
	PYR	25.3	13.7	39.0	37.8	1.3
55	PHE	22.8	8.5	31.3		
	PYR	21.3	7.6	28.9	30.1	1.2
81	PHE	12.8	70.6	83.4		
	PYR	30.4	30.7	61.1	72.3	11.1
109	PHE	11.1	75.6	86.7		
	PYR	11.1	70.1	81.2	84.0	2.7
132	PHE	13.0	73.6	86.6		
	PYR	13.2	71.5	84.7	85.7	0.9

[¹⁴C]Residue water phase:sediment ratios.

Day	Water % AR	Sed % AR	Ratio		W:S ratio		S:W ratio	
			W:S	S:W	Mean	s.d.	Mean	s.d.
			0	97.2	0.8	122	0	
11	98.0	0.9	109	0	115	6	0	0
	66.7	31.6	2	0				
26	64.9	33.3	2	1	2	0	0	0
	63.5	36.5	2	1				
55	61.1	39.0	2	1	2	0	1	0
	68.8	31.3	2	0				
81	69.9	28.9	2	0	2	0	0	0
	12.8	83.4	0	7				
109	33.5	61.1	1	2	0	0	4	2
	7.4	86.7	0	12				
132	8.3	81.2	0	10	0	0	11	1
	8.0	86.6	0	11				
	6.2	84.7	0	14	0	0	12	1

**Kansas (Lawrence) pond water-silty clay loam sediment/both labels.
 Total [¹⁴C]residues in sediment.**

Day	Label	Sediment				
		Ext.	Nonext	Total in Sediment		
		% AR	% AR	% AR	Mean	s.d.
0	PHE	1.0	0.4	1.4		
	PYR	0.9	0.5	1.4	1.4	0.0
21	PHE	17.6	8.2	25.8		
	PYR	17.1	8.7	25.8	25.8	0.0
42	PHE	20.0	8.3	28.3		
	PYR	18.0	11.5	29.5	28.9	0.6
64	PHE	19.2	11.4	30.6		
	PYR	19.1	11.4	30.5	30.6	0.1
83	PHE	16.8	11.3	28.1		
	PYR	18.0	14.4	32.4	30.3	2.2
104	PHE	19.9	10.3	30.2		
	PYR	17.0	15.9	32.9	31.6	1.4
131	PHE	24.2	10.0	34.2		
	PYR	23.2	11.1	34.3	34.3	0.0

[¹⁴C]Residue water phase:sediment ratios.

Day	Water % AR	Sed % AR	Ratio		W:S ratio		S:W ratio	
			W:S	S:W	Mean	s.d.	Mean	s.d.
			0	101.0	1.4	72	0	
21	100.8	1.4	72	0	72	0	0	0
	75.3	25.8	3	0				
42	75.5	25.8	3	0	3	0	0	0
	70.4	28.3	2	0				
64	71.4	29.5	2	0	2	0	0	0
	68.8	30.6	2	0				
83	70.8	30.5	2	0	2	0	0	0
	71.0	28.1	3	0				
104	67.1	32.4	2	0	2	0	0	0
	67.8	30.2	2	0				
131	65.5	32.9	2	1	2	0	0	0
	64.6	34.2	2	1				
	64.2	34.3	2	1	2	0	1	0

Results from Table 5B, p. 53; Table 5D, p. 55 of the study report.

Means and standard deviations calculated using Microsoft program functions @average(A1:A2) and stdevp(A1:A2).

Chemical: Pyrasulfotole (AE 0317309)

PC: 000692

MRID: 46801713

Guideline: 162-4

Aerobic metabolism of [¹⁴C]pyrasulfotole in two pond water-sediment systems.

Confirmation/determination of means/std.dev. for pyrasulfotole.

North Carolina (Pikeville) pond water-sandy loam sediment.

Day	Label	Pyrasulfotole					
		Water			Total system		
		% AR	mean	s.d.	% AR	mean	s.d.
0	PHE	97.2			98.0		
	PYR	98.0	97.6	0.4	98.9	98.5	0.4
11	PHE	66.7			83.9		
	PYR	64.9	65.8	0.9	82.6	83.3	0.7
26	PHE	62.4			87.2		
	PYR	61.1	61.8	0.6	86.4	86.8	0.4
55	PHE	66.6			88.9		
	PYR	69.9	68.3	1.6	92.6	90.8	1.9
81	PHE	12.2			25.0		
	PYR	33.5	22.9	10.7	63.9	44.5	19.5
109	PHE	4.1			15.2		
	PYR	6.3	5.2	1.1	17.4	16.3	1.1
132	PHE	5.8			18.0		
	PYR	5.1	5.5	0.4	18.3	18.2	0.2

Kansas (Lawrence) pond water-silty clay loam sediment.

Day	Label	Pyrasulfotole					
		Water			Total system		
		% AR	mean	s.d.	% AR	mean	s.d.
0	PHE	101.0			101.0		
	PYR	100.8	100.9	0.1	101.7	101.4	0.4
21	PHE	75.3			92.9		
	PYR	75.5	75.4	0.1	92.6	92.8	0.2
42	PHE	69.6			89.1		
	PYR	71.4	70.5	0.9	89.4	89.3	0.2
64	PHE	67.5			86.1		
	PYR	70.8	69.2	1.6	89.9	88.0	1.9
83	PHE	70.0			86.4		
	PYR	67.1	68.6	1.4	85.1	85.8	0.7
104	PHE	66.2			82.1		
	PYR	65.5	65.9	0.4	82.5	82.3	0.2
131	PHE	63.5			88.2		
	PYR	64.2	63.9	0.3	87.4	87.8	0.4

Results from Figures 21-24, pp. 82-85 of the study report; only results from sampling intervals for definitive study were used.

In Figures 21-24, first replicate at each interval pyrazole-label and second phenyl-label.

Means and standard deviations calculated using Microsoft program functions @average(A1:A2) and stdevp(A1:A2).

[¹⁴C]Pyrasulfotole (both labels) in aerobic NC pond water-sandy loam:
total system, nonlinear regression (MRID 46801713)

